



UNIVERSITEIT VAN PRETORIA  
UNIVERSITY OF PRETORIA  
YUNIBESITHI YA PRETORIA

# **PROCESS DEVELOPMENT FOR THE PRODUCTION OF BENEFICIATED TITANIA SLAG**

by

**Jacobus Philippus van Dyk**

Submitted in fulfillment of the requirements for the degree

**PHILOSOPHIAE DOCTOR**

In the Faculty Engineering

University of Pretoria

Pretoria

Study leader: PC Pistorius

5 November 1999

## ACKNOWLEDGEMENTS

I would like to thank and herewith express my sincere appreciation to the following people:

- Nanne Vegter, my colleague and friend, for the numerous discussions during the project the ideas that were generated during those discussions.
- Corelie Visser, for providing mineralogical and moral support during the project.
- Marie Nel, Bes Bester and Jaco Vermaak for performing numerous leach tests.
- Willem van Niekerk for having the vision and perseverance to support this project financially.
- John Winter and Ernie Walpole, my Australian friends, for generating some of the initial ideas and performing most of the initial test work.
- Chris Pistorius, my supervisor, for his support and advice during this project
- My family for their tolerance and support during this project

## TABLE OF CONTENTS

<b>LIST OF FIGURES</b>	vii
<b>LIST OF TABLES</b>	xiv
<b>ABSTRACT</b>	xviii
<b>OPSOMMING</b>	xx
<b>CHAPTER 1</b>	
<b>INTRODUCTION</b>	
1.1 The occurrence and uses of titanium dioxide	1
1.2 Pigment production processes	1
1.2.1 The sulphate process	2
1.2.2 The chloride process	2
1.2.3 Feedstock requirements for the chloride process	3
1.3 The production of titania slag	4
1.4 Slag upgrading processes	5
1.4.1 Oxidation and reduction roasting followed by leaching	5
1.4.2 Oxidation roasting followed by leaching	6
1.4.3 Salt roasting followed by leaching	7
1.4.4 Oxidation and fluxing of molten slag followed by leaching	7
1.4.5 Sulphidation and/or sulphation roasting followed by leaching	8
1.4.6 Chlorination	9
1.5 The motivation for upgrading chloride grade titania slag	9
<b>CHAPTER 2</b>	
<b>PRELIMINARY INVESTIGATION</b>	
2.1 Experimental design	11
2.1.1 Feed material and experimental plan	11
2.2 Experimental procedure	12
2.2.1 Slag pretreatment	12
2.2.2 Leaching	12
2.3 Results and discussion	13
2.4 Conclusions	16
<b>CHAPTER 3</b>	
<b>PROCESS DEVELOPMENT PHASE 1</b>	
3.1 Introduction	17
3.2 Experimental design	17
3.2.1 Feed material	17



3.2.2 Experimental plan	17
3.2.3 Experimental procedure	18
3.2.3.1 Roast procedure	18
3.2.3.1 Leach procedure	18
3.3. Results and discussion	19
3.3.1 Roast investigation	19
3.3.2 Leach investigation	20
3.3.3 Mineralogical investigation	22
3.3.3.1 As-cast titania feed slag	22
3.3.3.2 Oxidation	23
3.3.3.3 Reduction	30
3.3.3.4 Leaching	34
3.3.4 Summary of the mineralogical changes that occur during roasting	38
3.3.4.1 Standard slag	38
3.3.4.2 High iron slag	40
3.3.4.3 High magnesia slag	40
3.4 Conclusions	41
 <b>CHAPTER 4</b> <b>PROCESS DEVELOPMENT PHASE 2</b>	
4.1 Introduction	42
4.2 Experimental design	42
4.2.1 Feed material	42
4.2.2 Experimental plan	42
4.2.3 Experimental procedure	43
4.2.3.1 Roast procedure	43
4.2.3.2 Leach procedure	44
4.2.3.3 Electron microprobe analysis procedure	44
4.3 Results and discussion	45
4.3.1 Standard titania slag	45
4.3.1.1 Oxidation	45
4.3.1.2 Reduction	57
4.3.1.3 Particle size	61
4.3.2 High iron titania slag	63
4.3.2.1 Oxidation	63
4.3.2.2 Particle size	72
4.4 Conclusions	73
 <b>CHAPTER 5</b> <b>THE OXIDATION MECHANISM OF TITANIA SLAG</b>	
5.1 Introduction	74
5.2 Background	74
5.2.1 Segregation and diffusion of elements in oxide systems	74





5.2.2 Diffusion of Fe in TiO <sub>2</sub>	74
5.3 Oxidation of titaniferous materials	75
5.3.1 Thermodynamics	75
5.3.2 Kinetics of titania slag oxidation	78
5.3.3 Kinetics of ilmenite oxidation	78
5.3.4 Kinetics of titanomagnetite oxidation	78
5.4 Proposed mechanism of titania slag oxidation	79
5.5 Experimental plan	81
5.6 Experimental procedure	81
5.6.1 Roasting	81
5.6.2 Leaching	81
5.7 Results and discussion	81
5.7.1 Investigation into the roasting conditions required for iron migration	81
5.7.2 Porosity and particle size changes during roasting	83
5.7.3 Investigation into the oxidation of coated slag particles	84
5.7.4 Investigation into the oxidation state of iron at various positions in oxidised slag particles	86
5.7.4.1 WDS point chemical analysis	86
5.7.4.2 Leach investigation	87
5.7.5 Investigation into the influence of iron-rich rims on the mechanism of oxidation	90
5.7.6 Investigation into the influence of higher roasting temperatures on the mechanism of oxidation	94
5.7.7 Investigation into the influence of interrupted roasting on the mechanism of oxidation	98
5.8 Quantitative WDS analyses of selected phases in oxidised slag	103
5.9 Conclusions	104
<b>SUMMARY</b>	105
<b>REFERENCES</b>	106
<b>APPENDICES</b>	108
<b>Appendix I</b> Chemical analysis of the feed slags used for the preliminary investigation	109
<b>Appendix II</b> Log sheets for the preliminary investigation experiments	110
<b>Appendix III</b> Chemical analyses of the feed slags used for process development phase 1	129
<b>Appendix IV</b> Results of the process development phase 1 roast investigation	130
<b>Appendix V</b> Results of the process development phase 1 leach investigation	133
<b>Appendix VI</b> Titration procedure used to determine the Fe(II), Fe(III) and HCl concentrations of the leach liquors	143
<b>Appendix VII</b> Chemical analyses of the feed slags used for process	144

	development phase 2	
<b>Appendix VIII</b>	List of experiments conducted for process development phase 2	145
<b>Appendix IX</b>	Phase 2, Series 1 – Logsheets	149
<b>Appendix X</b>	Phase 2, Series 2 – Logsheets	169
<b>Appendix XI</b>	Phase 2, Series 3 – Logsheets	189
<b>Appendix XII</b>	Phase 2, Series 4 – Logsheets	223
<b>Appendix XIII</b>	Calculation of the gas flow rate necessary for fluidisation	231
<b>Appendix XIV</b>	Chemical composition profile data	234
<b>Appendix XV</b>	Reduction leach logsheets	276
<b>Appendix XVI</b>	Estimation of the oxygen isobars for oxidation and reduction at 850°C	285
<b>Appendix XVII</b>	Formation of hematite or ferric pseudobrookite during oxidation	288
<b>Appendix XVIII</b>	Mössbauer data	290

## LIST OF FIGURES

### CHAPTER 1

Figure 1	Flow diagram of the proposed IHM ilmenite smelting plant	4
Figure 2	Titaniferous feedstock prices	9

### CHAPTER 2

Figure 3	The leach kinetics of titanium and iron in 20% HCl from: <b>A</b> Standard as-cast slag at 95°C; <b>B</b> High iron granulated slag containing at 95°C; <b>C</b> Oxidised and reduced slag at 107°C and; <b>D</b> phosphate fluxed slag at 95°C	15
----------	---	----

### CHAPTER 3

Figure 4	Summary of the results from the tests conducted to evaluate the effect of leach time and feed slag composition	20
Figure 5	The effect of excess hydrochloric acid on iron extraction at different initial hydrochloric acid concentrations	21
Figure 6	The effect of initial hydrochloric acid concentration on iron extraction at different levels of excess hydrochloric acid	21
Figure 7	The effect of acid concentration and the level of excess acid on the final product quality after 12 h of leaching	22
Figure 8	As-cast standard titania slag	23
Figure 9	Standard slag oxidised for 1 h at 850 °C, displaying iron migration towards the edges of cracks and the outer rims of the particles	25
Figure 10	Standard slag oxidised for 3 h at 800 °C, contained dense particles that displayed iron enrichment to the outsides of the particles	25
Figure 11	(Left side) Longer oxidation times resulted in a decrease in the size of the unreacted cores in the particles and an increase in the amount of iron migration to the outsides of the particles. (Right side) Two distinct phases were visible in the iron-enriched rim on the outsides of the oxidised particles	26
Figure 12	High iron slag particle oxidised for 1 h at 850 °C displaying iron migration towards the edges of cracks leaving the adjacent areas enriched in titania and slightly porous. Particle core consisted of the $M_3O_5$ -solid solution. The glass phase depicted in micrograph (b) contained ilmenite	28
Figure 13	High iron slag particle oxidised at 850 °C for 3 h displaying a well defined zoned texture with $M_3O_5$ -rich inner core, $TiO_2$ -rich mantle and porous, iron-enriched outer rim	28

Figure 14	High magnesium slag (PFE418) particle oxidised for 1 h at 850 °C displaying $M_3O_5$ -rich core and $TiO_2$ -rich mantle with iron enrichment towards the edges of cracks and outer rim of the slag particle. Metallic iron precipitates were evident in the vicinity of internal cracks	29
Figure 15	High magnesium slag (PFE418) particle oxidised for 1 h at 850 °C displaying $M_3O_5$ -rich core with metallic iron precipitates associated with rutile along internal cracks extending through the particle	30
Figure 16	Standard slag (PFE437) particle, which had been oxidised at 850 °C for 1 h and reduced for 40 min at 800 °C displaying porosity and iron migration towards the outer margins of the particle. This particular slag particle had no unreacted core	31
Figure 17	High iron slag oxidised at 850°C for 3 h and reduced at 800°C for 30 minutes containing small unreacted $M_3O_5$ cores and broad $TiO_2$ mantles. Iron enrichment towards the outer margins of the particles can be observed	33
Figure 18	Oxidised and reduced high iron slag particle displaying iron enrichment towards the particle rim and along the edges of cracks extending through the particle	33
Figure 19	Optical micrograph of high magnesia slag which had been oxidised at 850 °C for 2 h and reduced at 800 °C for 30 min. Precipitated carbon associated with the particle is clearly visible	34
Figure 20	Standard slag, oxidised for 1 h at 850 °C; reduced at 800 °C for 40 min; leached for 5 h and calcined at 800°C for 2 h	35
Figure 21	High iron slag that was oxidised, reduced and then leached for 12 h	36
Figure 22	Optical micrograph of the high iron slag which was oxidised for 2 h at 850 °C, reduced for 30 min. at 800 °C, leached for 12 h and calcined. The exterior of the particles consisted predominantly of rutile and the interior predominantly of anatase	37
Figure 23	High magnesia slag, leached for 1 h; the effect of leaching is visible mainly at the outer margins of the individual slag particles	38
Figure 24	Optical micrograph of the high magnesia slag, which was oxidised for 2 h at 850 °C, reduced for 30 min. at 800 °C, leached for 12 h and calcined. The particle display a zoned appearance, in the center is an unreacted core surrounded by a mantle of anatase, while the rims consist of rutile	38
Figure 25	Summary of the morphological changes that occur during the production of BTS	39

## CHAPTER 4

Figure 26	Experimental set-up used for the roast experiments	43
Figure 27	Standard titania slag oxidised for ½ h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	46
Figure 28	Standard titania slag oxidised for 1 h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	46
Figure 29	Standard titania slag oxidised for 2 h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	47
Figure 30	Standard titania slag oxidised for 4 h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	47
Figure 31	Standard titania slag oxidised for 2 h at 850 °C in 8 % O <sub>2</sub> and reduced for 20 min in 100 % CO. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	48
Figure 32	Standard titania slag oxidised for 2 h at 850 °C in 8 % O <sub>2</sub> , reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	49
Figure 33	The influence of oxidation time during roasting of standard slag on BTS product grade. The slag was oxidised at 850 °C in 8 % O <sub>2</sub> , reduced in 100 % CO for 20 min and leached in boiling HCl for 12 h	50
Figure 34	The influence of oxidation time, during roasting of standard slag, on iron extraction during leaching. The slag was oxidised at 850 °C in 8 % O <sub>2</sub> , reduced in 100 % CO for 20 min and leached in boiling HCl for 12 h	51
Figure 35	The change in the oxidation state of iron during oxidation roasting of standard titania slag as determined by Mössbauer analysis	52
Figure 36	Changes in the relative concentration of the iron containing phases during oxidation roasting of standard titania slag as determined by Mössbauer analysis	52
Figure 37	The influence of oxygen concentration and temperature during oxidation of standard slag on BTS product grade. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl. The slag contained the equivalent of 85% TiO <sub>2</sub> before treatment	55

Figure 38	The influence of oxygen concentration during oxidation of standard slag at 850 °C on the total iron extraction during leaching. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl	56
Figure 39	The influence of roasting temperature and oxygen concentration during oxidation of standard slag on the rate of iron extraction during leaching. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl	56
Figure 40	The influence of reduction time, during roasting of standard slag, on BTS product grade. The slag was oxidised for 2 h at 850 °C in 8 % O <sub>2</sub> , reduced in 100 % CO and leached for 12 h in boiling 20 % HCl	58
Figure 41	The influence of reduction time during roasting of standard slag on the rate of iron extraction during leaching. The slag was oxidised for 2 h at 850 °C in 8 % O <sub>2</sub> , reduced in 100 % CO and leached for 12 h in boiling 20 % HCl	59
Figure 42	The changes in the oxidation state of iron during reduction as determined by Mössbauer analysis	60
Figure 43	The changes in the relative concentration of the iron containing phases in oxidised standard titania slag during reduction for various times as determined by Mössbauer analysis	60
Figure 44	The influence of the particle size distribution of standard slag on BTS product grade. The slag was oxidised at 850 °C for 2 h in 8 % O <sub>2</sub> , reduced for 20 min in 100 % CO and leached for 12 h in 20 % HCl	62
Figure 45	The influence of the particle size distribution of standard slag on iron extraction during leaching. The slag was oxidised at 850 °C for 2 h in 8 % O <sub>2</sub> , reduced for 20 min in 100 % CO and leached for 12 h in boiling HCl	62
Figure 46	The influence of oxidation time and the particle size distribution of standard slag on the rate of iron extraction during leaching. The slag was oxidised at 850 °C in 8 % O <sub>2</sub> , reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl	63
Figure 47	High iron titania slag oxidised for ½ h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	65
Figure 48	High iron titania slag oxidised for 1 h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	65
Figure 49	High iron titania slag oxidised for 2 h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	66

Figure 50	High iron titania slag oxidised for 4 h at 850 °C in 8 % O <sub>2</sub> . SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown	66
Figure 51	High iron titania slag oxidised for 2 h at 850 °C in 8 % O <sub>2</sub> and reduced for 20 min in 100 % CO. SEM micrographs as well as a chemical composition profile through one of the particles are shown	68
Figure 52	High iron titania slag oxidised for 4 h at 850 °C in 8 % O <sub>2</sub> , reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl. SEM micrographs as well as a chemical composition profile through one of the particles are shown	68
Figure 53	The influence of oxidation time, during roasting of high iron slag, on BTS product grade. The slag was oxidised at 850 °C in 8 % O <sub>2</sub> , reduced in 100 % CO for 20 min and leached for 12 h in boiling 20 % HCl	69
Figure 54	The influence of oxidation time, during roasting of high iron slag, on the rate of iron extraction during leaching. The slag was oxidised at 850 °C in 8 % O <sub>2</sub> , reduced in 100 % CO for 20 min and leached for 12 h in boiling 20 % HCl	69
Figure 55	The influence of oxygen concentration and temperature (in air-CO <sub>2</sub> mixtures) during oxidation of high iron slag on BTS product grade. The slag was oxidised for 2 h, reduced for 20 min in 100% CO and leached for 12 h in boiling 20 % HCl. The slag contained and equivalent of 72% TiO <sub>2</sub> before treatment	71
Figure 56	The influence of temperature and oxygen concentration during oxidation of high iron slag on the rate of iron extraction during leaching. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in 20 % HCl	71
Figure 57	The effect of particle size distribution on final BTS grade. The slag was oxidised at 850°C for 2 h in 8% O <sub>2</sub> , reduced for 20 min in 100% CO and leached for 12 h in 20% HCl	72
Figure 58	The influence of the particle size distribution of high iron slag on the rate of iron extraction during leaching. The slag was oxidised at 850°C for 2 h in 8% O <sub>2</sub> , reduced for 20 min in 100% CO and leached for 12 h in 20% HCl	72

## CHAPTER 5

Figure 59	Part of the Ti-O-Fe phase diagram at 1000 °C (compiled from phase diagrams produced by Lindsley, 1976 and Ericksson and Pelton, 1996). A star indicates the chemical composition of as-cast slag and the oxidation path of this material is indicated by a dotted line	76
-----------	--	----



Figure 60	The effect of temperature on the $\text{TiO}_2\text{-FeO-Fe}_2\text{O}_3$ phase diagram (Haggerty, 1976)	76
Figure 61	The isotherm of the $\text{Fe-Fe}_2\text{O}_3\text{-TiO}_2$ system at 800 °C (after Borowiec and Rosenqvist, 1981)	77
Figure 62	Summary of the phase and chemical changes that occurs in titania slag during oxidation	79
Figure 63	Proposed mechanism for the oxidation of titania slag	80
Figure 64	Slag roasted for 2 h at 850 °C. Oxygen was used to roast sample A, air was used for sample B and argon was used for sample C	82
Figure 65	Particle size changes during roasting of titania slag	84
Figure 66	Micrographs of the slag sample coated with gold after roasting in air at 850°C for 30 min	85
Figure 67	Variation of iron concentration, iron oxidation state and titanium oxidation state along a line through an oxidised slag particle	87
Figure 68	Titania slag oxidised for 45 min in 10 % $\text{O}_2$ at 850 °C and leached for different times under reducing conditions	88
Figure 69	Iron speciation in solution during leaching of oxidised titania slag	89
Figure 70	Iron speciation in solution during leaching of slag that was previously oxidised and reduction leached	90
Figure 71	Titania slag that was oxidised for 45 min in air, reduction leached for 1 h and roasted again in air for 2 h at 850°C	91
Figure 72	Titania slag that was oxidised for 45 min in air, reduction leached for 1 h and roasted again in argon for 2 h at 850°C	92
Figure 73	Titania slag that was oxidised for 45 min in air, reduction leached for 1 h and roasted again in carbon monoxide for 2 h at 850°C	93
Figure 74	Micrographs of slag oxidised at 1050 °C for various times. Micrographs of the samples after reduction for 20 min at 850 °C are also shown	95
Figure 75	WDS Line chemical analysis through a particle of standard titania slag that was oxidised at 1050 °C for 30 min in 10 % $\text{O}_2$	97
Figure 76	WDS Line chemical analysis through a particle of standard titania slag that was oxidised at 1050 °C for 60 min in 10 % $\text{O}_2$	98
Figure 77	Micrographs of a titania slag sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 850 °C for 2 h	99
Figure 78	Micrographs of a titania slag sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 1050 °C for 2 h	101



Figure 79	Micrographs of a recrystallised titania slag particle observed in the slag sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 1050 °C for 2 h	102
Figure 80	Micrographs of sintered titania slag particles observed in a sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 1050 °C for 2 h	103

## LIST OF TABLES

### CHAPTER 1

Table 1	Impact of feedstock impurities on chloride process unit operations	3
Table 2	SORELSLAG™ composition	5
Table 3	Upgraded Slag composition	6
Table 4	SORELSLAG™ composition	7
Table 5	QIT salt roasting product composition	7
Table 6	US Bureau of Mines slag composition	7
Table 7	Synthetic rutile product composition	8
Table 8	Feed slag composition to the sulphiding-sulphation process	8
Table 9	Impurity content of the residue from the sulphiding-sulphation process	8
Table 10	Feed slag to the chlorination process	9
Table 11	Chlorination process product composition	9

### CHAPTER 2

Table 12	Chemical composition of the feed slags	11
Table 13	The effect of different pre-treatments and slag compositions on the leachability of impurities	13
Table 14	The effect of phosphate fluxing on the leachability of impurities from titania slag	14
Table 15	The leach results presented in Table 14 normalised to a silica free basis	14
Table 16	Phase-chemical composition of the slag, given in order of decreasing abundance	16

### CHAPTER 3

Table 17	Concentration of selected species in the feed slags used for this investigation	17
Table 18	Coal analysis	17
Table 19	Variables investigated during the roast study	18
Table 20	Variables investigated during the leach study	18
Table 21	Results of the roast investigation on feed slag PFE437	19
Table 22	Phase-chemical composition of the feed slag samples as determined by XRD, given in order of decreasing abundance. The chemical compositions used to classify the slags are also given	23
Table 23	Phase-chemical compositions of slag PFE437 after oxidation at different temperatures and times, given in order of decreasing abundance	24



Table 24	Phase chemical composition of slag PFE657 after oxidation at 850 °C for different times, given in order of decreasing abundance	24
Table 25	Phase-chemical composition of the high iron containing slag PFE436 after oxidation at 850 °C with increasing time, given in order of decreasing abundance	27
Table 26	Phase-chemical composition of the high magnesium slag PFE418 after oxidation at 850 °C with increasing time, given in order of decreasing abundance	29
Table 27	Phase-chemical composition of standard slag (PFE437) which had been oxidised and reduced at different temperatures and times, given in order of decreasing abundance	31
Table 28	Phase-chemical composition of the high iron slag (PFE436) which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min; given in order of decreasing abundance	32
Table 29	Phase-chemical composition of the high magnesia slag (PFE418) which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min; given in order of decreasing abundance	33
Table 30	Phase-chemical composition of standard slag that had been oxidised and reduced at different temperatures and times, leached for 5 h and calcined at 800 °C for 2 h, given in order of decreasing abundance	35
Table 31	Phase-chemical composition of high iron slag which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min, leached for different times and calcined; given in order of decreasing abundance	36
Table 32	Phase-chemical composition of high magnesia slag which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min, leached for different times and calcined; given in order of decreasing abundance	37

#### CHAPTER 4

Table 33	Chemical composition of the feed slags used in this investigation	42
Table 34	Phase-chemical composition of the feed slags used in this investigation	42
Table 35	The phase-chemical composition, as determined by XRD, of standard slag after oxidation. The samples are categorised by the oxidation time used. For the experiments listed the slag was oxidised at 850 °C in 8 % O <sub>2</sub>	45

Table 36	The phase-chemical composition, as determined by XRD, of standard slag after oxidation and reduction. The samples are categorised by the oxidation time used. For the experiments listed the slag was oxidised at 850 °C in 8 % O <sub>2</sub> and reduced in 100 % CO for 20 min	48
Table 37	The phase-chemical composition, as determined by XRD, of standard slag after oxidation, reduction and leaching. The samples are categorised by the oxidation time used. For the experiments listed the slag was oxidised at 850 °C in 8 % O <sub>2</sub> , reduced in 100 % CO for 20 min and leached in 20 % HCl for 12 h	49
Table 38	Phase chemical analysis, as determined by XRD, of the oxidation samples used for the Mössbauer investigation	51
Table 39	The phase chemical composition as determined by XRD of standard slag after oxidation, reduction and leaching. The samples are categorised by the oxidation atmosphere that was used. The slag was oxidised at 850 °C for 2 h, reduced in 100 % CO for 20 min and leached in boiling 20 % HCl for 12 h	53
Table 40	The phase-chemical composition as determined by XRD-analysis of standard slag after oxidation, reduction and leaching. The samples are categorised by the roasting temperature that was used. The slag was oxidised in 8 % O <sub>2</sub> for 2 h, reduced in 100 % CO and leached for 12 h in boiling 20 % HCl	54
Table 41	The phase-chemical composition, as determined by XRD, of standard slag after oxidation, reduction and leaching. The samples are categorised by the retention time during reduction. The slag was oxidised at 850 °C for 2 h in 8% O <sub>2</sub> , and reduced in 100 % CO	57
Table 42	Phase chemical composition, as determined by XRD, of the oxidised and reduced samples submitted for Mössbauer analysis	59
Table 43	The phase-chemical composition, as determined by XRD, of standard slag after oxidation, reduction and leaching. The samples are categorised by the size distribution used. The slag was oxidised at 850 °C for 2 h in 8 % O <sub>2</sub> , reduced in 100 % CO for 20 min and leached in boiling 20 % HCl for 12 h	61
Table 44	The phase-chemical composition of high iron titania slag after oxidation. The samples are categorised by the retention time during oxidation. The slag was oxidised at 850 °C in 8 % O <sub>2</sub>	63

Table 45	The phase-chemical composition of high iron titania slag after oxidation and reduction. The samples are categorised by the retention time during oxidation. The slag was oxidised at 850 °C in 8 % O <sub>2</sub> and reduced in 100 % CO for 20 min	67
Table 46	The phase-chemical composition, as determined by XRD, of high iron slag after oxidation, reduction and leaching. The samples are categorised by the retention time during oxidation. For the experiments listed the slag was oxidised at 850°C in 8 % O <sub>2</sub> , reduced for 20 min in 100 % CO and leached in 20 % HCl for 12 h	67

## CHAPTER 5

Table 47	Phase composition of the slag samples roasted in 100 % O <sub>2</sub> , air and argon at 850 °C for 2 h	81
Table 48	Porosity of slag particles before and after the roasting stages	84
Table 49	Phase composition of the gold coated titania slag roasted in air at 850 °C for 30 min	85
Table 50	Phase-chemical compositions as determined by XRD of oxidised and reduction leached titania slag after roasting in various atmospheres	94
Table 51	Phase-chemical compositions of slag samples roasted at 1050 °C	96
Table 52	Mössbauer analysis of slag samples oxidised at 1050 °C and reduced at 850 °C	97
Table 53	Phase-chemical composition as determined by XRD-analysis of the samples subjected to interrupted roasting	99
Table 54	Quantitative WDS analyses of selected phases in oxidised slag	104

# PROSESONTWIKKELING VIR DIE PRODUKSIE VAN OPGEGRADEERDE TITAAN SLAK

deur

**Jacobus Philippus van Dyk**

Vir die graad Philosophiae Doctor aan die Departement Materiaalkunde en  
Metallurgiese Ingenieurswese by die Universiteit van Pretoria

Studieleier: PC Pistorius

*Sleutelwoorde: titaanslak, chloriedproses, rutiel, opgegradeerde slak, pigment,  
ystermigrasie, oksidasie, titaandioksied, pigment, ilmeniet, anataas*

## OPSOMMING

Daar is 'n reeks voermateriale beskikbaar vir die produksie van  $\text{TiO}_2$  pigment. Dit wissel van natuurlike voermateriale soos ilmeniet en rutiel to sintetiese rutiel. Daar is 'n sterk toename in die prys van titaanryke voermateriale soos die  $\text{TiO}_2$  graad van die materiale toeneem. 'n Proses is ontwikkel om voordeel te trek uit die prysverskil tussen chloriedgraad slak en natuurlike rutiel. Die proses verhoog die  $\text{TiO}_2$  inhoud van die slak van ~85% na meer as 95%. Hierdie "beneficiated titania slag" (BTS) lyk na 'n ideale voermateriaal vir die chloried proses.

Aanvanklik is verskeie prosesse geëvalueer. Daar is veral klem gelê op die voorafbehandeling van die slak. Dit was nodig omdat die onsuiverhede in slak baie moeilik loog. Deur van 'n geskikte voorafbehandeling gebruik te maak kan die onsuiverhede maklik loogbaar gemaak word, terwyl die titaan grootliks nie-loogbaar bly. Die resultate het getoon dat 'n proses wat uit oksidasie- en reduksie roostering bestaan gevolg deur loging, die grootste kans op sukses het.

Die eerste deel van die prosesontwikkeling is in 'n steenkoolgevuurde fluidbedrooster gedoen. Die prosesparameters was gedeeltelik geoptimeer, omdat daar kon slegs BTS met 'n  $\text{TiO}_2$  inhoud van 94% gemaak kon word. Die daaropvolgende prosesontwikkeling is in 'n klein roostereaktor gedoen wat gekoppel was aan 'n gasmengstelsel. Dit het beter beheer oor die roostertoestand toegelaat. Die proses parameters is hiermee geoptimeer na: oksidasie by 850 °C vir 1.5 h in 8%  $\text{O}_2$ ; reduksie by 850 °C vir 10 min in 100% CO en loging in 20% kokende soutuur. Onder hierdie prosesondisies is BTS met 'n graad van > 97%  $\text{TiO}_2$  geproduseer.

# PROCESS DEVELOPMENT FOR THE PRODUCTION OF BENEFICIATED TITANIA SLAG

by

**Jacobus Philippus van Dyk**

For the degree Philosophiae Doctor in the Department Materials Science and  
Metallurgical Engineering at the University of Pretoria

Study leader: PC Pistorius

*Key words: titania slag, chloride process, rutile, upgraded slag, beneficiation,  
pigment, iron migration, oxidation, titanium dioxide, ilmenite*

## ABSTRACT

There is a range of feed materials available for the production of TiO<sub>2</sub> pigment. These range from natural materials like ilmenite and rutile to synthetic materials like synthetic rutile. There is a large increase in the price of titaniferous feed materials as the TiO<sub>2</sub> content of the material increases. To take advantage of the difference in price between chloride grade slag and natural rutile a process was developed to increase the TiO<sub>2</sub> content of chloride grade slag from ~85% to more than 95%. This beneficiated titania slag product (BTS) should be ideal as feed material to the chloride pigment process.

Initially several processes were evaluated. Particular emphasis was placed on the slag pre-treatment procedure. This was necessary as impurities could only be leached with difficulty from as-cast slag. A suitable pre-treatment procedure would render the impurities easily leachable, while the titanium is retained in an insoluble form. The results indicated that a process consisting of oxidation and reduction roasting would satisfy these requirements.

Detailed process development was then undertaken on this process. The first phase of the process development was conducted in a coal fired fluid bed roaster. This allowed a set of semi optimised process parameters to be established, but the highest TiO<sub>2</sub> content that could be achieved was 94%. A second stage of process development was under taken under more controlled conditions, using a small fluid bed reactor connected to a gas mixing system. Based on the results in this phase of the process development a new set of optimum process parameters was established. They are oxidation at 850 °C for 1.5 h in an atmosphere containing 8% O<sub>2</sub>; reduction at 850 °C for 10 min in a 100% CO atmosphere and leaching in boiling 20 % hydrochloric acid for 12 h. Under these conditions it was possible to produce BTS containing > 97% TiO<sub>2</sub>.

During oxidation of titania slag several important morphological changes occur. These are the conversion of the original  $M_3O_5$  phase in the slag to a mixture of rutile/anatase, hematite and ferric  $M_3O_5$ . In the process the iron in the slag migrates to the outside surfaces of the slag particles where it is easily accessible during leaching. The iron containing phases are converted to ilmenite during reduction and during leaching the ilmenite is removed. This yields the BTS product. As the oxidation roast appeared to be a very important of the BTS process it was decided to investigate the mechanism of titania slag oxidation. A mechanism based on the nucleation energy that is required to form the relevant phases during oxidation was proposed. This mechanism was tentatively confirmed through selected experiments.



## 1. INTRODUCTION

Iscor Heavy Minerals (IHM) commissioned Iscor Consulting Services to do a feasibility study on the production of Beneficiated Titania Slag (BTS) in July 1997. BTS is a rutile substitute product that contains more than 95 %  $\text{TiO}_2$ . IHM's business strategy currently entails the production of a number of different products including concentrates of rutile, ilmenite, monazite and zircon as well as pig iron and titania slag from the smelting of ilmenite. BTS will be a natural addition to this product range and it will provide IHM with a premium product that is highly sought after in the market. This study was initiated as part of the feasibility study to develop a process for the production Beneficiated Titania Slag.

### 1.1 The occurrence and uses of titanium dioxide

Titanium is one of the most abundant elements in the earth's crust but deposits of titanium that are of sufficient concentration to be commercially viable are scarce (Lurie, 1987). The main ore minerals are rutile and ilmenite. Rutile is a crystalline form of titanium dioxide ( $\text{TiO}_2$ ) and contains around 95 %  $\text{TiO}_2$ . Commercially viable deposits of rutile are relatively scarce. Ilmenite ( $\text{FeTiO}_3$ ) deposits are far more abundant but they have a significantly lower  $\text{TiO}_2$  content. Depending on the geological history of the deposit ores can range in composition from 40 to 80 %  $\text{TiO}_2$ . Most deposits being mined produce concentrates with a  $\text{TiO}_2$  content between 59 and 67 %. The main impurity is iron but chromium, manganese, vanadium and magnesium can also be present among others. The ores from deposits with a  $\text{TiO}_2$  content higher than 70 % are weathered ilmenites known as leucoxene. Leucoxene is exploited on a limited commercial basis. Deposits of anatase, a polymorph of rutile, have been discovered but they have not yet been commercially exploited. Other natural occurring minerals that contain titanium are brookite (rhombic  $\text{TiO}_2$ ), perovskite ( $\text{CaTiO}_3$ ), sphene ( $\text{CaTiSiO}_5$ ) and geikielite ( $\text{MgTiO}_3$ ).

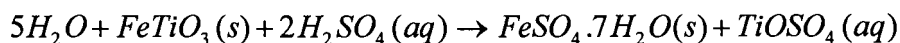
The primary use of titanium is as titanium dioxide ( $\text{TiO}_2$ ) in white pigment (Gambogi, 1991). This is used in paints, plastics and paper. Titanium dioxide pigment is superior as a white pigment due to its high refractive index and the resulting light scattering ability that provides brightness. Two pigments with a tetragonal crystal structure are produced, rutile and anatase. Rutile pigment is less reactive with other paint components when exposed to sunlight and is preferred in outdoor paints. Anatase, with a bluer tone, is used in indoor paints and paper.  $\text{TiO}_2$  also finds other minor applications in welding rod coatings, in fluxes used for continuous casting of steel, in heavy aggregates and in ceramics.

### 1.2 Pigment production processes

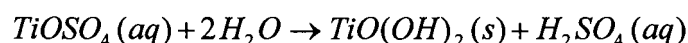
Two processes have been developed to purify naturally occurring minerals or titaniferous slag to the required pigment purity (> 99.9 %  $\text{TiO}_2$ ). These processes are the sulphate process and the chloride process. The sulphate process is the oldest and was developed in the 1930s while the chloride process was developed in the early 1950s.

### 1.2.1 The sulphate process

The raw materials required by this process, ilmenite or titania slag, are reacted with sulphuric acid at 150-180 °C in a batch process according to the following reaction (Gambogi, 1991):



If ilmenite is used as a raw material, some ferric sulphate usually forms during leaching and this is reduced to ferrous sulphate with the addition of scrap iron. The undissolved solids are removed during clarification and the liquid is cooled and concentrated by evaporation under vacuum. Copperas ( $FeSO_4 \cdot 7H_2O$ ) is precipitated during this treatment. This part of the process is omitted when slag is used as a starting material. The concentrated solution is heated to 90 °C to hydrolyse the titanyl sulphate to insoluble titanyl hydroxide,

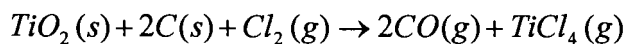


The precipitate is washed with sulphuric acid and water to remove all traces of discolouring elements such as chromium, iron, manganese and vanadium. Seed crystals are then added to ensure rutile is formed. If seed crystals are not added anatase is obtained. Finally the  $TiO(OH)_2$  is calcined at 1000°C to yield  $TiO_2$  pigment.

A sulphate process plant is easier to operate and maintain than a chloride process plant and it is able to use feedstock with a relatively low  $TiO_2$  content. However due to the larger number of equipment pieces required the capital cost for a modern sulphate process plant can be higher than a chloride process plant of the same pigment capacity. There is also a higher volume of waste products to treat and dispose of due to the use of impure feedstock and the lack of economic sulphate recycling processes. A final consideration is that rutile is insoluble in sulphuric acid. This rules out a large number of potential feedstocks (for example the various synthetic rutile products).

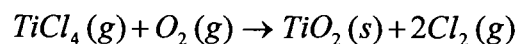
### 1.2.2 The chloride process

The chloride process is a modern continuous chemical process that uses a small number of complex unit operations. The feedstock to the process, high grade titania slag or rutile, is chlorinated in a fluidised-bed reactor at 925-1010 °C in the presence of coke (Bull, 1992),



This is an exothermic reaction that sustains itself. The resulting off-gas, which contains titanium tetrachloride, is purified by distillation. The off-gas is cooled to a temperature just above the boiling point of titanium tetrachloride. Most of the chlorides with low volatilities (such as iron, chromium and manganese) condense and are removed. Titanium tetrachloride condenses during cooling and the CO and CO<sub>2</sub> gases are scrubbed before being released to the atmosphere. Some impurities, like vanadium oxychloride, are not removed during distillation. These impurities are

removed further downstream. Distillation is repeated to selectively remove chlorides with high volatilities. The purified titanium tetrachloride is oxidised to  $TiO_2$  and chlorine is liberated,



The oxidation reaction must be executed above  $1000^\circ C$  to produce rutile of the correct particle size distribution. It is necessary to produce a very narrow particle size distribution in the oxidation reactor to minimise sintering of particles. Growth on the walls of the reactor must also be prevented because it leads to oversized particles. Another function of the reactor is to dissipate the heat of the exothermic reaction to allow the oxidation reaction to proceed with a high efficiency.

Following oxidation, the gas is cooled and the pigment particles separated and collected. The chlorine gas is recycled to the chlorination stage and the pigment particles are forwarded to a post-treatment stage to give special properties to different grades. The post-treatment stage has recently become very important because it enhances the properties of the base pigment by surface treatment or coating processes. These processes are very complex and account for about a third of the total capital cost of a manufacturing plant.

### 1.2.3 Feedstock requirements for the chloride process

The chloride process requires feedstock with a lower impurity content than those suitable for the sulphate process. This is necessary as a result of the complex and non-selective nature of the unit processes performed at high temperatures under aggressive conditions. Table 1 summarises the impact of various feedstock impurities on the chloride process unit operations (Hollitt, 1995 and Fisher, 1997).

**Table 1.** Impact of Feedstock Impurities on Chloride Process Unit Operations

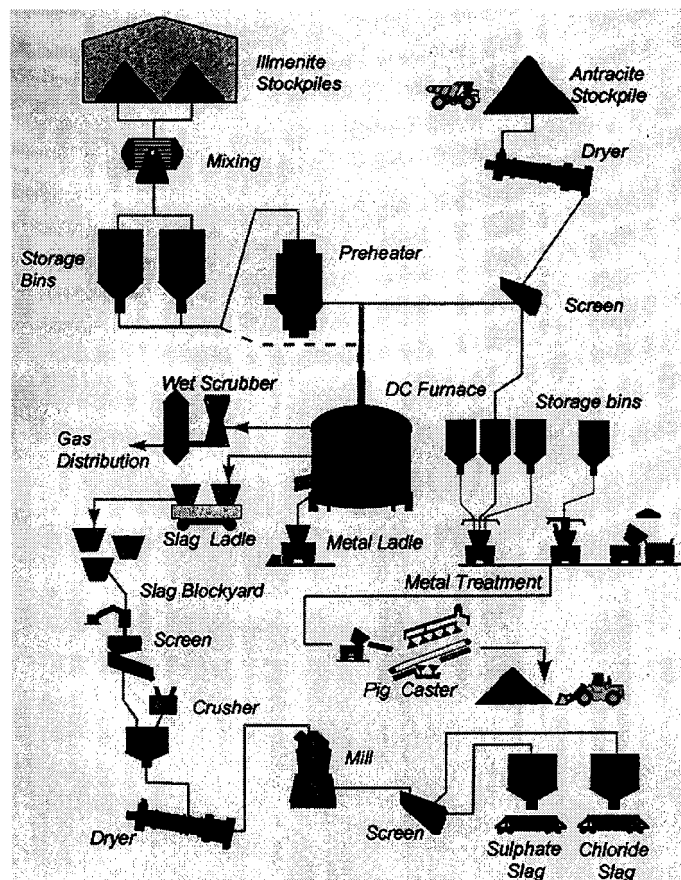
Impurity	Impact on Chloride Process Unit Operation
FeO, MnO	Consume chlorine, coke and increase gas volumes. Form solid/liquid chloride sludge which fouls the ducting.
Na <sub>2</sub> O, K <sub>2</sub> O, CaO, MgO	Form liquid chlorides that consume coke and chlorine and defluidise the fluid bed reactor by the formation of a viscous mass that plugs the chlorinator.
Al <sub>2</sub> O <sub>3</sub>	Consumes chlorine and coke, causes corrosion and forms sludge. A process step to convert Al impurities to a solid is usually included.
SiO <sub>2</sub>	Accumulates in the chlorinator, reducing campaign life and throughput, encourages blockages in the ducting, condenses partly with TiCl <sub>4</sub> and requires product distillation.
V <sub>2</sub> O <sub>5</sub>	Follows TiCl <sub>4</sub> , therefore it requires additional chemical treatment and distillation.
SnO <sub>2</sub>	SnCl <sub>4</sub> builds up during the oxidation step and can cause problems in the TiCl <sub>4</sub> recycle streams. Restricts sales outlets for TiCl <sub>4</sub> .
Other heavy metal oxides	Show up in solid or liquid waste streams and create problems with economic upgrading and sale of co-products, or with their disposal in land fill sites.
As <sub>2</sub> O <sub>5</sub>	Follows TiCl <sub>4</sub> and ends up in the final TiO <sub>2</sub> product, rendering the pigment unsuitable for some applications.
Th, Ra	Accumulate in the chlorinator brickwork and the radioactive nature causes disposal difficulties.

The characteristics of the preferred feedstock to the chloride process can be summarised as follows (Fisher, 1997):

- $\text{TiO}_2$  content as high as possible (ideally more than 95 %  $\text{TiO}_2$ );
- $\text{SiO}_2$  content as low as possible (ideally none);
- Bulk density of  $2.5 \text{ g/cm}^3$ ;
- Less than 50 ppm of uranium and thorium;
- Alkaline oxide content very low (ideally none) and;
- Heavy metals, arsenic and tin present only in trace amounts.

### 1.3 The production of titania slag

The  $\text{TiO}_2$  content of ilmenite can be increased by various upgrading processes before it is used as a feedstock to one of the pigment processes. The oldest ilmenite concentration process is thermal reduction (Minckler and Baroch, 1981) and involves smelting the ilmenite ore to produce pig iron and a titanium rich slag. Smelting is currently being done by Richardsbay Minerals and Namakwa Sands in South Africa, Tinfos in Norway and Quebec Iron and Titanium (QIT) in Canada (Fisher, 1997). Titania slag produced from South African ilmenite is low in deleterious impurities and is acceptable as a feedstock to the chloride process. In contrast the slag produced from Norwegian and Canadian ilmenite has high levels of alkaline earth impurities such as Ca and Mg and is only suitable as feedstock to the sulphate process.



**Figure 1.** Flow diagram of the proposed IHM ilmenite smelting plant (IHM brochure, 1997).

Figure 1 shows a flow diagram of the smelting plant proposed by IHM. Ilmenite and anthracite will be fed into a 50 MVA DC plasma furnace operating at temperatures in excess of 1600 °C. Fluidity in the slag is maintained by keeping around 10 % FeO in the slag. The FeO in the slag may also have the possible additional benefit of assisting in the control of the oxygen potential in the furnace. Strongly reducing conditions will result in the undesirable reduction of TiO<sub>2</sub> to Ti<sub>2</sub>O<sub>3</sub>, Ti<sub>3</sub>O<sub>5</sub> and even TiO. The formation of these suboxides may increase the viscosity of the slag substantially which can cause operational difficulties like frothing. Molten iron and slag will be periodically tapped from the furnace. After tapping the molten iron will be cleaned at a metal treatment station before being cast into pigs. The slag will be cast into blocks and left to cool for up to 10 days. Thereafter the slag will be crushed, dried, milled and screened to produce two size fractions. The larger fraction (+106 µm-850 µm) will be sold as feedstock to the chloride process pigment producers, while the smaller fraction (-106 µm) will be sold to sulphate process pigment producers.

#### 1.4 Slag upgrading processes

Several processes have been proposed in the past to upgrade low grade titania slag to a product that is a suitable feed stock for the chloride process. The processes can be divided into the following groups:

- Oxidation and reduction roasting followed by leaching;
- Oxidation roasting followed by leaching;
- Salt roasting followed by leaching;
- Oxidation and fluxing of molten slag followed by leaching;
- Sulphation and sulphidation roasting followed by leaching and;
- Chlorination.

These processes will be discussed in more detail below.

##### 1.4.1 Oxidation and reduction roasting followed by leaching

In 1996 a plant based on oxidation and reduction roasting followed by leaching was commissioned in Canada by QIT for the upgrading of SORELSLAG™ slag (Doan, 1996). SORELSLAG™ is produced from Allard Lake ilmenite that contains relatively high levels of alkaline earth impurities such as CaO and MgO. Table 2 gives the chemical composition of SORELSLAG™ (Borowiec et al., 1996).

**Table 2. SORELSLAG™ composition (wt %)**

TiO <sub>2</sub>	Fe <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	SiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
84.8	3.76	3.62	0.47	5.89	0.26	3.06	0.027	0.65

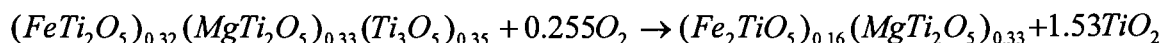
(\* total Ti reported as TiO<sub>2</sub> regardless of valence state)

SORELSLAG™ consists mainly of the pseudobrookite solid solution with a minor amount of glassy silicate. Pseudobrookite is a solid solution of iron and titanium oxides with the general formula M<sub>3</sub>O<sub>5</sub>. The MgO impurity is present mostly in the pseudobrookite phase, while the CaO impurity occurs in the glassy silicate phase.

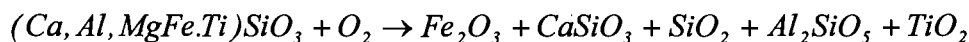


These phases are inherently inert toward the action of mineral acids and this makes the slag difficult to upgrade. The Upgraded Slag (UGS) process modifies the phase composition of the slag to increase the leachability of the impurities.

The first step of the process consists of sizing the slag by grinding, screening and classification to the 75-850  $\mu\text{m}$  size range with a mean particle size between 250 and 350  $\mu\text{m}$ . The slag is then oxidised in a fluid bed roaster at 1025  $^{\circ}\text{C}$  for 1 h. During oxidation all the Ti(III)-oxide in the slag is converted to Ti(IV)-oxide and the Fe(II) oxide is converted to Fe(III) oxide. These reactions can be represented by the following equation:



The oxidation results in a major rutile ( $TiO_2$ ) phase and a minor pseudobrookite phase ( $M_3O_5$ ). The glassy silicate phase decomposes into wollastonite ( $CaSiO_3$ ) and tridymite ( $SiO_2$ ). The decomposition of the glassy silicate phase is triggered by the oxidation of FeO and can be represented by the following equation:



Following oxidation the slag is reduced in a fluid bed roaster at 850  $^{\circ}\text{C}$  for 1 h. Reduction of the oxidised slag takes place in two stages. In the initial stage the Fe(III) oxide is converted to Fe(II) oxide. In the second stage an MgO-enriched ilmenite-geikielite solid solution and a MgO deficient residual pseudobrookite phase and a rutile phase are formed. These changes are accompanied by the creation of a large number of pores and other defects in the crystal lattice.

Next the roasted slag is cooled before it is leached with 18-20 % HCl at 150  $^{\circ}\text{C}$  in a pressure vessel for 7 h. During leaching the impurities are removed to form soluble chlorides leaving an upgraded residue.

The leach residue is separated from the spent leach liquor, washed and calcined at 800  $^{\circ}\text{C}$  to remove moisture and residual acid. The resulting upgraded slag is a granular product with  $TiO_2$  content around 95 %  $TiO_2$ . Table 3 gives a typical composition of UGS.

**Table 3.** Upgraded Slag composition (wt%)

$TiO_2$	FeO	$Al_2O_3$	CaO	MgO	MnO	$SiO_2$	$Cr_2O_3$	$V_2O_5$
94.9	2.47	0.46	0.06	0.67	0.03	1.69	0.06	0.35

#### 1.4.2 Oxidation roasting followed by leaching

Two processes have been proposed to upgrade titania slag by oxidation followed by leaching. In the first process (Leddy and Schecter, 1962) SORELSLAG<sup>TM</sup> is oxidised at 900  $^{\circ}\text{C}$  for 6 h to oxidise all the Ti(III)-oxide to Ti(IV)-oxide. The oxidised slag is then pressure leached at 200  $^{\circ}\text{C}$  in 33 % HCl for 6 h. By using this process SORELSLAG<sup>TM</sup> with a  $TiO_2$  content of 70 % was upgraded to slag product containing 90 %  $TiO_2$  and less than 0.2 % CaO and MgO. A serious disadvantage of this

process is that the feed slag has to be crushed to below 75  $\mu\text{m}$  before leaching. This makes the material too fine for use in the chloride pigment process.

In second process titania slag is roasted at 800  $^{\circ}\text{C}$  - 1200  $^{\circ}\text{C}$  to oxidise the Ti(III)-oxide to Ti(IV)-oxide and the Fe(II)-oxide to Fe(III)-oxide (Tikkanen and Tholand, 1960; Tikkanen et. al., 1964). This induces cracks in the grains. The slag is then leached at 100  $^{\circ}\text{C}$  with 50 %  $\text{H}_2\text{SO}_4$  in the presence of a reducing agent such as Cu for 3 h. This process has not found any commercial application due to the fact that a cost effective reducing agent still has to be found.

### 1.4.3 Salt roasting followed by leaching

QIT proposed a process for the upgrading of SORELSLAG<sup>TM</sup> based on roasting with an alkali salt followed by leaching (Jarish, 1977). Table 4 gives the composition of SORELSLAG<sup>TM</sup> used for this process.

**Table 4.** SORELSLAG<sup>TM</sup> composition (wt %)

TiO <sub>2</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	SiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
70.3	12.21	6.0	1.0	5.1	0.24	5.7	0.18	0.58

At first the slag is ground to  $-45 \mu\text{m}$  before it is mixed with an alkali salt such as NaOH in the ratio 0.3:1 to 0.6:1. The mixture is then roasted for 2 h at 900  $^{\circ}\text{C}$ . This converts most of the impurities to alkali compounds, some of which are soluble in water and others which are soluble in mineral acids. After roasting the agglomerates that formed are dispersed by wet milling for 30 min. At the same time the water soluble alkali chromate and vanadate dissolve. The remaining impurities are then removed by leaching in two stages with  $\text{H}_2\text{SO}_4$ . Finally the residue is separated from the spent leach solution, washed and calcined at 900  $^{\circ}\text{C}$  for 30 min. Table 5 gives the composition of the calcined slag.

**Table 5.** QIT salt roasting product composition (wt %)

TiO <sub>2</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	SiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
96.2	2.12	0.47	0.26	0.43	0.20	0.40	0.02	0.03

### 1.4.4 Oxidation and fluxing of molten slag followed by leaching

The U.S. Bureau of Mines (Elger et al., 1974) developed a modified ilmenite smelting process in the 1970s that produces a synthetic rutile product. In the first stage of the process ilmenite ore is smelted in an electric arc furnace in the presence of CaO as a fluxing agent at temperatures around 1350  $^{\circ}\text{C}$ . This results in titania slag that contains mainly pseudobrookite ( $\text{M}_3\text{O}_5$ ) and perovskite ( $\text{CaTiO}_3$ ) along with some glassy silicates. Table 6 gives a typical slag composition.

**Table 6.** U.S. Bureau of Mines slag composition (wt %)

TiO <sub>2</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	SiO <sub>2</sub>
67.4	3.41	4.59	4.64	3.33	7.04	6.68

(\* total Ti reported as TiO<sub>2</sub> regardless of valence state)

The molten slag is then oxidised before its is fluxed with a phosphate compound (for example  $P_2O_5$ ) at around 1400 °C. After the fluxing the slag is quenched to produce a slag that consists only of rutile crystals embedded in a phosphate-rich glass phase. During the fluxing all of the impurities in the slag are partitioned to the glass phase. The glass along with the impurities is removed during an atmospheric leaching step with either  $H_3PO_4$  or  $H_2SO_4$ . This leaves a synthetic rutile product that is a suitable feedstock for the chloride pigment process. Table 7 gives a typical composition of the final product. This process has been demonstrated on a pilot plant scale, but it is not used commercially.

**Table 7.** Synthetic rutile product composition (wt %)

TiO <sub>2</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>
96.5	1.26	1.5	0.06	0.03	0.001	0.04	0.04

#### 1.4.5 Sulphidation and/or sulphation roasting followed by leaching

The U.S. Bureau of Mines proposed a process for the upgrading of titania slag (Elger and Holmes, 1982) based on sulphating the impurities. The process consists of grinding the slag to below 220 µm, mixing it with  $NaCO_3$  and agglomerating it with a rotating disc pelletiser. The pellets are then reacted with  $SO_3$  or a mixture of  $SO_2$  and  $O_2$  in vertical shaft furnace at 700 °C for 7 h. This converts most of the alkaline earth oxides in the slag to sulphates. These impurities are consequently removed during a water leach procedure. The final product is suitable as feed to the chloride pigment process. Nafziger and Elger (1987) reported that this process was most successful at upgrading slag with an FeO content below 5 %. They also found that the removal of Mg and Mn was difficult and depended strongly on sufficient oxidation of the slag during roasting.

Borowiec et al. (1987) proposed a sulphiding-sulphation process for the upgrading of titania slag. Table 8. gives a typical composition of the feed slag to this process.

**Table 8.** Feed slag composition to the sulphiding-sulphation process (wt %)

TiO <sub>2</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	SiO <sub>2</sub>	C <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
75	7.6	1.2	1.0	4.7	5.3	0.1	0.3

(\* total Ti reported as TiO<sub>2</sub> regardless of valence state)

In the first part of the process the molten slag is treated with about 5 %  $Na_2S$ . This produces a sulphide phase in the slag that contains most of the Fe and Mn, along with the normal pseudobrookite and glass phases. After cooling and crushing of the slag the sulphide phase is removed by leaching the slag with sulphurous acid at room temperature for 1 h. The second part of the process consists of sulphating the leached slag in a vertical tube furnace with a mixture of  $SO_2$  and air at 800 °C for 4 h. During this procedure sulphates of the remaining impurities are formed. The sulphates are removed in by leaching at room temperature with water. The residual impurity content is given in Table 9.

**Table 9.** Impurity content of the residue from the sulphiding-sulphation process (wt%)

FeO	CaO	MgO	V <sub>2</sub> O <sub>5</sub>
0.47	1.3	0.47	0.07



### 1.4.6 Chlorination

QIT proposed a process for the upgrading of titania slag based on chlorination of the impurities (Gueguin, 1986; 1990; 1991; 1995). Titania slag, containing alkaline earth impurities and some titanium as  $Ti_2O_3$  (Table 10), is preheated in an inert atmosphere to 850 °C before it is contacted with chlorine gas in a fluid bed reactor.

**Table 10.** Feed slag to the chlorination process (wt %)

TiO <sub>2</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	SiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
81.4	8.97	2.91	0.20	5.27	0.27	1.98	0.18	0.64

(\* total Ti reported as TiO<sub>2</sub> regardless of valence state)

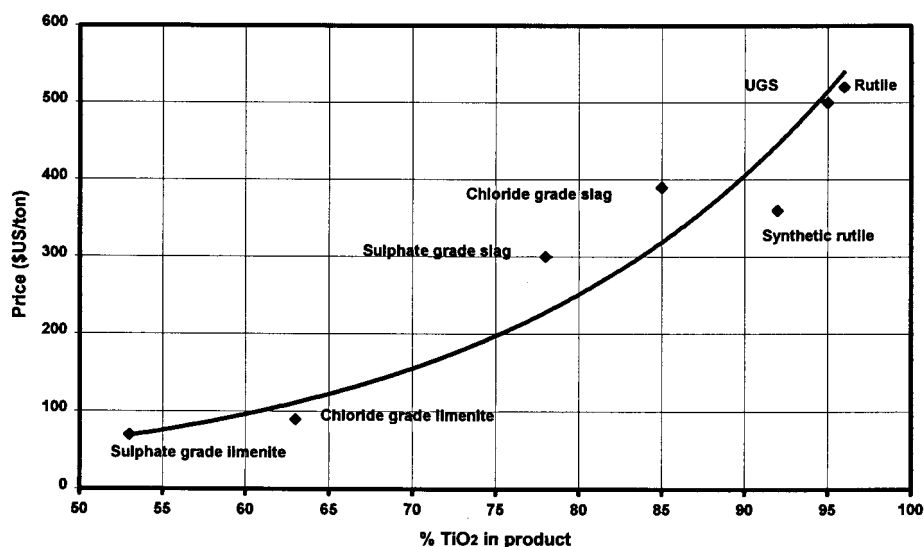
The reaction of  $Ti_2O_3$  with the chlorine and the impurities is an exothermic reaction that lowers the energy cost required for the chlorination reaction. During chlorination the iron and other impurities are distilled as chlorides. After chlorination the product is cooled to room temperature. The resulting product has a porous texture and consists mainly of rutile along with small amounts of aluminium and magnesium titanates. The titanates are removed by leaching the chlorinated slag with 33 % HCl at 210 °C for 4 h. Table 11 gives the final product composition.

**Table 11.** Chlorination process product composition (wt %)

TiO <sub>2</sub>	FeO	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	SiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
96.4	0.45	1.02	0.07	0.51	0.04	0.78	0.07	0.29

### 1.5 The motivation for upgrading chloride grade titania slag

All the known slag upgrading processes have as their aim the upgrading of sulphate grade titania slag to chloride grade titania slag for economic and environmental reasons. The main reason for upgrading chloride grade titania slag to a synthetic rutile product (as proposed here) is also an economic one. Figure 2 gives the 1997 prices for titaniferous feedstocks (Gambogi, 1998).



**Figure 2.** Titaniferous feedstock prices

There is a \$ 110 price difference between the price of chloride grade slag (85 %  $\text{TiO}_2$ ) and UGS (95 %  $\text{TiO}_2$ ). The reason for this price difference can be related to the quantity of effluent generated by the chloride process when these feedstocks are used. A higher purity feedstock generates less waste and this makes it easier to comply with environmental regulations. Based on this analysis there appears to be a need for a chloride grade slag upgrading process. This study reports on the development of such a process.

## 2. PRELIMINARY INVESTIGATION

Several possible process routes for the production of beneficiated titania slag were identified in the previous chapter. All of these processes were however designed to upgrade sulphate grade titania slag. The aim of the investigation reported in this chapter was to determine the potential of some of these processes in upgrading chloride grade titania slag. The main difference between chloride and sulphate grade slag is the levels of alkaline earth elements that are present. In chloride grade slag the % MgO + % CaO < 1.2, while it exceeds 6% in sulphate grade slag (Borowiec et al., 1996). Special emphasis was placed on slag preparation and slag pre-treatment procedures.

### 2.1 Experimental design

#### 2.1.1 Feed material and experimental plan

The slags used for this investigation were obtained from the 5<sup>th</sup> IHM plasma furnace campaign (conducted in a 3MVA pilot scale furnace) of July 1996. The complete slag analyses are listed in Appendix I. Table 12 gives the concentration of selected elements in the feed slags.

**Table 12.** Chemical composition of the feed slags (wt %)

Reference no.	Classification	TiO <sub>2</sub>	FeO	P <sub>2</sub> O <sub>5</sub>
YS3812	Standard slag: As-cast	87.7	10.04	-
YS17	Standard slag: Granulated	83.0	10.66	-
YS16	High iron slag: Granulated	72.5	18.27	-
YS3813	Standard slag: Oxidised in the solid state	85.1	10.66	-
YS13	High iron slag: Oxidised in the solid state	78.3	16.60	-
-	Standard slag: Oxidised in the molten state	80.1	15.31	-
-	High iron slag: Oxidised in the molten state	74.6	21.61	-
-	Standard slag: Phosphate treated	69.5	8.83	7.74
-	Standard slag: Oxidised en reduced	88.1	8.75	-

\* Ti is reported as total TiO<sub>2</sub> regardless of oxidation state

As-cast titania slag contains mainly oxides of titanium and iron. Titanium is present in the Ti(III) and the Ti(IV) oxidation states and iron is present in the Fe(II) oxidation state. Slags subjected to various pre-treatments were chosen for this investigation. This was done to investigate the effect of slag pre-treatment on the leachability of impurities from the slag. Another important variable that was identified was the FeO content of the slag. Slags with high FeO levels can be produced with a lower energy input to the smelting furnace. This may be a process advantage and feed slags with high and low FeO contents were therefore evaluated. The effects of the various slag pre-treatment procedures on the leachability of impurities from the slag were evaluated by leaching most of the materials in hydrochloric and sulphuric acid. The phosphate treated slag was leached in hydrochloric acid, sulphuric acid and phosphoric acid, while the oxidised and reduced slag was only leached in hydrochloric acid. The increase in TiO<sub>2</sub> content of the leach residues compared to the feed was used to evaluate the effectiveness of the upgrading procedures.

## **2.2 Experimental procedure**

### **2.2.1 Slag pretreatment**

The standard as-cast slag (YS3812) was produced by smelting ilmenite in the presence of carbon in a 3 MVA plasma furnace. An as-cast slag block was crushed and screened to the +106-850  $\mu\text{m}$  range.

The two granulated slag samples (YS16 and YS17) were produced from molten titania slag with an air granulator. These samples were also screened to the +106-850  $\mu\text{m}$  size range.

Two samples of slag were produced to imitate slag that had been oxidised in the molten state; slag samples were melted in a tundish and oxidised by blowing oxygen through the molten charge with a steel lance.

Two sized slag samples (YS3813 and YS13) were oxidised in a vertical thermogravimetric furnace for 2 h at 800°C with air. The roasted product was cooled inside the furnace without any gas circulation.

The phosphate treated slag was produced by grinding 8260 g slag, 330 g lime and 1410 g ammonium phosphate in a rotating mill for 2 h. Alumina crucibles, charged with this mixture of fine material, were loaded into a muffle furnace and heated to 1300 °C. The crucibles were held at this temperature for 12 h while oxygen was circulated through the furnace. After fluxing the furnace was opened and the crucibles and their content allowed to cool rapidly in air. The solidified mass was removed from the crucible and crushed and sized to +106-850  $\mu\text{m}$  prior to leaching.

The oxidised and reduced slag was produced by charging a 1000 g batch of sized slag to a Linn rotating furnace equipped with external heating and a tube with lifter bars. The slag was heated to 850 °C and held at this temperature for 2 h while air flowed through the furnace at a rate of 67.2 L/min; the gas flow was changed to a mixture of carbon monoxide and nitrogen at a rate of 11.8 and 42.5 L/min respectively for a period of 20 min prior to cooling the slag down under nitrogen flow. A second run was performed using the same procedure but using a gas mixture consisting of 67 % CO and 33 % CO<sub>2</sub> for the 20 min reducing part of the cycle.

### **2.2.2 Leaching**

Most of the leach experiments were conducted in 2 L batch stirred tank reactors. The reactors were placed in thermostatically controlled water baths at 95°C. The glass reactors were closed and equipped with condensers to limit evaporation losses. Stirring at 500 rpm was provided by flat paddle type impellers driven by overhead motors and the reactors were equipped with 4 baffles each. Each reactor was filled with 1 L solution and once the reactor stabilised at the desired temperature 500 g dry feed material was charged. 20 % Hydrochloric acid, 26 % sulphuric acid and 18 % phosphoric acid solutions were respectively used for the various experiments. 20 mL Pulp samples were taken at 0; ½; 1; 2 and 4 h. These samples were filtered immediately and the filtrate was saved for chemical analysis. At the start and end of

an experiment the reactor and its contents were weighed to determine the total mass loss due to sampling and evaporation. At the end of the leach the slurry was filtered and repeatedly repulped and washed until the pH of the wash water was neutral. Special care was taken to limit losses of solids during the bulk filtration and washing operations. The washed filter cake was dried overnight at 120 °C, weighed and sampled for full chemical analysis. Solution samples were analysed by ICP-AES to determine the concentrations of respectively: Ti, Fe, Mg, Al, Mn, Cr, Ca, Si and V and by acid titration to determine the free acid concentrations. The solid samples were analysed by ICP-AES to determine the concentrations of respectively: Ti, Fe, Mg, Al, Mn, Cr, Ca, Si, V and P in the feed material and the washed leach residues.

The oxidised and reduced slag was leached in a 2 L Erlenmeyer flask fitted with a reflux condenser to limit evaporation losses. The reactor was placed on a hot plate that kept the leach solution at boiling point. 20% Hydrochloric acid was used to leach the slag for up to six hours. The rest of the procedure was similar to the experiments conducted in the water bath.

## 2.3 Results and discussion

The effect of the different slag pre-treatments on slag leachability is presented in Table 13. It shows the chemical analysis of the feed materials as well as the chemical analysis of the residues after leaching in hydrochloric and sulphuric acid. The log sheets for the experiments are listed in Appendix II.

**Table 13.** The effect of different pre-treatments and slag compositions on the leachability of impurities

Chemical composition (wt %)		TiO <sub>2</sub>	FeO	MgO	CaO	SiO <sub>2</sub>
Standard slag: As-cast	Feed	87.7	10.05	0.93	0.13	1.47
	HCl leach residue	88.4	9.08	0.98	0.10	1.26
	H <sub>2</sub> SO <sub>4</sub> leach residue	88.0	8.95	0.97	0.10	1.26
Standard slag: Granulated	Feed	83.0	10.66	1.90	0.34	1.73
	HCl leach residue	84.9	9.85	1.83	0.17	1.61
	H <sub>2</sub> SO <sub>4</sub> leach residue	84.1	10.05	1.90	0.19	1.60
High iron slag: Granulated	Feed	72.5	18.27	1.84	0.99	2.11
	HCl leach residue	79.5	14.66	1.91	0.13	1.81
	H <sub>2</sub> SO <sub>4</sub> leach residue	79.9	14.41	1.88	0.13	1.83
Standard slag: Oxidised in the solid state	Feed	85.1	10.66	0.91	0.14	1.50
	HCl leach residue	87.5	8.27	0.97	0.09	1.26
	H <sub>2</sub> SO <sub>4</sub> leach residue	86.7	8.84	0.94	0.12	1.30
High iron slag: Oxidised in the solid state	Feed	78.3	16.59	1.29	0.20	1.20
	HCl leach residue	80.1	15.57	1.32	0.19	1.32
	H <sub>2</sub> SO <sub>4</sub> leach residue	79.4	15.69	1.30	0.18	1.22
Standard slag: Oxidised in the molten state	Feed	80.1	15.31	1.66	0.33	1.62
	HCl leach residue	79.4	15.69	1.30	0.18	1.22
	H <sub>2</sub> SO <sub>4</sub> leach residue	79.9	14.15	1.64	0.26	1.67
High iron slag: Oxidised in the molten state	Feed	74.6	21.61	1.74	0.23	1.37
	HCl leach residue	74.3	20.84	1.75	0.21	1.48
	H <sub>2</sub> SO <sub>4</sub> leach residue	74.4	20.97	1.75	0.20	1.42
Standard slag: Oxidised and reduced in the solid state	Feed	85.4	11.12	0.96	0.12	0.16
	HCl Leach residue	92.1	2.86	0.41	0.08	0.12

The results presented in Tables 13 clearly indicate that the proper pre-treatment of titania slag is critical to ensure good leachability of impurities. With no slag pre-treatment the increase in TiO<sub>2</sub> content of the slag after leaching was less than 1 %. Granulation in air oxidises the Ti(III) in the slag to Ti(IV) and the Fe(II) to Fe(III). The implications of this pretreatment process is unclear as no significant upgrading could be achieved for the high titanium slag, but the TiO<sub>2</sub> content of the high iron slag increased from 72.5 % to 79.9 %. Oxidation in the solid as well as molten states was not beneficial and very little upgrading was achieved. As a result of the poor results achieved with the granulation and oxidation pretreatment steps it was decided to evaluate an oxidation and reduction pretreatment. This pretreatment has the advantage that during oxidation Ti(III) and Fe(II) are oxidised to insoluble phases containing Ti(IV) and Fe(III). During reduction the insoluble Fe(III)-containing phases are converted to a soluble phase containing Fe(II). In addition the experiment was conducted at a high temperature and for a longer time than the previous experiments. The results in Table 13 show that this pretreatment led to a significant increase in TiO<sub>2</sub> concentration after leaching.

**Table 14.** The effect of phosphate fluxing on the leachability of impurities from titania slag

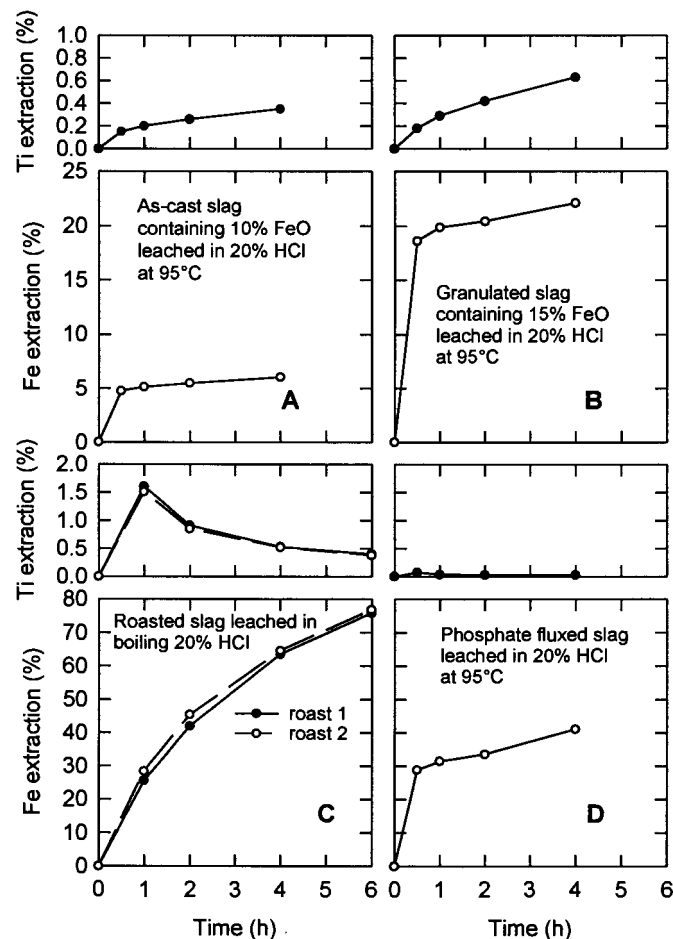
Chemical composition (wt%)	TiO <sub>2</sub>	FeO	MgO	CaO	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>
Slag before phosphate fluxing	87.3	8.49	0.98	0.11	1.35	<0.1
Lime	0.04	0.32	1.05	62.7	1.35	<0.1
Ammonium phosphate	<0.01	0.13	0.02	0.02	<0.01	63.2
Slag after phosphate fluxing	69.5	8.82	0.80	2.07	6.62	7.74
H <sub>3</sub> PO <sub>4</sub> leach residue	82.3	6.51	0.09	0.13	7.21	1.51
H <sub>2</sub> SO <sub>4</sub> leach residue	82.4	6.15	0.07	1.16	7.34	0.62
HCl leach residue	83.4	6.03	0.09	0.13	7.21	1.51

**Table 15.** The leach results presented in Table 14 normalised to a silica free basis

Chemical composition (wt%)	TiO <sub>2</sub>	FeO	MgO	CaO	P <sub>2</sub> O <sub>5</sub>
Slag after phosphate fluxing	74.4	9.45	0.86	2.22	8.29
H <sub>3</sub> PO <sub>4</sub> leach residue	88.7	7.02	0.10	0.14	1.62
H <sub>2</sub> SO <sub>4</sub> leach residue	88.9	6.63	0.08	1.25	0.67
HCl leach residue	90.2	6.52	0.10	0.04	0.77

The phosphate fluxing pretreatment was attempted because the morphology of the slag is modified to rutile crystals set in a phosphate glass that contains all the impurities. The results of phosphate fluxing experiment are presented in Table 14. It shows the chemical analysis of the slag before and after the phosphate fluxing procedure and that of the leach residues after leaching in phosphoric acid, sulphuric acid and hydrochloric acid respectively. It is immediately obvious from the high silica values that contamination of the slag occurred during the slag preparation procedure. This is confirmed by the low silica contents of the different feed materials used in the preparation procedure. The leach results were normalised to a silica free basis by recalculating the chemical compositions without any silica. The adjusted values are presented in Table 15. This shows that the TiO<sub>2</sub> content of the slag increased to more than 90% after phosphate fluxing followed by leaching in hydrochloric acid.





**Figure 3.** The leach kinetics of titanium and iron in 20% HCl from: **A** Standard as-cast slag at 95°C; **B** High iron granulated slag containing at 95°C; **C** Oxidised and reduced slag at 107°C and; **D** Phosphate fluxed slag at 95°C

Figure 3-A shows the extraction curves for Fe and Ti from as-cast slag and it is evident that with no pre-treatment very little iron or titanium are leached. Most of the initial slag pre-treatment procedures did not enhance the leachability of the slag (Table 13). Granulation of the slag which contained 15 % FeO did however increase the leachability of the slag markedly. Figure 3-B shows that almost 25 % of the iron was leached from this slag in a hydrochloric acid solution at 95 °C. The best pre-treatment process proved to be the oxidation-reduction roast procedure; Figure 3-C shows that almost 80 % of the iron was extracted in a boiling hydrochloric acid solution. Figure 3-C also shows the titanium extraction with leaching time; it increases to more than 1.5 % in the first half an hour before it decreases to less than a half percent after four hours. This result is interesting as it is similar to the results published by Sinha (1984) for ilmenite leaching in HCl. He explained this phenomenon by noting that both iron and titanium initially go into solution but as leaching proceeds the amount of titanium in solution decreases as a result of hydrolysis and precipitation. He also described how fines are formed during this

reaction. Apparently factors such as agitation rate, acid concentration, temperature and ferrous chloride concentration dictate if the hydrolysis and precipitation of titanium occur in the bulk solution or in the particles. If it occurs in the bulk solution fines are formed. This occurrence of this phenomenon in the experiments is confirmed by the presence of white fines in the leach solutions of the oxidised and reduced slag. Figure 3-D shows that more than 40 % of the iron was extracted from the slag and that almost no titanium was dissolved. These results were achieved despite a high FeO value of ~ 9 % in the feed material. Oden, Summer and Howe (1973) found that high iron values were detrimental to the leachability of impurities from phosphate fluxed slag. Van Dyk (1996) achieved a high iron extraction from a slag containing ~6 % FeO. This suggests that the TiO<sub>2</sub> content can be increased even further if a slag with a low iron content is subjected to the phosphate fluxing procedure.

A mineralogical investigation was conducted on the standard as-cast slag as well as the slag oxidised in air at 850 °C for 2 h and reduced for 20 min in a 66 % CO and 33 % CO<sub>2</sub> atmosphere. The leach residue of the roasted slag was also investigated. Table 16 gives the phase compositions of the samples as determined by X-ray diffraction (XRD). The primary phase in the as-cast slag is an iron titanium solid solution (M<sub>3</sub>O<sub>5</sub>). Trace amounts of rutile could also be detected. After oxidation and reduction the main phase in the slag was rutile, while the M<sub>3</sub>O<sub>5</sub> phase was reduced to a trace component. A trace of ilmenite was also present. Leaching did not alter the phase composition dramatically as only the ilmenite phase disappeared.

**Table 16.** Phase-chemical composition of the slag, given in order of decreasing abundance.

Sample	Mineralogical composition		
	Main phases	Minor phases	Trace phases
Standard as cast slag	FeTi-Oxide (M <sub>3</sub> O <sub>5</sub> )	-	Rutile
Oxidised and reduced slag	Rutile	-	Ilmenite; FeTi-Oxide (M <sub>3</sub> O <sub>5</sub> )
Oxidised, reduced and leached slag	Rutile	-	FeTi-Oxide (M <sub>3</sub> O <sub>5</sub> )

Legend :Rutile - TiO<sub>2</sub>; FeTi-Oxide - M<sub>3</sub>O<sub>5</sub>-solid solution and Ilmenite - FeTiO<sub>3</sub>

## 2.4 Conclusions

This study showed that slag pre-treatment is vital to achieve a high extraction of iron from titania slag during leaching. The two pre-treatment processes that showed the most promise were an oxidation-reduction roast procedure and phosphate fluxing of the slag. Although high extraction of iron was obtained it was not high enough to yield a beneficiated slag product containing more than 95 % TiO<sub>2</sub>.



### 3. PROCESS DEVELOPMENT PHASE 1

#### 3.1 Introduction

The preliminary investigation on processes for the production of BTS indicated that a process consisting of oxidation-reduction roasting followed by atmospheric leaching in boiling hydrochloric extracted most of the impurities from the slag. The aim of this phase of the process development was to determine the optimum roasting and leaching conditions to obtain a BTS grade of >95 % TiO<sub>2</sub>. The flexibility of the process with regard to feed slag composition was also investigated.

#### 3.2 Experimental design

##### 3.2.1 Feed material

The slag used for this investigation was obtained from the 6<sup>th</sup> IHM 3MVA plasma furnace campaign of July 1997. The complete chemical analyses of the slags are listed in Appendix III. Table 17 shows the concentration of selected species in the feed slags.

**Table 17.** Concentration of selected species in the feed slags used for this investigation.

Sample no.	Cast no.	TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe	FeO	Cr <sub>2</sub> O <sub>3</sub>	MgO
PFE418	K6-S-26	51.80	30.00	-	8.58	0.30	2.65
PFE436	K6-S-02	58.40	13.60	-	21.90	0.11	1.52
PFE437	K6-S-29	50.90	33.40	-	8.70	0.17	1.33
PFE467	K6-S-08	57.00	21.20	0.82	14.30	0.11	1.17
PFE489	K6-S-35	52.30	32.10	0.03	9.34	0.12	1.17
PFE655	K6-S-03	66.10	9.10	1.58	15.60	0.11	3.55
PFE656	K6-S-26	58.00	22.20	0.35	9.45	0.28	2.50
PFE657	K6-S-30	50.70	34.20	0.21	7.65	0.15	1.42
PFE658	K6-S-46	53.90	28.70	0.27	9.71	0.12	1.95

An Australian coal from Wallarah was used for the reduction experiments. The analysis for this coal is given in Table 18.

**Table 18.** Coal analysis.

Total moisture	9.0%
Inherent moisture	3.5%
Volatile matter	30.0%
Fixed carbon	53.0%
Sulphur	0.33%
Ash	13.5%
Calorific value	27 MJ/kg

##### 3.2.2 Experimental plan

The variables that were investigated during the roast study are listed in Table 19. The roast conditions were evaluated by conducting a 5 h atmospheric leach with boiling 20 % HCl and determining the TiO<sub>2</sub> content of the leach residue.

**Table 19. Variables investigated during the roast study**

Variable	Conditions
Roast temperature	750 °C; 800 °C; 850 °C; 950 °C
Oxidation time	60 min; 120 min; 180 min; 240 min
Reduction time	0 min; 10 min; 20 min; 40 min

The variables that were investigated during the leach study are listed in Table 20.

**Table 20. Variables investigated during the leach study**

Variable	Conditions
Feed slag composition	< 10 % FeO; >10 % FeO; >2 % MgO
Acid concentration	18 % HCl; 20 % HCl; 33 % HCl
Excess Acid	20 %; 40 %; 70 %
Leach time	0 h; ½ h; 1 h; 2 h; 4 h; 6 h; 8 h; 12 h

### 3.2.3 Experimental procedure

#### 3.2.3.1 Roast procedure

A 100 mm diameter fluidised bed roaster was used for roasting. The roaster was fitted with an externally gas fired muffle that was used for starting-up the furnace and to control the temperature inside the furnace. The fluidisation gases were passed through rotameters. Char or coal depending on the requirement was added to the roaster with a variable speed feeder. This did not allow close control over the oxygen potential in the roaster, but it does replicate the operation of an industrial roaster. During oxidation air at a rate of 1.35 m<sup>3</sup>/h (STP) was used and char, produced by a previous reduction roast, was fed into the roaster at 0.4 kg/h. For reduction air at a rate of 0.14 m<sup>3</sup>/h (STP) was mixed with nitrogen at a rate of 0.67 m<sup>3</sup>/h (STP). Coal was fed into the roaster at a rate of 1.0 kg/h. A test was started by passing the fluidisation gas through the reactor. 5 kg slag was then added while the gas-fired muffle was used to heat the reactor. At 650°C the char feed was started. Oxidation was taken to have commenced once the reactor temperature reached a value 50 °C below the set value. The bed temperature was controlled by adjusting the gas burner and the flue damper. During oxidation the roaster ran autothermally. At the completion of the oxidation roast a sample was taken and the reduction conditions were implemented. This was accomplished by reducing the airflow, adding nitrogen and changing the char feed to coal. The reduction temperature was maintained at 50 °C below the oxidation temperature. The roaster did not operate auto thermally during reduction and the gas fired muffle had to be used to maintain the required temperature. At the end of an experiment the slag was cooled rapidly by inserting a water-cooled lance into the bed and by changing the fluidisation gas to nitrogen alone. Char and ash were removed from the slag samples by screening at 1 mm followed by panning. The wet slag was dried at 110 °C.

#### 3.2.3.2 Leach procedure

Roasted slag was leached in boiling 20 wt % hydrochloric acid for 5 to 12 h (unless otherwise specified). Conical Quickfit flasks equipped with condensers were heated on a hot plate. No stirring was provided. At the end of the leach the solution was assayed titrimetrically for Fe (II) and Fe (III). The method used for this titration is

described in the Appendix VI. The leach liquor was decanted from the leach residue before the residue was washed. Slimes were separated from the leach solution by flocculation with a commercial flocculation agent called magnafloc. The leach residue and slimes were calcined at 850 °C for 1 h before weighing. The leach residues were analysed by inductively coupled plasma optical emission spectroscopy (ICP-OES) at the ITEC Services laboratories in Pretoria. Some solution samples were analysed by ALS, an Australian laboratory.

### 3.3 Results and discussion

#### 3.3.1 Roast investigation

A series of experiments were conducted on feed slag PFE437 (88% TiO<sub>2</sub>) to determine the optimum roast conditions. The results of the roast investigation are summarised in Table 21. The complete chemical analyses of the samples are listed in Appendix IV.

**Table 21.** Results of the roast investigation on feed slag PFE437.

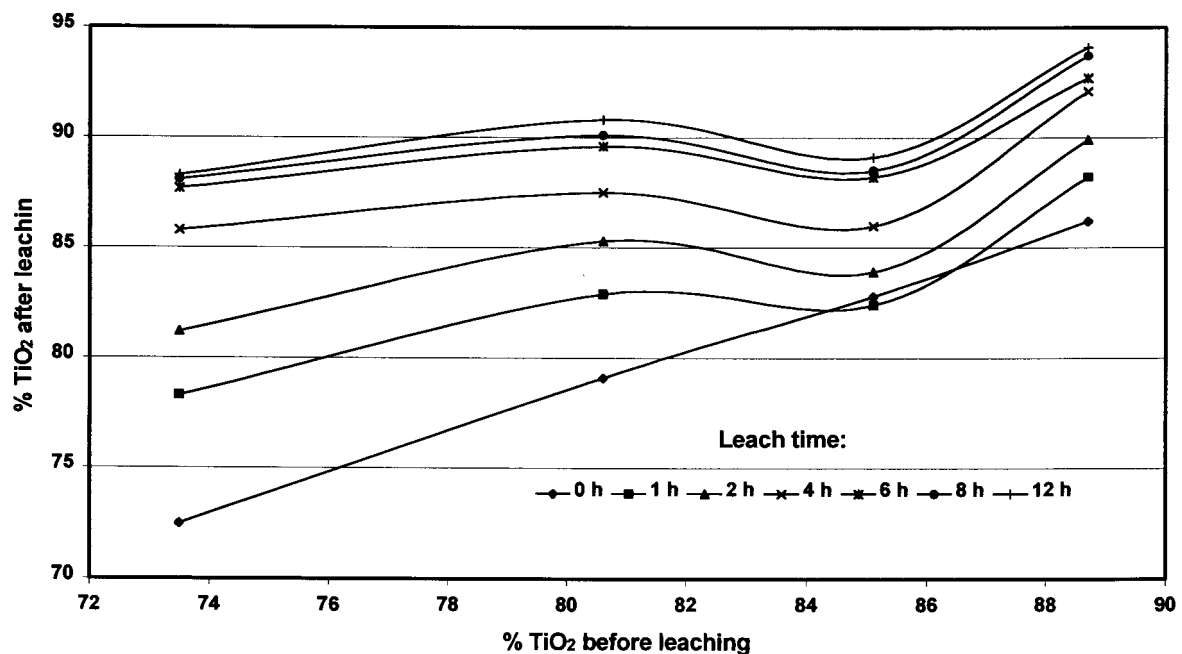
Sample No.	Roast No.	Sample No.	Oxidation Temp. (°C)	Oxidation Time (min)	Reduction Temp. (°C)	Reduction Time (min)	% TiO <sub>2</sub> in leach residue
696	1	1	950	60	900	0	85.50
698		2	950	60	900	10	86.60
700		3	950	60	900	20	87.10
702		4	950	60	900	40	87.00
704	2	5	950	120	900	0	85.40
706		6	950	120	900	10	87.90
708		7	950	120	900	20	88.00
710		8	950	120	900	40	87.60
712	3	9	950	240	900	0	84.60
714		10	950	240	900	10	87.00
716		11	950	240	900	20	87.40
718		12	950	240	900	40	87.30
720	4	13	850	60	800	0	86.80
722		14	850	60	800	10	90.10
724		15	850	60	800	20	90.70
726		16	850	60	800	40	90.20
728	5	17	850	120	800	0	86.70
730		18	850	120	800	10	91.10
733		19	850	120	800	20	92.40
734		20	850	120	800	40	91.50
736	6	21	850	240	800	0	85.70
738		22	850	240	800	10	89.00
740		23	850	240	800	20	91.00
742		24	850	240	800	40	85.40
744	7	25	800	180	750	0	86.60
746		26	800	180	750	10	89.70
748		27	800	180	750	20	88.60
750		28	800	180	750	40	89.40
752	8	29	800	240	750	0	86.70
754		30	800	240	750	10	89.50
756		31	800	240	750	20	89.20
758		32	800	240	750	40	88.90
760	9	33	750	240	700	0	87.90
762		34	750	240	700	10	87.40

The results presented in Table 21 were analysed with BDMP, a statistical computer package developed by the Department of Biomathematics, School of Medicine, University of California, as a factorial analysis. This showed that the optimum roast conditions are:

- Oxidation temperature: 850 °C
- Oxidation time: 3 h
- Reduction temperature: 800 °C
- Reduction time: 30 min

### 3.3.2 Leach investigation

The effects of leach time and feed slag composition were evaluated by roasting four different slags under the optimum conditions identified during the roast investigation. Separate leach experiments were then performed on the slags for times varying between ½ h and 12 h. Boiling 20% HCl, 70 % above the stoichiometric amount of acid required to dissolve the impurities, was used. The complete chemical analyses of the leach investigation samples are listed in Appendix V. A summary of the results is presented in Figure 4.

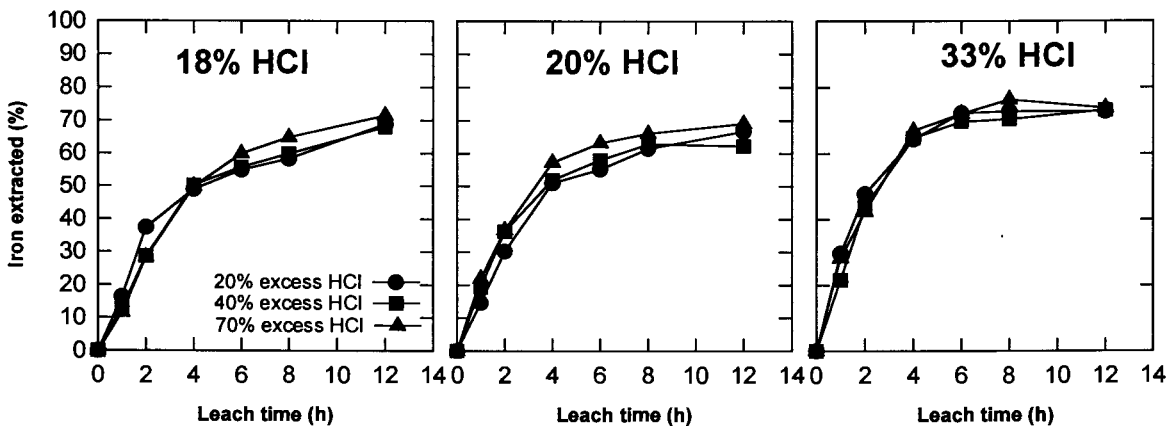


**Figure 4.** Summary of the results from the tests conducted to evaluate the effect of leach time and feed slag composition.

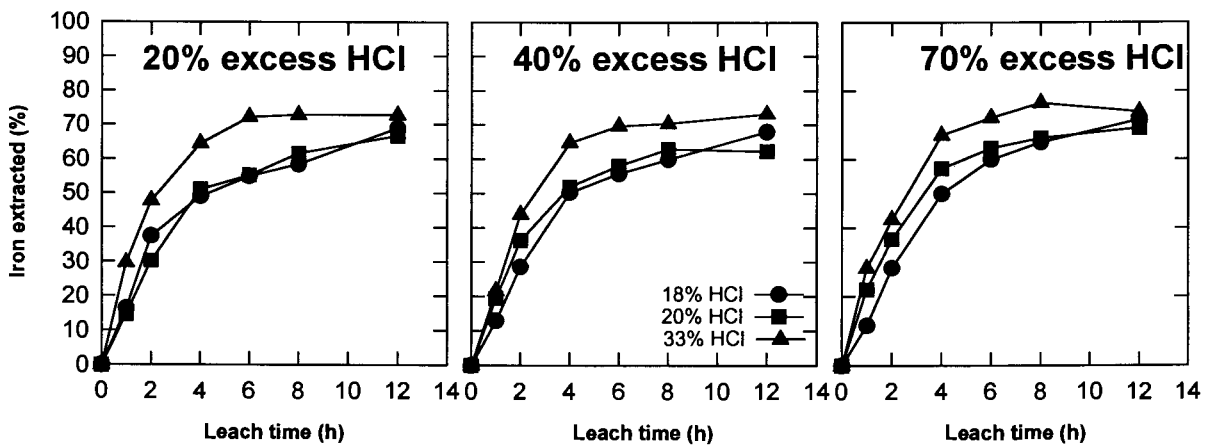
Figure 4 shows that slag composition influenced the BTS product quality significantly. The product with highest TiO<sub>2</sub> content was made from PFE657 (88.7 % TiO<sub>2</sub>). This slag contained less than 2 % MgO and around 8 % FeO. The slag containing high levels of MgO and FeO showed up as a local minimum on the graph as they were not sufficiently upgraded (85.13 % TiO<sub>2</sub>). Figure 4 also shows that long leach times are necessary to produce BTS of the required grade.



The influence of excess acid and acid concentration on the extraction of iron was investigated using PFE467 (a relatively high iron slag – see Table 12) which was oxidised for 3 h at 850 °C and reduced at 800 °C for 30 min. Three initial acid concentrations, 18%, 20% and 33% HCl were selected as these were the acid concentrations that could readily be supplied by the enhanced acid regeneration system (EARS) (Walpole, 1993) in a full scale plant. The level of excess acid was varied by keeping the solid mass constant and varying the solution volume. Solution samples were regularly taken and analysed for iron and acid. After 12 h of leaching the leach residue was analysed for TiO<sub>2</sub>. The complete results are presented in the Appendix V. Figures 5 and 6 show the effect of acid concentration and level of excess acid on iron extraction. Figure 7 shows the effect of acid concentration and level of excess acid on the final product quality.

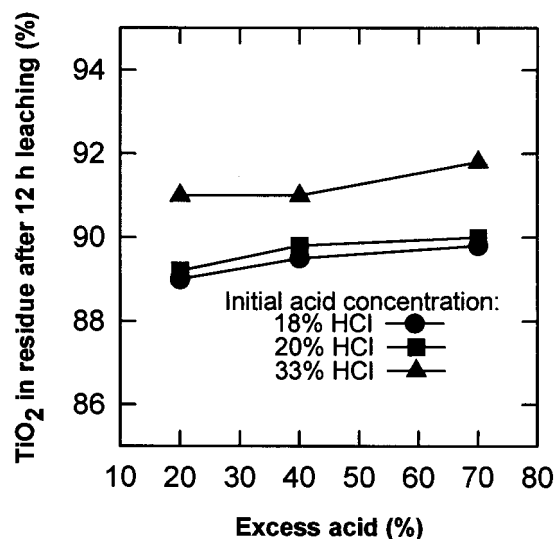


**Figure 5.** The effect of excess hydrochloric acid on iron extraction at different initial hydrochloric acid concentrations



**Figure 6.** The effect of initial hydrochloric acid concentration on iron extraction at different levels of excess hydrochloric acid

b17337392  
i18808748



**Figure 7.** The effect of acid concentration and the level of excess acid on the final product quality after 12 h of leaching.

Figure 5 indicates that the rate of iron leaching was not significantly improved by increasing excess acid at constant initial acid concentration. On the other hand, the rate of iron leaching increased with increasing initial acid concentration at constant excess acid level (Figure 6). Final product quality improved only marginally with increasing excess acid level while it improved with increasing initial acid concentration (Figure 7). Thus, higher acid strengths improved both the kinetics of leaching and the final product quality.

### 3.3.3 Mineralogical investigation

A mineralogical investigation was conducted to determine what morphological changes occur in the slag during roasting and leaching. The investigation was conducted using three techniques: (a) X-ray diffraction analysis (XRD) (b) Optical microscopy and (c) Scanning electron microscope (SEM) analysis, using back-scattered electron imaging. The results are presented in the context of the relevant process stages of the BTS process.

#### 3.3.3.1 As-cast titania feed slag

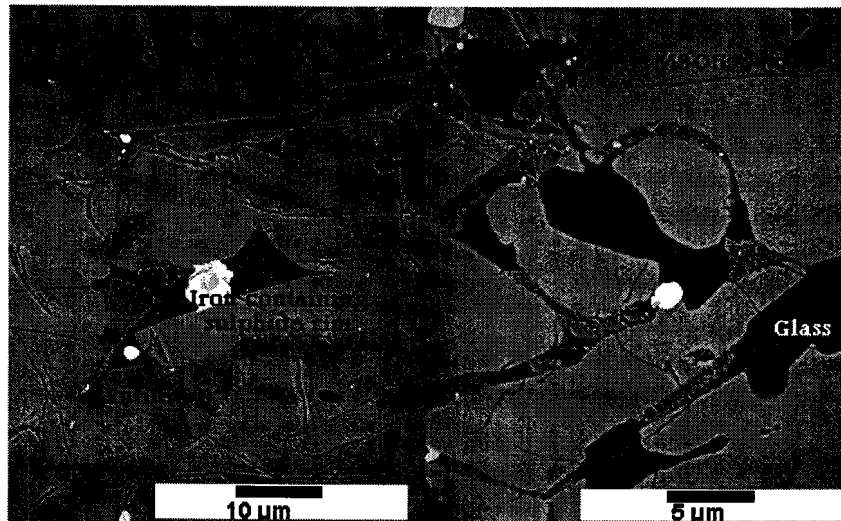
The phase-chemical compositions of the feed slag samples, as determined by XRD are shown in Table 22. The slag samples were all of similar mineralogical composition with the  $M_3O_5$  solid solution identified as primary constituent.

**Table 22.** Phase-chemical composition of the feed slag samples as determined by XRD, given in order of decreasing abundance. The chemical compositions used to classify the slags are also given.

PFE	Description	Chemical Composition (%)		Mineralogical composition		
		FeO	MgO	Main	Minor	Trace
418	High MgO	9.68	2.65	FeTi-Oxide	-	-
436	High Iron	24.30	1.52	FeTi-Oxide	-	Ilmenite
437	Standard	8.55	1.60	FeTi-Oxide	-	Rutile
467	High Iron	14.30	1.85	FeTi-Oxide	-	-
657	Standard	7.65	1.42	FeTi-Oxide	-	-

*Legend : FeTi-Oxide -  $M_3O_5$ -solid solution; Rutile -  $TiO_2$ ; Ilmenite -  $FeTiO_3$*

The standard, high iron and high MgO slag all had a similar optical appearance. The individual slag particles consisted mainly of the  $M_3O_5$ -solid solution. The  $M_3O_5$ -phase occurred as fine- to coarse-grained, angular to sub-rounded, greyish coloured particles with a smooth appearance (Figure 8). Small amounts of a silicate-rich glassy phase were visible, situated at the grain boundaries of the individual  $M_3O_5$  crystals. This glass contained a second silicate-enriched glass. The silicate-rich glassy phase had a smooth appearance and contained finely disseminated metallic iron precipitates. The major glassy phase was fine crystalline with needle-like titanium oxide crystallites as well as small and large metallic iron precipitates. The larger metallic precipitates were characterised by an iron sulphide outer rim.



**Figure 8.** As-cast standard titania slag.

### 3.3.3.2 Oxidation

The feed slags, listed in Table 17, were all oxidised at 850 °C with exception of slag PFE437 which was oxidised respectively at 750 °C, 850 °C and 950 °C. Samples were taken after 1, 2, 3 and 4 h oxidation. The oxidised products of samples PFE437, PFE657, PFE436 and PFE418 were examined mineralogically. For the sake of the discussion they were placed in the following categories:

- Standard slag: PFE437 and PFE657
- High iron slag: PFE436
- High magnesia slag: PFE418

### Standard slag

The phase-chemical compositions of the individual slag samples, as obtained by XRD are shown in Tables 23 (PFE437) and 24 (PFE657). The slags consisted mainly of rutile, anatase and smaller amounts of the  $M_3O_5$ -solid solution after the oxidation roast. The effects of temperature and time of oxidation on the phase composition of the slag could be deduced from the data given in Tables 23 and 24. At 950 °C rutile was the main titanium dioxide-containing phase while anatase became more dominant at lower temperatures. At 850 °C both anatase and rutile occurred in more or less equal amounts and at 800 °C anatase was the main phase and rutile was present only in minor amounts. At 850 °C and reaction times of less than 1½ h, anatase appeared to be the main crystalline phase present in the samples while at longer reaction times, rutile occurred together with anatase as main crystalline phases. After a reaction period of 3 h, more rutile was present in the slag than anatase. Thus at lower temperatures the  $M_3O_5$ -phase transforms to anatase and a new (presumably iron-rich)  $M_3O_5$ -phase, while at higher temperatures the presumed new iron-rich  $M_3O_5$  phase forms in conjunction with rutile. At lower temperatures the anatase transforms slowly to rutile with longer reaction times.

**Table 23.** Phase-chemical compositions of slag PFE437 after oxidation at different temperatures and times, given in order of decreasing abundance.

PFE	Temperature (°C)	Time (h)	Mineralogical composition		
			Main	Minor	Trace
696	950	1	Rutile	-	FeTi-Oxide; Anatase
712	950	4	Rutile	-	FeTi-Oxide
720	850	1	Anatase; Rutile	-	FeTi-Oxide
736	850	4	Rutile; Anatase	-	FeTi-Oxide
744	800	3	Anatase	Rutile	FeTi-Oxide
752	800	4	Anatase,	Rutile	FeTi-Oxide
760	750	4	Anatase	Rutile	FeTi-Oxide

Legend : FeTi-Oxide -  $M_3O_5$ -solid solution; Rutile -  $TiO_2$ ; Anatase -  $TiO_2$ ; Ilmenite -  $FeTiO_3$ .

**Table 24.** Phase chemical composition of slag PFE657 after oxidation at 850 °C for different times, given in order of decreasing abundance.

PFE	Time (h)	Mineralogical composition		
		Main	Minor	Trace
924	½	Anatase	Rutile; FeTi-Oxide	-
925	1	Anatase	Rutile	FeTi-Oxide
926	1½	Anatase	Rutile	FeTi-Oxide
927	2	Anatase; Rutile	-	FeTi-Oxide
928	2½	Anatase; Rutile	-	FeTi-Oxide
929	3	Rutile; Anatase	-	FeTi-Oxide

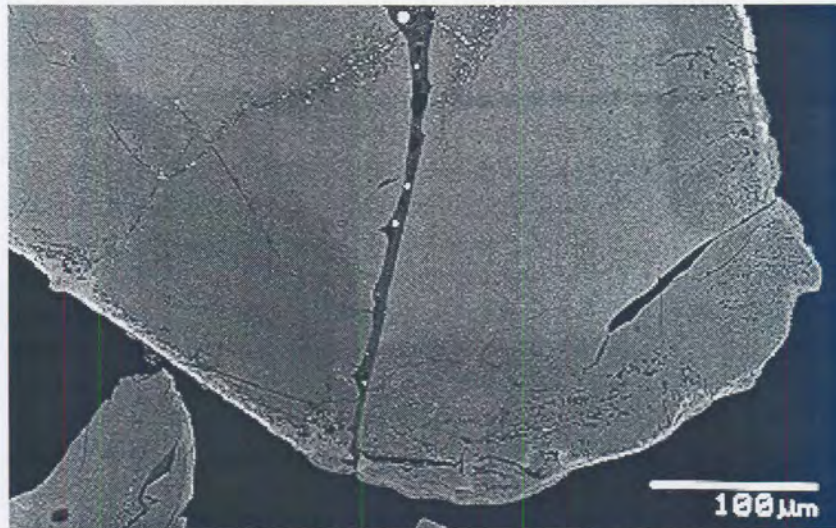
Legend : FeTi-Oxide -  $M_3O_5$ -solid solution; Rutile -  $TiO_2$ ; Anatase -  $TiO_2$ ; Ilmenite -  $FeTiO_3$ .

An optical as well as a SEM investigation were conducted on polished blocks of the oxidised samples. The SEM images were generated from back scattered electrons. This allowed areas of iron concentration to be visible in micrographs of the slag as compositional differences were highlighted by a change in tone. Areas rich in iron were very light and appeared almost white.

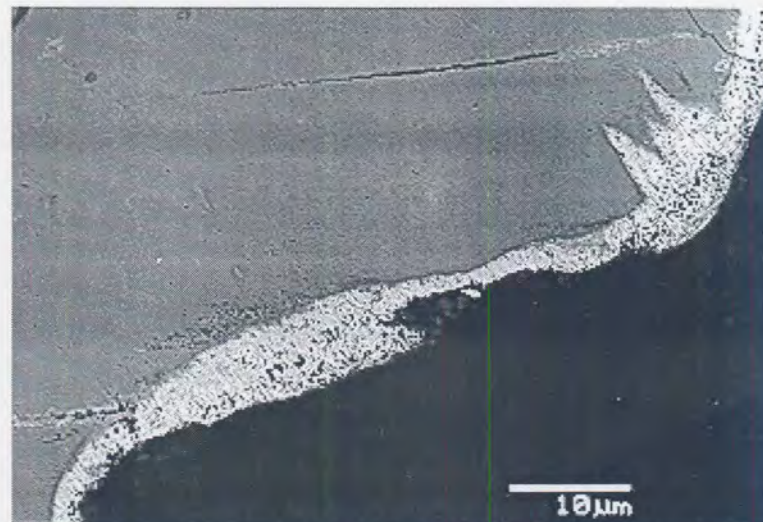
The majority of the slag particles had a zoned appearance. In the centre of the particles was a  $M_3O_5$  core surrounded by a  $TiO_2$ -rich mantle. Most of the iron migrated outwards to form an iron-enriched zone on the outsides of the particles. This marginal zone was slightly porous and rutile was also present (Figure 9). Iron migration towards the edges of cracks also occurred. The unreacted  $M_3O_5$ -cores



contained fine metallic iron precipitates situated at the edges of cracks that extended through the slag particles. As in the unreacted slag, small amounts of silicate-rich glass were present at the grain boundaries. This glass contained fine needle-like titania-rich crystallites as well a second silica-enriched glassy phase. Small and large metallic iron precipitates were present. The larger metallic precipitates contained iron-sulphide outer rims.



**Figure 9.** Standard slag oxidised for 1 h at 850 °C, displaying iron migration towards the edges of cracks and the outer rims of the particles.

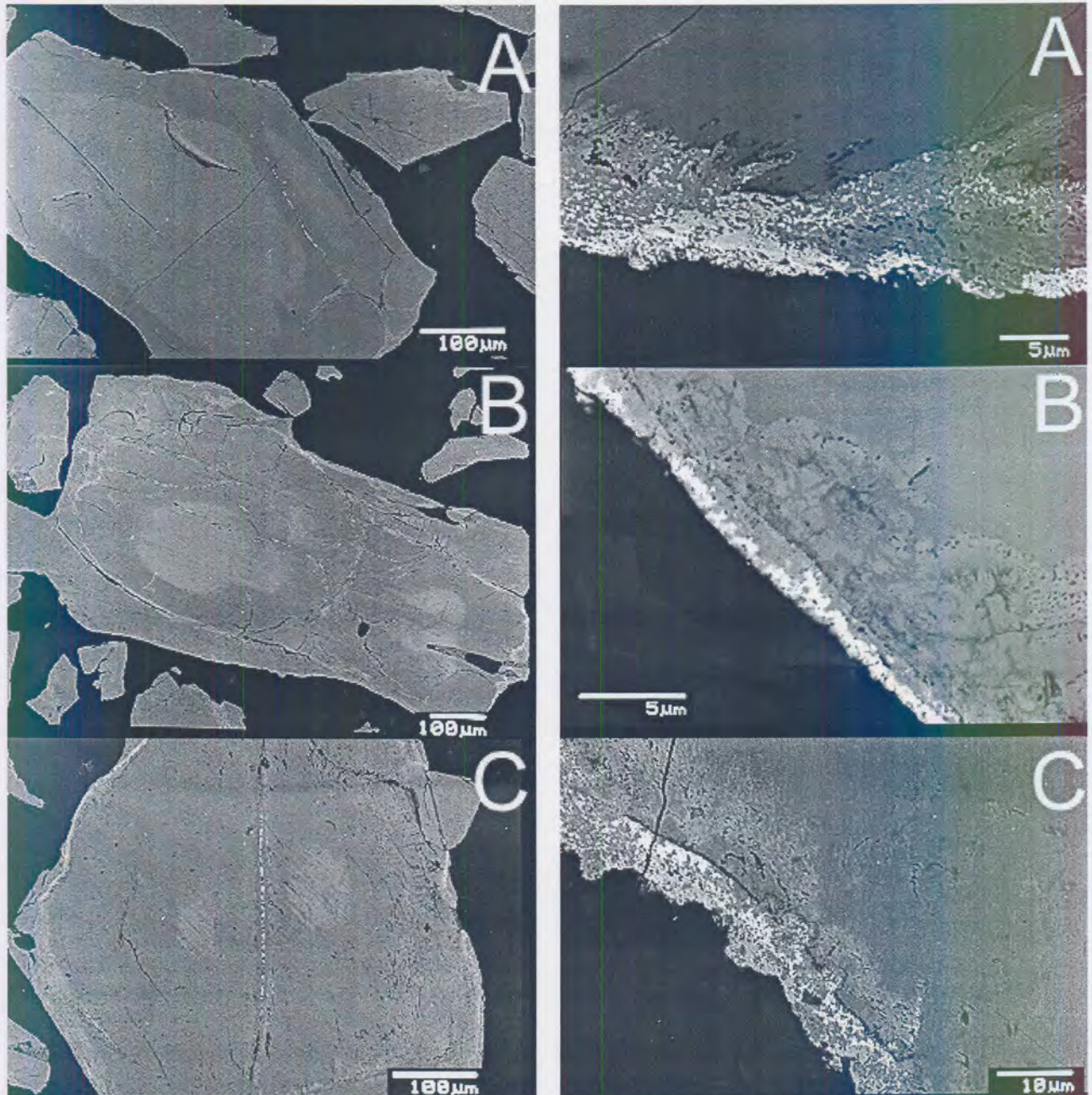


**Figure 10.** Standard slag oxidised for 3 h at 800 °C, contained dense particles that displayed iron enrichment to the outsides of the particles.

Minor differences were observed between the different oxidation samples concerning the degree of reaction and general appearance of the individual slag particles. Only the largest slag particles contained unreacted cores, in the sample roasted for 1 h at 950 °C, and these cores were smaller compared with samples roasted at lower temperatures. The smaller particles were completely oxidised. A few particles contained thin, dense rutile rims. Slag oxidised at 850 °C for respectively 1 and 4 h demonstrated the effect of longer oxidation times. Both slag samples contained coarse-grained particles with unreacted  $M_3O_5$ -cores surrounded by  $TiO_2$ -rich mantles



and porous outer rims. Particles displaying incomplete transformation were less abundant in the sample oxidised for 4 h. Slag oxidised for 3 h at 800 °C contained many particles displaying well-defined unreacted  $M_3O_5$ -cores. Most of the oxidised slag particles appeared to be dense and only a small portion of the particles was slightly porous. Iron migration occurred towards the outer rims of the slag particles as well as the edges of cracks extending through the particles (Figure 10). Fine metallic iron precipitates were present along the edges of these cracks.



**Figure 11.** (Left side) Longer oxidation times resulted in a decrease in the size of the unreacted cores in the particles and an increase in the amount of iron migration to the outsides of the particles. (Right side) Two distinct phases were visible in the iron-enriched rim on the outsides of the oxidised particles.

- A:** Standard slag oxidised for 1 h at 850 °C
- B:** Standard slag oxidised for 1½ h at 850 °C
- C:** Standard slag oxidised for 3 h at 850 °C



The effect of oxidation time was evident from samples roasted at 850°C for times ranging between ½ h and 3 h (Table 24 and Figure 11). The slag samples oxidised for ½, 1 and 1½ h had a zoned appearance, with large unreacted  $M_3O_5$  cores in the particles centres surrounded by thin  $TiO_2$ -rich mantles. The outer margins of the particles were enriched in iron. These particles were dense overall and only the outer margins were slightly porous. Longer oxidation times resulted in a decrease in the size of the unreacted cores and in an increase in the amount of iron migration to the outer margins of the slag particles (Figure 11). An interesting observation was the presence of two distinct phases in the iron-enriched zone on the outsides of the particles.

### High iron slag

The phase-chemical compositions of the slag samples as obtained by XRD analysis, are listed in Table 25. The oxidised slag contained mainly  $M_3O_5$  and anatase. Rutile was present in minor amounts. Trace amounts of ilmenite present in the as-cast feed slag (PFE436) were still present after oxidation for respectively 1, 2 and 3 h at 850 °C. With increasing oxidation time, anatase appears to convert to rutile. This is evident in the sample oxidised for three hours (PFE769) where rutile is present as a main component.

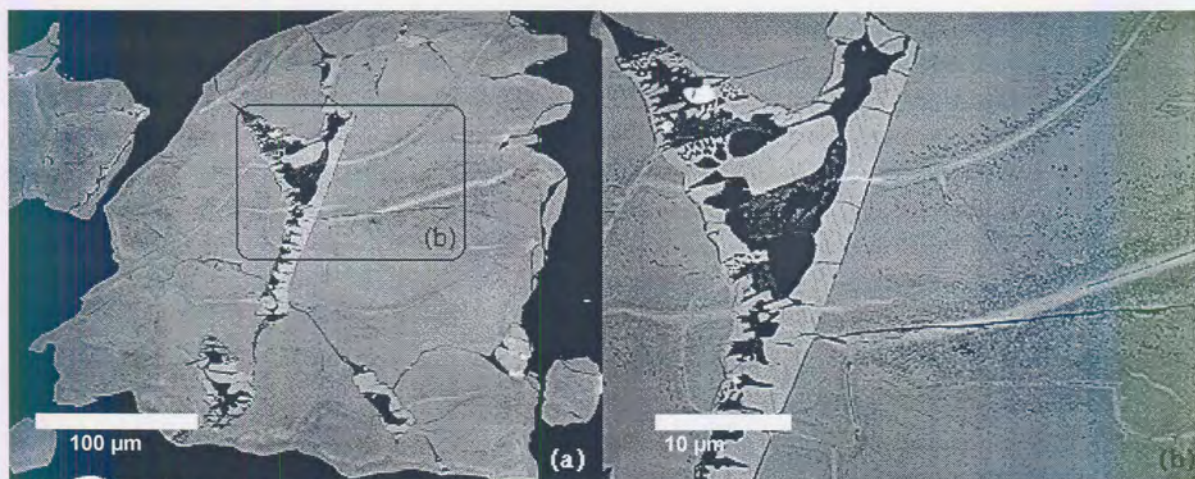
**Table 25.** Phase-chemical composition of the high iron containing slag PFE436 after oxidation at 850 °C with increasing time, given in order of decreasing abundance.

PFE	Time (h)	Mineralogical composition		
		Main	Minor	Trace
765	1	FeTi-Oxide/Anatase	Rutile	Ilmenite
767	2	FeTi-Oxide/Anatase	Rutile	Ilmenite
769	3	FeTi-Oxide/Anatase; Rutile	-	Ilmenite

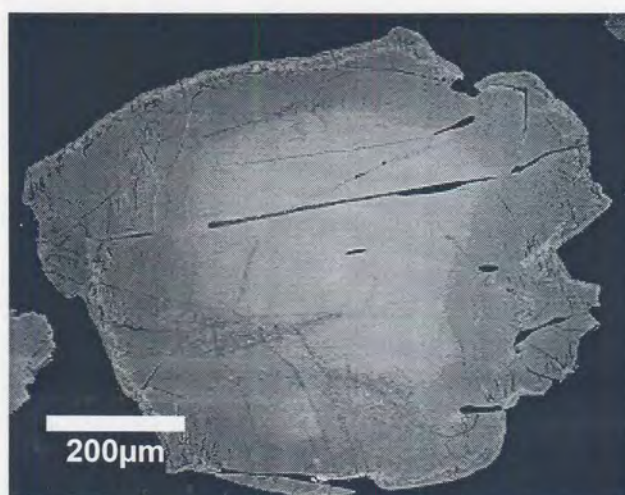
**Legend :** FeTi-Oxide -  $M_3O_5$ -solid solution; Rutile -  $TiO_2$ ; Anatase -  $TiO_2$ ; Ilmenite -  $FeTiO_3$ .

The optical and SEM analysis revealed similar features in the high iron slag compared to the standard slag. The most significant feature was the iron migration towards the outer margins of the individual slag particles as well as the edges of cracks (Figure 12). This feature was clearly recognised in all the samples. The sample oxidised for 1 h contained a number of the slag particles with large unreacted  $M_3O_5$ -cores and relatively “thin”  $TiO_2$ -rich mantles. Longer oxidation times resulted in a more complete conversion of the slag to rutile/anatase (Figure 13). After 3 h oxidation the size of the unreacted cores had diminished resulting in broader  $TiO_2$ -rich mantles. The finer-grained particles were completely oxidised and transformed to rutile/anatase. The effects of oxidation were also visible along cracks extending through the slag particles, as rutile and finely disseminated metallic iron precipitates were visible adjacent to cracks in the unreacted  $M_3O_5$  cores. With longer oxidation times the metallic iron appeared more abundant in the cracks. During oxidation the transformed mantles became slightly porous, while the unreacted cores remained dense.





**Figure 12.** High iron slag particle oxidised for 1 h at 850 °C displaying iron migration towards the edges of cracks leaving the adjacent areas enriched in titania and slightly porous. Particle core consisted of the  $M_3O_5$ -solid solution. The glass phase depicted in micrograph (b) contained ilmenite.



**Figure 13.** High iron slag particle oxidised at 850 °C for 3 h displaying a well defined zoned texture with  $M_3O_5$ -rich inner core,  $TiO_2$ -rich mantle and porous, iron-enriched outer rim.

#### High magnesia slag

The phase-chemical compositions of the slag samples as obtained by XRD analysis, are listed in Table 26. This shows that the  $M_3O_5$ -solid solution, anatase and rutile were present as main crystalline phases in the slag oxidised for 1 h at 850 °C. After 2 h of oxidation the amount of rutile increased to such an extent that the  $M_3O_5$  and anatase phases were reduced to minor phases. Trace amounts of ilmenite were present in this sample. This might be an intermediate product that formed during oxidation and it may be consumed with further oxidation. In the sample oxidised for three hours the amount of  $M_3O_5$  decreased to below that of anatase, while rutile was still present as the main phase.



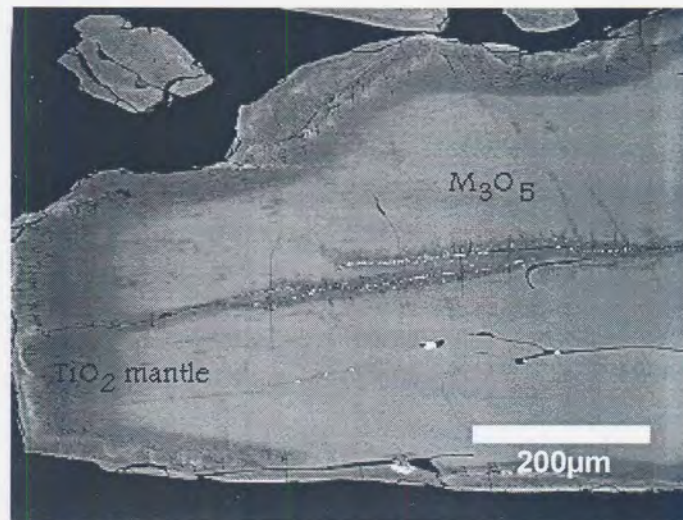
**Table 26.** Phase-chemical composition of the high magnesium slag PFE418 after oxidation at 850 °C with increasing time, given in order of decreasing abundance.

PFE	Time (h)	Mineralogical composition		
		Main	Minor	Trace
771	1	FeTi-Oxide/Anatase; Rutile	-	-
773	2	Rutile	FeTi-Oxide/Anatase	Ilmenite
775	3	Rutile	Anatase/FeTi-Oxide	-

**Legend :** FeTi-Oxide -  $M_3O_5$ -solid solution; Rutile -  $TiO_2$ ; Anatase -  $TiO_2$ ; Ilmenite -  $FeTiO_3$ .

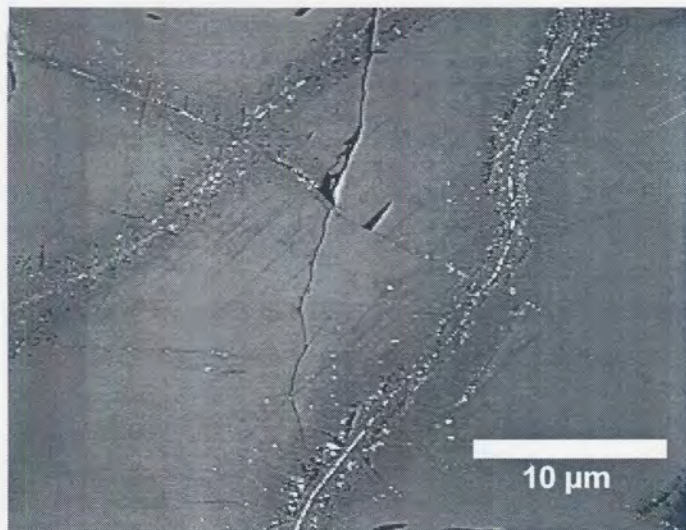
The optical and SEM analysis showed that the characteristic features of the high magnesium slag were similar to those of the standard slag. Most notably, iron migration also occurred towards the particle rims, but the amount of migration seemed less than that observed for the other slag compositions.

After 1 h oxidation the majority of the particles, including the finer-grained particles, contained relatively large unreacted  $M_3O_5$ -cores with only thin  $TiO_2$ -rich mantles. After 2 h oxidation the slag particles contained slightly smaller unreacted  $M_3O_5$ -cores and broader  $TiO_2$ -rich mantles. A large percentage of the finer-grained slag particles were completely oxidised, consisting mainly of rutile/anatase. After 3 h oxidation, unreacted  $M_3O_5$ -cores were evident only in the coarser-grained slag particles. All three oxidised slag samples contained some fine metallic iron precipitates along cracks extending through the slag particles (Figures 14 and 15). These precipitates were associated with rutile.



**Figure 14.** High magnesium slag (PFE418) particle oxidised for 1 h at 850 °C displaying  $M_3O_5$ -rich core and  $TiO_2$ -rich mantle with iron enrichment towards the edges of cracks and outer rim of the slag particle. Metallic iron precipitates were evident in the vicinity of internal cracks.





**Figure 15.** High magnesium slag (PFE418) particle oxidised for 1 h at 850 °C displaying  $M_3O_5$ -rich core with metallic iron precipitates associated with rutile along internal cracks extending through the particle.

### 3.3.3.3 Reduction

The different as-cast slags, which had been oxidised for respectively 1, 2, 3 and 4 h, were subjected to a reducing roast at 800°C. Samples, large enough for a leach test as well as mineralogical investigation, were taken at respectively 10, 20, 30 and 40 min.

#### *Standard slag*

The phase-chemical compositions of the individual reduced slag samples from the oxidised feed slag PFE437, as determined by XRD are shown in Table 27. The reduced slag samples consisted mainly of rutile and anatase with trace amounts of the  $M_3O_5$ -solid solution and ilmenite. When these results are compared with those presented in Table 23 for the oxidised samples it appears that no dramatic changes in the mineralogical composition of the samples occurred during the reduction process. The only noticeable difference is the presence of trace quantities of ilmenite in all of the reduced slag samples.

The reduced slag samples appeared very similar to the oxidised samples. The larger particles had unreacted  $M_3O_5$  cores surrounded by reacted mantles of  $TiO_2$ . Iron-enriched rims were present on the outsides of the particles. The smaller particles were completely reacted and did not contain  $M_3O_5$  cores. A significant difference between the oxidised samples and the reduced samples is that the iron-enriched rims converted to ilmenite in the reduced samples. The ilmenite was not visible during the optical analysis, but its presence was confirmed with EDS analysis based on the Ti/Fe ratio in the iron-enriched areas. Differences in the general appearance between slag particles that had been reduced at different temperatures for different time intervals were noticed. Particles containing unreacted cores were less abundant in the slag samples that had been reduced for respectively 20 and 40 min in comparison to those reduced for only 10 min.

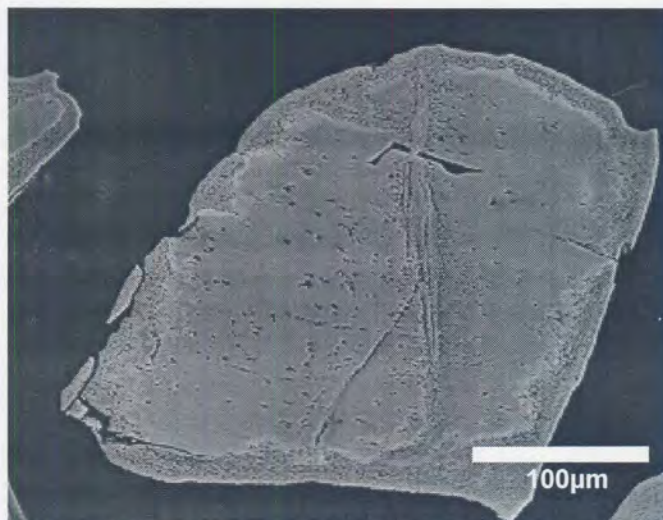


**Table 27.** Phase-chemical composition of standard slag (PFE437) which had been oxidised and reduced at different temperatures and times, given in order of decreasing abundance.

PFE	Roast conditions		Mineralogical composition		
			Main	Minor	Trace
698	1h 950°C	10 min	Rutile	-	FeTi-Oxide; Ilmenite; Anatase
700	Ox. (PFE696);	20 min	Rutile	-	FeTi-Oxide; Ilmenite
702	900°C red. for:	40 min	Rutile	-	FeTi-Oxide; Ilmenite
722	1h 850°C	10 min	Anatase; Rutile	-	FeTi-Oxide
724	Ox. (PFE720);	20 min	Anatase; Rutile	-	Ilmenite; FeTi-Oxide
726	800°C red. for:	40 min	Rutile; Anatase	-	Ilmenite; FeTi-Oxide
738	4h 850°C	10 min	Rutile; Anatase	-	FeTi-Oxide; Ilmenite
740	Ox. (PFE736);	20 min	Rutile; Anatase	-	FeTi-Oxide; Ilmenite
742	800°C red. for:	40 min	Rutile; Anatase	-	FeTi-Oxide
746	3h 800°C	10 min	Anatase	Rutile	FeTi-Oxide; Ilmenite
748	Ox. (PFE744);	20 min	Anatase	Rutile	FeTi-Oxide; Ilmenite
750	750°C red. for:	40 min	Anatase	Rutile	Ilmenite; FeTi-Oxide
827	3h 850°C Ox. (PFE929); 800°C red. for:	30 min	Anatase; Rutile	-	Ilmenite; FeTi-Oxide

**Legend:** Anatase -  $TiO_2$ ; Rutile -  $TiO_2$ ; FeTi-Oxide -  $M_3O_5$ -solid solution; Ilmenite -  $FeTiO_3$

The samples which had been oxidised at 950 °C, appeared almost completely reacted as very few particles containing unreacted  $M_3O_5$ -cores were present after reduction intervals of 10, 20 and 40 min. Some of the particles contained thin outer rims of densely packed rutile crystallites. Iron migration towards the edges of cracks as well as outer margins of the particles was noticeable.



**Figure 16.** Standard slag (PFE437) particle, which had been oxidised at 850 °C for 1 h and reduced for 40 min at 800 °C displaying porosity and iron migration towards the outer margins of the particle. This particular slag particle had no unreacted core.

The slag oxidised for 1 h at 850 °C and reduced for respectively 10, 20 and 40 min at 800 °C, contained particles with unreacted cores and  $TiO_2$ -rich mantles. Cracks extending through the unreacted cores were characterised by the presence of rutile and fine metallic iron precipitates. The metal appeared to be more abundant in the cracks in the sample reduced for 40 min. The slag particles varied from slightly porous to dense. The reacted marginal zones were generally more porous (Figure 16). The samples which were oxidised for 4 h at 850 °C and reduced at 800 °C for



10, 20 and 40 min, looked very similar to the samples oxidised for 1 h but there were fewer unreacted  $M_3O_5$  cores present.

The slag samples oxidised at 800 °C for 3 h and reduced at 750°C for respectively 10, 20 and 40 min, contained a large number of particles with unreacted cores and thin  $TiO_2$ -rich mantles. The outer margins were slightly porous and the remainder of the particles appeared to be dense. Metallic iron precipitates, associated with rutile, were present along cracks extending through the unreacted cores.

The slag sample oxidised at 850°C for 3 h and reduced at 800°C for 30 min (PFE827) contained dense unreacted  $M_3O_5$  cores in only a few larger particles. The rest of the particles were fully reacted. Precipitated carbon associated with the finer-grained slag particles was present in this sample.

### High iron slag

The phase-chemical results, as obtained by XRD, for the high iron slag subjected to oxidation and reduction, are presented in Table 28. This shows that ilmenite formed during the reduction roast. The amount of ilmenite that formed is significantly more than that formed during reduction of the standard slag. Ilmenite was present as a minor phase in the reduced high iron slag and it occurred in trace quantities in the reduced standard slag.

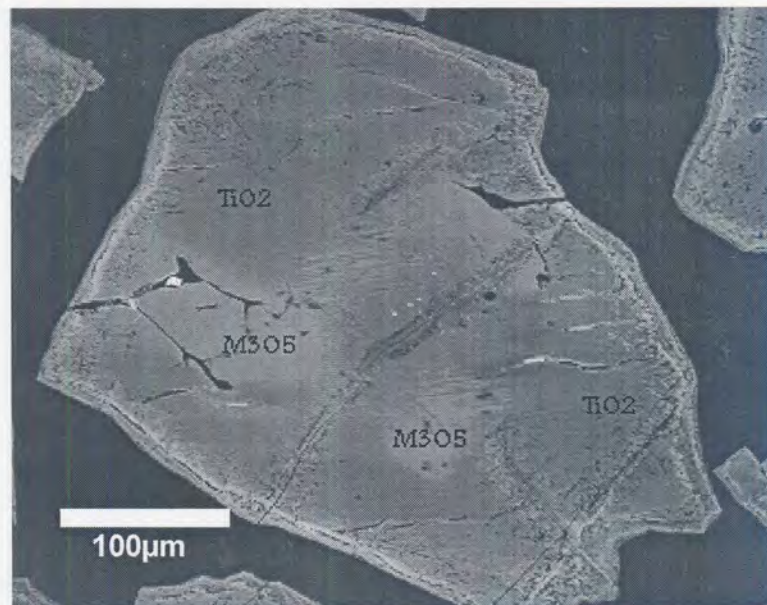
**Table 28.** Phase-chemical composition of the high iron slag (PFE436) which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min; given in order of decreasing abundance.

PFE	Oxidised slag	Mineralogical composition		
		Main	Minor	Trace
833	PFE769	Anatase; Rutile	Ilmenite	FeTi-Oxide

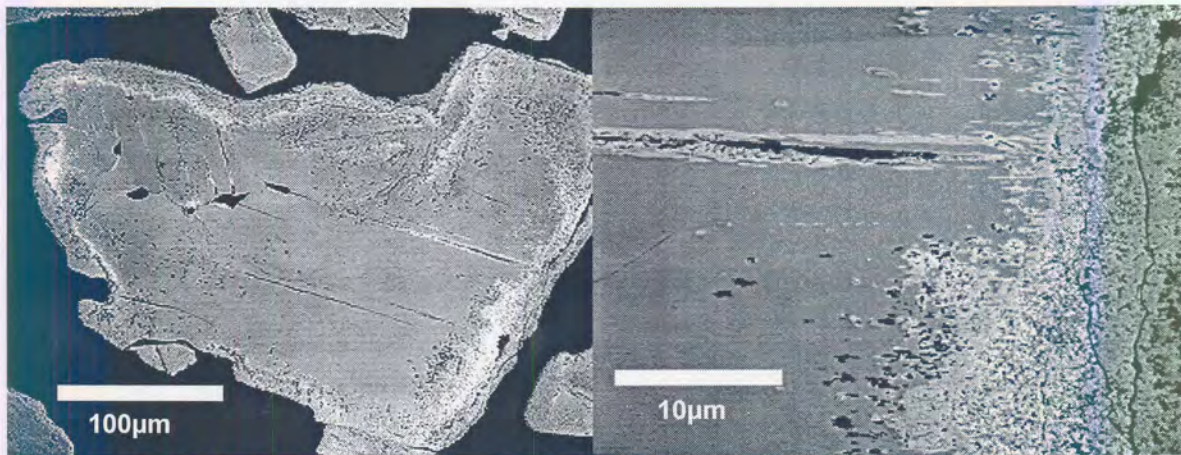
**Legend:** Anatase -  $TiO_2$ ; Rutile -  $TiO_2$ ; FeTi-Oxide -  $M_3O_5$ -solid solution; Ilmenite -  $FeTiO_3$

The sample had a zoned appearance. The centre of the particles consisted of the  $M_3O_5$  phase surrounded by a  $TiO_2$  mantle. A iron enriched layer was present on the outer margins of the particles. The iron in the iron-rich layer was present as ilmenite. The iron-rich layers appeared wider than those observed in the standard slag (Figures 17 and 18). The sample contained large quantities of glass at the grain and crystal boundaries of the original  $M_3O_5$ -crystals. The outer margins of the slag particles were porous with the core-areas, slightly denser. A few of the slag particles contained thin, dense rutile-rich outer rims. Some of the slag particles displayed fine, metallic iron precipitates associated with rutile along cracks extending through the slag particles. A few of the smaller particles had graphite borders.





**Figure 17.** High iron slag oxidised at 850°C for 3 h and reduced at 800°C for 30 minutes containing small unreacted  $M_3O_5$  cores and broad  $TiO_2$  mantles. Iron enrichment towards the outer margins of the particles can be observed.



**Figure 18.** Oxidised and reduced high iron slag particle displaying iron enrichment towards the particle rim and along the edges of cracks extending through the particle.

#### *High magnesia slag*

The phase-chemical composition of oxidised and reduced high magnesia slag, as determined by XRD, is shown in Table 29. The main phases were rutile and anatase. The  $M_3O_5$  phase was present in trace quantities along with the ilmenite that formed during reduction.

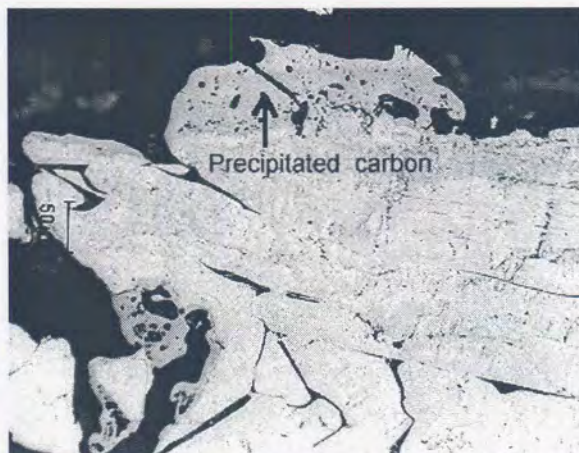
**Table 29.** Phase-chemical composition of the high magnesia slag (PFE418) which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min; given in order of decreasing abundance.

PFE	Oxidised slag	Mineralogical composition		
		Main	Minor	Trace
830	PFE775	Rutile; Anatase		Ilmenite; FeTi-Oxide

**Legend:** Anatase -  $TiO_2$ ; Rutile -  $TiO_2$ ; FeTi-Oxide -  $M_3O_5$ -solid solution; Ilmenite -  $FeTiO_3$



The SEM investigation revealed the presence of ilmenite on the rims of the particles. The metallic iron-rutile association was still present in the few unreacted cores. Precipitated carbon was present as a thin fragile rim around some of the smaller particles (Figure 19).



**Figure 19.** Optical micrograph of high magnesia slag which had been oxidised at 850 °C for 2 h and reduced at 800 °C for 30 min. Precipitated carbon associated with the particle is clearly visible.

#### 3.3.3.4 Leaching

All the reduced samples were subjected to leaching in boiling 20% HCl. After 5 h of leaching the solids and liquids were separated. The solids were then calcined at 800°C for 2 h.

##### *Standard slag*

The phase-chemical compositions of the leach residues after calcination, as determined by XRD, are shown in Table 30. The phase compositions of the leach residues were similar to that of the oxidised products presented in Table 23. This means that the ilmenite phase that formed during reduction is removed during leaching.

Macroscopically the leached residues displayed a brownish to yellowish tint, compared to the dull black appearance of the reduced slag samples. Optically, the leached residues had a severely “weathered” appearance compared to the oxidised as well as the reduced products. Several zones could be recognised in the particles (Figure 20). The centres of the larger particles consisted of unreacted  $M_3O_5$ . This was surrounded by a  $TiO_2$  layer that had a bluish appearance which is characteristic of anatase. The outer margins of the particles were affected by leaching and were very porous. This area had an amber appearance which is characteristic of rutile. The iron-enriched rims that were observed in the oxidised and reduced particles were almost completely removed in the leach residues. Small differences in microscopic appearance of the various leach residues were noticed. The slag reduced for 20 to 40 min appeared to be more completely leached compared to the slag samples reduced for 10 min. These particles displayed a more distinct “weathered” appearance with much broader leach affected rims. Leaching also occurred along cracks extending through the particles. Slag particles with unreacted  $M_3O_5$ -cores

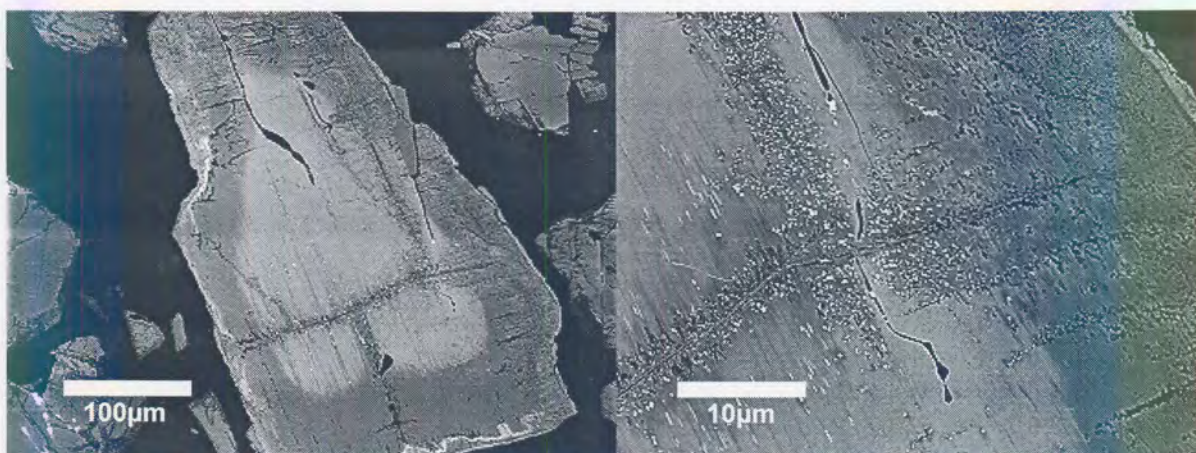


were mainly present in the samples reduced for 10 min. The glass phase in all the samples appeared to be unaffected by leaching.

**Table 30.** Phase-chemical composition of standard slag that had been oxidised and reduced at different temperatures and times, leached for 5 h and calcined at 800 °C for 2 h, given in order of decreasing abundance.

PFE	Roast conditions		Final TiO <sub>2</sub> content (%)	Mineralogical composition		
				Main	Minor	Trace
697	Ox: 1h at 950°C Red: at 900°C for:	0 min	85.50	Rutile		FeTi-Oxide; Anatase
699		10 min	86.60	Rutile		FeTi-Oxide
701		20 min	87.10	Rutile		FeTi-Oxide; Anatase
703		40 min	87.00	Rutile		FeTi-Oxide
721	Ox: 1h at 850°C Red: at 800°C for:	0 min	86.80	Anatase; Rutile		FeTi-Oxide
723		10 min	90.10	Anatase; Rutile		FeTi-Oxide
725		20 min	90.70	Anatase; Rutile		FeTi-Oxide
727		40 min	90.20	Rutile; Anatase		FeTi-Oxide
737	Ox: 4h at 850°C Red: at 800°C for:	0 min	85.70	Rutile; Anatase		FeTi-Oxide
739		10 min	89.00	Rutile; Anatase		FeTi-Oxide; Ilmenite
741		20 min	91.00	Rutile; Anatase		FeTi-Oxide
743		40 min	85.40	Rutile; Anatase		FeTi-Oxide
751	Ox: 4h at 800°C Red: at 750°C for:	40 min	89.40	Anatase; Rutile		FeTi-Oxide

**Legend:** Anatase - TiO<sub>2</sub>; Rutile - TiO<sub>2</sub>; FeTi-Oxide - M<sub>3</sub>O<sub>5</sub>-solid solution; Ilmenite - FeTiO<sub>3</sub>



**Figure 20.** Standard slag, oxidised for 1 h at 850 °C; reduced at 800 °C for 40 min; leached for 5 h and calcined at 800°C for 2 h.

#### High iron slag

The phase-chemical composition, as determined by XRD analysis, of the high iron slag (PFE436) after it had been oxidised at 850 °C for 3 h, reduced at 800 °C for 30 min, leached for times varying between 1 and 12 h and calcined are given in Table 31. This shows that the ilmenite phase decreased from a minor phase to a phase present in trace quantities after 1 h of leaching. No ilmenite was detected after 4 h of leaching and the anatase decreased from a major phase to a minor phase. Additional leaching up to 12 h did not change the phase composition of the slag, but the TiO<sub>2</sub> content increased.

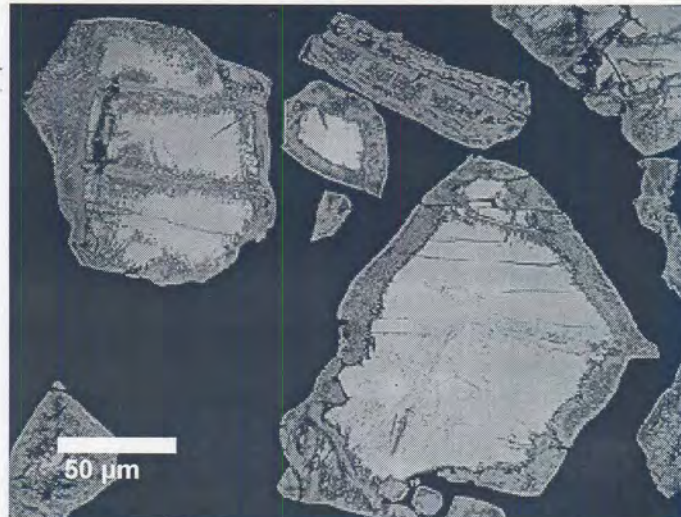


**Table 31.** Phase-chemical composition of high iron slag which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min, leached for different times and calcined; given in order of decreasing abundance.

PFE	Time (h)	Final TiO <sub>2</sub> content (%)	Mineralogical composition		
			Main	Minor	Trace
789	1	78.30	Rutile; Anatase	FeTi-Oxide	Ilmenite
791	4	85.80	Rutile	Anatase	FeTi-Oxide
794	12	88.30	Rutile	Anatase	FeTi-Oxide

**Legend:** Anatase - TiO<sub>2</sub>; Rutile - TiO<sub>2</sub>; FeTi-Oxide - M<sub>3</sub>O<sub>5</sub>-solid solution; Ilmenite - FeTiO<sub>3</sub>

These samples looked optically very similar to the leach residues of the standard slag. The centres of the particles were very dense and consisted primarily of anatase, while the rims of the particles, as a result of the leach procedure, were very porous. The areas affected by leaching consisted mainly of rutile. The sample which was leached for 12 h (Figure 21), was much more severely affected compared to sample which was leached for only 1 h. Leaching was especially effective at the outer rims of the slag particles as well as along cracks extending through the particles. The characteristic optical appearance of the leach residues can be seen in Figure 22. The interior of the particles appeared bluish, while the areas severely affected by leaching had an amber appearance. The predominance of rutile along the outsides of the particles might have been due to the calcination treatment. The finer-grained particles were more completely leached and appeared more porous than the coarser-grained particles. The unreacted M<sub>3</sub>O<sub>5</sub> cores that were present in the particles before leaching were still present after leaching. The most significant observation from the SEM analysis was that the ilmenite rims, observed in the reduced slag, were not visible in any of the leach residues. The ilmenite that was present in the glass phase also appeared to have been leached.



**Figure 21.** High iron slag that was oxidised, reduced and then leached for 12 h.





**Figure 22.** Optical micrograph of the high iron slag which was oxidised for 2 h at 850 °C, reduced for 30 min. at 800 °C, leached for 12 h and calcined. The exterior of the particles consisted predominantly of rutile and the interior predominantly of anatase.

#### *High magnesia slag*

The phase-chemical compositions of the leach residues from the high magnesia slag (PFE418), as obtained by XRD, are presented in Table 32. This shows that the leach residues consisted mainly of rutile. Anatase was present as a minor phase and the  $M_3O_5$  phase occurred in trace amounts. No change in the phase composition of the slag was noticed with increasing leach times. A comparison between the phase compositions of the leach residues and the reduced slag reveals that the ilmenite phase is removed during leaching. Anatase also declined from a major phase to a minor phase. This may have occurred as a result of calcination; before calcination the particles appeared greyish, but after calcination all the particles had a whitish-brown appearance.

**Table 32.** Phase-chemical composition of high magnesia slag which had been oxidised at 850 °C for 3 h and reduced at 800 °C for 30 min, leached for different times and calcined; given in order of decreasing abundance.

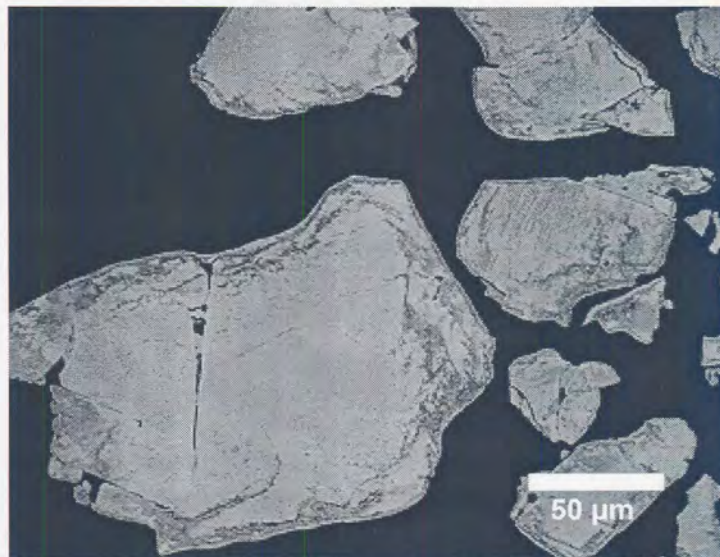
PFE	Time (h)	Final $TiO_2$ content (%)	Mineralogical composition		
			Main	Minor	Trace
796	1	82.40	Rutile	Anatase	FeTi-Oxide
798	4	86.00	Rutile	Anatase	FeTi-Oxide
801	12	89.10	Rutile	Anatase	FeTi-Oxide

**Legend:** Anatase -  $TiO_2$ ; Rutile -  $TiO_2$ ; FeTi-Oxide -  $M_3O_5$ -solid solution; Ilmenite -  $FeTiO_3$

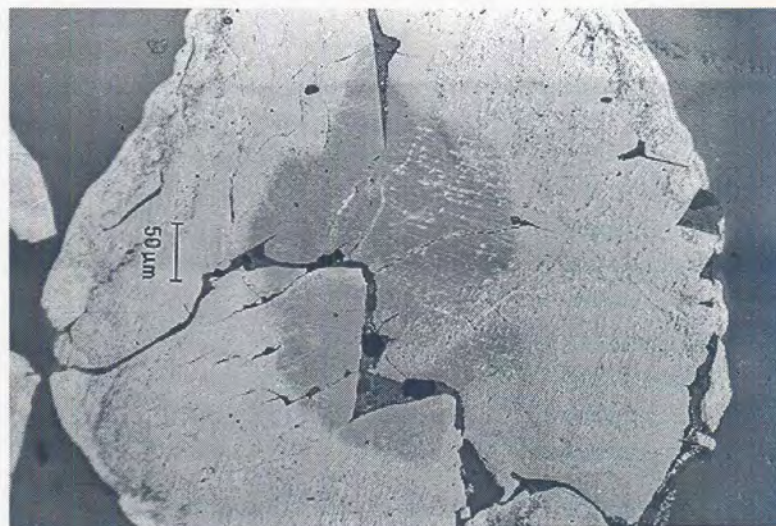
The individual slag particles of the high magnesia slag (Figure 23), oxidised for 3 h at 850 °C, reduced at 800 °C for 30 minutes, leached for 1 h and calcined, appeared very dense and only a very thin rim on the outside of the particles was affected by leaching. Some of the particles contained unreacted  $M_3O_5$  cores. Fine metallic iron precipitates associated with rutile, could still be recognised situated along cracks extending through the unreacted cores. After 12 h of leaching the slag particles appeared more “weathered” and the affected outer margins were broader (Figure 24). The SEM analysis confirmed that, similarly to the standard and high iron slag, the iron-enriched layer on the outsides of the particles had been removed during



leaching. It also showed that the effect of leaching was much less severe on the high magnesia slag than on the standard slag and the high iron slag.



**Figure 23.** High magnesia slag, leached for 1 h; the effect of leaching is visible mainly at the outer margins of the individual slag particles.

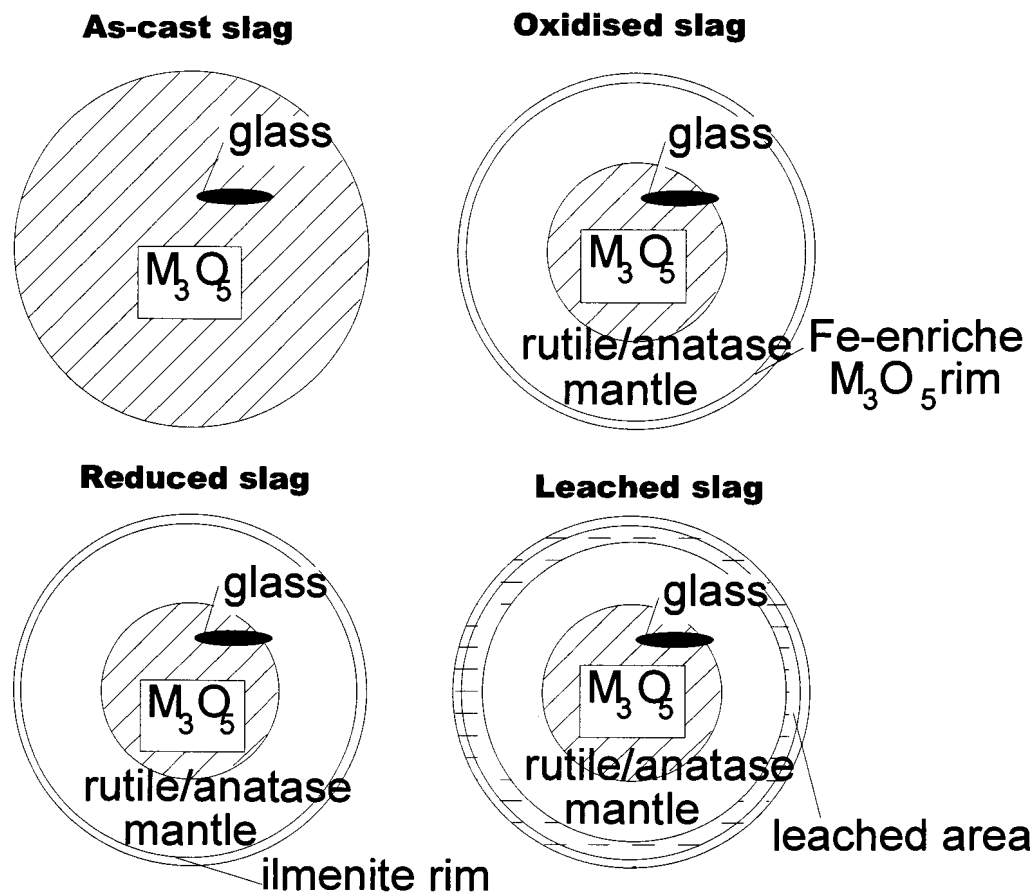


**Figure 24.** Optical micrograph of the high magnesia slag, which was oxidised for 2 h at 850 °C, reduced for 30 min. at 800 °C, leached for 12 h and calcined. The particle display a zoned appearance, in the center is an unreacted core surrounded by a mantle of anatase, while the rims consist of rutile.

### 3.3.4 Summary of the mineralogical changes that occur during roasting

#### 3.3.4.1 Standard slag

A standard slag by definition contains about 10 % FeO and about 85 % TiO<sub>2</sub>. In the **as-cast** form the slag consist mainly of the M<sub>3</sub>O<sub>5</sub> solid solution phase (Figure 25). During **oxidation** the M<sub>3</sub>O<sub>5</sub> phase converted to anatase, rutile and a new iron rich M<sub>3</sub>O<sub>5</sub> phase.



**Figure 25.** Summary of the morphological changes that occur during the production of BTS

Some of the larger slag particles retained unreacted  $M_3O_5$  cores after oxidation. Iron migrated to the outside rims of the particles and probably existed as a  $M_3O_5$  type solid solution as the XRD results indicated that the  $M_3O_5$  phase was still the only iron-containing phase present in the slag. Two distinct phases could be recognised in this zone. From the SEM photographs there appears to be a difference in the iron content of the two phases. The reacted mantles of the particles were porous and consisted of a mixture of rutile and anatase. In the unreacted cores metallic iron precipitates in association with rutile occurred around cracks. Variation of the oxidation time and temperature changed the ratio of anatase to rutile in the slag. With short oxidation times anatase was dominant, while at longer oxidation times rutile became more dominant. Rutile was also dominant at higher oxidation temperatures and with increased oxidation times at the high temperature anatase completely disappeared. At lower oxidation temperatures anatase was dominant but the kinetics of oxidation slowed down significantly. **Reduction** resulted in the formation of ilmenite. The ilmenite formed in the iron-enriched rims on the outsides of the particles. The metallic iron/rutile association was still present in the unreacted cores. Rutile seemed to occur mainly on the outsides of the particles, while anatase dominated in the centres. **Leaching** removed mainly the ilmenite phase. The leached parts of the particles consisted predominantly of rutile, while the un-leached parts consisted of anatase. This observation could have been due to calcination of all the samples prior to analysis. The smaller particles were leached completely and had a “weathered”



appearance. Only the rims of the larger particles were leached and some still had unreacted  $M_3O_5$  cores. The metallic iron-rutile association was still present in these cores. The glass phase in all the particles were unaffected by leaching.

#### 3.3.4.2 High iron slag

High iron slag by definition contains more than 10 % FeO. In the **as-cast** form the slag consisted mainly of the  $M_3O_5$  solid solution phase, but rutile and ilmenite were also present in small quantities. The ilmenite occurred as crystals in a silica glass phase. **Oxidation** of the high iron slag also proceeded in a shrinking core fashion. This led to unreacted  $M_3O_5$  cores in the particle centres surrounded by mantles of  $TiO_2$ . Oxidation resulted in iron migration to the outer rims of the slag particles as well as the edges of cracks extending through the particles. The amount of iron migration seemed to be more than that observed in the standard slag, probably because there was more iron present in the slag. After the oxidation procedure the  $M_3O_5$  phase, rutile and anatase occurred as major phases in the slag, while the ilmenite was unaffected. The appearance of the unreacted  $M_3O_5$  cores changed to a paler colour during **reduction** and they seemed somewhat smaller than in the oxidised slag. The amount of ilmenite increased from trace quantities to minor quantities as some of the iron in the iron-enriched zones was converted to ilmenite. At the same time the  $M_3O_5$  phase decreased from a major phase to a phase that was only present in trace amounts. The slag particles became porous with a “weathered” appearance during **leaching**. The leach procedure removed mainly the ilmenite phase (including the ilmenite in the glass phase) and after 1 h of leaching there was no more ilmenite present in the slag. The leached areas consisted of rutile, while the unleached areas consisted mainly of anatase.

#### 3.3.4.3 High magnesia slag

High magnesia slag by definition contains more than 2 % MgO. This slag could result from poor slag making practice when the refractory lining of the slag furnace is eroded or from high MgO levels in the ilmenite feedstock to the slag furnace. The slag in the **as-cast** form also consists of the  $M_3O_5$  solid solution phase. The **oxidised** slag had the same zoned appearance as the standard slag with unreacted cores in the particle centres surrounded by a mantle of  $TiO_2$ . Iron migration also occurred towards the outside rims of the particles and to cracks extending through the particles, but the amount of iron migration was much lower than that observed in the standard slag. At the start of the oxidation procedure anatase and rutile were the dominant phases along with the  $M_3O_5$  solid solution. With increased oxidation times rutile became the major phase and anatase and  $M_3O_5$  occurred as minor phases. Ilmenite was present as a minor phase after 2 h oxidation, but it disappeared with longer oxidation times. The amount of anatase increased to a main phase during **reduction** and the  $M_3O_5$  phase decreased to trace quantities. Ilmenite also formed in the iron-enriched zones at the particle rims. Iron sulphide was picked up in the reduced sample. **Leaching** removed the ilmenite phase.

### 3.4 Conclusions

- The optimum roast conditions are oxidation at 850 °C for 3 h and reduction at 800 °C for 30 min.
- The oxidation roast is the most crucial part of the BTS process as important structural changes occur in the slag during this process stage that influence all the subsequent BTS process stages. These structural changes are the conversion of the original  $M_3O_5$  phase to rutile and a new iron rich  $M_3O_5$  phase and the migration of iron to the outside rims of the slag particles.
- The effectiveness of the oxidation roast is influenced by the roasting temperature and the chemical composition of the slag.
- High iron and high magnesia slags do not yield BTS of the required grade.
- During oxidation iron migration occurs towards the outsides of the slag particles where reduction converts it to ilmenite. The ilmenite phase is removed during leaching.
- The rate of leaching of iron from the roasted slag is a strong function of hydrochloric acid concentration. The final purity of the BTS product increased slightly with increasing acid strength after extended leaching.
- The rate of leaching is independent of the level of stoichiometric excess hydrochloric acid. The level of stoichiometric excess did not affect the final purity of the BTS product.
- The optimum leaching conditions are 12 h in boiling 20 % HCl at a 20 % stoichiometric excess.
- The highest BTS grade produced was 94 %  $TiO_2$ .

## 4. PROCESS DEVELOPMENT PHASE 2

### 4.1 Introduction

Phase 1 of the process development for the production of BTS was conducted with a coal fired fluid bed roaster. This did not allow close control over parameters such as oxygen potential. The aim of the second phase of the process development was to refine the roast conditions necessary for the production of BTS with a 95 % TiO<sub>2</sub> content. The previous work showed that oxidation was the most critical part of the BTS process. The bulk of the work during this phase was therefore concentrated on oxidation of standard titania slag. Some experiments were also conducted on high iron titania slag as the high iron content magnifies the mechanisms that operate during roasting.

### 4.2 Experimental design

#### 4.2.1 Feed material

The slag used for this investigation was obtained from the 6<sup>th</sup> IHM 3MVA plasma furnace campaign of July 1997. The complete analyses are listed in Appendix VII. Table 33 shows the concentration of selected elements in the feed slag. For the purposes of this investigation the feed slags were placed in two categories based on their chemical composition namely standard slag and high iron slag. Table 34 gives the phase composition of the slag as determined by XRD.

**Table 33.** Chemical composition of the feed slags used in this investigation.

PFE no.	Classification	TiO <sub>2</sub> %	Ti <sub>2</sub> O <sub>3</sub> %	Fe %	FeO %	Fe <sub>2</sub> O <sub>3</sub> %
469	standard	52.3	32.1	0.03	9.3	-
437	high iron	58.40	13.60	-	21.90	-

**Table 34.** Phase-chemical composition of the feed slags used in this investigation.

PFE no.	Classification	Major Phases	Minor Phases	Trace Phases
469	Standard	FeTi-Oxide	-	Rutile
437	High iron	FeTi-Oxide	-	Ilmenite, Iron

*Legend: FeTi-Oxide – M<sub>2</sub>O<sub>3</sub> solid solution; Rutile – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>*

#### 4.2.2 Experimental plan

In previous investigations it was shown that oxidation is the most important stage of the process for the production of BTS. Therefore it was decided to focus on investigating variables that have an influence on this process stage. To evaluate the effectiveness of the oxidation roast it was decided to subject most of the samples to a standard reducing roast and leach procedure. This procedure was only modified in the case of the reduction time investigation where a standard oxidation roast and leach procedure were used. The final TiO<sub>2</sub> content or the amount of iron extracted from the slag after leaching was used as an indication of the effectiveness of the oxidation roast. The following parameters were investigated:

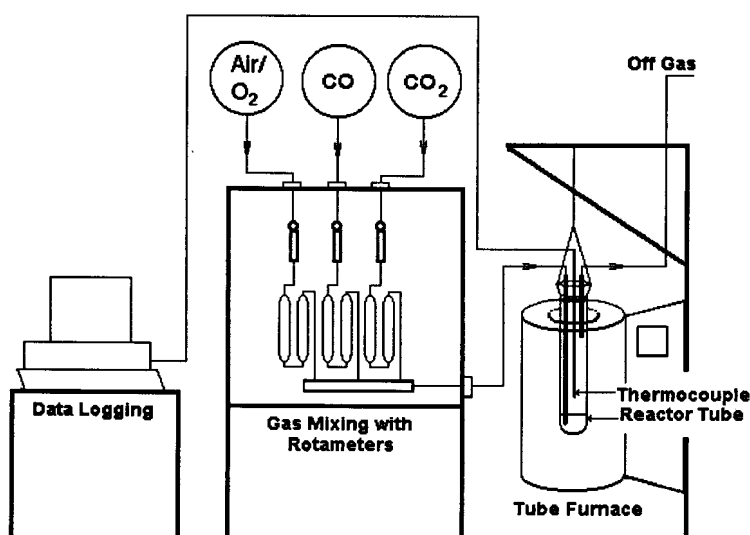


- Oxidation atmosphere;
- Oxidation temperature;
- Oxidation time;
- Reduction time and;
- Particle size.

The experimental plan was designed to investigate the effect of all the above-mentioned parameters on the standard slag, but the effects of only some of them were evaluated for the high iron slag. A list of all the experiments that were conducted on the various feed slags is given in Appendix VIII.

### 4.2.3 Experimental procedure

#### 4.2.3.1 Roast procedure



**Figure 26.** Experimental set-up used for the roast experiments.

Roasting was conducted in a small fluid bed roaster. The set-up consisted of a 20 mm diameter glass reactor, a tube furnace and a gas mixing system (Figure 26). The feed material was titania slag, separated in several narrow size fractions. About 20 – 50 g slag was loaded into the reactor before each experiment. The gas mixing system was then used to provide the oxidation atmosphere inside the reactor with air, carbon dioxide, oxygen or mixtures of these gases. The flow rates of the gases were controlled with rotameters to a total flow of 11 L/min at 20 °C. This flow rate was selected after the minimum and maximum gas flow rates for fluidisation were calculated (Appendix XIII).

The experiment started as soon as the reactor was lowered into the pre-heated tube furnace. The temperature inside the fluidised bed was measured with a thermocouple. The temperature inside the fluid bed initially increased sharply by about 150 to 200 °C, but it started to decline after about 5 min. After about 10 min the

bed temperature was within 5 °C of the target value and it remain there for the duration of the experiment. After the oxidation roast the experiment was either stopped and cooled to room temperature, to allow a sample to be taken, before the reduction roast, or the oxidation gases were directly changed to carbon monoxide gas. Previous experiments had indicated no noticeable difference in the roasting behaviour of titania slag between experiments that were interrupted after oxidation (with cooling to room temperature) and experiments where no interruption occurred. Once the reducing roast was finished the reactor was removed from the furnace and allowed to cool in air. The slag sample was then removed from the reactor. Part of the sample was submitted for chemical and mineralogical analysis and the rest of the slag sample was subjected to a leach experiment.

#### 4.2.3.2 Leach procedure

Leach experiments were conducted in 2 L Erlenmeyer flasks fitted with reflux condensers. The purpose of the condensers was to limit evaporation losses. The reactors were placed on a hot plate that kept the leach solution at boiling point. No additional stirring was provided. The concentration of the feed solutions was 20 weight % hydrochloric acid. The reactor was filled with 0.5 L solution and once boiling point was reached about 15-30 g dry roasted feed material was charged. Pulp samples were taken at timed intervals, filtered and reserved for chemical analysis. The experiments were stopped after 12 h. The intermediate and final solution samples were submitted for a complete chemical analysis. At the end of the leach the slurry was filtered and repeatedly repulped and washed until the pH of the wash water was neutral. Special care was taken to limit losses of solids during the bulk filtration and washing operations. The filter cake was dried at 110 °C overnight, weighed and submitted for full chemical analysis.

#### 4.2.3.3 Electron microprobe analysis procedure

Selected slag samples were mounted in resin before they were mechanically ground and polished to a surface roughness of around 1 µm. An optical microscope was then used to select three particles in each sample for analysis. The positions of the selected particles were marked with a diamond tip marker and a layer of carbon was deposited onto the polished blocks to prevent the build up of an electrostatic charge on the surface during analysis. Following this, the coated samples were placed into an electron microprobe (ARL SEMQ-37) fitted with a wavelength dispersive spectrometer (WDS). Thereafter the marked particles were located with an optical microscope fitted to the microprobe and their positions were stored on a computer. The microprobe then performed a series of point chemical analysis on each of the marked particles, starting at the outside surface and moving to the centre in 5 µm intervals. The electron beam that was used had a spot size of 1 to 3 µm, while the operational accelerating voltage was 15 kV and the beam current was 0.03 µA. Each analysis took approximately 20 s to complete.

#### 4.2.3.4 Mössbauer analysis procedure

$^{57}\text{Fe}$  Mössbauer-effect spectroscopy (MES) is a (nuclear)  $\gamma$ -radiation resonance technique involving the recoil-free emission of probing radiation by the source and the subsequent recoil-free absorption of this  $\gamma$ -radiation by  $^{57}\text{Fe}$  nuclei in the absorber i.e. sample of interest. As such it is extremely sensitive to the local surroundings of  $^{57}\text{Fe}$  atoms; these have a 2% natural abundance and are therefore present in all iron-bearing compounds. The local surroundings of the iron nuclei are primarily constituted by the electronic structure of the Fe atom, the compositional make-up of neighbouring atoms and by neighbouring defect structures. Therefore in the case of iron-bearing compounds the local-probe nature of the technique makes it a powerful non-destructive analytical tool for, establishing the oxidation state of iron (e.g., ferrous or ferric), quantifying the abundance's of different iron compounds within the same sample (i.e., phase analysis) and establishing the overall crystal chemistry when used in conjunction with complementary techniques like XRD and SEM. Typically iron-phase abundance's to as low as 5% may be detected with high accuracy. Moreover, as an atomic-scale local probe it is equally effective in the analysis of poorly crystallised or amorphous materials where conventional macroscopic-type probes like XRD have considerable limitations. The spectral parameters that serve as fingerprints in conventional transmission MES are the, isomer shift  $\delta$ , quadrupole splitting  $\Delta$  and internal magnetic field  $B_{\text{hf}}$ , all of which serve to characterise the chemical state of Fe in the solid phase.

The titania slag samples were prepared by milling it to a fine powder. MES measurements were conducted at room-temperature in the conventional transmission geometry using a 5-10 mCi  $^{57}\text{Co}(\text{Rh})$  radioactive Mössbauer source. Typically 40-50  $\text{mg}/\text{cm}^2$  of sample was used, corresponding to 5-10  $\text{mg}/\text{cm}^2$  of Fe. This is considered to be an optimal thickness to avoid spectral lineshape distortions, ensure acceptable (i.e., at least 30 %) transmission of the incident radiation when taking into consideration electronic absorption effects, and to have adequate quantities of the  $^{57}\text{Fe}$  isotope in the sample to ensure a satisfactory signal-to-noise ratios after typical 12-24 hour data accumulation periods. Some samples showed evidence of very low iron content in which case data accumulation was extended for 3-4 days to obtain an adequate signal-to-noise ratio for a reliable analysis. Data analysis was effected by using the non-linear least squares Mössbauer analysing programme NORMOS (supplied by WISSEL Starnberg-Germany) using a minimum number of spectral components, each having a Lorentzian lineshape. Final parameters of the fitted components were compared with the accepted literature values for various iron-oxides and iron-titanium-oxides to make a phase identification. Oxidation states of Fe in the various phases were unambiguously determined from the combination of values of fitted  $\delta$  and  $\Delta$  parameters obtained for quadrupole doublets or from the  $B_{\text{hf}}$  value of magnetically split sextets.

## 4.3 Results and Discussion

### 4.3.1 Standard titania slag

#### 4.3.1.1 Oxidation

##### *The effect of oxidation time*

The main purpose of the oxidation roast is to oxidise all the easily leachable Ti(III) in the slag to largely insoluble Ti(IV). During this procedure all the Fe(II) that is present in the slag is oxidised to Fe(III). The effect of oxidation on the slag was studied by varying the duration of the oxidation roast from 0.5 to 8 h. Table 35 gives the phase-chemical composition of the samples after oxidation. This shows that the amount of  $M_3O_5$  in slag declines and the amount of rutile increases with an increase in oxidation time. Traces of anatase are present in all the samples.

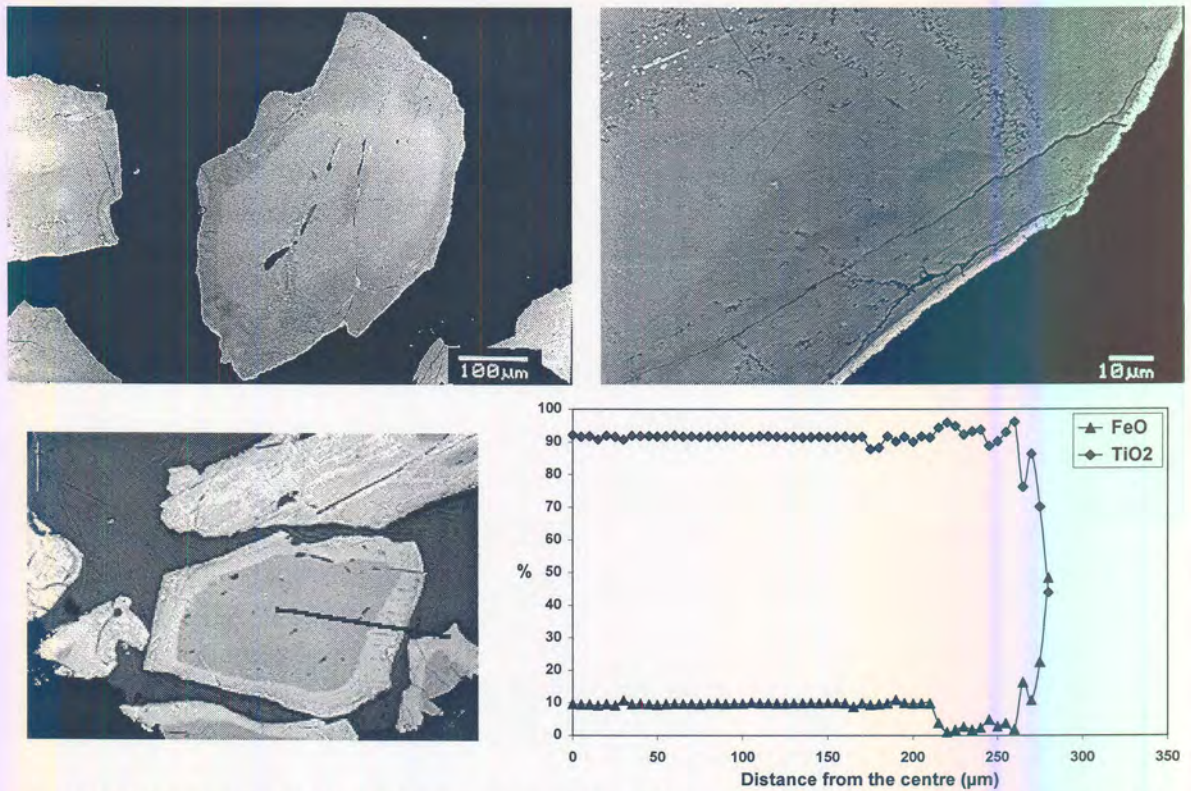
**Table 35.** The phase-chemical composition, as determined by XRD, of standard slag after oxidation. The samples are categorised by the oxidation time used. For the experiments listed the slag was oxidised at 850 °C in 8 %  $O_2$ .

Phase composition after oxidation		Major Phases	Minor Phases	Trace Phases
Sample	PFE			
0.5 h	1172/1382	FeTi-Oxide	Rutile	Anatase
1 h	1173/1383	FeTi-Oxide; Rutile	-	Anatase
2 h	986/1375	Rutile; FeTi-Oxide	-	Anatase
3 h	1384	Rutile	FeTi-Oxide	Anatase
4 h	1174/1377	Rutile	FeTi-Oxide	Anatase
8 h	1378	Rutile	FeTi-Oxide	Anatase

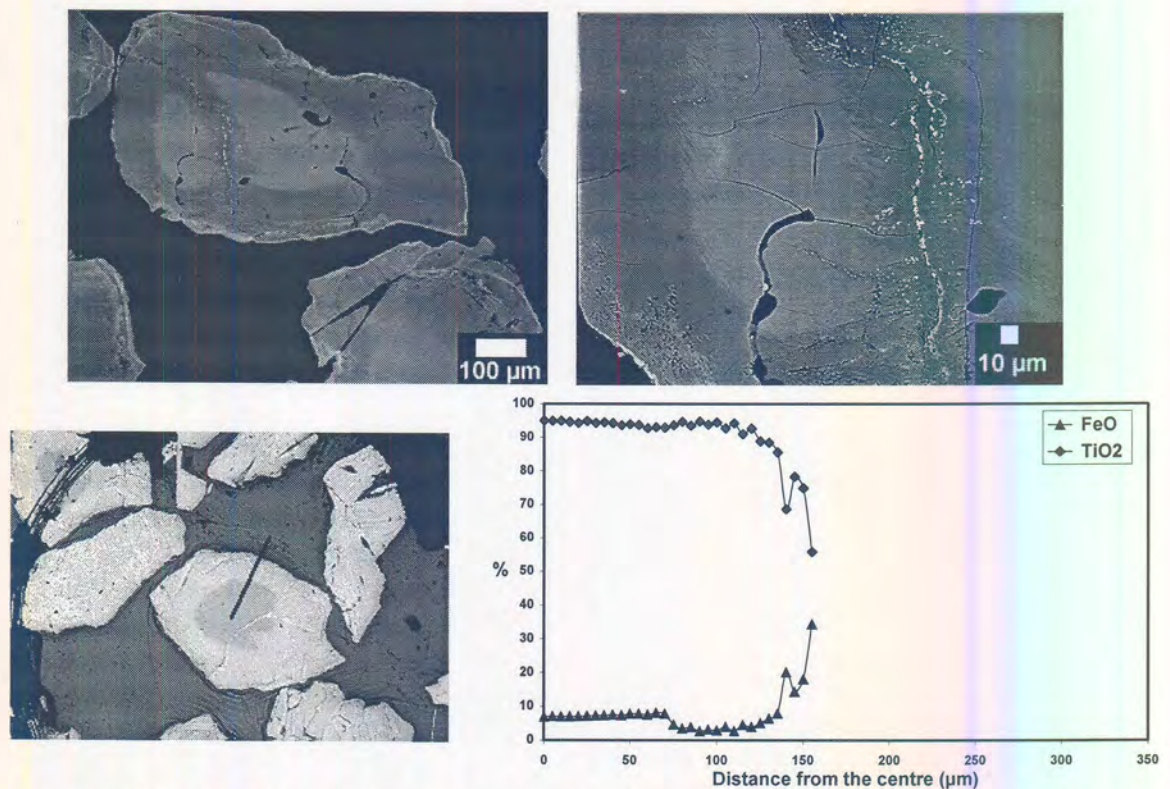
*Legend: FeTi-Oxide –  $M_3O_5$  solid solution; Rutile –  $TiO_2$ ; Anatase –  $TiO_2$ ; Ilmenite –  $FeTiO_3$ ; Iron –  $Fe^0$*

An optical and SEM investigation of the samples revealed that oxidation proceeded in a shrinking core fashion. The slag samples oxidised for 0.5 and 1 h (Figures 27 and 28) contained many particles with a zoned appearance. The cores of the particles were still unreacted and appeared relatively dense, but in the vicinity of cracks passing through the cores metallic iron particles were visible in association with rutile. The unreacted cores were surrounded by oxidised zones: These mantle zones were slightly porous and enriched in  $TiO_2$ . Another zone could be distinguished on the outsides of the particles. This outer shell was characterised by a very high concentration of iron. The slag samples oxidised for 2 and 3 h (Figure 29) appeared slightly different from the slag oxidised for shorter periods. The majority of the particles were completely oxidised, but some of the coarser-grained particles had small, unreacted,  $M_3O_5$  cores. The slag particles were in general slightly porous with iron-enriched rims. All the slag particles appeared to be completely oxidised after 4 and 8 h (Figure 30). Optically no particles with unreacted  $M_3O_5$ -cores could be recognised. Fine pores were present throughout the majority of slag particles. Some particles, however, had a dense appearance. This was accompanied by the presence of mainly anatase in the centres of the individual slag particles. Rutile was in general abundant in the mantle zones and outer margins of the slag particles as well as being associated with cracks extending through the particles. There were also particles with well-defined rutile-rich outer rims. From the chemical composition profiles it is clear that almost all of the iron had migrated to the outsides of the particles and that the cores consisted of almost pure  $TiO_2$ .



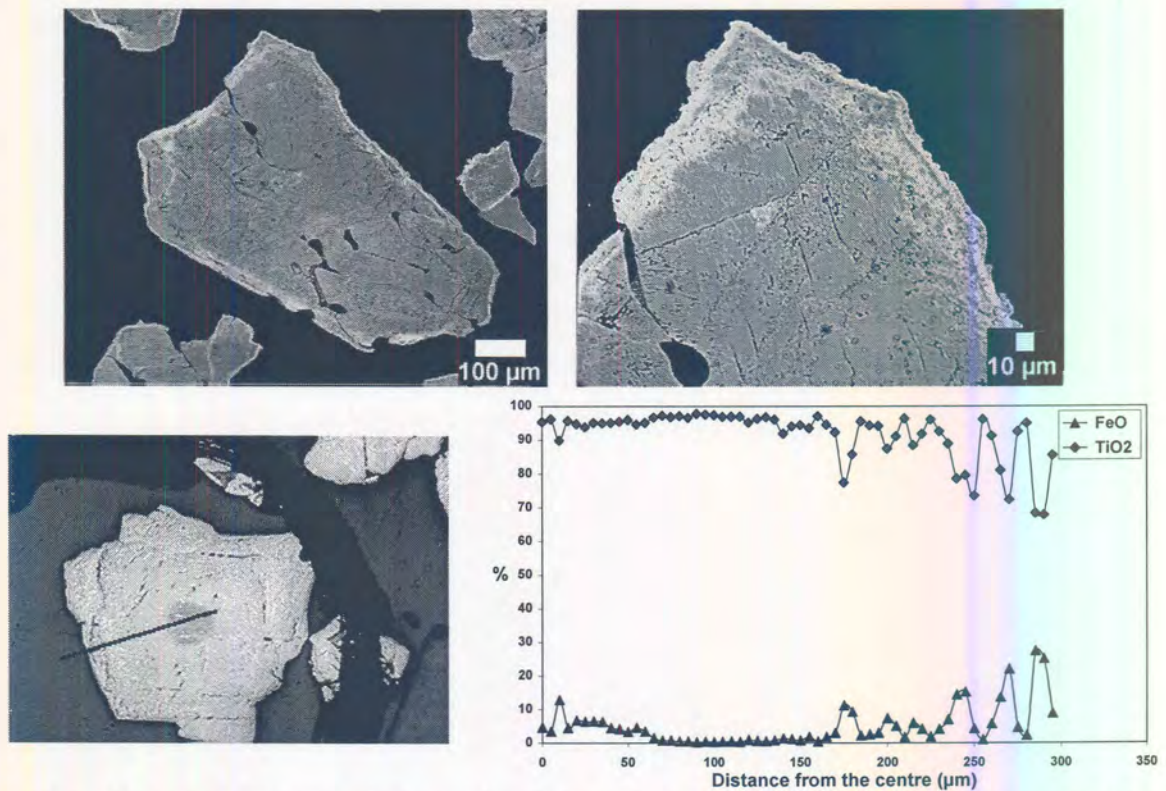


**Figure 27.** Standard titania slag oxidised for ½ h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.

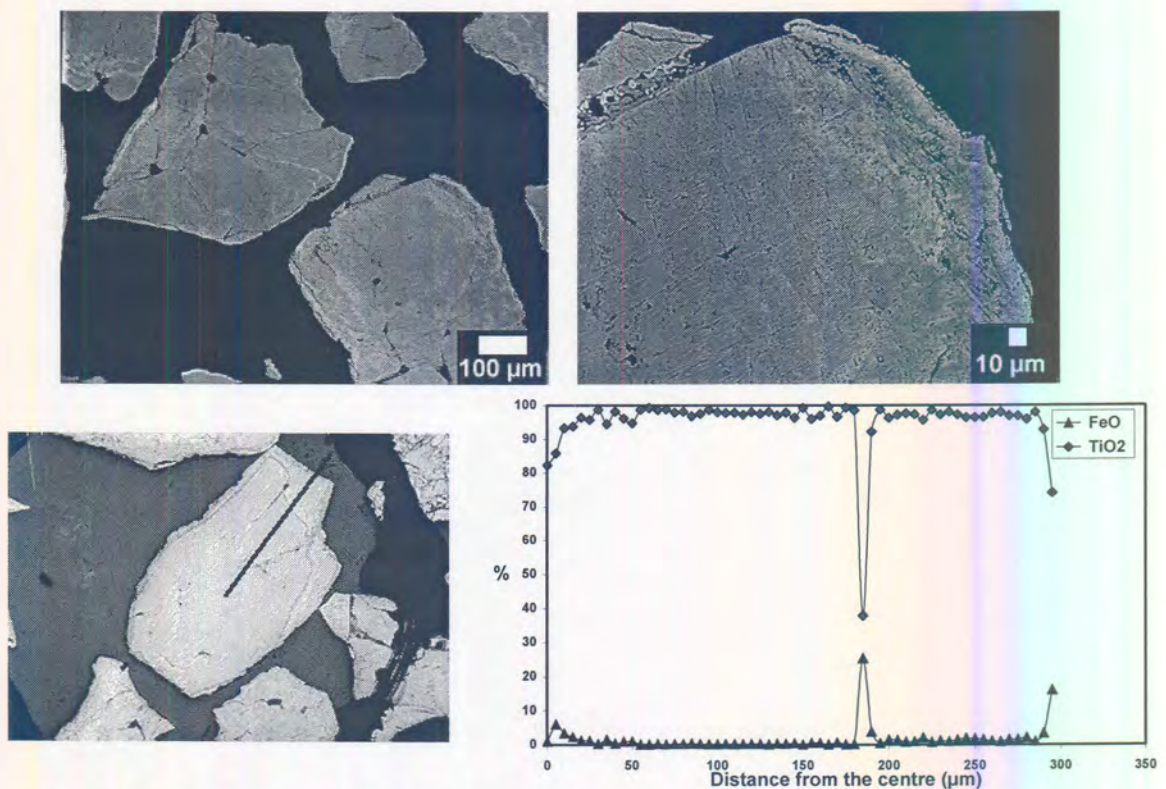


**Figure 28.** Standard titania slag oxidised for 1 h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.





**Figure 29.** Standard titania slag oxidised for 2 h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.



**Figure 30.** Standard titania slag oxidised for 4 h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.

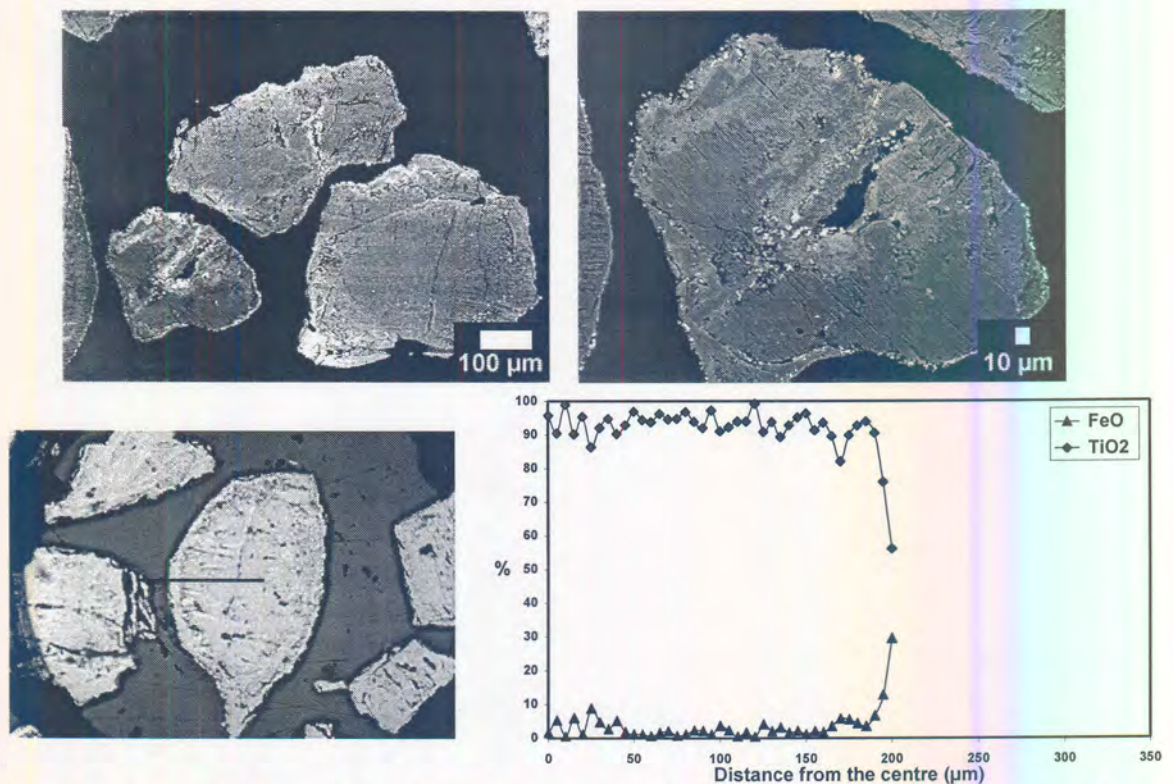


During reduction no dramatic structural changes occurred in the slag, but an important phase change took place. Table 36 shows that ilmenite formed. The optical and SEM investigation indicated that the ilmenite formed in the areas of high iron concentration, on the rims of the particles (Figure 31). It is important to note that not all of the iron was converted to ilmenite and that some of the iron remained in the  $M_3O_5$  phase in the unreacted cores of the particles, as indicated by XRD.

**Table 36** The phase-chemical composition, as determined by XRD, of standard slag after oxidation and reduction. The samples are categorised by the oxidation time used. For the experiments listed the slag was oxidised at 850 °C in 8 %  $O_2$  and reduced in 100 % CO for 20 min

Phase composition after reduction		Major Phases	Minor Phases	Trace Phases
Sample	PFE			
0.5 h	1421	FeTi-Oxide; Rutile	-	Ilmenite; Anatase
1 h	1427	Rutile; FeTi Oxide	Anatase	Ilmenite
2 h	1356	Rutile	FeTi-Oxide	Ilmenite; Anatase
3 h	1433	Rutile	-	FeTi-Oxide; Ilmenite; Anatase
4 h	1362	Rutile	-	FeTi-Oxide; Ilmenite; Anatase
8 h	1368	Rutile	-	FeTi-Oxide; Ilmenite

**Legend:** FeTi-Oxide –  $M_3O_5$  solid solution; Rutile –  $TiO_2$ ; Anatase –  $TiO_2$ ; Ilmenite –  $FeTiO_3$ ; Iron –  $Fe^0$



**Figure 31.** Standard titania slag oxidised for 2 h at 850 °C in 8 %  $O_2$  and reduced for 20 min in 100 % CO. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.

Table 37 gives the phase composition of the samples after oxidation, reduction and leaching. The absence of ilmenite suggests that ilmenite is the main phase that leaches, but the increase in the relative amount of anatase also suggests that some iron is leached from the  $M_3O_5$  phase. The optical and SEM investigation showed that the particles became very porous during leaching (Figure 32), while the chemical

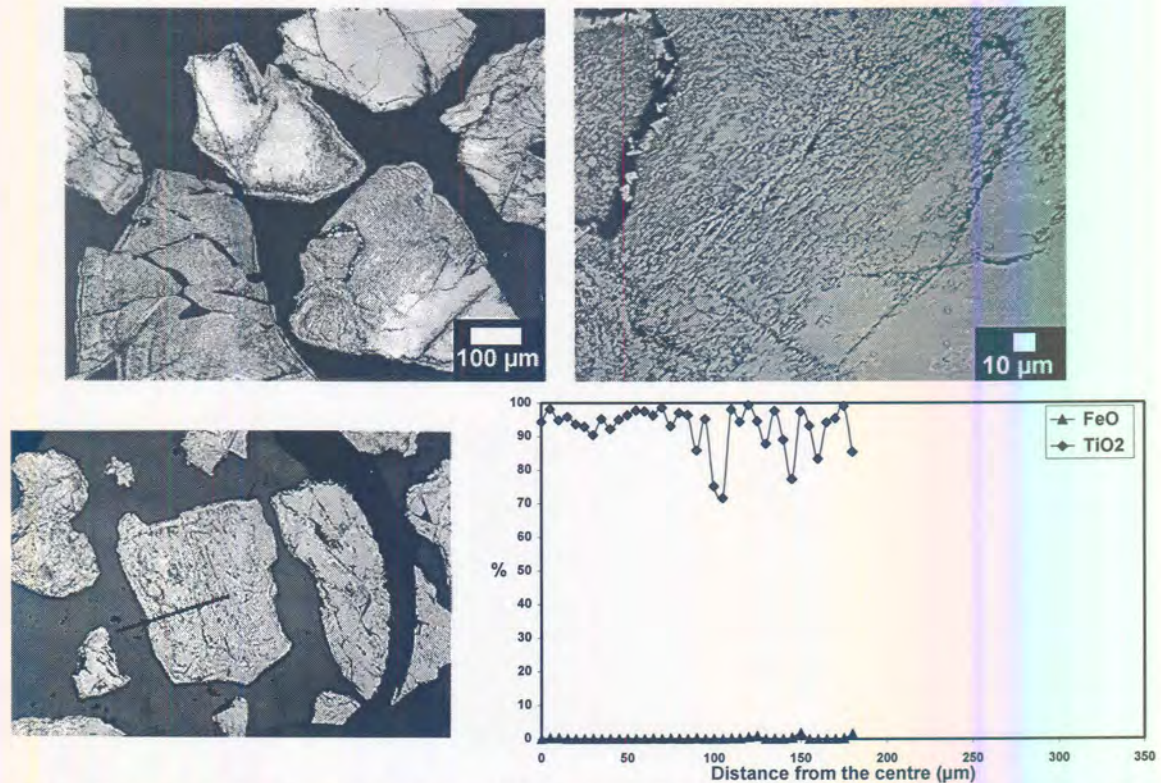


composition profiles indicates that most of the iron had been removed from the sample oxidised for 2 h.

**Table 37.** The phase-chemical composition, as determined by XRD, of standard slag after oxidation, reduction and leaching. The samples are categorised by the oxidation time used. For the experiments listed the slag was oxidised at 850 °C in 8 % O<sub>2</sub>, reduced in 100 % CO for 20 min and leached in 20 % HCl for 12 h.

Phase composition after leaching		Major Phases	Minor Phases	Trace Phases
Sample	PFE			
0.5 h	1426	Rutile	Anatase; FeTi-Oxide	-
1 h	1432	Rutile	Anatase	FeTi-Oxide
2 h	1361	Rutile	Anatase	FeTi-Oxide
3 h	1438	Rutile	-	Anatase
4 h	1367	Rutile	Anatase	FeTi-Oxide
8 h	1373	Rutile	Anatase	FeTi-Oxide

Legend: FeTi-Oxide – M<sub>3</sub>O<sub>5</sub> solid solution; Rutile – TiO<sub>2</sub>; Anatase – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>; Iron – Fe<sup>0</sup>



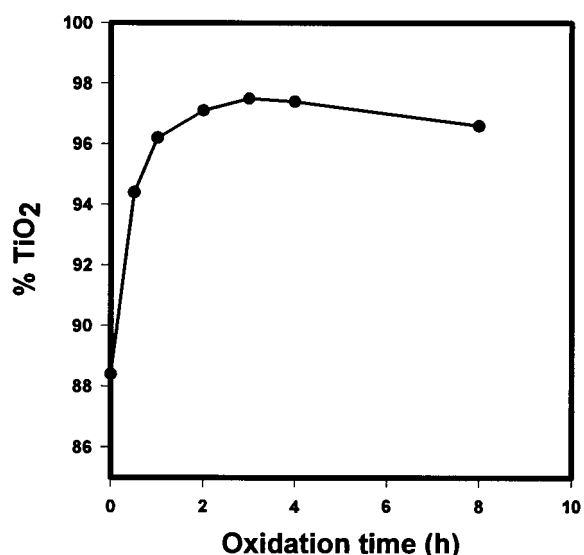
**Figure 32.** Standard titania slag oxidised for 2 h at 850 °C in 8 % O<sub>2</sub>, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.

The sample oxidised for 0.5 h, reduced for 20 min in 100 % CO at 850 °C and leached for 12 h in boiling HCl had a weathered appearance but contained abundant particles with unaltered M<sub>3</sub>O<sub>5</sub>-cores. The outer margins appeared to be in general, relatively well leached and porous while the particles centres appeared to be unaffected. The finer-grained particles appeared to be more porous compared to the coarser-grained particles thus also slightly better leached. Fine metallic iron particles, associated with rutile along cracks extending through the slag particles, were still present and could be observed optically in some of the slag particles.

The samples oxidised for 1 and 2 h respectively, reduced in a 100 % CO atmosphere for 20 min at 850 °C and leached for 12 h in boiling HCl contained only a small number of slag particles with unreacted  $M_3O_5$  cores. The slag particles displayed a weathered appearance and were porous due to leaching. The sample oxidised for 3 h; reduced in a 100 % CO atmosphere for 20 min at 850 °C and leached for 12 h in boiling HCl had a severely weathered appearance. The slag appeared porous but the porosity was concentrated at the outer rims and mantle zones of the individual slag particles.

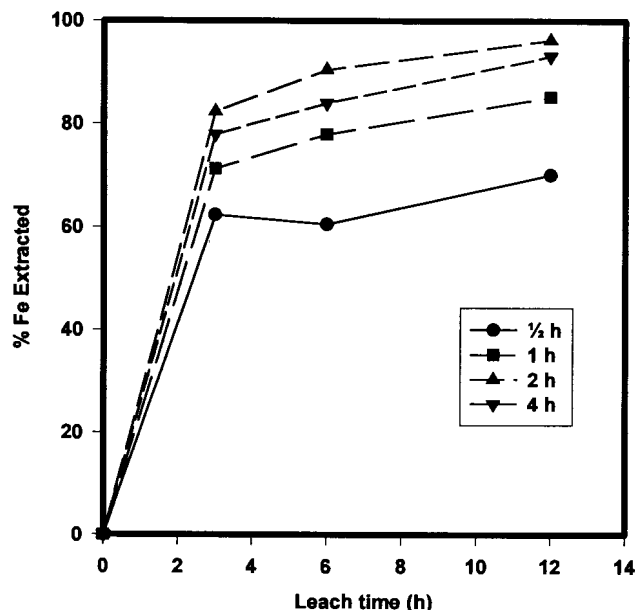
The samples oxidised for 4 and 8 h respectively reduced in a 100 % CO atmosphere for 20 min at 850 °C and leached for 12 h in boiling HCl appeared to be completely leached; the majority of the particles had a severely weathered appearance. Slag particles displaying a relatively dense texture were also present. These dense particles consisted mainly of anatase.

Figures 33 and 34 show the effect of oxidation time on the final  $TiO_2$  content of the slag and the percentage iron extracted during leaching. The log sheets of the experiments are given in Appendices IX to XII. Increasing the oxidation time from ½ to 3 h increased the iron extraction and the final  $TiO_2$  content. This is probably a result of the decrease in the size of the unreacted  $M_3O_5$  cores in the particles. When the slag is oxidised for more than 4 h the iron extraction and final  $TiO_2$  content decreased slightly.



**Figure 33.** The influence of oxidation time during roasting of standard slag on BTS product grade. The slag was oxidised at 850 °C in 8 %  $O_2$ , reduced in 100 % CO for 20 min and leached in boiling HCl for 12 h.





**Figure 34.** The influence of oxidation time, during roasting of standard slag, on iron extraction during leaching. The slag was oxidised at 850 °C in 8 % O<sub>2</sub>, reduced in 100 % CO for 20 min and leached in boiling HCl for 12 h.

To quantify the phase changes that occur during oxidation a Mössbauer investigation was conducted (See Appendix XVIII). Standard titania slag was oxidised for different times at 850 °C in a 10 % O<sub>2</sub> atmosphere and characterised by XRD and Mössbauer spectroscopy. Table 38 shows the phase chemical composition of the samples. The results are similar to that of the oxidised samples presented in Table 35 except for the fact that more anatase seems to be present in these samples. The Mössbauer results indicated the presence of the ilmenite-hematite solid solution that were not detected by XRD; this is probably as a result of the fact that the concentrations of these phases were below the detection limit of the XRD technique. The Mössbauer results also differentiated between the two end members of the FeO.2TiO<sub>2</sub>-Fe<sub>2</sub>O<sub>3</sub>.TiO<sub>2</sub> solid solution phase.

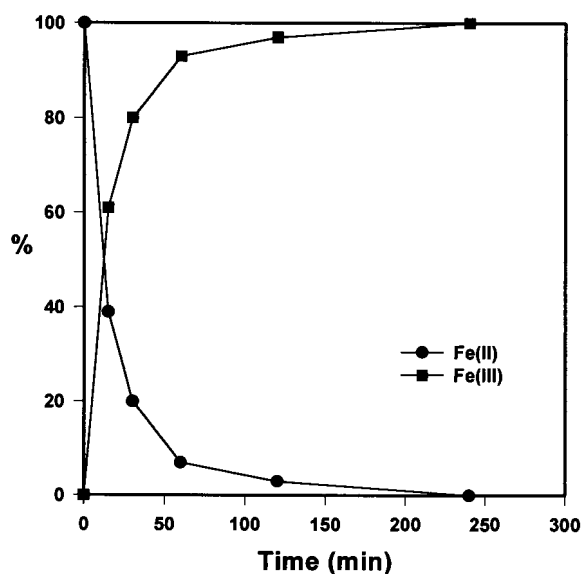
**Table 38.** Phase chemical analysis, as determined by XRD, of the oxidation samples used for the Mössbauer investigation.

Oxidised for:	PFE	Mineralogical composition		
		Main	Minor	Trace
0 min	-	FeTi-Oxide	Rutile	-
15 min	3060	Anatase, Rutile	Rutile, FeTi-Oxide	-
30 min	3061	Anatase, Rutile	FeTi-Oxide	-
60 min	3062	Anatase, Rutile	FeTi-Oxide	-
120 min	3063	Rutile, Anatase	FeTi-Oxide	-
240 min	3064	Rutile, Anatase	FeTi-Oxide	-

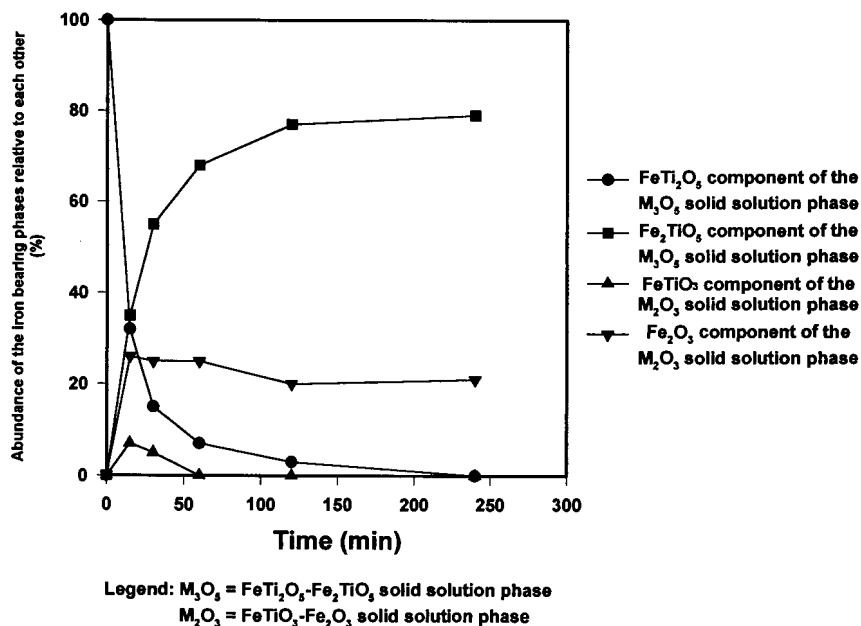
**Legend:** FeTi-Oxide – M<sub>3</sub>O<sub>5</sub> solid solution; Rutile – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>; Iron – Fe<sup>0</sup>

The change in oxidation state of iron in the samples relative to the total amount of iron in the slag is presented in Figure 35. This shows that 95 % of the Fe(II) in the

slag is oxidised to Fe(III) after 2 h of roasting. The changes in the abundance of the iron bearing phases relative to each other are shown in Figure 36. This indicates that the  $\text{FeO} \cdot 2\text{TiO}_2$  phase, originally present in the slag, is rapidly converted to  $\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$ , the hematite-ilmenite solid solution phase. The ilmenite component of the  $\text{M}_2\text{O}_3$  solid solution forms initially and then declines to zero after 60 min. The hematite part of the  $\text{M}_2\text{O}_3$  solid solution also declines with increasing oxidation time, but a significant quantity was still present after 4 h.



**Figure 35.** The change in the oxidation state of iron during oxidation roasting of standard titania slag as determined by Mössbauer analysis.



**Figure 36.** Changes in the relative concentration of the iron containing phases during oxidation roasting of standard titania slag as determined by Mössbauer analysis.



### The effect of oxidation atmosphere and temperature

The final  $TiO_2$  content of the slag and the iron extraction after leaching are also dependent on the oxidation atmosphere and the oxidation temperature. The influence of the oxidation atmosphere was evaluated by varying the oxygen content of the fluidising gas.

Table 39 shows the phases present in the slag after oxidation. This data indicates that the amount of  $M_3O_5$  in the slag declines and the amount of rutile increases as the oxygen content of the gas during oxidation increases. It also indicates that the oxygen content of the oxidation gas influences the kinetics of oxidation. In an atmosphere containing 100 %  $CO_2$  there is still a large amount of  $M_3O_5$  left after oxidation, while in an atmosphere containing 4 %  $O_2$   $M_3O_5$  is still a main phase in the oxidised product although more rutile than  $M_3O_5$  is present in the slag. In samples oxidised in an atmosphere containing 8% or more  $O_2$  the  $M_3O_5$  phase is only present as a minor phase. Table 39 also shows that ilmenite formed during reduction along with some iron. The phase-chemical compositions of the leach residues indicates that ilmenite is removed during leaching. It also shows that the sample oxidised in  $CO_2$  did not leach well as there was still a large amount of  $M_3O_5$  present in the leach residue. In the other leach residues the  $M_3O_5$  phase was relegated to a trace component. This suggests that the  $M_3O_5$  phase is partially removed during leaching.

**Table 39.** The phase chemical composition as determined by XRD of standard slag after oxidation, reduction and leaching. The samples are categorised by the oxidation atmosphere that was used. The slag was oxidised at 850 °C for 2 h, reduced in 100 % CO for 20 min and leached in boiling 20 % HCl for 12 h.

Phase composition after oxidation		Main phases	Minor phases	Trace phases
Sample	PFE			
$CO_2$	985	FeTi-Oxide; Rutile	-	Anatase
4% $O_2$	988	Rutile; FeTi-Oxide	-	Anatase
8% $O_2$	986	Rutile	FeTi-Oxide	Anatase
12% $O_2$	987	Rutile	FeTi-Oxide	Anatase
21% (Air)	984	Rutile	FeTi-Oxide	Anatase
100% $O_2$	1499	Rutile	FeTi-Oxide	Anatase
Phase composition after reduction		Main phases	Minor phases	Trace phases
Sample	PFE			
$CO_2$	-	-	-	-
4% $O_2$	1052	Rutile	FeTi-Oxide	Ilmenite; Anatase; Iron
8% $O_2$	1356	Rutile	FeTi-Oxide; Anatase	Ilmenite
12% $O_2$	1047/1314	Rutile	FeTi-Oxide	Ilmenite; Anatase; Iron
21% (Air)	-	-	-	-
100% $O_2$	1469	Rutile	FeTi-Oxide	Ilmenite; Anatase; Iron
Phase composition after leaching		Main phases	Minor phases	Trace phases
Sample	PFE			
$CO_2$	1042	Rutile	FeTi-Oxide	-
4% $O_2$	1056	Rutile; Anatase	-	FeTi-Oxide
8% $O_2$	1046/1361	Rutile; Anatase	-	FeTi-Oxide
12% $O_2$	1051/1319	Rutile	Anatase	FeTi-Oxide
21% (Air)	1038	Rutile	FeTi-Oxide	Anatase
100% $O_2$	1474	Rutile; Anatase	-	FeTi-Oxide

Legend: FeTi-Oxide –  $M_3O_5$  solid solution; Rutile –  $TiO_2$ ; Anatase –  $TiO_2$ ; Ilmenite –  $FeTiO_3$ ; Iron –  $Fe^0$

The influence of temperature was investigated by varying the roasting temperature between 750 °C and 950 °C. Table 40 gives the phase-chemical composition of the samples after oxidation, reduction and leaching. From this data it is clear that the kinetics of oxidation is influenced by roasting temperature. At 750 °C the oxidation of the  $M_3O_5$  phase is only partially completed after 2 h, while it is substantially completed at 850 °C after the same time period. The amount of rutile relative to the amount of anatase in the slag also seems to increase as the roasting temperature increases. The presence of ilmenite in the reduced samples is once again shown in Table 40. No other major phase changes seem to have taken place during reduction. The phase composition of the leach residues also confirms that roasting temperature influences the kinetics of oxidation. In the samples oxidised at 750 °C and 800 °C there was still a large amount of  $M_3O_5$  present that was not removed during leaching. There was no ilmenite present in the leach residues. It also seems that the amount of rutile relative to the amount of anatase increased as the roasting temperature increased.

**Table 40.** The phase-chemical composition as determined by XRD-analysis of standard slag after oxidation, reduction and leaching. The samples are categorised by the roasting temperature that was used. The slag was oxidised in 8 %  $O_2$  for 2 h, reduced in 100 % CO and leached for 12 h in boiling 20 % HCl.

Phase composition after oxidation		Main phases	Minor phases	Trace phases
Sample	PFE			
750°C	981	FeTi-Oxide	Rutile	Anatase
800°C	-	-	-	-
850°C	986/1375	Rutile	Anatase; FeTi-Oxide	Iron
950°C	-	-	-	-
Phase composition after reduction		Main phases	Minor phases	Trace phases
Sample	PFE			
750°C	-	-	-	-
800°C	1403	FeTi-Oxide	Rutile	Anatase
850°C	1067/1356	Rutile; Anatase	FeTi-Oxide	Ilmenite; Iron
950°C	-	-	-	-
Phase composition after leaching		Main phases	Minor phases	Trace phases
Sample	PFE			
750°C	1026	FeTi-Oxide	Rutile	Anatase
800°C	1408	FeTi-Oxide	Rutile; Anatase	-
850°C	1046/1361	Rutile; Anatase	-	FeTi-Oxide
950°C	1071	Rutile	-	FeTi-Oxide

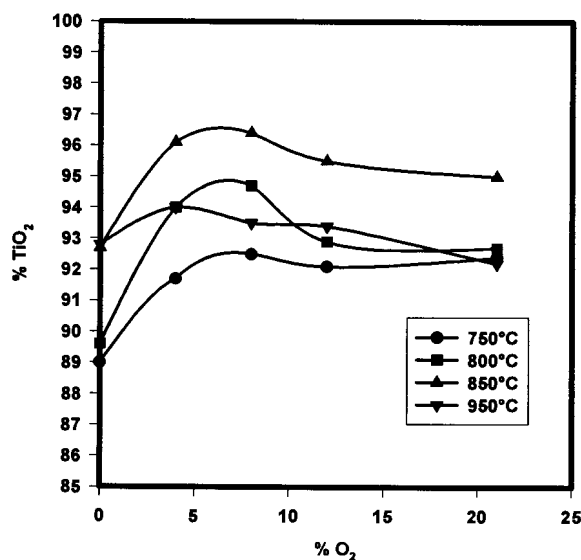
Legend: FeTi-Oxide –  $M_3O_5$  solid solution; Rutile –  $TiO_2$ ; Anatase –  $TiO_2$ ; Ilmenite –  $FeTiO_3$ ; Iron – Fe

The influence of the oxygen content of the oxidation atmosphere and the roasting temperature on the final  $TiO_2$  content and the amount of iron extracted during leaching are shown in Figures 37, 38 and 39. The log sheets of the experiments are given in Appendices IX to XII. The oxygen content of the oxidation gas was varied by mixing air and  $CO_2$ .

Figures 37, 38 and 39 show that the final  $TiO_2$  content and the iron extraction are influenced by the roasting temperature and the oxygen content of the oxidation atmosphere. When roasting is performed in  $CO_2$  the kinetics of oxidation is very slow as a result of the kinetics of the  $CO_2$  dissociation reaction on the surface of the particles (Abuluwefa, et al., 1997). This results in a low iron extraction and a low final  $TiO_2$  content in the slag. In air/ $CO_2$  mixtures with an oxygen content between 4 % and 12 %  $O_2$  the kinetics of oxidation improves dramatically and the iron extraction and final  $TiO_2$  content of the slag are much higher than for slag oxidised in  $CO_2$ . The

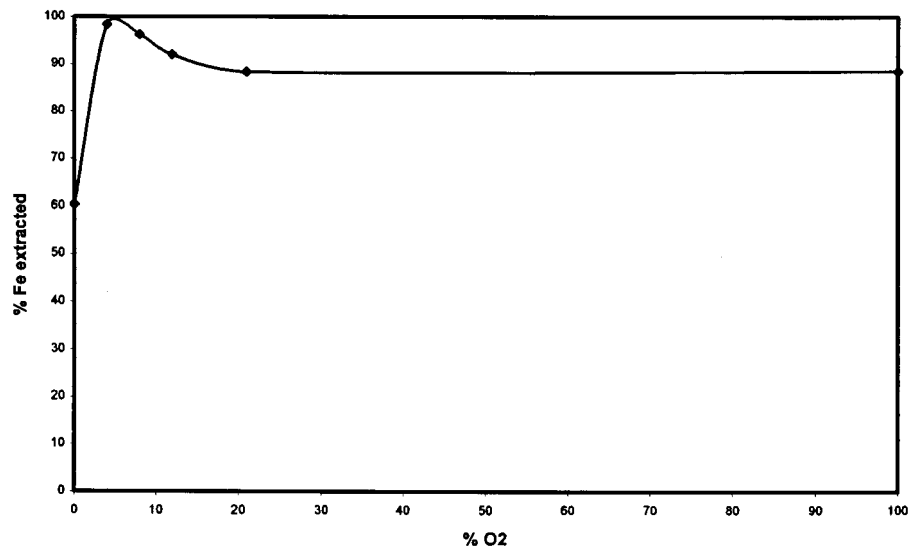
effect of oxidation for 2 h appears to be very similar at 750 °C, 800 °C and 850 °C. In this temperature range the final TiO<sub>2</sub> content generally increases with a increase in the roasting temperature. There also appears to be an optimum oxygen concentration in this temperature range between 4 % and 8 % O<sub>2</sub>. This optimum is much sharper at 800°C, in terms of the final TiO<sub>2</sub> content, than it is at 750 °C and 850 °C. This behaviour is unexpected as the difference between the PO<sub>2</sub> of a gas mixture containing 8% O<sub>2</sub> and air containing 21 % O<sub>2</sub> is very small. This suggests a kinetic effect – rather than an equilibrium one – in the achievement of the optimal structure during oxidation for subsequent removal of iron. This possible effect is discussed further in Chapter 5.

When the iron extraction curves in Figure 39 are considered the similarity between the curves at 750°C, 800°C and 850°C is also apparent. There is a very high initial rate of iron extraction up to 2 h of leaching followed by a sharp decline in the rate of leaching. The apparent optimum at oxygen concentrations between 4 % and 8 % O<sub>2</sub> is not as evident from the iron extraction curves, but there does appear to be a larger variation in the final amount of iron extracted between samples oxidised in the various gas mixtures at 800 °C versus the other roasting temperatures.

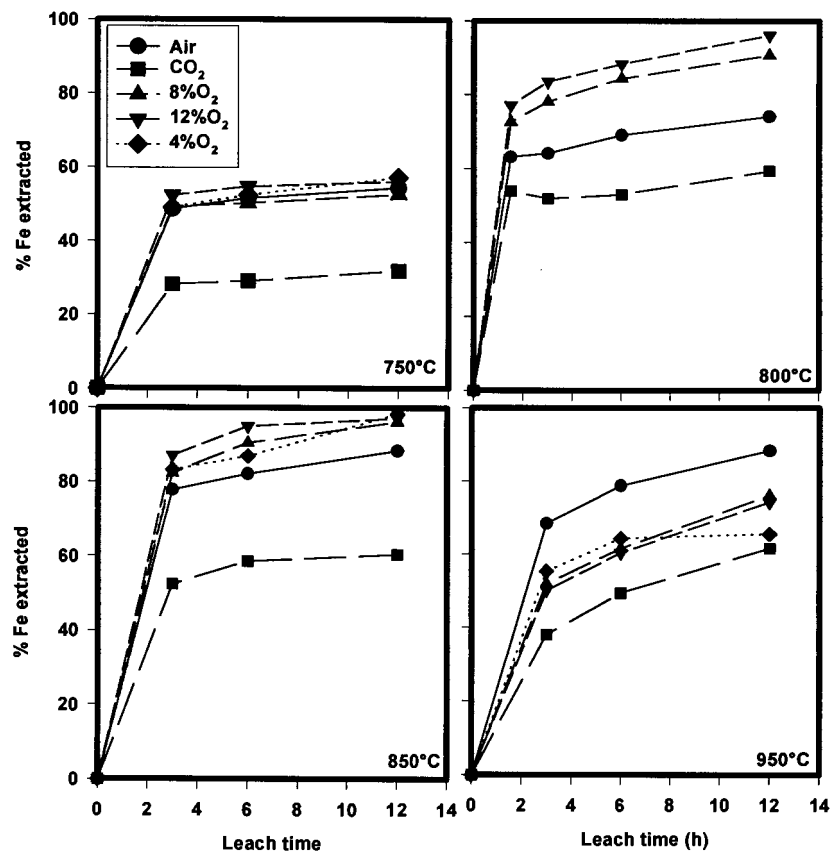


**Figure 37.** The influence of oxygen concentration and temperature during oxidation of standard slag on BTS product grade. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl. The slag contained the equivalent of 85% TiO<sub>2</sub> before treatment.





**Figure 38.** The influence of oxygen concentration during oxidation of standard slag at 850 °C on the total iron extraction during leaching. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl.



**Figure 39.** The influence of roasting temperature and oxygen concentration during oxidation of standard slag on the rate of iron extraction during leaching. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl.

At 950°C the oxidation behaviour changes dramatically as the final TiO<sub>2</sub> content and iron extraction decrease sharply relative to slag oxidised at 850 °C. The leach curves of slag roasted at 950 °C also differ from slag roasted at lower temperatures. The initial rate of iron extraction is still high, but it is lower compared to slag roasted at 850 °C and this is followed by a gradual decline in the rate of iron extraction after 2 h of leaching.

#### 4.3.1.2 Reduction

Oxidised titania slag contains iron in the Fe(III) form. Iron in this form is notoriously difficult to leach and as a result the slag is subjected to a partial reduction roast to reduce the Fe(III) back to Fe(II) without reducing the Ti(IV) to Ti(III). The influence of reduction was studied by varying the reduction time between 5 and 80 min. Table 41 gives the phase-chemical composition of the slag after oxidation, reduction and leaching.

**Table 41.** The phase-chemical composition, as determined by XRD, of standard slag after oxidation, reduction and leaching. The samples are categorised by the retention time during reduction. The slag was oxidised at 850 °C for 2 h in 8% O<sub>2</sub>, and reduced in 100 % CO.

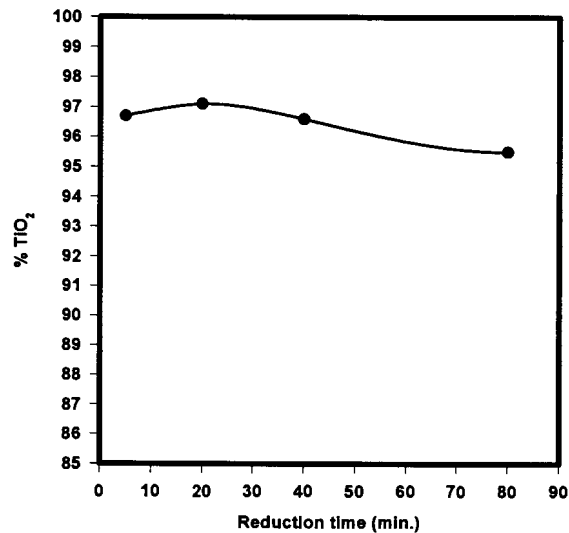
Phase composition after oxidation		Major phase	Minor phase	Trace phase
Sample	PFE			
Feed	986	Rutile	FeTi-Oxide	Anatase
Phase composition after reduction		Major phase	Minor phase	Trace phase
Sample	PFE			
5 min	1439	Rutile	FeTi-Oxide; Anatase	Ilmenite
10 min	1475	Rutile	FeTi-Oxide; Anatase	Ilmenite
20 min	1356	Rutile	FeTi-Oxide; Anatase	Ilmenite
40 min	1481	Rutile	FeTi-Oxide; Anatase	Ilmenite; Iron
80 min	1487	Rutile	FeTi-Oxide; Anatase	Iron; Ilmenite
Phase composition after leaching		Major phase	Minor phase	Trace phase
Sample	PFE			
5 min	1444	Anatase; Rutile		FeTi-Oxide
10 min	1480	Anatase; Rutile		FeTi-Oxide
20 min	1361	Rutile	Anatase	FeTi-Oxide
40 min	1486	Rutile; Anatase		FeTi-Oxide
80 min	1492	Rutile	Anatase	

Legend: FeTi-Oxide – M<sub>3</sub>O<sub>5</sub> solid solution; Rutile – TiO<sub>2</sub>; Anatase – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>; Iron – Fe<sup>0</sup>

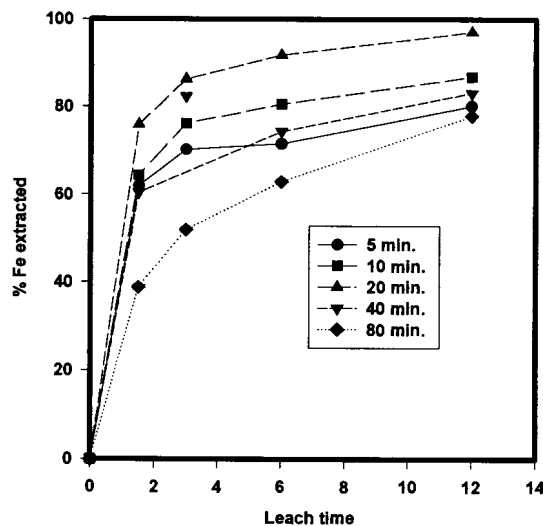
Table 41 shows that ilmenite formed during reduction. Table 41 further shows that metallic iron formed when the reduction period is extended to 40 min. A reduction period of 80 min was not sufficient to reduce all of the iron to the metallic form. All the ilmenite is removed during leaching. The lowering of M<sub>3</sub>O<sub>5</sub> to a trace component indicates that this phase is also partially removed during leaching. Anatase appears to be the dominant phase in the leach residues of samples reduced for 10 min or less, while rutile becomes the dominant phase in the leach residues of samples reduced for longer than 10 min. This might be related to the previously noted observation that TiO<sub>2</sub> initially forms as anatase during oxidation, but that it transforms to rutile as the duration of roasting is extended.

Figures 40 and 41 show the effect of reduction time on the final TiO<sub>2</sub> content of the slag and the % Fe extracted during leaching. The log sheets of the experiments are

given in Appendices IX to XIII. There appears to be no real difference in the final  $\text{TiO}_2$  content of samples oxidised for 5 to 40 min, but the final  $\text{TiO}_2$  content does decline if the reduction period is extended to 80 min. According to the leach curves the highest iron extraction was achieved after a reduction period of 20 min. It is also interesting to note that leach behaviour changes when the slag is reduced for 80 min. When the slag is reduced for shorter periods the initial leach rate is very high, but it declines sharply after 2 h. In contrast the initial leach rate of slag reduced for 80 min is lower, but the decline in rate after 2 h is much more gradual.



**Figure 40.** The influence of reduction time, during roasting of standard slag, on BTS product grade. The slag was oxidised for 2 h at 850 °C in 8 %  $\text{O}_2$ , reduced in 100 % CO and leached for 12 h in boiling 20 % HCl.



**Figure 41.** The influence of reduction time during roasting of standard slag on the rate of iron extraction during leaching. The slag was oxidised for 2 h at 850 °C in 8 %  $\text{O}_2$ , reduced in 100 % CO and leached for 12 h in boiling 20 % HCl.



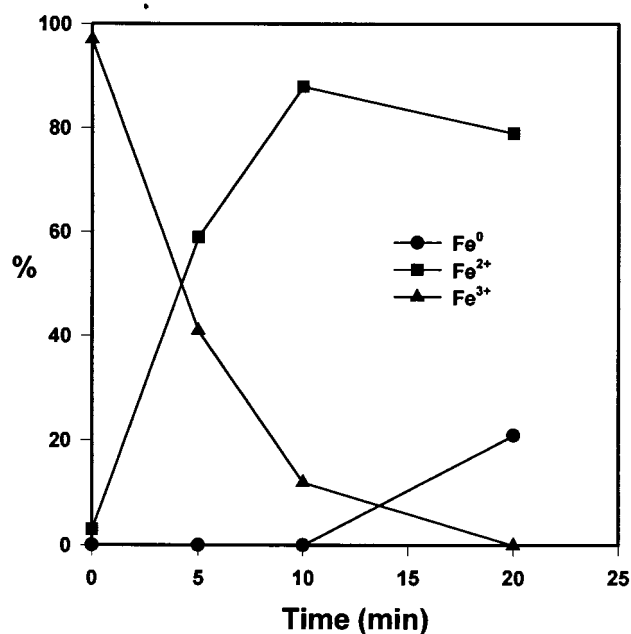
To quantify the phase changes that occur during reduction a Mössbauer investigation was conducted (See Appendix XVIII for the detailed results). The phase chemical compositions of the samples submitted for analysis are presented in Table 42. The samples were all oxidised for 2 h at 850°C in 10 % O<sub>2</sub> before they were reduced in 100 % CO for various times. The results presented in Table 42 are similar to the reduction results presented in Table 41.

**Table 42.** Phase chemical composition, as determined by XRD, of the oxidised and reduced samples submitted for Mössbauer analysis.

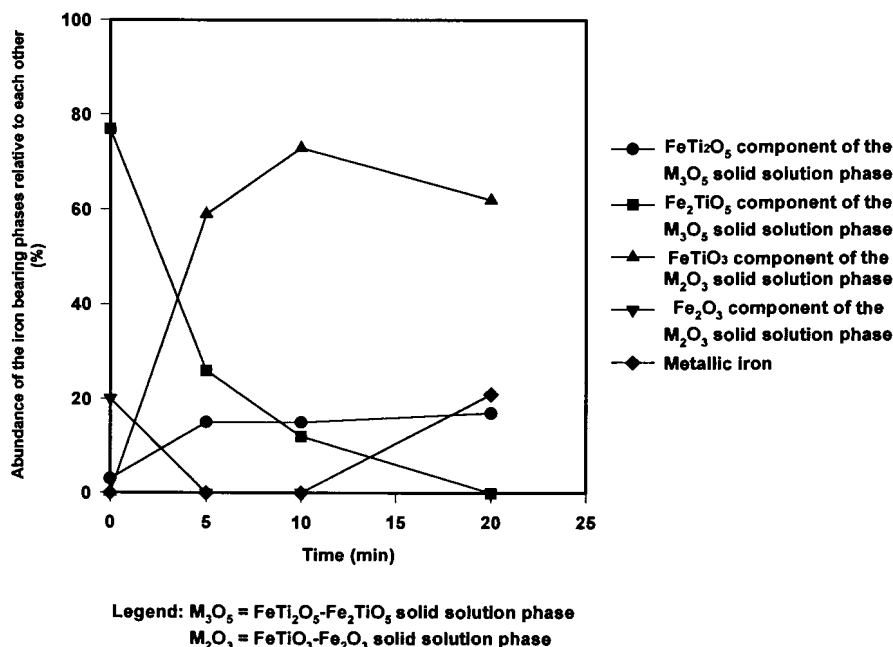
Reduced for:	PFE	Mineralogical composition		
		Main	Minor	Trace
5 min	3068	Rutile, Anatase	FeTi-Oxide	Ilmenite
10 min	3069	Rutile	Anatase, FeTi-Oxide	Ilmenite
20 min	3070	Rutile	Anatase, FeTi-Oxide	Ilmenite, Iron

**Legend:** FeTi-Oxide – M<sub>3</sub>O<sub>5</sub> solid solution; Rutile – TiO<sub>2</sub>; Anatase – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>; Iron – Fe<sup>0</sup>

The changes in the oxidation state of iron during reduction are presented in Figure 42. This shows that all of the Fe(III) has been reduced to either Fe(II) or Fe<sup>0</sup> after 20 min. Metallic iron started to appear after 10 min of reduction. Figure 43 shows the changes in the abundance of the iron containing phases relative to each other. The Fe<sub>2</sub>TiO<sub>5</sub> component of the M<sub>3</sub>O<sub>5</sub> solid solution and the hematite component of the M<sub>2</sub>O<sub>3</sub> solid solution decline rapidly and are converted to FeTi<sub>2</sub>O<sub>5</sub> and ilmenite. With extended roasting times metallic iron starts to appear.



**Figure 42.** The changes in the oxidation state of iron during reduction as determined by Mössbauer analysis.



**Figure 43.** The changes in the relative concentration of the iron containing phases in oxidised standard titania slag during reduction for various times as determined by Mössbauer analysis.

#### 4.3.1.3 Particle size

The influence of particle size was studied by subjecting three different size fractions, +150-300  $\mu\text{m}$ , +300-500  $\mu\text{m}$  and +500-700  $\mu\text{m}$ , to the same oxidation, reduction and leach procedure. Table 43 gives the phase-chemical composition of the various size fractions after the various processing stages.

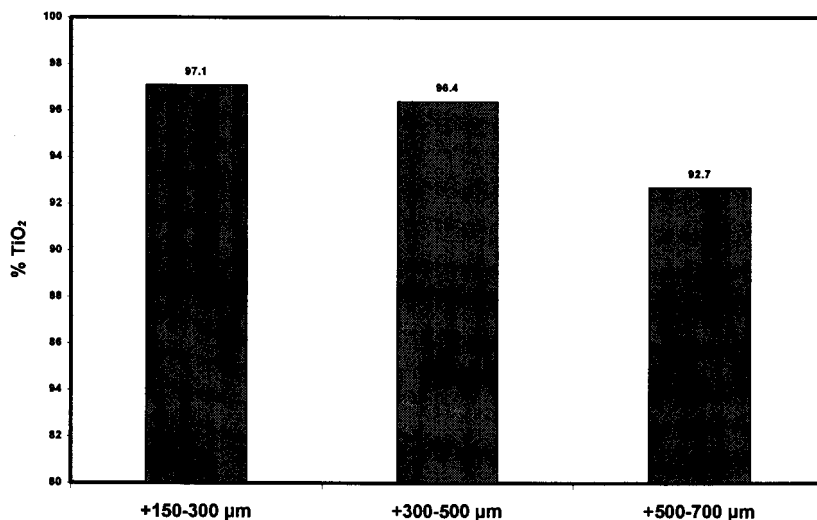
**Table 43.** The phase-chemical composition, as determined by XRD, of standard slag after oxidation, reduction and leaching. The samples are categorised by the size distribution used. The slag was oxidised at 850 °C for 2 h in 8 % O<sub>2</sub>, reduced in 100 % CO for 20 min and leached in boiling 20 % HCl for 12 h.

Phase composition after oxidation		Major phases	Minor phases	Trace phases
Sample	PFE			
+150-300 $\mu\text{m}$	1374	Rutile	FeTi-Oxide; Anatase	-
+300-500 $\mu\text{m}$	1375	Rutile	FeTi-Oxide; Anatase	-
+500-700 $\mu\text{m}$	1376	Rutile	-	FeTi-Oxide; Anatase
Phase composition after reduction		Major phases	Minor phases	Trace phases
Sample	PFE			
+150-300 $\mu\text{m}$	1350	Rutile	FeTi-Oxide	Ilmenite; Anatase
+300-500 $\mu\text{m}$	1356	Rutile	FeTi-Oxide	Ilmenite; Anatase
+500-700 $\mu\text{m}$	1385	Rutile	-	FeTi-Oxide; Ilmenite; Anatase
Phase composition after leaching		Major phases	Minor phases	Trace phases
Sample	PFE			
+150-300 $\mu\text{m}$	1355	Rutile	-	Anatase; FeTi-Oxide
+300-500 $\mu\text{m}$	1361	Rutile	Anatase	FeTi-Oxide
+500-700 $\mu\text{m}$	1390	Rutile	-	Anatase; FeTi-Oxide

Legend: FeTi-Oxide –  $M_3O_5$  solid solution; Rutile –  $TiO_2$ ; Anatase –  $TiO_2$ ; Ilmenite –  $FeTiO_3$ ; Iron –  $Fe^0$

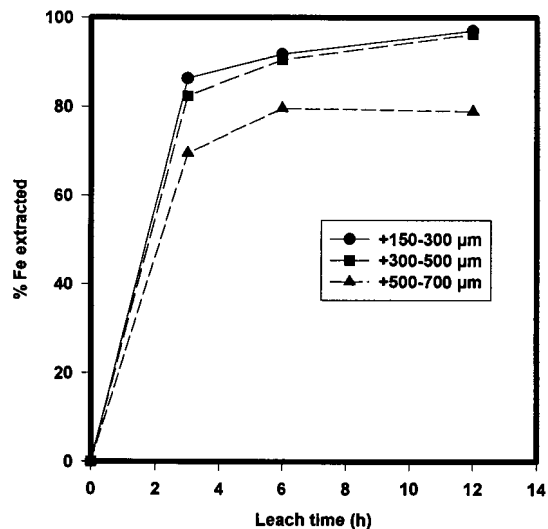
Table 43 shows that the slag samples contain mostly rutile after oxidation, but that the relative amount of rutile is much larger in the coarser size fraction. This larger amount of rutile in the coarser size fraction might be a result of the presence of primary rutile crystals from the smelting stage. Ilmenite formed during reduction. The ilmenite phase was removed during leaching and the relative amount of  $M_3O_5$  in the slag also declined as a result of leaching. Figures 44, 45 and 46 show the effect of particle size on the final  $TiO_2$  content and the iron extraction during leaching.

Figures 44 and 45 show that iron extraction and final  $TiO_2$  content increase with decreasing particle size, but there is a much smaller difference between the iron extraction and the final  $TiO_2$  content of the +150-300  $\mu m$  fraction and the +300-500  $\mu m$  fraction compared to the +500-700  $\mu m$  fraction. Figure 46 shows that an increase in the oxidation time does not have a big influence on the amount of iron extracted from the fine +150+300  $\mu m$  size fraction, the amount of iron extracted increases drastically for the coarser size fractions when the oxidation time is increased. This can probably be explained by the observation that the relative rate of oxidation increases as the particle size of the slag decreases (based on the observed shrinking core behaviour). As a result there are large unreacted cores present in the coarse particles that do not leach well.

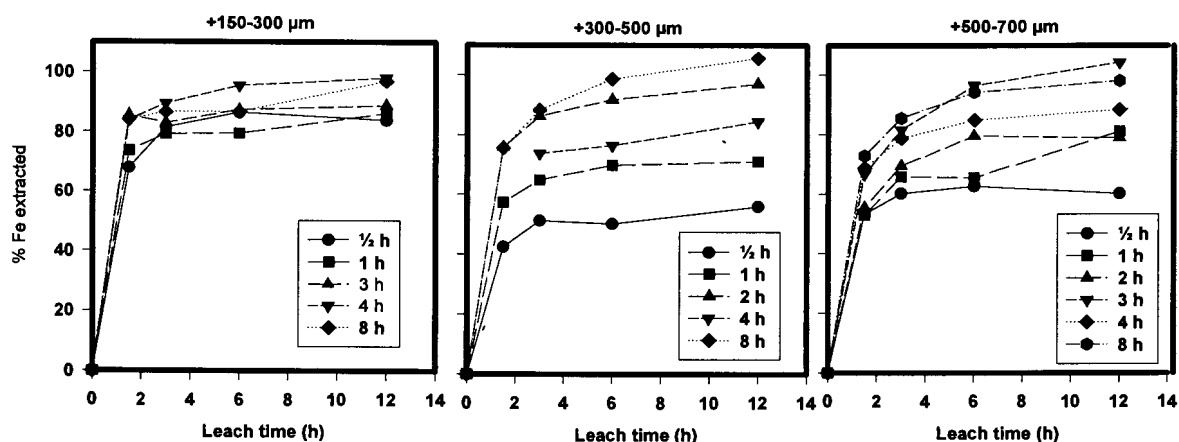


**Figure 44.** The influence of the particle size distribution of standard slag on BTS product grade. The slag was oxidised at 850 °C for 2 h in 8 %  $O_2$ , reduced for 20 min in 100 % CO and leached for 12 h in 20 % HCl.





**Figure 45.** The influence of the particle size distribution of standard slag on iron extraction during leaching. The slag was oxidised at 850 °C for 2 h in 8 % O<sub>2</sub>, reduced for 20 min in 100 % CO and leached for 12 h in boiling HCl.



**Figure 46.** The influence of oxidation time and the particle size distribution of standard slag on the rate of iron extraction during leaching. The slag was oxidised at 850 °C in 8 % O<sub>2</sub>, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl.

### 4.3.2 High iron titania slag

#### 4.3.2.1 Oxidation

##### *The effect of oxidation time*

The high iron titania slag that was studied contained about 22 % FeO compared to the 9.3 % FeO in standard titania slag. The influence of oxidation on high iron slag was studied in a similar fashion to the standard slag by varying the duration of the

oxidation roast from 0.5 to 8 h. Table 44 gives the phase-chemical composition of the samples after oxidation.

**Table 44.** The phase-chemical composition of high iron titania slag after oxidation. The samples are categorised by the retention time during oxidation. The slag was oxidised at 850 °C in 8 % O<sub>2</sub>

Phase composition		Major phase	Minor phase	Trace phase
Sample	PFE			
Feed	437	FeTi-Oxide	-	Ilmenite, Iron
Phase composition after oxidation		Major phase	Minor phase	Trace phase
Sample	PFE			
0.5 h	1167/1500	FeTi-Oxide	Rutile	Ilmenite; Anatase; Iron
1 h	1168/1501	FeTi-Oxide; Rutile	-	Anatase
2 h	1001/1502	FeTi-Oxide; Rutile	-	Anatase; Ilmenite
3 h	-	-	-	-
4 h	1169/1504	FeTi-Oxide; Rutile	Anatase	-
8 h	1505	FeTi-Oxide; Rutile	Anatase	-

Legend: FeTi-Oxide – M<sub>3</sub>O<sub>5</sub> solid solution; Rutile – TiO<sub>2</sub>; Anatase – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>; Iron – Fe<sup>0</sup>

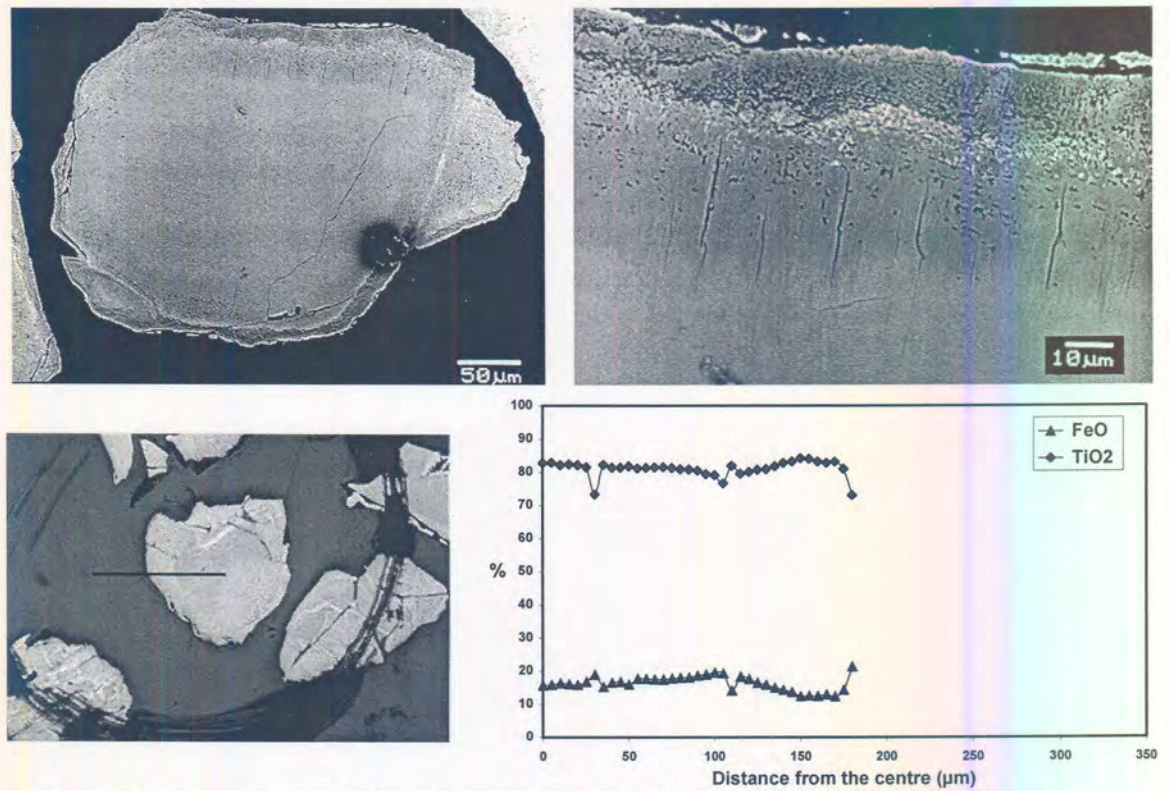
Table 44 shows that the M<sub>3</sub>O<sub>5</sub> phase transforms to rutile/anatase during oxidation. In contrast to the standard slag FeTi-Oxide remains as a major phase in the slag after oxidation. This is a result of the larger amount of iron in the slag. An optical and SEM investigation revealed that oxidation proceeded in a similar fashion to the standard slag. Samples which were oxidised for 0.5 h at 850 °C in 8 % O<sub>2</sub> (Figure 47), contained abundant partially reacted slag particles with a zoned appearance. There were - in contrast with the single iron-rim in the standard slag - two iron-rich rims in every particle, separated by a rutile-rich zone. The cores consisted primarily of the original M<sub>3</sub>O<sub>5</sub>-solid solution phase. The TiO<sub>2</sub>-rich marginal zones appeared to be slightly porous and seemed to consist of a mixture of anatase and rutile. Rutile was present along cracks extending through the unreacted M<sub>3</sub>O<sub>5</sub> particle cores. Finely disseminated metallic iron particles were also present in association with the rutile along cracks. The slag particles contained minor quantities of a silicate-rich glassy phase, usually situated at the grain boundaries of the individual titania crystals. The glass was characterised by the appearance of ilmenite crystallites as well as metallic iron. Disseminated, relatively coarse-grained metallic iron globules, associated with Fe-sulphide were present in the silicate-rich glass. Rounded “blebs” of a silicate-enriched glassy phase were also present embedded in the main glass phase.

The sample oxidised for 1 h at 850°C in 8% O<sub>2</sub> (Figure 48) appeared similar to samples that were oxidised for 0.5 h. The slag particles had a zoned texture with unreacted M<sub>3</sub>O<sub>5</sub>-rich cores and TiO<sub>2</sub>-enriched, slightly porous marginal zones. The marginal zones might be slightly broader compared to the slag subjected to 0.5 hours oxidation. The fine-grained slag particles appeared to be more completely transformed to TiO<sub>2</sub>.

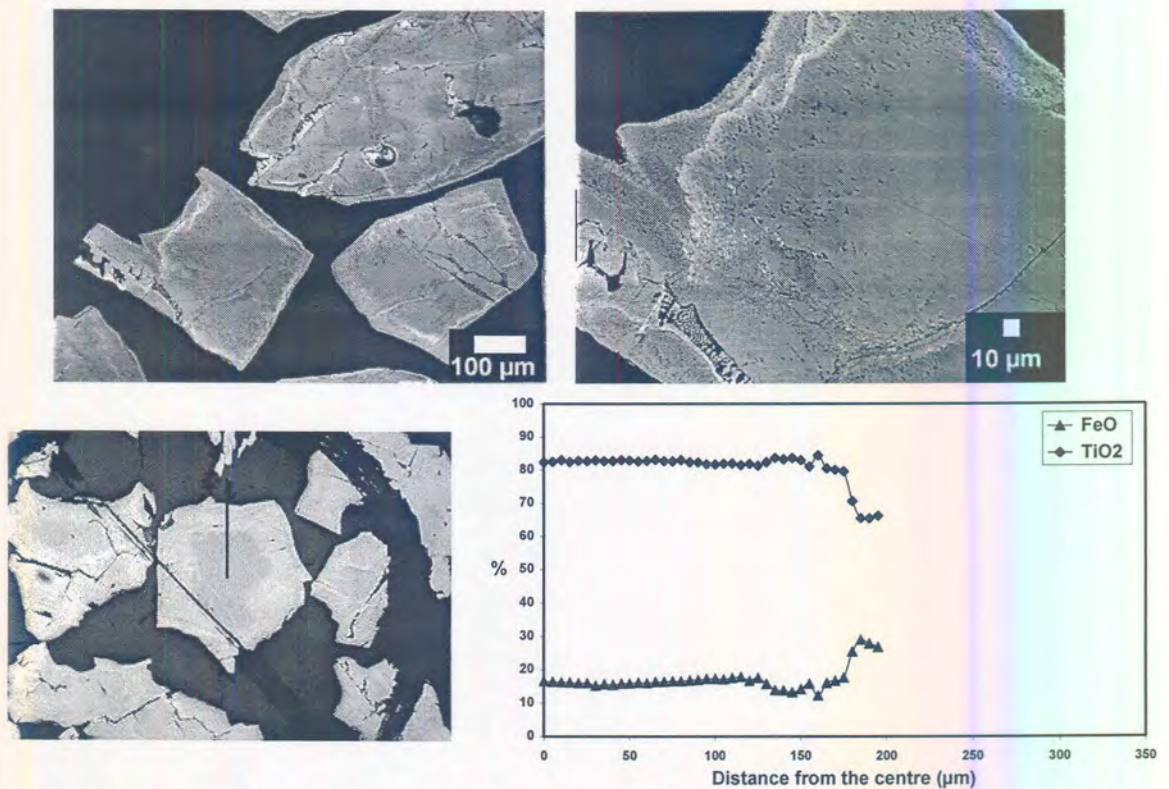
The samples oxidised for 2 h at 850°C in 8% O<sub>2</sub> (Figure 49) appeared to be almost completely oxidised. Some coarser-grained slag particles with small unreacted M<sub>3</sub>O<sub>5</sub>-cores and broad TiO<sub>2</sub>-rich outer margins were however still present.

The samples oxidised for 4 and 8 hours at 850°C in 8% O<sub>2</sub> (Figure 50) appeared to be completely oxidised (there were no unreacted cores). The marginal zones of the slag particles were characterised by the presence of rutile while the particle centres contained a mixture of sub-microscopic rutile and anatase. Some of the slag particles had dense anatase-rich centres.



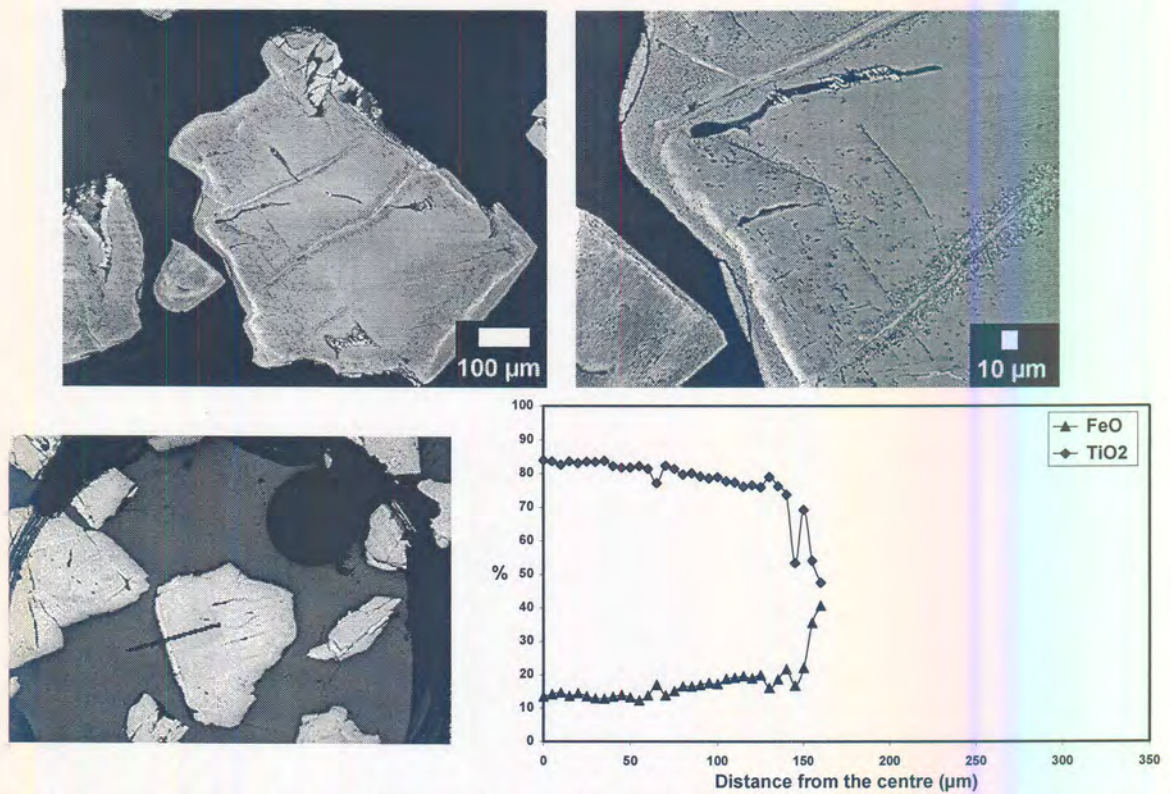


**Figure 47.** High iron titania slag oxidised for ½ h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.

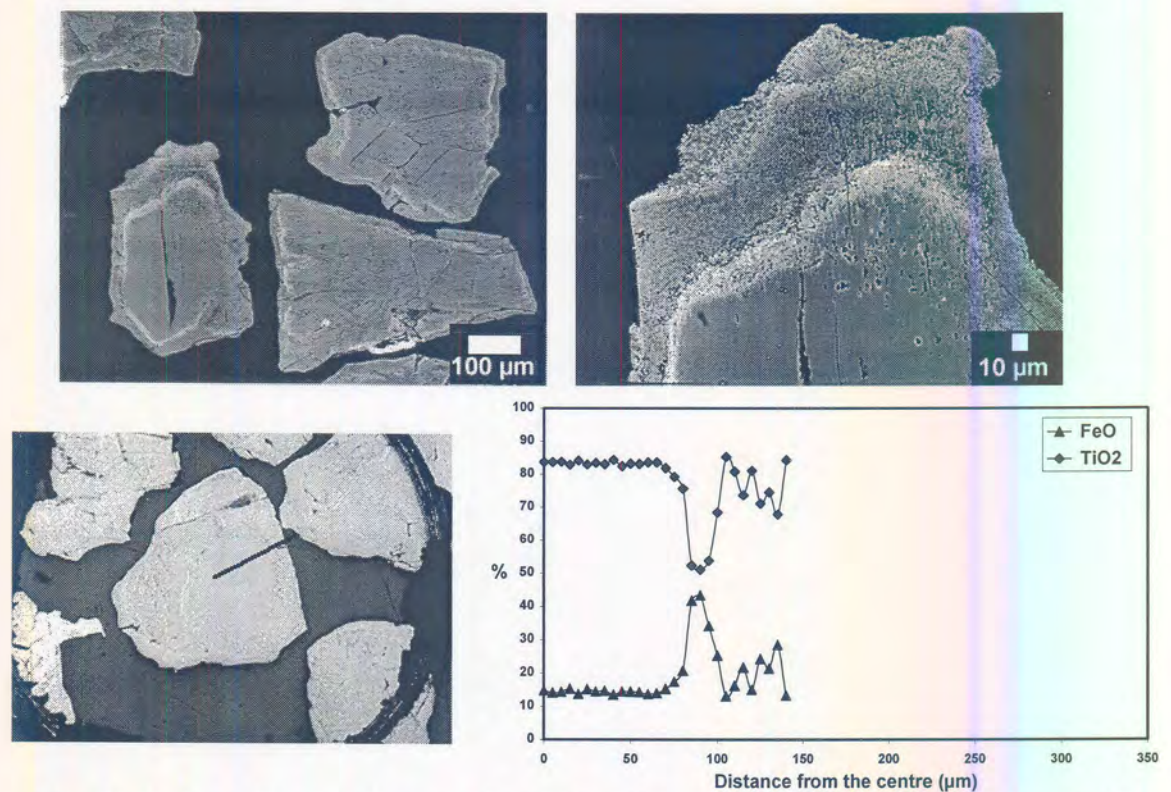


**Figure 48.** High iron titania slag oxidised for 1 h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.





**Figure 49.** High iron titania slag oxidised for 2 h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.



**Figure 50.** High iron titania slag oxidised for 4 h at 850 °C in 8 % O<sub>2</sub>. SEM micrographs as well as a chemical composition profile (weight %) through one of the particles are shown.





**Table 45.** The phase-chemical composition of high iron titania slag after oxidation and reduction. The samples are categorised by the retention time during oxidation. The slag was oxidised at 850 °C in 8 % O<sub>2</sub> and reduced in 100 % CO for 20 min.

Phase composition after reduction		Major phase	Minor phase	Trace phase
Sample	PFE			
0.5 h	1197/ 1564	FeTi-Oxide; Rutile	Ilmenite	Anatase
1 h	1202/1606	Rutile; FeTi-Oxide	Ilmenite	Iron; Anatase
2 h	1109 /1612	Rutile	FeTi-Oxide; Ilmenite	Iron; Anatase
3 h	1618	Rutile	FeTi-Oxide; Ilmenite; Iron	Anatase
4 h	1207/ 1624	Rutile	FeTi-Oxide; Ilmenite	Anatase; Iron
8 h	1630	Rutile	FeTi-Oxide; Ilmenite	Iron; Anatase

**Legend:** FeTi-Oxide – M<sub>3</sub>O<sub>5</sub> solid solution; Rutile – TiO<sub>2</sub>; Anatase – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>; Iron – Fe<sup>0</sup>

Table 45 shows that ilmenite formed during reduction. Ilmenite is present in all the samples as a minor phase compared to the standard oxidised and reduced slag, where it occurred only as a trace component. The large amount of ilmenite in high iron slag may be as a result of the high iron content of the slag. The presence of M<sub>3</sub>O<sub>5</sub> as a major phase in the samples oxidised for ½ h and 1 h indicates that these samples have not been completely oxidised. In the samples oxidised for 2 h or longer the M<sub>3</sub>O<sub>5</sub> phase is only present as a minor phase after oxidation. This suggests that oxidation proceeded much further in these samples compared to the samples roasted for shorter periods. The optical and SEM investigation did not reveal any major morphological changes as a result of the reduction roast (Figure 51).

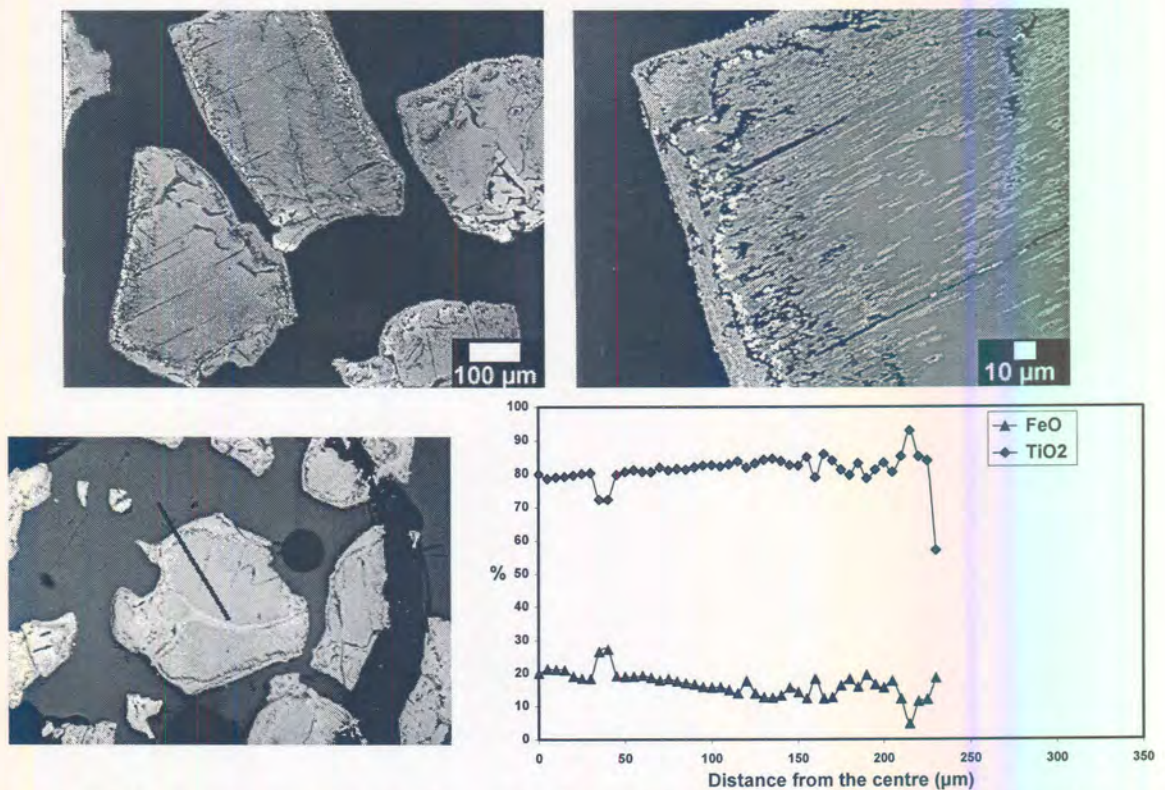
**Table 46.** The phase-chemical composition, as determined by XRD, of high iron slag after oxidation, reduction and leaching. The samples are categorised by the retention time during oxidation. For the experiments listed the slag was oxidised at 850°C in 8 % O<sub>2</sub>, reduced for 20 min in 100 % CO and leached in 20 % HCl for 12 h.

Phase composition after leaching		Major phase	Minor phase	Trace phase
Sample	PFE			
0.5 h	1201/ 1569	Rutile	FeTi-Oxide	Anatase
1 h	1206 / 1611	Rutile	FeTi-Oxide	Anatase
2 h	1113 / 1617	Rutile;	FeTi-Oxide	Anatase
3 h	1623	Rutile	-	Anatase; FeTi-Oxide
4 h	1211/ 1629	Rutile	-	Anatase; FeTi-Oxide
8 h	1635	Rutile	-	Anatase; FeTi-Oxide

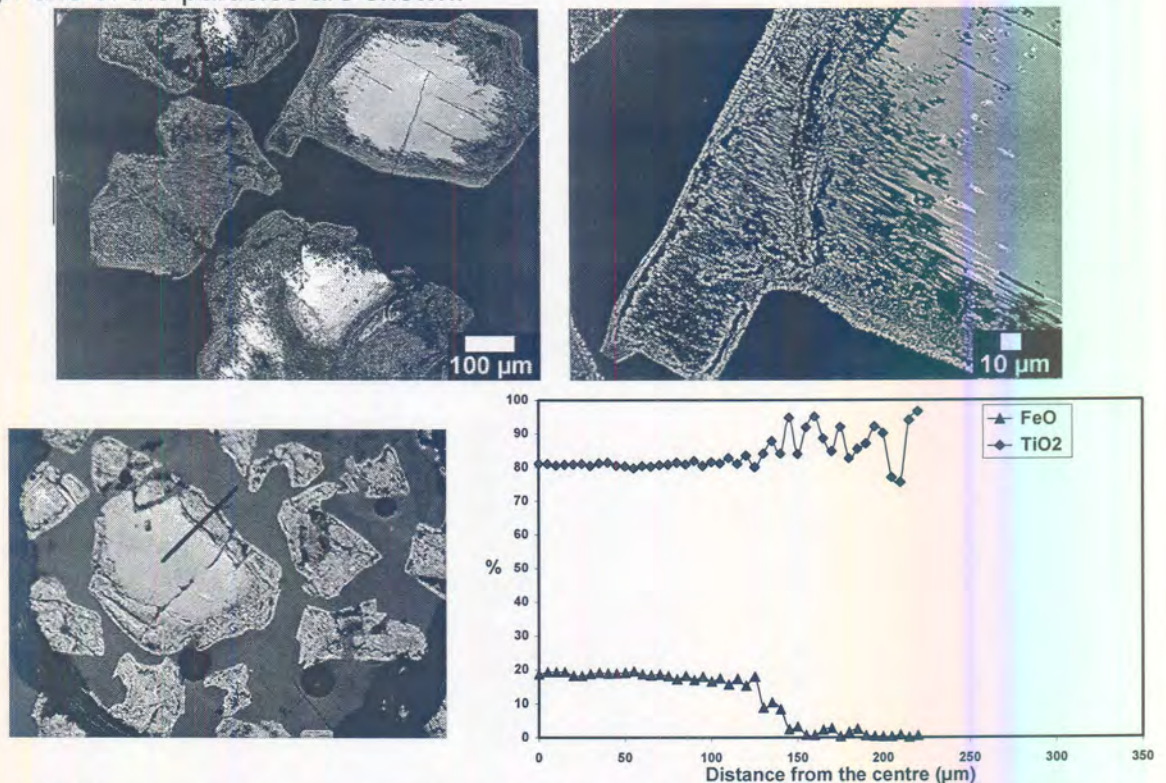
**Legend:** FeTi-Oxide – M<sub>3</sub>O<sub>5</sub> solid solution; Rutile – TiO<sub>2</sub>; Anatase – TiO<sub>2</sub>; Ilmenite – FeTiO<sub>3</sub>; Iron – Fe<sup>0</sup>

From Table 46 it appears that all of the ilmenite and a large amount of M<sub>3</sub>O<sub>5</sub> are removed during leaching. In the samples oxidised for less than 2 h the M<sub>3</sub>O<sub>5</sub> are present in minor amounts as a result of incomplete oxidation, while in samples oxidised for longer periods it is only present in trace quantities. The optical and SEM investigation revealed that structure of the slag changed from relatively dense to very porous during leaching. Only the outsides of the particles oxidised for periods up to 2 h were affected by leaching (Figure 52), while the particles oxidised for longer periods were leached more completely.





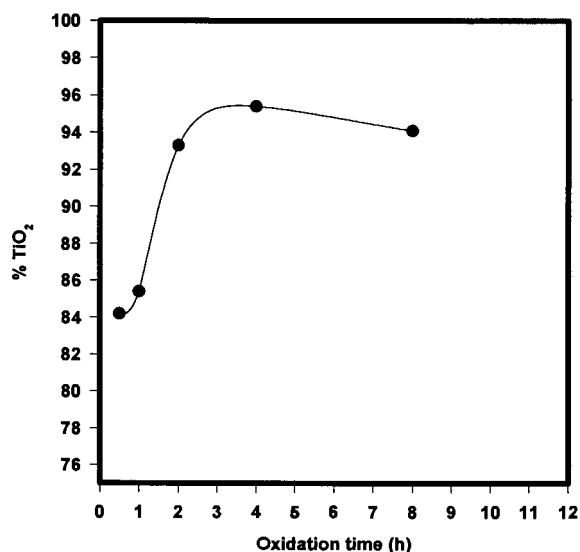
**Figure 51.** High iron titania slag oxidised for 2 h at 850 °C in 8 % O<sub>2</sub> and reduced for 20 min in 100 % CO. SEM micrographs as well as a chemical composition profile through one of the particles are shown.



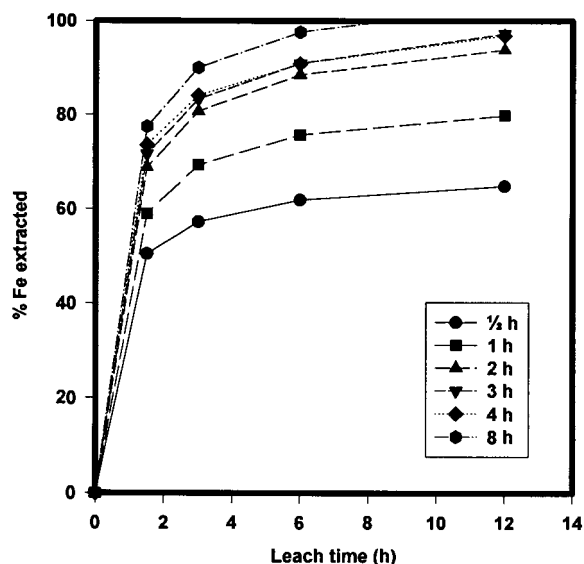
**Figure 52.** High iron titania slag oxidised for 4 h at 850 °C in 8 % O<sub>2</sub>, reduced for 20 min in 100 % CO and leached for 12 h in boiling 20 % HCl. SEM micrographs as well as a chemical composition profile through one of the particles are shown.



The influence of oxidation time on the final  $\text{TiO}_2$  content of the slag as well as the percentage of iron extracted during leaching are shown in Figures 53 and 54. The log sheets of the experiments are given in Appendices IX to XIII. The iron extraction for some of the experiments exceeded 100 % as a result of a poor accountability over these experiments. The poor accountability is due to the very small amount of slag available for leaching (5-10 g) after roasting samples were taken.



**Figure 53.** The influence of oxidation time, during roasting of high iron slag, on BTS product grade. The slag was oxidised at 850 °C in 8 %  $\text{O}_2$ , reduced in 100 % CO for 20 min and leached for 12 h in boiling 20 % HCl.



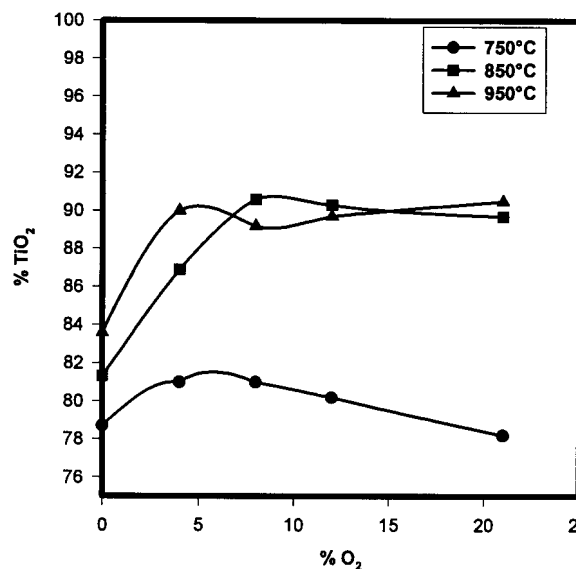
**Figure 54.** The influence of oxidation time, during roasting of high iron slag, on the rate of iron extraction during leaching. The slag was oxidised at 850 °C in 8 %  $\text{O}_2$ , reduced in 100 % CO for 20 min and leached for 12 h in boiling 20 % HCl.

The final  $\text{TiO}_2$  content of the slag increases as the oxidation time is increased up to 4 h. When the slag has been oxidised for 8 h there seems to be a slight decrease in the final  $\text{TiO}_2$  content of the slag. The iron extraction follows a similar pattern to the final  $\text{TiO}_2$  content and increases as the oxidation time increases, but in contrast to the final  $\text{TiO}_2$  content there is no decrease as the oxidation time is extended to 8 h. This might be related to poor accountability of this particular experiment. As a result of a large liquid to solid ratio the concentration of metals in solution was very low. This introduced a large error on the liquor analyses. These results do suggest that the same mechanisms operate during the oxidation of high iron slag as during the oxidation of standard slag.

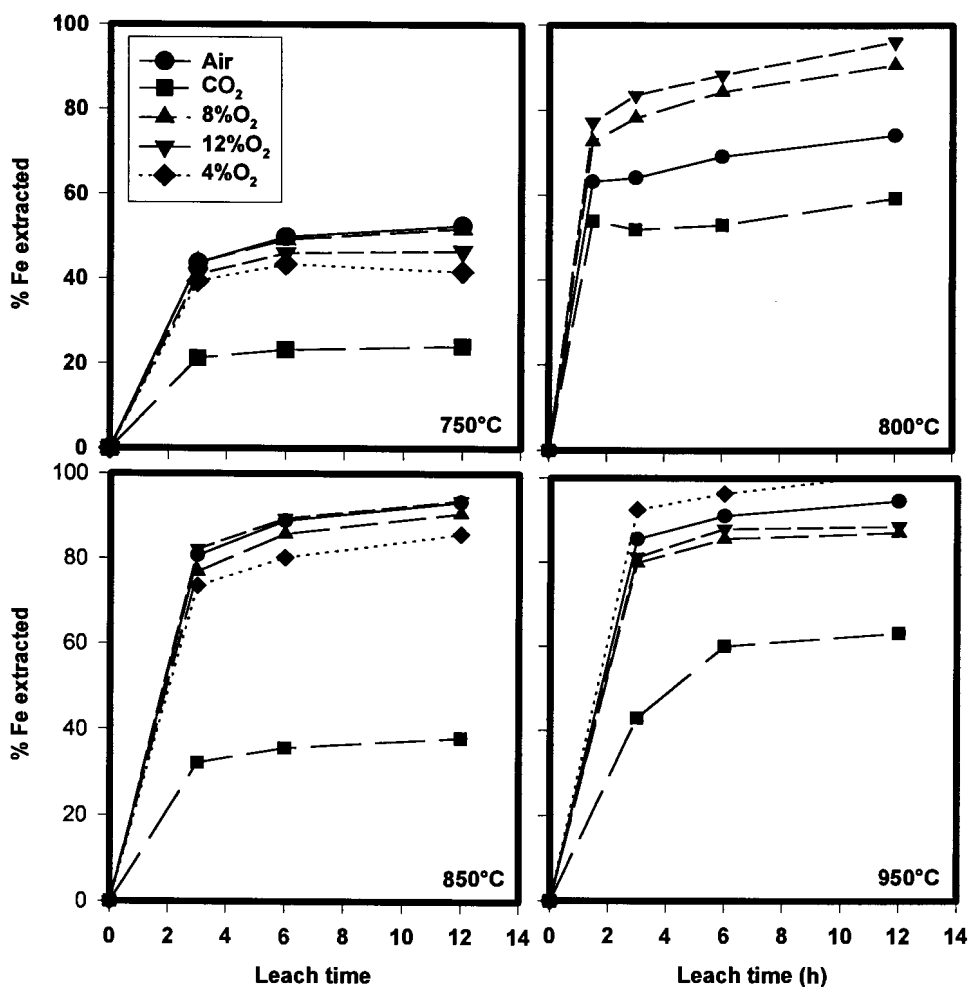
#### *The effect of oxidation atmosphere and temperature*

Oxidation is similarly influenced by the roasting temperature and the oxygen concentration of the oxidation atmosphere. The influence of the oxidation atmosphere was evaluated by varying the oxygen content of the fluidising gas, while the effect of roasting temperature was studied by varying it between 750 °C and 950 °C. The effects of oxidation atmosphere and roasting temperature on the final  $\text{TiO}_2$  content and the iron extraction are shown in Figures 55 and 56. The log sheets of the experiments are given in Appendices IX to XIII.

The rate of oxidation in  $\text{CO}_2$  is, as expected, very slow. This results in a final  $\text{TiO}_2$  content below 85 %  $\text{TiO}_2$  and a correspondingly low iron extraction. The final  $\text{TiO}_2$  content and iron extraction increase dramatically when oxidation is conducted in air/ $\text{CO}_2$  gas mixtures. There does not appear to be an optimum oxygen concentration between 4 % and 21 %  $\text{O}_2$  for the roasts conducted at 850 °C and 950 °C as there are no significant differences in the final  $\text{TiO}_2$  content and the iron extraction when oxidation is conducted in these atmospheres. This is in contrast to the oxidation of standard slag where there appeared to be an optimum oxygen concentration for oxidation between 4 % and 8 %  $\text{O}_2$ . The roasting temperature has a much more dramatic influence on oxidation compared to the effect of oxidation atmosphere. At 850°C and 950°C the final  $\text{TiO}_2$  content is in the region of 90 %, but at 750 °C the maximum  $\text{TiO}_2$  content is only 80 %. The iron extraction from the samples follows in general the pattern of the residual  $\text{TiO}_2$  content, but once again there are some accountability problems that introduce some discrepancies. For example the high iron extraction in air at 750 °C is in contrast to the residual  $\text{TiO}_2$  content of this experiment. Extractions in excess of 100 % were also calculated.



**Figure 55.** The influence of oxygen concentration and temperature (in air-CO<sub>2</sub> mixtures) during oxidation of high iron slag on BTS product grade. The slag was oxidised for 2 h, reduced for 20 min in 100% CO and leached for 12 h in boiling 20 % HCl. The slag contained and equivalent of 72% TiO<sub>2</sub> before treatment.

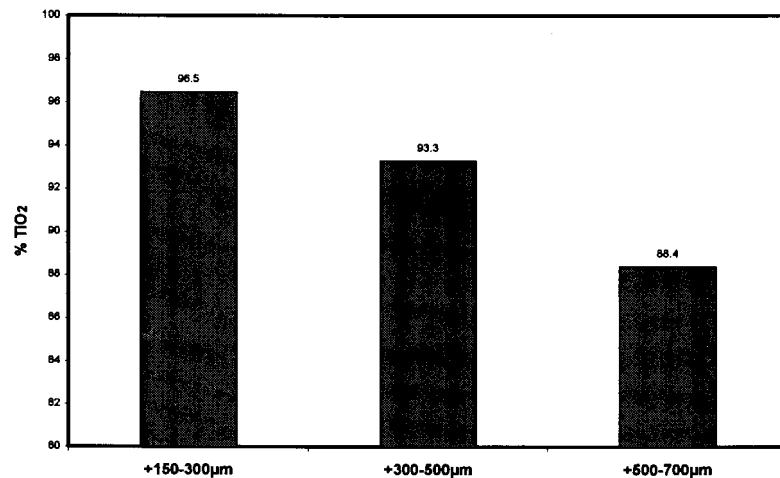


**Figure 56.** The influence of temperature and oxygen concentration during oxidation of high iron slag on the rate of iron extraction during leaching. The slag was oxidised for 2 h, reduced for 20 min in 100 % CO and leached for 12 h in 20 % HCl.

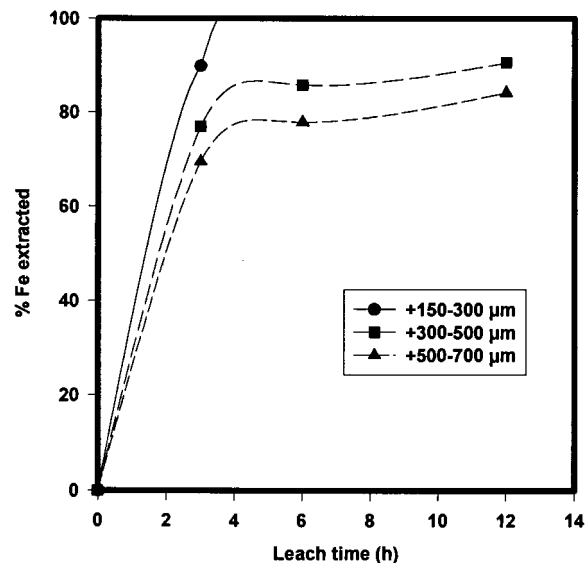


#### 4.3.2.2 Particle size

The influence of particle size was studied by subjecting three different size fractions, +150-300  $\mu\text{m}$ , +300-500  $\mu\text{m}$  and +500-700  $\mu\text{m}$  to the same oxidation, reduction and leach procedure. Figures 57 and 58 show the effect of particle size on the final  $\text{TiO}_2$  content of the slag and the iron extraction during leaching. The final  $\text{TiO}_2$  content of the slag increases as the particle size decreases. The difference between the final  $\text{TiO}_2$  content of the finest and the coarsest size fractions is 8 %  $\text{TiO}_2$ . These results suggest that the performance of high iron slag in the BTS process is highly dependent on the particle size distribution of the slag.



**Figure 57.** The effect of particle size distribution on final BTS grade. The slag was oxidised at 850°C for 2 h in 8%  $\text{O}_2$ , reduced for 20 min in 100%  $\text{CO}$  and leached for 12 h in 20%  $\text{HCl}$ .



**Figure 58.** The influence of the particle size distribution of high iron slag on the rate of iron extraction during leaching. The slag was oxidised at 850°C for 2 h in 8%  $\text{O}_2$ , reduced for 20 min in 100%  $\text{CO}$  and leached for 12 h in 20%  $\text{HCl}$ .

#### 4.4 Conclusions

- Oxidation converts the original solid solution phase in the as-cast slag ( $M_3O_5$ ) to  $TiO_2$  and an iron-rich solid solution phase. This phase change is accompanied by the migration of iron to the outside rims of the particles.
- Reduction converts the iron-rich solid solution phase to ilmenite. The ilmenite is located on the particle rims in the areas of iron enrichment.
- The ilmenite phase is removed during leaching along with part of the iron-rich solid solution phase. This results in a beneficiated titania slag product.
- The optimum roasting conditions specified in Chapter 3 were oxidation at 850 °C for 3 h and reduction at 800 °C for 30 min. The best BTS grade produced in process development phase 1 was 94 %  $TiO_2$ . Much better results were achieved during process development phase 2 and BTS with a  $TiO_2$  content in excess of 97 %  $TiO_2$  was produced. This may be related to the smaller sample sizes and the more controlled roasting conditions employed during process development phase 2. This allowed a good repeatability between experiments. The optimum process parameters were also refined through a large number of experiments.
- The oxidation rate (in terms of the relative amount of the  $M_3O_5$  phase converted to  $TiO_2$  and an iron-enriched rim) is lower for the higher-FeO slags.
- Two different roasting specifications can be drawn up from the results that have been presented. The conditions for producing a BTS product containing more than 95 %  $TiO_2$  can be specified or the conditions for producing BTS with the highest possible  $TiO_2$  content can be specified. In the following table these two specifications are listed for the materials that were investigated.

Standard titania slag	
Minimum specification	Maximum specification
<ul style="list-style-type: none"> <li>• Oxidation: 1½ h at 850 °C in 8 % <math>O_2</math></li> <li>• Reduction: 10 min at 850 °C in 100 % CO</li> </ul>	<ul style="list-style-type: none"> <li>• Oxidation: 3 h at 850 °C in 8 % <math>O_2</math></li> <li>• Reduction: 20 min at 850 °C in 100 % CO</li> </ul>
Expected grade: 96.2 % $TiO_2$ 1.52 % FeO	Expected grade: 97.5 % $TiO_2$ 0.72 % FeO
High iron titania slag	
Minimum specification	Maximum specification
<ul style="list-style-type: none"> <li>• Oxidation: 4 h at 850 °C in 8 % <math>O_2</math></li> <li>• Reduction: 20 min at 850 °C in 100 % CO</li> </ul>	Similar to the minimum specification
Expected grade: 95.4 % $TiO_2$ 1.90 % FeO	

## 5. THE OXIDATION MECHANISM OF TITANIA SLAG

### 5.1 Introduction

The previous chapters detail the development of the BTS process. The most important part of the BTS process is the oxidation roast because significant phase and chemical changes occur during this process stage that determine the success of the entire process. During oxidation the  $M_3O_5$  phase in the as-cast slag is transformed to rutile/anatase ( $TiO_2$ ), iron-rich  $M_3O_5$  and  $M_2O_3$ . In the process the Ti(III) and Fe(II) that are present in the as-cast slag are oxidised to Ti(IV) and Fe(III). Iron migration to the outside rims of the slag particles is another important morphological change that occurs during the oxidation of titania slag. This renders the iron easily accessible to the leach solution in subsequent processing. This chapter focuses on the mechanism of titania slag oxidation in an attempt to explain the observed chemical and morphological changes.

### 5.2 Background

#### 5.2.1 Segregation and diffusion of elements in oxide systems

The segregation of impurities in oxides to grain boundaries and surfaces is a common occurrence. Kingery (1984) found that Ca segregates in MgO as a result of the misfit strains generated by the Ca ion substituting for MgO. Cook (1988) found that Ca also segregates in  $Al_2O_3$  for the same reason. When aliovalent impurities (impurities having more than one oxidation state) are present in ionic solids the driving force for segregation can be the electrostatic potential present at grain boundaries and surfaces. Due to the formation of cation and anion vacancies the boundaries of an ionic solid will carry an electric charge caused by the presence of excess ions of one sign. A compensating charge of the opposite sign adjacent to the boundary would then be created. An example of segregation by this mechanism can be found in the Sc(III)-MgO system (Chiang, 1981). Pint (1998) investigated the diffusion of rare earth impurities (RE) in  $Al_2O_3$ . He found that the RE impurities segregated to the grain boundaries from where it diffused to the oxide gas interface. There it accumulated until the solubility limit was exceeded. Following that the R.E. impurities were precipitated as separate particles. Pint found that the segregation of the R.E. impurities to the grain boundaries occurred as a result of a misfit of ions, but that the migration to the surface of the oxide occurred as a result of an oxygen potential gradient through the  $Al_2O_3$ .

#### 5.2.2 Diffusion of Fe in $TiO_2$

The iron-titanium mixed oxide system has been the subject of several investigations as a result of its use as a photocatalyst (Bickley et al., 1991, Bickley et al., 1994 and Nobile and Davis, 1989). Amorelli et al. (1987) prepared polycrystalline  $TiO_2$  powders treated with low levels (~200 ppm) of Fe(III). They showed that Fe(III) placed on the surface of rutile will migrate into the lattice when subjected to temperatures in excess of 400 °C. They also found that Fe(III) placed on the surface of anatase migrated into the lattice at high temperatures, but the migration did not take place to the same degree as found for rutile. This suggested that the structure of anatase is permeable to entry by Fe(III) but it was more difficult in comparison with rutile. The reason for the migration of the iron was



the concentration gradient of iron that existed between the insides and the outsides of the particles. Bickley et al. (1994) also prepared TiO<sub>2</sub> powders with 0.5 to 5 atom % Fe(III) ions on the surfaces of the particles. They found that the Fe(III) migrated into the particles at 500 °C. At temperatures in excess of 500 °C they found that Fe(III) that had previously migrated into the particles segregated back to the particle surfaces to form ferric pseudobrookite and hematite in addition to the solid solution of Fe(III) in TiO<sub>2</sub>. They suggested that this surface enrichment occurred because the solubility limit of Fe(III) in TiO<sub>2</sub> had been exceeded. Bickley et al. (1991) reported that the solubility limit of Fe(III) in TiO<sub>2</sub> at 500 °C is in the region of 3 atom %. They found that Fe(III) is incorporated substitutionally in the TiO<sub>2</sub> matrix and that the solubility limit of Fe(III) is higher in rutile than it is in anatase.

### 5.3 Oxidation of titaniferous materials

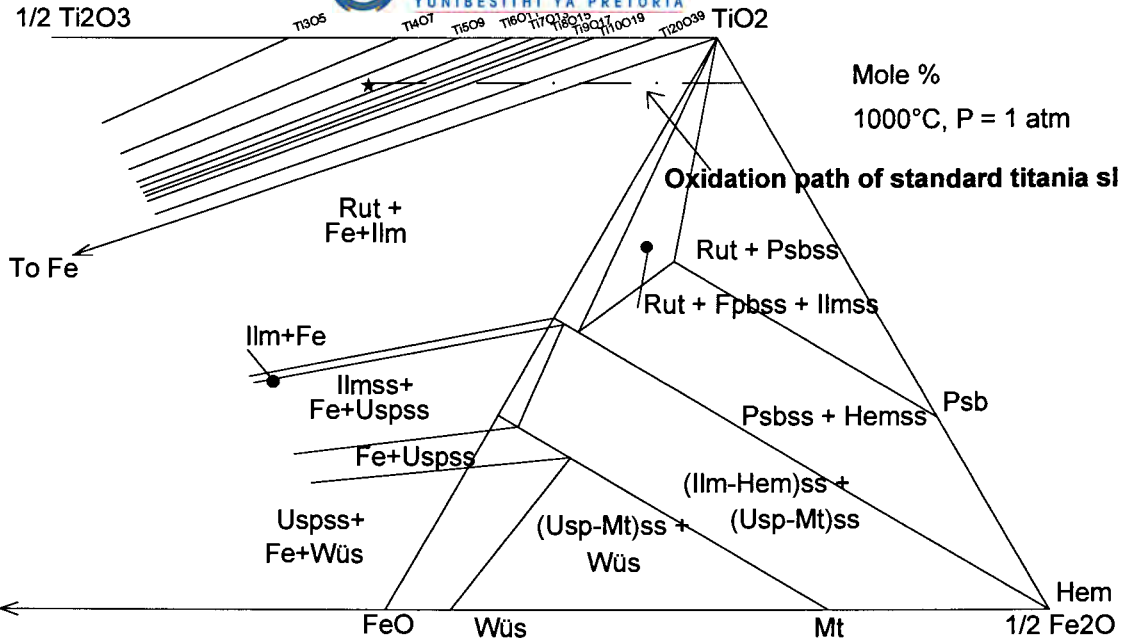
#### 5.3.1 Thermodynamics

As-cast titania slag contains mainly the FeTi<sub>2</sub>O<sub>5</sub>-Ti<sub>3</sub>O<sub>5</sub> solid solution (ferrous M<sub>3</sub>O<sub>5</sub>) phase because the slag is quenched during casting. This phase is thermodynamically stable at the casting temperature of the slag (~1700 °C), but the phase composition of the slag is meta-stable at the roasting temperatures that were investigated during the process development for the production of BTS. This fact is illustrated by Figure 59, a part of the phase diagram for the system Ti-O-Fe at 1000 °C. The chemical composition of as-cast titania slag is indicated with a star on the diagram. At this temperature the thermodynamically stable phases for titania slag may be reduced rutile and metallic iron at a certain oxygen potential and not the ferrous M<sub>3</sub>O<sub>5</sub> that is present in the slag. During oxidation the phase composition of the slag changes along the dotted line as oxygen is added to the material. This is a tie line between the chemical composition of the as-cast slag and oxygen. For oxidation in dilute air the phase composition of the slag will change along this line until it reaches the oxygen isobar in the rutile-pseudobrookite phase field that corresponds with the oxygen potential of the oxidising gas. The intersection between the tie line and the oxygen isobar representing an oxidising gas containing 5 % oxygen was calculated (Appendix XVI). This indicated that a partial oxygen pressure of 0.043 atm (corresponding to 5% oxygen at 0.86 atm) yields an equilibrium M<sub>3</sub>O<sub>5</sub> solid solution with the mole fraction of ferric pseudobrookite in the Fe<sub>2</sub>TiO<sub>5</sub>-FeTi<sub>2</sub>O<sub>5</sub> mixture equal to 0.997. The equilibrium products of slag oxidation in this gas hence are essentially pure ferric pseudobrookite and rutile.

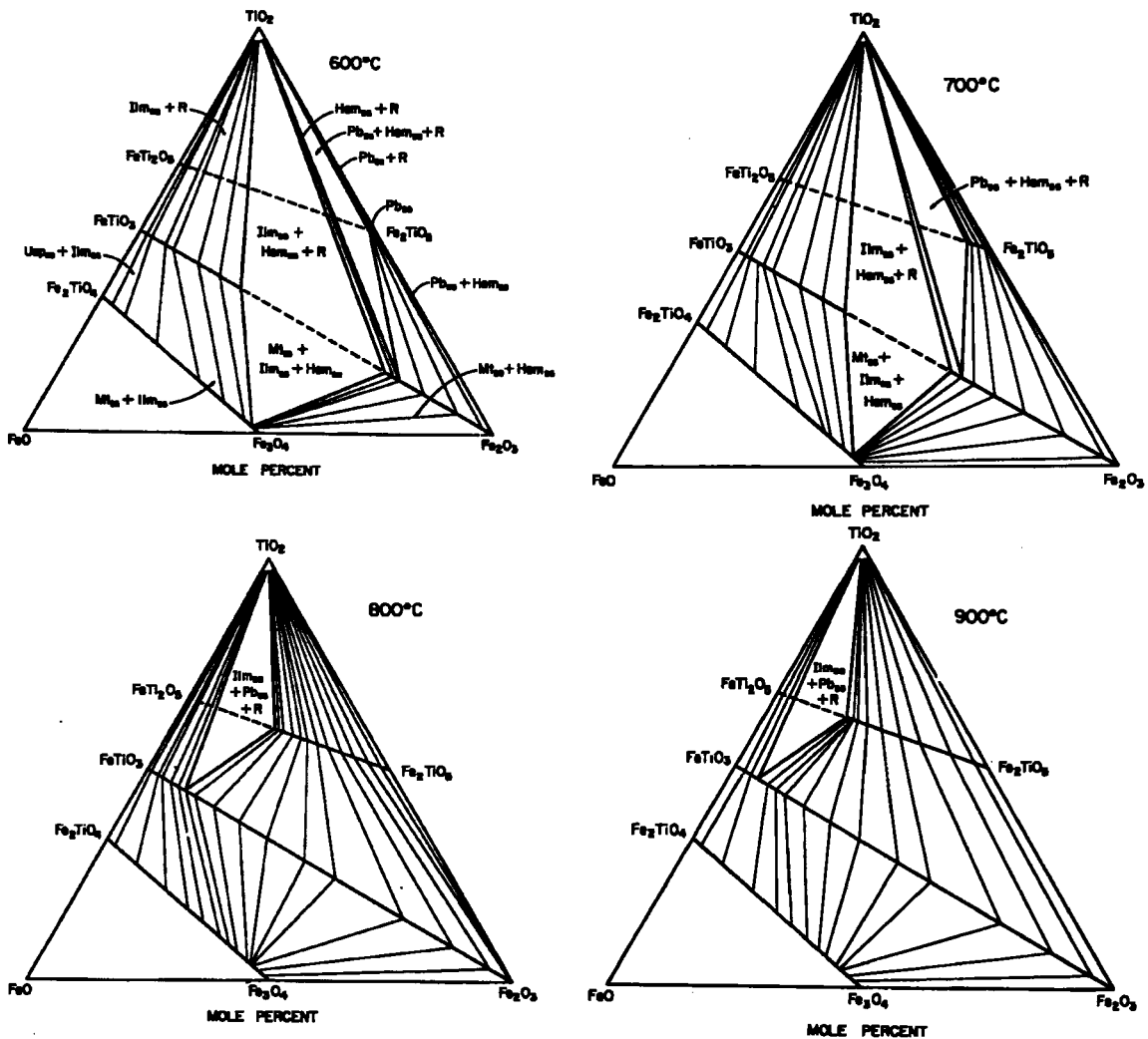
From Figure 59 it can be predicted that the phase composition of as-cast titania slag may change during oxidation from ferrous M<sub>3</sub>O<sub>5</sub> to:

1. a mixture of rutile, ilmenite and metallic iron;
2. a mixture of rutile and ilmenite-hematite solid solution;
3. a mixture of rutile, ilmenite-hematite solid solution and ferrous pseudobrookite solid solution and;
4. a mixture of rutile, and pseudobrookite solid solution.

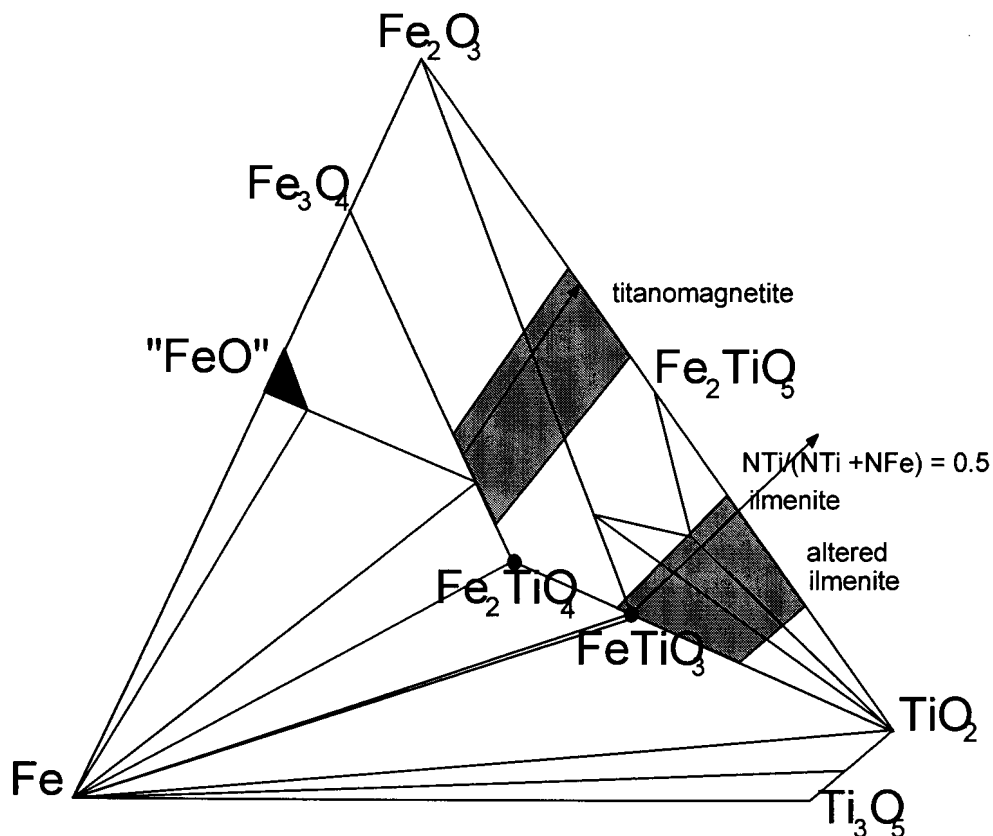
The effect of temperature on the phase diagram is shown by Figure 60. It can be seen that the size of the rutile, ilmenite-hematite solid solution stability area increases dramatically as the temperature decreases. Another important effect is the formation, at lower temperatures, of an area where pseudobrookite solid solution, hematite solid solution and rutile are the stable phases. These changes in the phase stability areas can influence the phase changes that occur in titania slag during oxidation and reduction.



**Figure 59.** Part of the Ti-O-Fe phase diagram at 1000 °C (compiled from phase diagrams produced by Lindsley, 1976 and Ericksson and Pelton, 1996). A star indicates the chemical composition of as-cast slag and the oxidation path of this material is indicated by a dotted line.



**Figure 60.** The effect of temperature on the TiO<sub>2</sub>-FeO-Fe<sub>2</sub>O<sub>3</sub> phase diagram (Haggerty, 1976).



**Figure 61.** The isotherm of the Fe-Fe<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> system at 800 °C (after Borowiec and Rosenqvist, 1981).

Literature data on ilmenite and titanomagnetite oxidation may shed some light on the possible phase transformations and hence are briefly reviewed here. The phase changes that occur during oxidation of ilmenite and titanomagnetite can be compared by examining Figure 61. This shows a part of the Fe-O-Ti phase diagram at 800 °C.

The theoretical composition of ilmenite, indicated by FeTiO<sub>3</sub>, is shown in Figure 61. During oxidation oxygen is added to the material while the ratio of titanium to iron remains constant. Hence, the equilibrium phase composition of theoretically pure ilmenite subjected to oxidation  $N_{Ti} / (N_{Ti} + N_{Fe}) = 0.5$  (with  $N$  the molar concentration). For other ilmenite ore compositions it moves along the band of lines with  $N_{Ti} / (N_{Ti} + N_{Fe})$  varying between 0.48 - 0.68 depending on the degree of alteration of the ore (Lynd et al, 1954). During oxidation at 800 °C the composition changes upward to the right, along the lines. The equilibrium phase composition of material with a composition close to that of theoretical ilmenite changes from ilmenite to:

1. a mixture containing rutile/anatase (TiO<sub>2</sub>) and ilmenite-hematite solid solution (M<sub>2</sub>O<sub>3</sub>);
2. a mixture containing rutile/anatase (TiO<sub>2</sub>), pseudobrookite (FeTi<sub>2</sub>O<sub>5</sub>-FeTi<sub>2</sub>O<sub>5</sub>) solid solution and ilmenite-hematite solid solution (M<sub>2</sub>O<sub>3</sub>) and;
3. a mixture containing pseudobrookite solid solution (FeTi<sub>2</sub>O<sub>5</sub>-Fe<sub>2</sub>TiO<sub>5</sub>) and rutile/anatase (TiO<sub>2</sub>).

Titanomagnetite is a solid solution between ulvöspinel and magnetite with the following formula:  $xFe_2TiO_4 \cdot (1-x)Fe_3O_4$ ,  $x$  varies between 0.547 for unreacted material and 0.838 for oxidised material (Akimoto, 1984). This lies in the band of lines indicated by  $N_{Ti} / (N_{Ti} + N_{Fe}) = 0.18-0.28$ . During oxidation at 800°C the phase composition changes from titanomagnetite along a constant titanium to iron line to:



1. a mixture containing titanomagnetite ( $M_3O_4$ ), and ilmenite-hematite solid solution ( $M_2O_3$ ) and;
2. a mixture containing ilmenite-hematite solid solution ( $M_2O_3$ ) and pseudobrookite solid solution ( $M_3O_5$ ).

### **5.3.2 Kinetics of titania slag oxidation**

Kinetic factors during oxidation result in phase and morphological changes that can not be predicted by phase equilibria. A detailed study on the oxidation of titania slag between 750 °C and 950 °C was conducted in Chapters 3 and 4. It was found that iron diffused to exposed surfaces of the slag particles during oxidation. This resulted in particles with  $TiO_2$  rich cores surrounded by iron rich rims. XRD-analysis indicated that the original ferrous  $M_3O_5$  is converted to rutile, anatase and  $Fe_2TiO_5$ - $FeTi_2O_5$  solid solution ( $M_3O_5$ ) phase. Mössbauer spectroscopy indicated that the intermediate transformation phase ilmenite-hematite solid solution that is predicted by the phase diagram also form during oxidation, but contrary to the phase diagram the  $M_2O_3$  phase in the form of hematite remains in the particles after extended reaction times as a meta-stable phase. The morphological investigation showed that the iron rich rims surrounding the particles consisted of two distinct phases that were probably ferric  $M_3O_5$  and hematite. A phase change that was not expected was the formation of metallic iron and rutile around cracks in the unreacted particle cores. This appeared to form as a result of the oxidation of the Ti(III), that was in excess of the solubility limit for pseudobrookite at the roasting temperature, by Fe(II) and is predicted by the phase diagram in Figure 56 for a situation where no oxidation occurs.

### **5.3.3 Kinetics of ilmenite oxidation**

Rao and Rigaud (1974) investigated the product morphology of oxidised ilmenite. Hematite was formed on the surface of the particles during oxidation below 770 °C. They proposed that Fe(II) migrated through the ilmenite to the hematite-ilmenite interface where it was oxidised. During oxidation between 770 °C and 900 °C hematite still formed on the surface, but a pseudorutile ( $Fe_2Ti_3O_9$ ) layer was formed directly under the hematite. They proposed that the Fe(II) was oxidised to Fe(III) in the pseudorutile layer before it was precipitated as hematite on the outer surface. During oxidation above 900 °C ferric pseudobrookite was found close to the surface under a layer of rutile. They suggested that the mobility of titanium increase above 900 °C and that this result in  $TiO_2$  on the surface. Briggs and Sacco (1993) investigated the oxidation of ilmenite with synthetic ilmenite discs at temperatures between 775 °C and 1000 °C. They did not find pseudorutile in any of their samples but they did find that rutile and ilmenite-hematite solid solution formed on the insides of the discs, while a pseudobrookite-rich layer formed closer to the surface of the discs. The outer surface of the discs always consisted of hematite. They suggested that the hematite layer formed by outward cation diffusion.

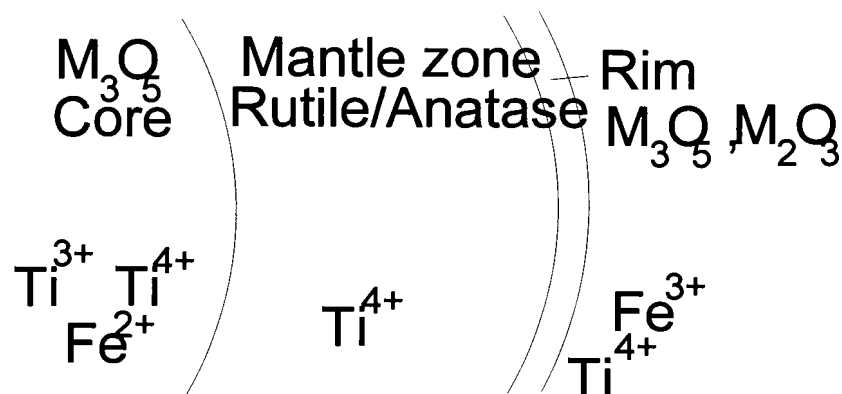
### **5.3.4 Kinetics of titanomagnetite oxidation**

The oxidation of titanomagnetite has proved to be an integral part in studies about magnetic anomalies on the sea floor (Rayll and Hall, 1980). O'Reilly and Banerjee (1966) and Readman and O'Reilly (1972) found that oxidation was not a simple process without any changes in the relative cation content. Instead the process is accompanied

by iron diffusion out of the original oxide lattice. They suggested the following oxidation mechanism: Cations diffusing through the crystal lattice combine with adsorbed oxygen atoms at the surface which are ionised by the extra electrons from the Fe(II) ions. The speed at which the reaction occurs depends on the rate of diffusion of the cations. The cation distribution of oxidised single phase spinel is such that the tetrahedral sites are shared by Fe(II) and Fe(III). The octahedral sites are occupied by Ti (IV), Fe(II) and vacancies. Chemical bonding in the tetrahedral sites is not ionic but covalent as a result of the fact that covalent bonding at these sites lowers the lattice energy. This means that oxidation occurs largely at the expense of octahedral Fe(II). During the adsorption and ionisation of each oxygen atom on average 3/4 new tetrahedral sites and 1/2 new octahedral sites are formed that must be partially filled by cations. Those most likely to fill the new sites are the Fe(II) which have diffused to surface and transformed to Fe(III) cations during ionisation of the oxygen atom. When all octahedral Fe(II) ions have been oxidised the reaction can only proceed by the oxidation of tetrahedral Fe(II) and this results in the decomposition of the cation deficient spinel.

#### 5.4 Proposed mechanism of titania slag oxidation

A summary of the phase and chemical changes that occur in titania slag during oxidation is shown in Figure 62. The three main zones represented are the unreacted core, the TiO<sub>2</sub>-rich mantle zone and the iron-rich rim. The unreacted core consists of ferrous M<sub>3</sub>O<sub>5</sub>. The TiO<sub>2</sub>-rich zone consists predominantly of anatase and rutile and the iron-rich zone consists predominantly of ferric pseudobrookite and hematite. In the process iron migrates to the outside rims of the slag particles, while Ti(III) and Fe(II) are oxidised to Ti(IV) and Fe(III).

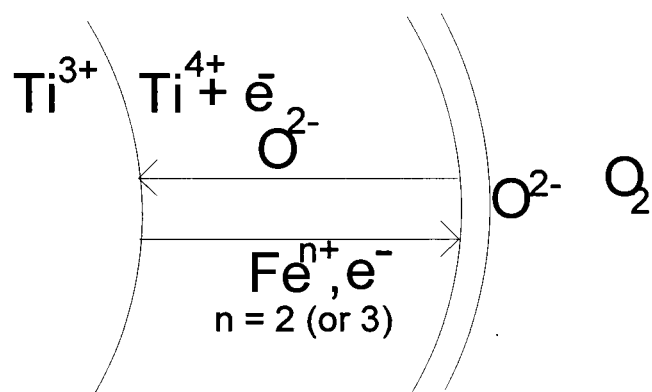


**Figure 62.** Summary of the phase and chemical changes that occurs in titania slag during oxidation.

Iron migration during oxidation of titania slag is a significant phenomenon. Three causes for the segregation or diffusion of elements in oxide systems have been identified in the background study:

1. The presence of bulk strains generated by the misfit of impurity ions in the crystal structure;
2. The existence of space charge effects as a result of the presence of aliovalent impurities in the oxide;
3. The presence of a chemical potential gradient between the insides and the outsides of the particles.

The first two causes listed above do not seem to be applicable in this system. For segregation to occur by the misfit of impurity ions the phase composition of the sample should stay the same. During oxidation of titania slag the original  $M_3O_5$  phase is completely destroyed. Segregation of iron therefore cannot occur in the original  $M_3O_5$  phase. Segregation by space charge effects is also not plausible as segregation by this mechanism only occurs over relatively short distances within the same phase. Migration of iron in titania slag takes place over several hundred microns through more than one phase. The most probable cause for the migration of iron to the outside surfaces of titania slag particles during oxidation is the presence of a chemical potential gradient between the insides and outsides of the particles.



**Figure 63.** Proposed mechanism for the oxidation of titania slag.

Based on the summary of the chemical and morphological changes presented in Figure 62, the movement of cations and ions in the slag during oxidation can be inferred (Figure 63). Oxygen is reduced on the surface of the particles and diffuses into the interior, where the Ti(III) is oxidised and precipitated as rutile or anatase. The iron migrates to the surface of the particles as either Fe(II) or Fe(III) depending on where it is oxidised.

One possible reason for the observed morphology might be as a result of a large difference in the relative mobility of iron cations and oxygen anions, i.e. the iron cations precipitate at the outer surface because of their inherently higher diffusivity. This however seems unlikely, as the oxygen anions are able to diffuse through the product layer to precipitate rutile or anatase inside the particles, indicating substantial oxygen anion mobility. Another reason might be related to the formation of the  $M_2O_3$  phase. The phase diagram predicts that this phase will form as an intermediate product during oxidation (See also Appendix XVII). The Mössbauer results shown in Chapter 4 confirmed that  $M_2O_3$  formed. It was however still present in significant quantities in the slag at the end of the oxidation roast. The thermodynamic evaluation in paragraph 5.3.1 showed that this phase is more stable at lower roasting temperatures. The crystal structure of the  $M_2O_3$  phase differs radically from the rutile and pseudobrookite phases that are present in the slag. Nucleation of the  $M_2O_3$  phase would therefore require a large amount of nucleation energy. The nucleation energy that is required can be reduced if the  $M_2O_3$  phase forms on a free surface. This can only occur on the outsides of the slag particles. Migration of iron cations to the surface of the particles therefore presumably determines where  $M_2O_3$  precipitates.



## 5.5 Experimental plan

The experiments were planned around the following concepts:

- Roasting in the presence and in the absence of oxygen to determine whether oxygen is required for iron migration;
- Investigation into size and porosity changes during oxidation and reduction of titania slag;
- Coating slag particles with a marker material - to investigate the way oxygen is added to the particles;
- Evaluation of previously conducted line chemical analysis on roasted slag to determine the speciation of iron and titanium in the slag through phase fitting of the data;
- Leaching roasted slag under various conditions and measuring the speciation of iron in solution to determine the speciation of iron in the slag at different positions;
- Testing the influence of the iron rich rim on the mechanism of oxidation;
- Roasting at higher temperatures - to determine if there is a change in roasting mechanism and;
- Interrupted roasting - to test if this changes the mechanism.

## 5.6 Experimental procedure

### 5.6.1 Roasting

The roasting procedure was similar to that described in Chapter 4.

### 5.6.2 Leaching

The leach experimental procedure was similar to that described in Chapter 4 except for the reduction leach experiments where 31.5 g  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  were added to the leach solution per 50 g slag.

## 5.7 Results and Discussion

### 5.7.1 Investigation into the roasting conditions required for iron migration

Three experiments were designed to test whether a high oxygen potential is required for iron migration to occur in titania slag. For the first experiment slag was roasted in 100 % oxygen for 2 h at 850 °C. The second roast experiment was conducted in air and the third roast experiment was conducted in argon (containing < 2 ppm  $\text{O}_2$ ). Micrographs of the slags after roasting are shown in Figure 64 and Table 47 gives the phase compositions.

**Table 47.** Phase composition of the slag samples roasted in 100 %  $\text{O}_2$ , air and argon at 850 °C for 2 h

Description	Mineralogical composition		
	Main	Minor	Trace
Slag roasted in 100% $\text{O}_2$	Anatase; Rutile	FeTi-Oxide	-
Slag roasted in air	Rutile, Anatase	FeTi-Oxide	-
Slag roasted in argon	FeTi-Oxide	Rutile	Iron

**Legend:** FeTi-Oxide –  $\text{M}_3\text{O}_5$  solid solution; Rutile –  $\text{TiO}_2$ ; Anatase –  $\text{TiO}_2$ ; Ilmenite –  $\text{FeTiO}_3$ ; Iron – Fe



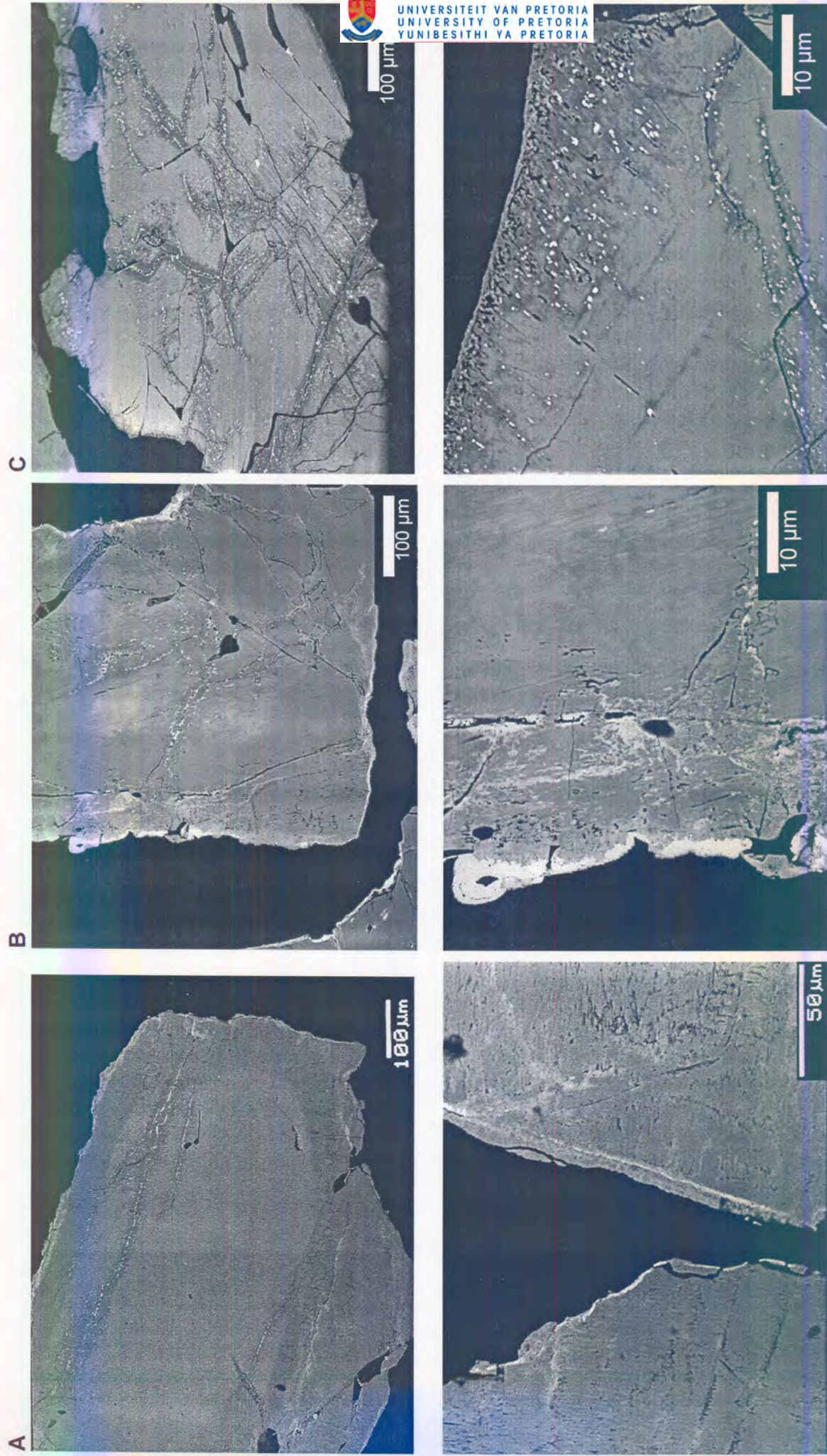


Figure 64. Slag roasted for 2 h at 850 °C. Oxygen was used to roast sample A, air was used for sample B and argon was used for sample C.



According to the results obtained by XRD-analysis, the slag roasted at 850 °C for 2 h in a 100 % O<sub>2</sub> atmosphere, consisted predominantly of anatase and rutile with lesser amounts of the M<sub>3</sub>O<sub>5</sub> solid solution phase. The individual slag particles had a similar optical appearance to the titania slag particles roasted in air at 850 °C. The majority of slag particles displayed a well-defined zoned texture with unreacted M<sub>3</sub>O<sub>5</sub>-rich cores, TiO<sub>2</sub>-rich mantle zones and thin iron enriched outer rims. Some of the slag particles were however completely transformed to TiO<sub>2</sub> with finely porous structure and iron enriched outer rims.

The slag particles roasted in air also had a similar well-defined zoned texture with iron enrichment towards the outer rims of the particles, a broad TiO<sub>2</sub>-rich mantle zone and relatively small unreacted M<sub>3</sub>O<sub>5</sub>-rich cores.

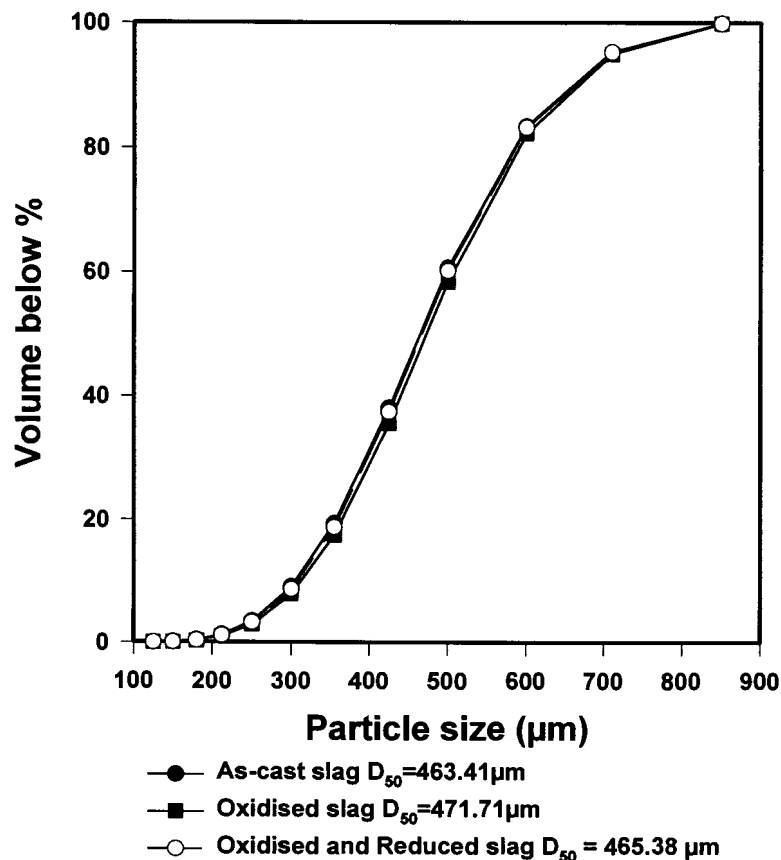
The optical appearance of slag roasted in argon was completely different from the slag roasted in oxygen and air. The slag particles contained large unreacted cores consisting of the M<sub>3</sub>O<sub>5</sub> solid solution phase displaying a relatively smooth and dense appearance. In addition, the slag particles contained relatively thin reaction zones along their outer margins as well as along cracks cutting through the unreacted M<sub>3</sub>O<sub>5</sub>-rich cores. These reaction zones consisted primarily of rutile as well as extremely fine-grained disseminated metallic iron precipitates. No iron migration occurred towards the outer rims of the slag particles.

These results can be explained by referring to the Ti-Fe-O phase diagram (Figure 59). The different phase compositional areas on the diagram are in equilibrium with gas mixtures of different oxygen potentials. In paragraph 5.3.1 it was shown that at equilibrium slag roasted in air will consist of the phases M<sub>3</sub>O<sub>5</sub> and rutile. On the other hand the oxygen potential in argon is essentially an inert gas and this means that the oxidation kinetics is very slow. Based on these results roasting in oxygen rich atmospheres appear to be necessary for iron migration to occur.

### **5.7.2 Porosity and particle size changes during roasting**

During oxidation oxygen is added to the slag and during reduction oxygen is removed. One would therefore expect the average particle size of the material to change during roasting. To investigate this the size distribution of the same slag sample was determined after crushing, oxidation and reduction. The results are presented in Figure 65. During oxidation the average particle size of the sample increased from a D<sub>50</sub> of 463 µm to 471 µm. After reduction the average particle size decreased again to a D<sub>50</sub> of 465 µm. An additional factor that influences the particle size is the introduction of pores in the material during roasting. Table 48 gives the porosity of the slag sample after crushing, oxidation and reduction. The as-cast slag is fairly dense with a porosity of only 0.0601 cm<sup>3</sup>/g. During oxidation the porosity increases to 0.1397 cm<sup>3</sup>/g and during reduction it increases further to 0.1433 cm<sup>3</sup>/g. The particle size and porosity data supports the proposed oxidation mechanism. As oxygen is added to the material and iron migrates to the outsides of the particles the average particle size of the slag increases. This increases the porosity in the slag.





**Figure 65.** Particle size changes during roasting of titania slag.

**Table 48.** Porosity of slag particles before and after the roasting stages.

Description	Porosity (cm <sup>3</sup> /g)
As-cast slag	0.0604
Oxidised slag	0.1397
Oxidised and reduced slag	0.1433

\* Determined with a Micromeritics 9300 mercury porosimeter

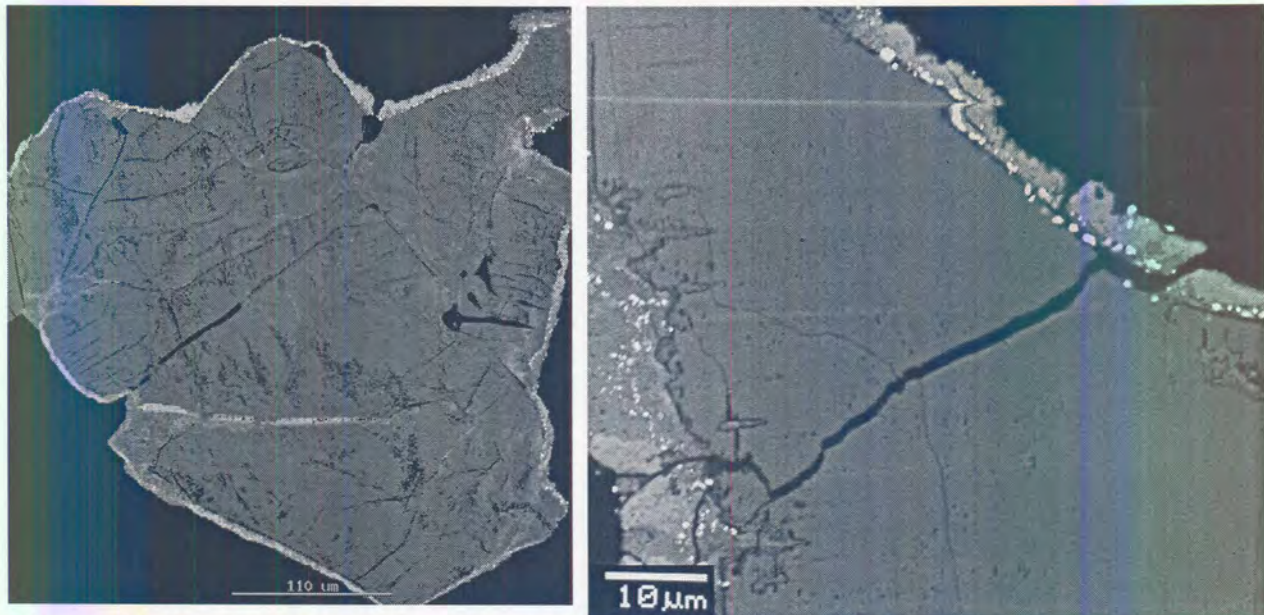
### 5.7.3 Investigation into the oxidation of coated slag particles

To investigate the migration of iron and titanium during oxidation an experiment was devised whereby the individual as-cast slag particles were coated with a marker material in order to observe the movement of cations. Gold was chosen as marker material and a few slag particles were sputter coated with gold (on average 0.5 to 1 µm thick). The gold-coated particles were mixed with uncoated particles that acted as a reference, and oxidised for 30 min at 850 °C in air. The phase chemical composition of the sample as determined by XRD-analysis is given in Table 49. Figure 66 shows micrographs of the sample after oxidation.

**Table 49.** Phase composition of the gold coated titania slag roasted in air at 850 °C for 30 min.

Description	Mineralogical composition		
	Main	Minor	Trace
Gold coated sample	Rutile; Anatase; FeTi-Oxide	-	Hematite

Legend : Rutile -  $TiO_2$ ; Anatase -  $TiO_2$ ; FeTi-Oxide -  $M_3O_5$ -solid solution; Hematite -  $Fe_2O_3$



**Figure 66.** Micrographs of the slag sample coated with gold after roasting in air at 850°C for 30 min.

Optically the gold-coated slag particles had a well-defined zoned texture and appeared to be finely porous. The majority of slag particles were characterised by the occurrence of small, partially oxidised,  $M_3O_5$ -rich cores with relatively broad  $TiO_2$ -rich mantle zones and thin iron enriched outer rims. Some of the slag particles were however completely oxidised and transformed to  $TiO_2$ . These completely oxidised slag particles still displayed thin, but well-defined iron enriched outer rims. Remnants of the original gold layer, sputter coated onto the outer surfaces of the slag particles, was preserved and was observed during both optical microscopy as well as SEM investigation. This gold coated layer, originally situated at the outer rims of the individual titania slag particles, was now situated at the outer rim of the  $TiO_2$ -rich mantle zones, between the mantle and iron enriched outer layer of the slag particles. The outer rims of the slag particles appeared to be dual phased as two optically different phases could be distinguished. According to the results obtained during SEM investigation the higher reflecting phase (abundant phase) situated at the outer rims of the oxidised slag particles, represents a very high iron containing phase, probably hematite and/or titano-hematite, with the lower reflecting phase containing lesser amounts of iron-oxide with appreciable amounts of titania. This result supports the suggested oxidation mechanism of iron migration towards the surfaces of the particles and oxygen anions towards the interiors of the particles where Ti(III) is oxidised.



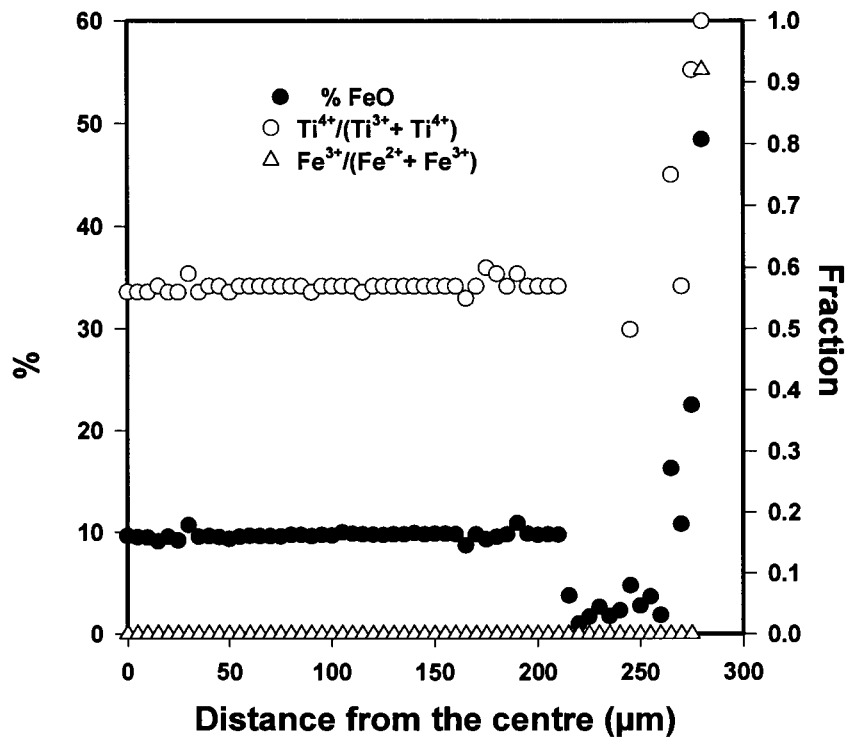
## **5.7.4 Investigation into the oxidation state of iron at various positions in oxidised slag particles**

### **5.7.4.1 WDS point chemical analysis**

Wavelength dispersive spectroscopy (WDS) analyses along lines through oxidised slag particles were conducted as part of the process development investigation in Chapter 4. These analyses showed that iron migrated to the outsides of the particles during oxidation. Based on the phases identified in the slag by XRD and the point chemical analyses it was possible to tentatively assign a phase composition to each of the points that were analysed. Basically one of three phases, glass, rutile or  $M_3O_5$  was assigned to a point. Glass was assigned if the  $SiO_2$  content exceeded 1 %. Rutile was assigned if the FeO content was below 4 % and all other analyses were assigned to  $M_3O_5$ . The chemical analyses of the points assigned to  $M_3O_5$  were mathematically forced to fit stoichiometrically into the  $M_3O_5$  formula. The amount of oxygen below or above the stoichiometric amount allowed the oxidation states of iron and titanium to be calculated. The results are presented in Figure 67. This shows that the unreacted cores contained about 10 % FeO. The iron is present in the Fe(II) oxidation state and the titanium in the Ti(IV) and Ti(III) oxidation states. In the  $TiO_2$ -rich mantle zone the FeO content varies between 0 % and 4 %. Iron is present in the Fe(II) oxidation state and titanium in the Ti(IV) and Ti(III) oxidation states. In the iron-rich rim the FeO content increases to about 50 %. The iron is present in rim in the Fe(II) oxidation state and the titanium in the Ti(IV) and Ti(III) states except for the extreme outer part where all iron is present in the Fe(III) oxidation state and all the titanium is in the Ti(IV) oxidation state.

This data showed that rutile precipitated from the original  $M_3O_5$  phase during oxidation. In the process the remaining ferrous  $M_3O_5$  phase gets enriched in iron and this can act as a driving force for the migration of iron to the outside surface of the particles or to local pores where ferric  $M_3O_5$  can precipitate by the oxidation of Fe(II) (and the associated extra precipitation of rutile). This is tentatively confirmed by the observed iron concentration gradient through the mantle zone (Chapter 4).





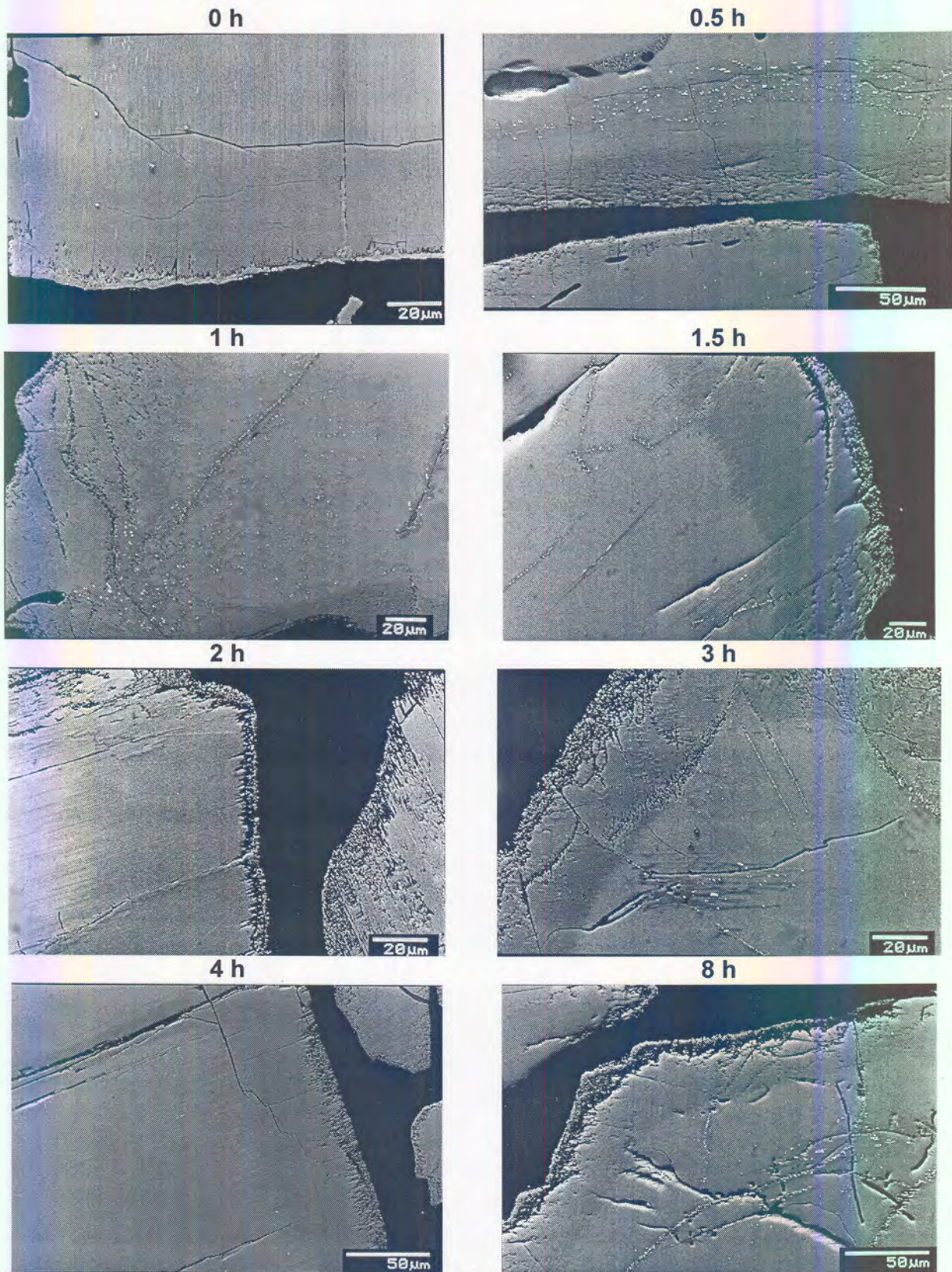
**Figure 67.** Variation of iron concentration, iron oxidation state and titanium oxidation state along a line through an oxidised slag particle.

#### 5.7.4.2 Leach investigation

These experiments were designed as an alternative to the WDS investigation to determine the oxidation state of iron in the  $TiO_2$ -rich mantle zones that exist between the cores of unreacted  $M_3O_5$  and the iron rich rims in oxidised titania slag particles. The areas in question had a relatively low iron concentration, but the iron migrates from the cores through this zone to the iron rich rims during oxidation.

Initially the following experiments were conducted: Titania slag was oxidised for 45 min at 850 °C in an atmosphere containing 10 %  $O_2$ . The slag was then subjected to leaching in boiling 20 % hydrochloric acid for various periods. To aid leaching  $SnCl_2$  was added as a reductant. The aim of these experiments was to determine the leach time that was required to remove the iron rich-rims from the slag particles. Figure 68 shows the effect of leaching on the morphology of the samples. The iron-rich rims appeared to have been removed in all the samples. With extended leaching times the mantle zones became slightly more porous, but the unreacted cores were unaffected even after 8 h of leaching.



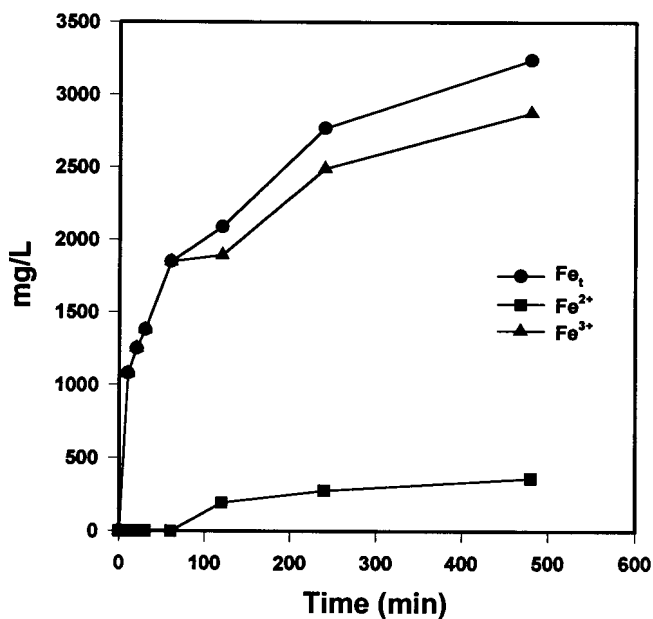


**Figure 68.** Titania slag oxidised for 45 min in 10 % O<sub>2</sub> at 850 °C and leached for different times under reducing conditions.



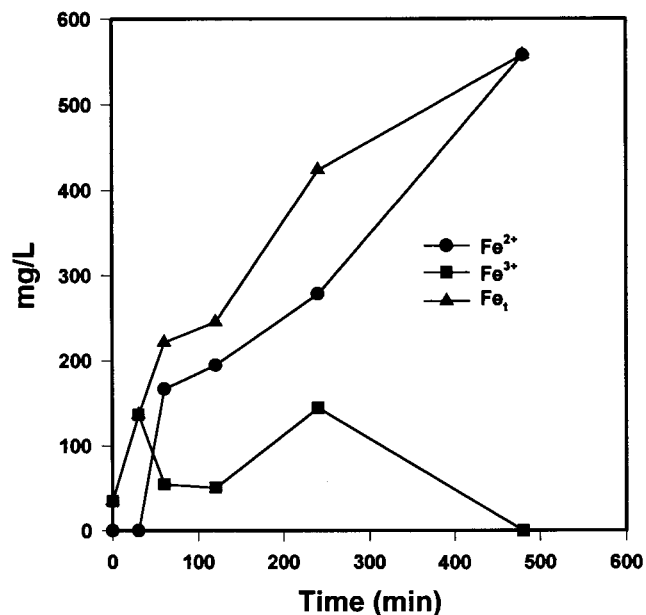
To provide information on the oxidation state of iron in the mantle zones two experiments were conducted. In the first experiment titania slag in the size range +710-850  $\mu\text{m}$  was oxidised for 90 min at 850  $^{\circ}\text{C}$  in 10 %  $\text{O}_2$  and leached for 8 h in boiling 20 %  $\text{HCl}$  without the addition of a reductant. The feed to the second experiment was roasted in the same manner as that for the first, but the slag was then subjected to a two stage leach procedure. Firstly the slag was leached for 1 h in boiling 20 %  $\text{HCl}$  with  $\text{SnCl}_2$  added as a reductant to remove the iron-rich rims on the slag particles. Thereafter the slag was separated from the leach solution, washed and dried before it was leached again for 8 h in boiling 20 %  $\text{HCl}$ , without addition of  $\text{SnCl}_2$ . In the leaches where  $\text{SnCl}_2$  was absent the speciation of iron in the leach solutions was determined by titration. The results of the experiments are listed in Appendix XV.

Figure 69 shows the speciation of iron in the leach solution of the slag where the iron-rich rims had not previously been removed. Initially iron goes into solution as  $\text{Fe(III)}$  and as leaching proceeds  $\text{Fe(II)}$  starts to appear along with  $\text{Fe(III)}$ . This supports the proposed oxidation mechanism as follows: The  $\text{Fe(III)}$  that initially goes into solution is located on the outsides of the particles. With time the leach solution start to penetrate into the particles and  $\text{Fe(II)}$  goes into solution.  $\text{Fe(III)}$  continues to be a major component of the leach solution as the rate of  $\text{Fe(III)}$  dissolution is very slow and this shields the majority of the  $\text{Fe(II)}$  located beneath the surface of the particles.



**Figure 69.** Iron speciation in solution during leaching of oxidised titania slag.





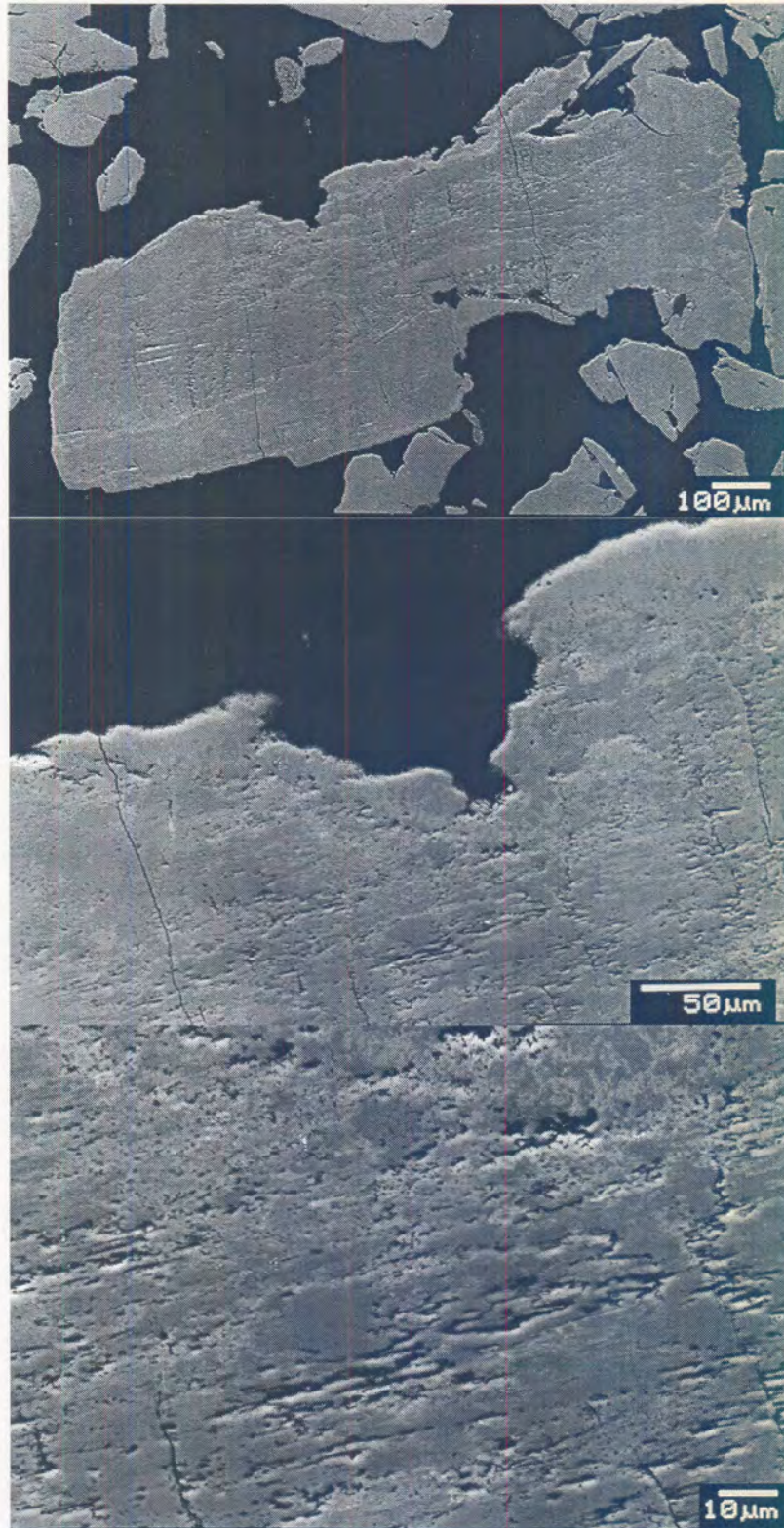
**Figure 70.** Iron speciation in solution during leaching of slag that was previously oxidised and reduction leached.

Figure 70 gives the speciation of iron in the leach solution of the slag where the iron-rich rims had previously been removed. Initially only Fe(III) is present in the leach solution, but soon the amount of Fe(III) in solution starts to decline and Fe(II) starts to appear. With extended leaching Fe(III) eventually disappears, while the amount of Fe(II) in solution continues to increase. This data supports the proposed oxidation mechanism as follows: The Fe(III) that initially goes into solution is a remnant of the iron-rich rims. With further leaching Fe(II) and Ti(III) comes into solution from the interiors of the particles. The Ti(III) in solution reduces the Fe(III) in solution until all the iron is present as Fe(II).

The results of these two experiments tentatively support the conclusion that iron in the enriched rim is present as Fe(III), but largely as Fe(II) deeper within the slag particles.

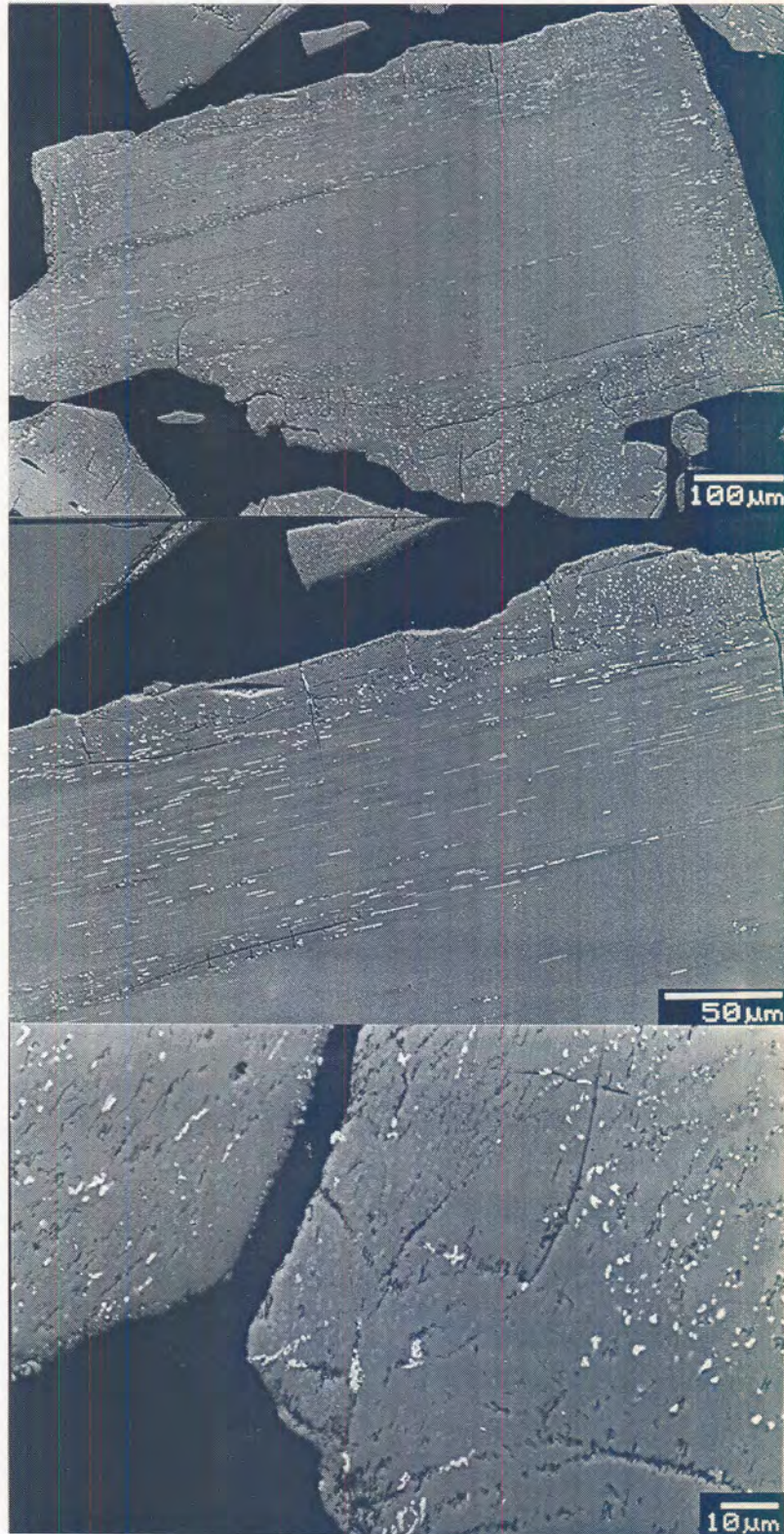
### **5.7.5 Investigation into influence of iron-rich rims on the mechanism of oxidation**

The iron rich rims on the outsides of oxidised slag particles can lower the nucleation energy that is needed to precipitate additional  $M_2O_3$ . This would explain why migrating iron cations would preferentially precipitate at the iron-rich rim rather than at any other location. To test this hypothesis titania slag was oxidised at 850 °C in 10 %  $O_2$  for 45 min and leached for 1 h under reducing conditions. After leaching the slag was washed and dried. The slag was then subjected to roasting for 2 h at 850 °C under three different atmospheres: air, argon and carbon monoxide. Micrographs of the different samples are presented in Figures 71 to 73. The phase compositions of the samples are given in Table 50.



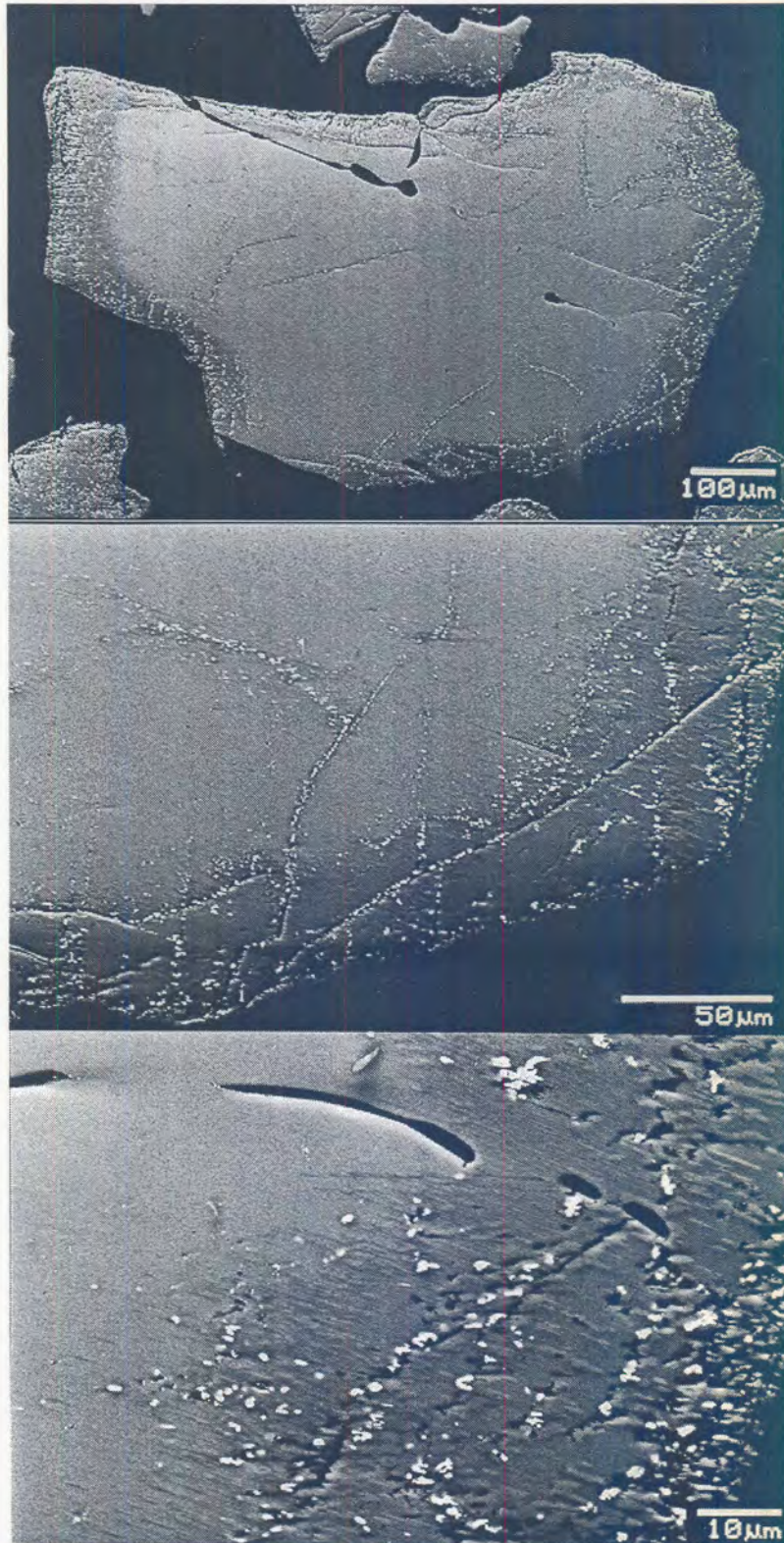
**Figure 71.** Titania slag that was oxidised for 45 min in air, reduction leached for 1 h and roasted again in air for 2 h at 850°C.





**Figure 72.** Titania slag that was oxidised for 45 min in air, reduction leached for 1 h and roasted again in argon for 2 h at 850°C.





**Figure 73.** Titania slag that was oxidised for 45 min in air, reduction leached for 1 h and roasted again in carbon monoxide for 2 h at 850°C.

**Table 50.** Phase-chemical compositions as determined by XRD of oxidised and reduction leached titania slag after roasting in various atmospheres.

Description	Mineralogical composition		
	Main	Minor	Trace
Sample after reduction leach	Rutile, Anatase	FeTi-Oxide	-
Sample roasted in air	Rutile	Anatase, FeTi-Oxide	-
Sample roasted in argon	Rutile	Anatase, FeTi-Oxide	Iron, Ilmenite
Sample roasted in carbon monoxide	Anatase, FeTi-Oxide, Rutile	-	Iron

**Legend:** Rutile -  $TiO_2$ ; Anatase -  $TiO_2$ ; FeTi-Oxide -  $M_3O_5$ -solid solution; Iron -  $Fe^0$ ; Ilmenite -  $FeTiO_3$

Figure 71 shows micrographs of the leached slag after roasting in air. No unreacted cores were present. The outer rims of the particles were porous due to the previous leaching. Iron did not migrate to the outer surfaces of the slag particles. Instead iron-rich areas intimately mixed and intergrown with titania-rich areas could be observed throughout the particles. The XRD results (Table 50) indicate that the iron-rich phase was the  $M_3O_5$  solid solution phase with rutile and anatase present as the titania-rich phases. This result shows that no iron migration towards the outer surfaces of the slag particles occurred in the absence of an iron-rich rim at the outer rims of the particles.

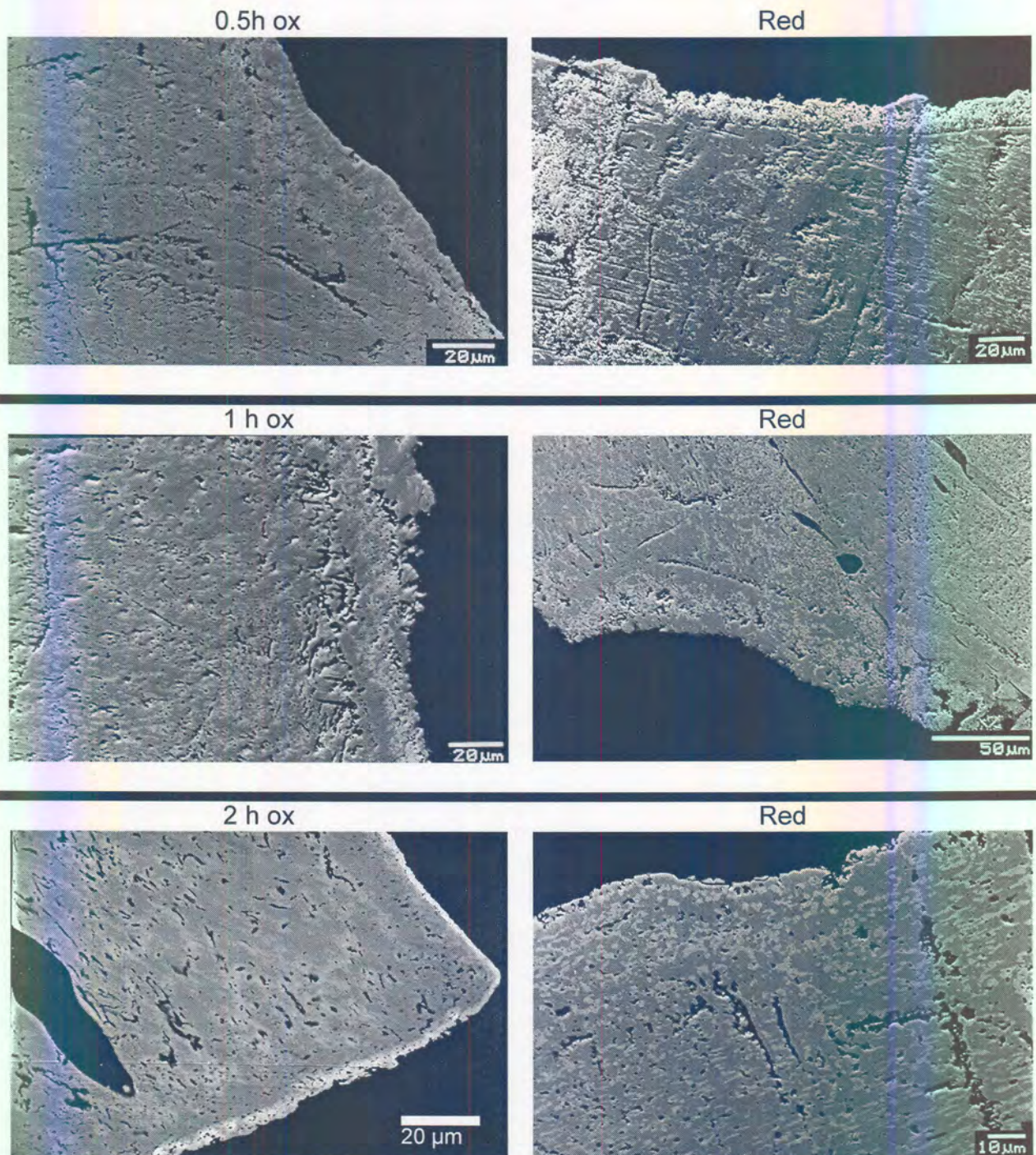
Figure 72 shows micrographs of the leached slag after roasting in argon. The slag particles were characterised by the occurrence of large unreacted  $M_3O_5$ -rich cores with a smooth and dense appearance. Fine-grained disseminated metallic iron precipitates were present and contained in the titania rich mantle zone. The XRD results (Table 50) show that small quantities of ilmenite were present in the particles. No iron migration occurred towards the previously leached outer rims of the slag particles. Metallic iron, rutile and ilmenite are the expected equilibrium phases for titania slag subjected to heat treatment in impure argon at  $850^\circ C$  (Figure 59). These phases were present towards the outer margins of the slag particles and it is assumed that if the slag was kept under these conditions for an optimum time period in order to reach equilibrium, the slag particles would consist entirely of these phases.

Figure 73 shows micrographs of the leached material after roasting in carbon monoxide. The slag particles were characterised by the occurrence of large  $M_3O_5$  rich cores and well-defined  $TiO_2$ -rich mantle zones. Metallic iron was present mainly in the mantle zones. No iron migration occurred towards the outer rims of the slag particles. The XRD results (Table 50) indicated that the  $M_3O_5$  solid solution phase increased to major phase after roasting. Anatase and rutile were also present as major phases in the slag. Metallic iron was present as a trace component. These phases correspond to those that are expected in titania slag in equilibrium with a carbon monoxide atmosphere at  $850^\circ C$  (Figure 59, Appendix XVI).

### **5.7.6 Investigation into the influence of higher roasting temperatures on the mechanism of oxidation**

Chapters 3 and 4 showed that the optimum roasting temperature for the production of BTS is  $850^\circ C$ . During oxidation at this temperature iron migrates from the insides of the slag particles towards the outsides where it is easily accessible during leaching.





**Figure 74.** Micrographs of slag oxidised at 1050 °C for various times. Micrographs of the samples after reduction for 20 min at 850 °C are also shown.

Borowiec et al. (1994) patented a process for the upgrading of titania slag. Part of the process consists of oxidation above 1000 °C. They found that fast diffusion of iron and titanium occurred in the pseudobrookite phase during oxidation. This resulted in a large number of pores in the slag particles with the iron cations concentrated around these pores. Two different morphologies therefore seem to result when slag is oxidised at



higher and lower temperatures. It was decided to investigate this effect and its relevance to the mechanism of titania slag oxidation.

Slag was oxidised at 1050 °C for various times. Following this the slag was reduced at 850 °C for 20 min. Tables 51 and 52 give the phase compositions and the Mössbauer analysis of the samples respectively (The detailed results are shown in Appendix XVIII). Micrographs of the samples are presented in Figure 74.

Figure 74 shows titania slag roasted at 1050 °C for 0.5 h. It appeared to be much more porous compared to slag roasted at 850 °C (see Figure 63 as a comparison). Noticeable as well was the absence of unreacted cores. Iron migration occurred towards the outer rims of the slag particles as well as the margins of pores. Abundant rutile rich areas closely associated and in contact with iron-rich, titania poor areas were present, randomly distributed throughout the slag particles.

After a reductive roast abundant iron-rich precipitates were present randomly distributed throughout the particles. The XRD results (Table 51) suggest that the precipitates were ilmenite.

**Table 51.** Phase-chemical compositions of slag samples roasted at 1050 °C.

Description	Mineralogical composition		
	Main	Minor	Trace
Oxidised for ½ h	Rutile	-	FeTi-Oxide
Oxidised for ½ h & reduced for 20 min	Rutile	-	Ilmenite, FeTi-Oxide
Oxidised for 1 h	Rutile	-	FeTi-Oxide
Oxidised for 1 h & reduced for 20 min	Rutile	-	Ilmenite, FeTi-Oxide
Oxidised for 2 h	Rutile	-	FeTi-Oxide
Oxidised for 2 h and reduced for 20 min	Rutile	-	Ilmenite, FeTi-Oxide

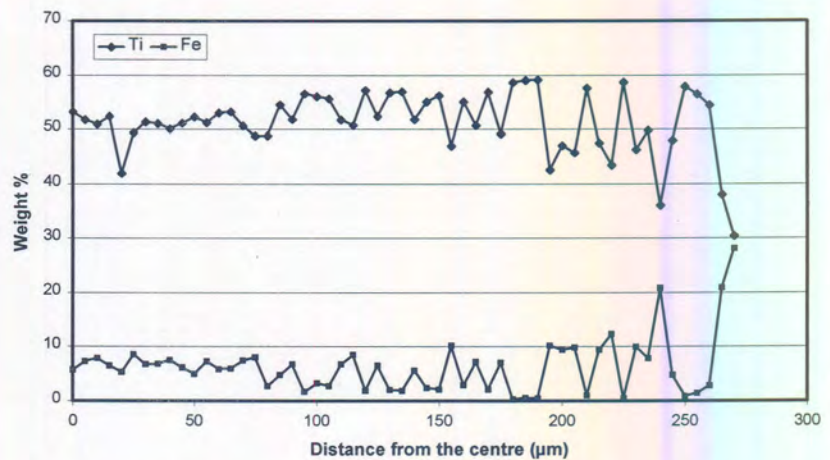
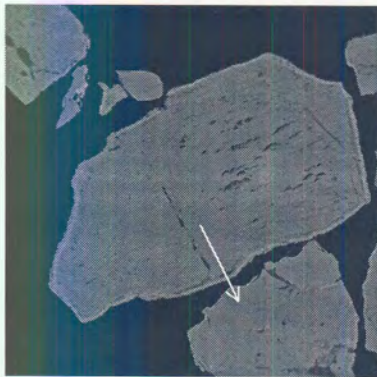
*Legend : Rutile - TiO<sub>2</sub>; Ilmenite - FeTiO<sub>3</sub>; FeTi-Oxide -M<sub>3</sub>O<sub>5</sub>-solid solution*

The Mössbauer results (Table 52, see APPENDIX XVIII for the detailed results) show that all the Fe (II) in the as-cast slag was oxidised to Fe(III) within 0.5 h during oxidation at 1050 °C. After 0.5 h of oxidation most of the iron was present in the Fe<sub>2</sub>TiO<sub>5</sub> phase, but some of the iron was also present in a hematite phase. The hematite phase disappeared as the oxidation time increased. After reduction all the samples contained iron in the Fe (III) and the Fe(II) oxidation states. Iron in the Fe(III) state was contained in the unconverted Fe<sub>2</sub>TiO<sub>5</sub> phase, while iron in the Fe(II) state was distributed between predominantly ilmenite along with some M<sub>3</sub>O<sub>5</sub>.

**Table 52.** Mössbauer analysis of slag samples oxidised at 1050 °C and reduced at 850 °C.

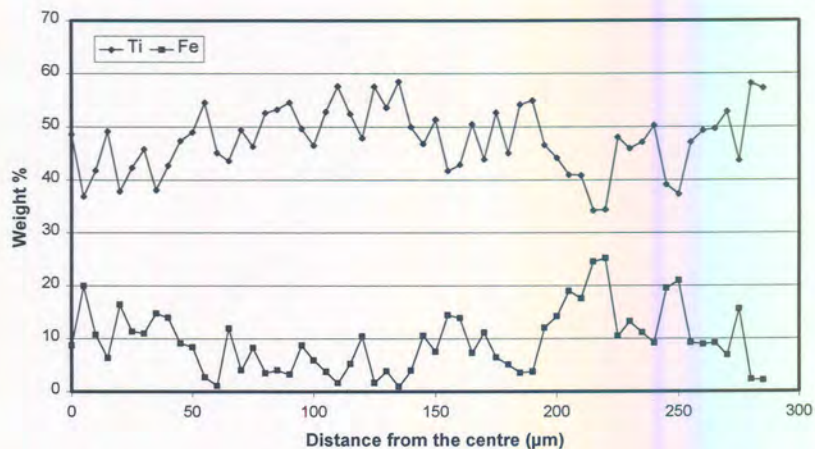
PFE	Description	% Abundance	Attribution
3065	Oxidised for ½ h	84	Fe <sub>2</sub> TiO <sub>5</sub>
		16	Hematite-like
3071	Oxidised for ½ h & reduced for 20 min	21	Fe <sub>2</sub> TiO <sub>5</sub>
		64	Ilmenite-like
		15	FeTi <sub>2</sub> O <sub>5</sub>
3066	Oxidised for 1 h	100	Fe <sub>2</sub> TiO <sub>5</sub>
3072	Oxidised for 1 h & reduced for 20 min	20	Fe <sub>2</sub> TiO <sub>5</sub>
		65	Ilmenite-like
		15	FeTi <sub>2</sub> O <sub>5</sub>
3067	Oxidised for 2 h	100	Fe <sub>2</sub> TiO <sub>5</sub>
3073	Oxidised for 2 h and reduced for 20 min	21	Fe <sub>2</sub> TiO <sub>5</sub>
		69	Ilmenite-like
		10	FeTi <sub>2</sub> O <sub>5</sub>

The samples oxidised at 1050 °C were submitted for WDS line chemical analysis through some of the particles. The aim was to compare these results with that of slag particles roasted at 850 °C (Chapter 4). The results are presented in Figures 75 and 76.



**Figure 75.** WDS Line chemical analysis through a particle of standard titania slag that was oxidised at 1050 °C for 30 min in 10 % O<sub>2</sub>.





**Figure 76.** WDS Line chemical analysis through a particle of standard titania slag that was oxidised at 1050 °C for 60 min in 10 % O<sub>2</sub>.

The results presented in Figures 75 and 76 show that unreacted cores were present in some particles after 30 min of oxidation, but after 60 min no cores could be observed. The line chemical analyses in the reacted zones of the particles showed a large variation in titanium and iron concentration with position. This behaviour can be related to the morphology of the particles. All of the particles were very porous and the large variation in iron and titanium concentrations suggests that iron migration occurred to the margins of the pores. This resulted in titanium rich areas intermingled with iron rich areas.

The morphology observed in the samples oxidised at 1050 °C was similar to that described by Borowiec et al. (1996). The difference in morphology between samples oxidised at 850 °C and 1050 °C can be explained by the proposed titania slag oxidation mechanism. According to the thermodynamic considerations discussed in paragraph 5.3.2 the stability area of hematite decreases with increasing temperature. There is consequently a much smaller amount of M<sub>2</sub>O<sub>3</sub> that precipitates at higher temperatures (See Appendix XVII). The nucleation energy that is needed to for ferric pseudobrookite is probably much less than that needed to precipitate hematite. Iron-rich pseudobrookite consequently precipitates through the slag particles at higher roasting temperatures.

### **5.7.7 Investigation into the influence of interrupted roasting on the mechanism of oxidation**

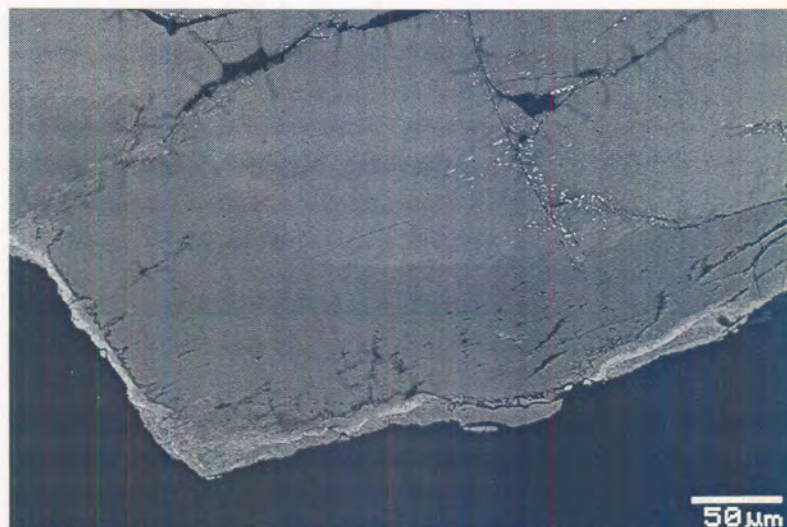
Two experiments were conducted to test whether an interruption in the roasting process changes the mechanism of oxidation. The first experiment was performed by subjecting titania slag to an oxidative roast in air for 30 min at 850 °C whereafter the slag was cooled down to room temperature and oxidised again in air for 2 h at 850 °C. For the second experiment titania slag was subjected to an oxidative roast in air for 30 min at 850 °C, cooled down to room temperature and subjected to a second oxidative roast in air for 2 h at 1050 °C. Micrographs of the two samples are shown in Figures 77 and 78. The phase chemical compositions of the samples are given in Table 53.



**Table 53.** Phase-chemical composition as determined by XRD-analysis of the samples subjected to interrupted roasting.

Sample No.	Roast procedure	Mineralogical composition		
		Main	Minor	Trace
A524-57	1 <sup>st</sup> Roast - 850 °C, 30 min, air 2 <sup>nd</sup> Roast - 850 °C, 2 h, air	Anatase; Rutile	FeTi-Oxide	-
A524-58	1 <sup>st</sup> Roast - 850 °C, 30 min, air 2 <sup>nd</sup> Roast - 1050 °C, 2 h, air	Rutile	FeTi-Oxide	-
A524-59	1 <sup>st</sup> Roast - 850 °C; 30 min, air 2 <sup>nd</sup> Roast - 1050 °C; 2 h; air 3 <sup>d</sup> Roast - 850 °C; 20 min; 100 % CO	Rutile	-	FeTi-Oxide; Ilmenite

Legend : Rutile -  $TiO_2$ ; FeTi-Oxide -  $M_3O_5$  solid solution ; Anatase -  $TiO_2$



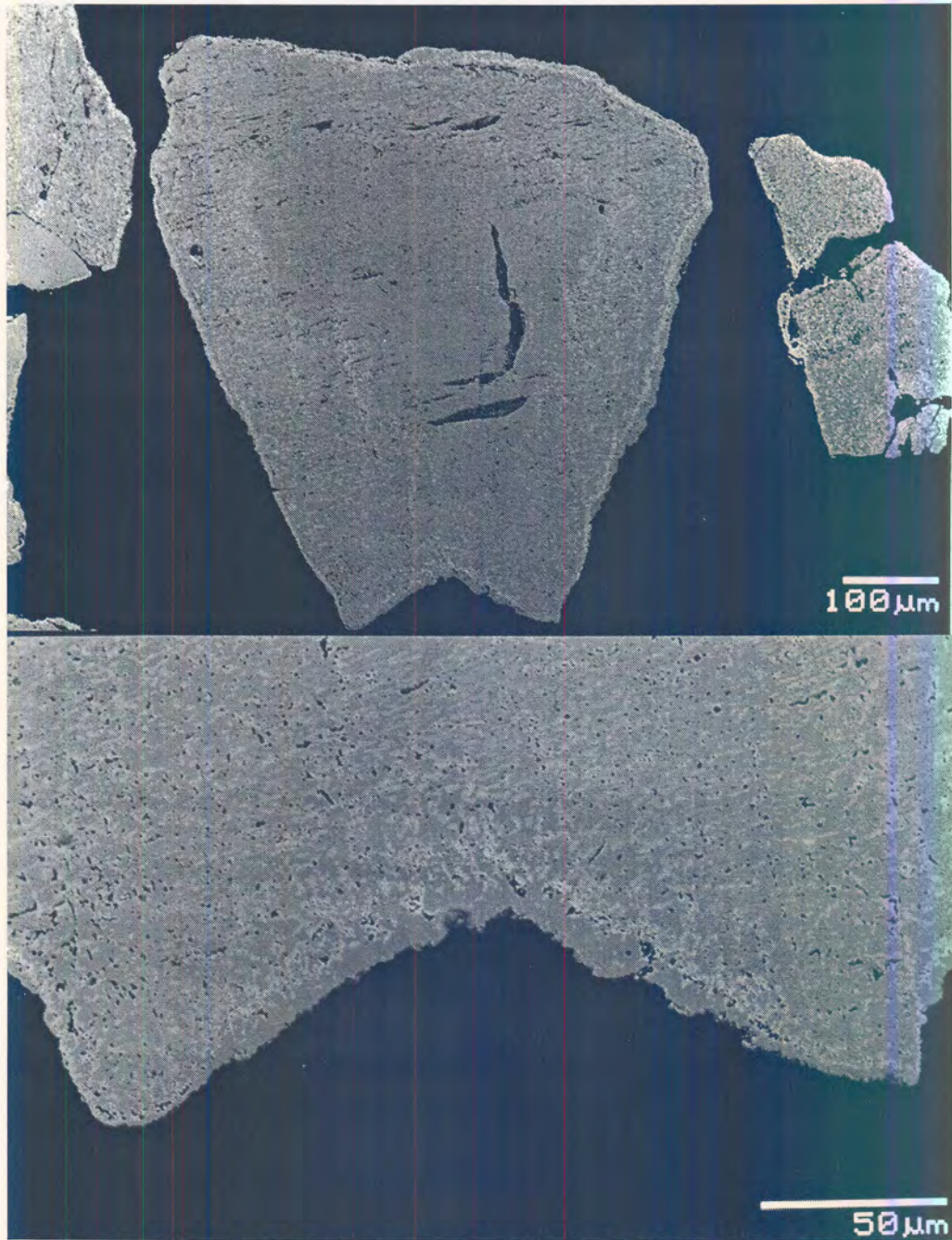
**Figure 77.** Micrographs of a titania slag sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 850 °C for 2 h.

The sample subjected to an oxidative roast in air at 850 °C for 30 min. and roasted again in air for 2 h at 850 °C consisted predominantly of anatase and rutile with lesser amounts of the  $M_3O_5$  solid solution phase. Optically the slag particles looked similar to slag samples that were roasted without interruption (Figure 77). The particles had a zoned texture and iron migration occurred towards the outer margins of the individual slag particles. Oxidation occurred along cracks cutting through the unreacted  $M_3O_5$ -rich cores with the crystallisation of rutile associated with the occurrence of fine metallic iron “blebs” and precipitates. An interruption in the normal roast procedure therefore did not change the oxidation mechanism.

The titania slag sample subjected to an oxidative roast in air at 850 °C for 30 min and roasted again in air at 1050 °C for 2 h consisted primarily of rutile with minor amounts of the  $M_3O_5$ -solid solution phase. No anatase was observed by XRD-analysis. Optically the slag particles seemed to be in general completely oxidised and transformed to rutile (Figure 78). Only a few of the coarser-grained slag particles displayed small oxidised  $M_3O_5$ -rich cores. Iron migration occurred towards the outer margins of the slag particles with the majority of slag particles displaying well-defined iron enriched outer rims. A thin relatively fragile rutile-rich layer seemed however to have formed beyond these iron-enriched rims. The mantle zones of the slag particles appeared to be very porous. The outer margins of the individual pores were characterised by iron enrichment as iron migration occurred towards the outer margins of the pores. The cores of the particle had in general a relatively dense appearance.

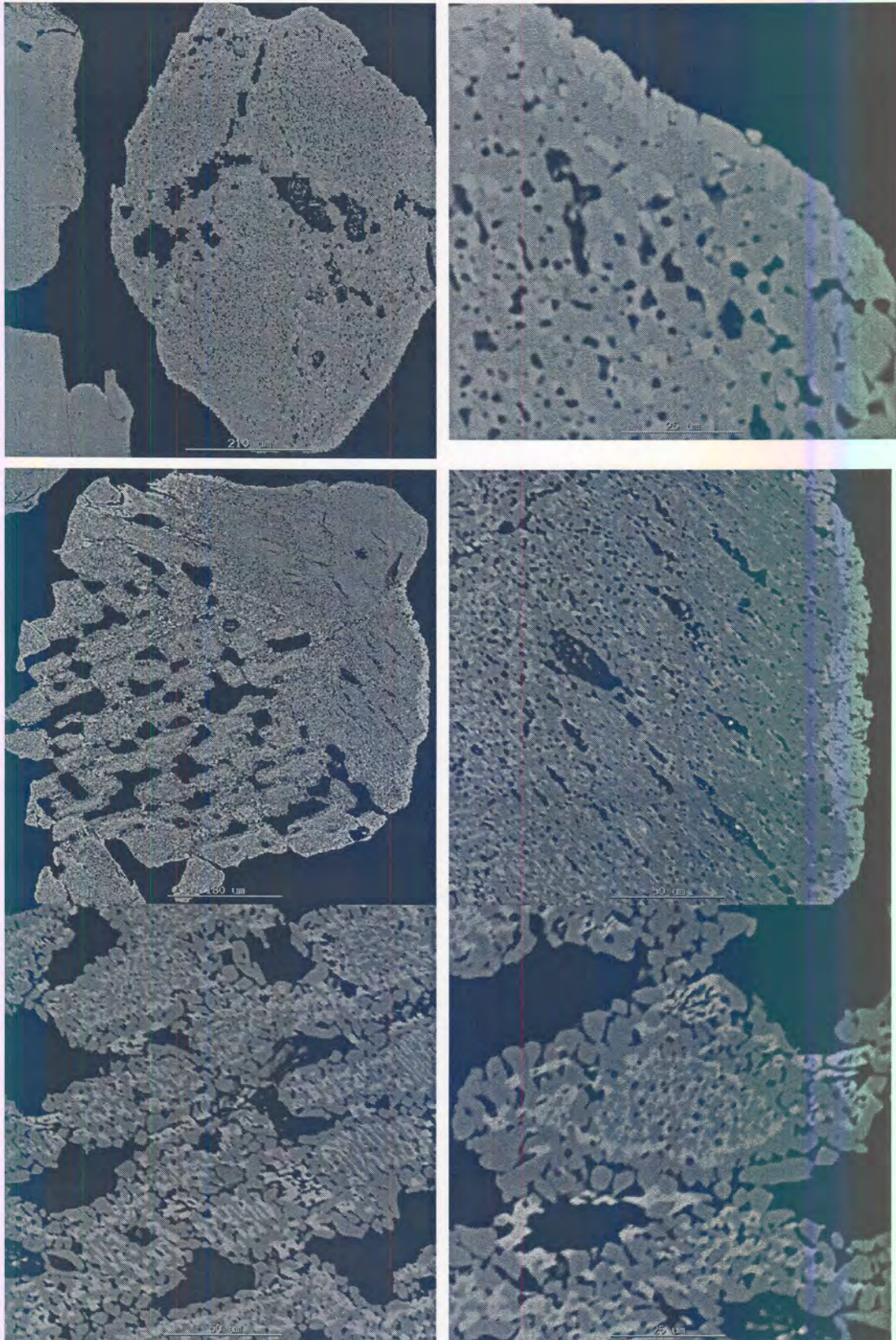
Some of the slag particles displayed a well-defined crystalline texture and seemed to be partially to completely recrystallised during the roasting procedure. The recrystallised slag particles appeared to be much more porous compared to the slightly denser appearance of the remainder of the slag particles. The recrystallised slag particles consisted primarily of densely packed, fine-grained, rounded to slightly elongated, rutile crystals whereas in other particles crystallisation seems to be uncompleted with coarse-grained rutile “blebs” and precipitates as well as needles and laths “embedded” in the oxidised  $M_3O_5$  rich matrix (Figure 79). These recrystallised particles displayed well-defined iron enriched outer margins. These iron-rich outer margins displayed a crystalline nature consisting of densely packed and interwoven fine-grained tabular-, needle- and lath-like crystals. The slag sample was furthermore characterised by the occurrence of several sintered slag particles as sintering occurred between the iron-enriched outer rims of the individual slag particles (Figure 80).





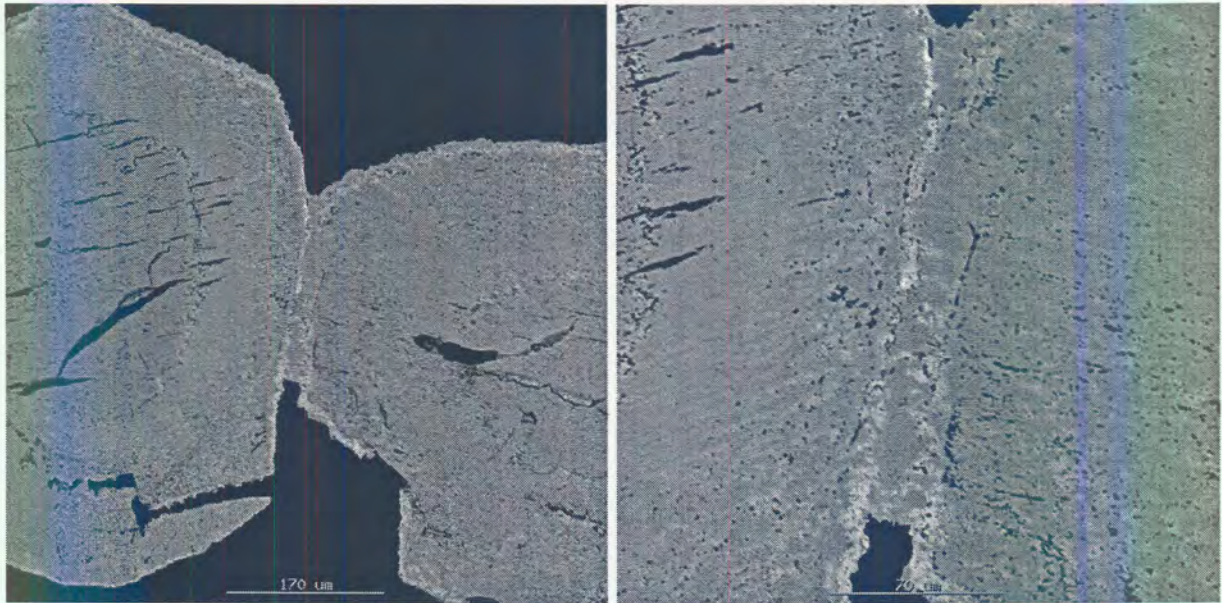
**Figure 78.** Micrographs of a titania slag sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 1050 °C for 2 h.





**Figure 79.** Micrographs of a recrystallised titania slag particle observed in the slag sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 1050 °C for 2 h





**Figure 80.** Micrographs of sintered titania slag particles observed in a sample that was oxidised at 850 °C for 30 min, then cooled to room temperature and oxidised again at 1050 °C for 2 h.

An explanation of the morphology observed in the sample oxidised at 850 °C for 30 min and then oxidised further at 1050 °C can be attempted by using the proposed oxidation mechanism. During oxidation at 850 °C iron migrates to the outside rims of the particles where it precipitates as  $M_2O_3$  and  $M_3O_5$ . This results in the formation of an iron-rich rim on the outsides of the particles. When the partially oxidised sample is then further oxidised at 1050 °C a different morphology result. This is as a result of the instability of  $M_2O_3$  at higher temperatures. The iron now preferentially precipitates as  $M_3O_5$  which is potentially much easier to nucleate. The hematite that previously precipitated on the outsides of the particles now also converts to pseudobrookite. The exothermic reaction heat that is generated during oxidation can explain the sintering that was observed between some of the particles. This heat in conjunction with the high furnace temperature resulted in the sintering. The recrystallisation that was observed in about a third of the particles cannot be explained by the proposed oxidation mechanism. This may have occurred as a result of the high temperature and the resulting high reaction rates that allowed the slag sample to move rapidly towards thermodynamic equilibrium.

### 5.8 Quantitative WDS analyses of selected phases in oxidised slag

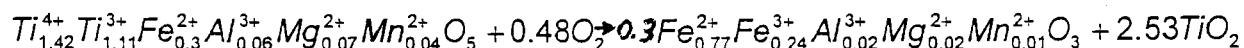
The composition of selected phases in oxidised titania slag were determined quantitatively by SEM-WDS. A summary of the data presented in Chapter 4, Figure 28 is shown in Table 54. These phases include  $TiO_2$  and the solid solution phases  $M_3O_5$  and  $M_2O_3$ . In the instances where  $M_3O_5$  and  $M_2O_3$  were identified the cation to oxygen stoichiometric ration was forced to be equal to either 3:5 or 2:3 whereby the relative amounts of Ti(IV), Ti(III), Fe(II) and Fe(III) in the  $M_3O_5$  and  $M_2O_3$  phases were calculated.



**Table 54.** Quantitative WDS analyses of selected phases in oxidised slag.

Location	Al <sub>2</sub> O <sub>3</sub>	MnO	MgO	TiO <sub>2</sub>	FeO	Total	Stoichiometry
Core	1.29	1.42	1.35	91.69	9.85	105.6	Ti <sup>4+</sup> <sub>1.42</sub> Ti <sup>3+</sup> <sub>1.11</sub> Fe <sup>2+</sup> <sub>0.3</sub> Al <sup>3+</sup> <sub>0.06</sub> Mg <sup>2+</sup> <sub>0.07</sub> Mn <sup>2+</sup> <sub>0.04</sub> O <sub>5</sub>
Mantle	0.71	0.49	0.51	96.15	1.85	99.71	Ti <sup>4+</sup> <sub>0.95</sub> Fe <sup>2+</sup> <sub>0.02</sub> Al <sup>3+</sup> <sub>0.01</sub> Mg <sup>2+</sup> <sub>0.01</sub> Mn <sup>2+</sup> <sub>0.01</sub> O <sub>2</sub>
Rim	3.68	0.41	1.25	43.96	48.45	97.75	Ti <sup>4+</sup> <sub>0.82</sub> Fe <sup>2+</sup> <sub>0.77</sub> Fe <sup>3+</sup> <sub>0.24</sub> Al <sup>3+</sup> <sub>0.11</sub> Mg <sup>2+</sup> <sub>0.05</sub> Mn <sup>2+</sup> <sub>0.01</sub> O <sub>3</sub>

Using the data in Table 54 a balanced chemical reaction can be written for the oxidation of titania slag:



## 5.9 Conclusions

An oxidation mechanism for titania slag was proposed: The mechanism is based on the phases that form during oxidation. At lower roasting temperatures rutile, M<sub>2</sub>O<sub>3</sub> and M<sub>3</sub>O<sub>5</sub> form in the slag. The nucleation energy that is needed for the precipitation of individual M<sub>2</sub>O<sub>3</sub> crystals is probably much higher than that needed to precipitate individual rutile and M<sub>3</sub>O<sub>5</sub> crystals, because the crystal structure of M<sub>2</sub>O<sub>3</sub> differs radically from M<sub>3</sub>O<sub>5</sub>. This results in the migration of iron to the outside surface of the slag particles where less nucleation energy is needed to precipitate M<sub>2</sub>O<sub>3</sub> on the existing free surfaces. At higher roasting temperatures the stability of M<sub>2</sub>O<sub>3</sub> declines and the iron precipitates as M<sub>3</sub>O<sub>5</sub> throughout the particles.

This mechanism was tentatively confirmed through selected experiments:

- It was shown that oxygen is required for iron migration to occur;
- Particle size measurements of oxidised slag as well as an experiment conducted with a gold marker on the slag confirmed that the particle size of the slag increases as the iron migrates to the outside surfaces of the particles;
- The role of the iron-rich rim, as a site where low nucleation energy is required for the continued precipitation of M<sub>2</sub>O<sub>3</sub>, was confirmed when the iron-rich rim was removed with reductive leaching. Under these conditions iron did not continue to migrate to the outside surfaces of the particles, but precipitated locally;
- Roasting at temperatures in excess of 1000°C changed the morphology of the oxidised slag and iron migration no longer occurred to the outside surfaces of the particles. This may be related to the instability of M<sub>2</sub>O<sub>3</sub> at higher temperatures and;
- An interruption in the oxidation process did not change the morphology of the oxidised slag, but partial oxidation at lower temperatures followed by oxidation at temperatures in excess of 1000°C did change the morphology of the slag drastically; Iron stopped migrating to the outside surfaces of the particles and rather precipitated locally.

To take advantage of iron migration the oxidation conditions need to be chosen very carefully as this phenomenon is not dependent of equilibrium behaviour, but rather on kinetic behaviour of the system.



## SUMMARY

Phase 1 of the process development for the production of beneficiated titania slag was conducted with a coal fired fluid bed. Based on the results from this investigation the following optimum processing conditions were recommended:

- Oxidation: 3 h at 850°C
- Reduction: 30 min at 800°C
- Leaching: 12 h with 20% HCl at 20% excess

It was also found that the most important process parameters were roasting temperature and the chemical composition of the slag. The highest TiO<sub>2</sub> content achieved was 94%.

Phase 2 of the process development was conducted under more stringent roasting conditions, where the gas atmosphere in the reactor was regulated with a gas mixing system. Based on the results from this investigation the following optimum process conditions are recommended:

- Oxidation: 1.5 h at 850° in 8% O<sub>2</sub>
- Reduction: 10 min at 850°C in 100% CO
- Leaching 12 h with 20 % HCl

As part of the process development the phase changes that occur were characterised; during oxidation the FeTi<sub>2</sub>O<sub>5</sub>-Ti<sub>2</sub>O<sub>3</sub> (ferrous M<sub>3</sub>O<sub>5</sub>) phase that is present in the as-cast slag is oxidised to rutile or anatase, M<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>TiO<sub>5</sub>-FeTi<sub>2</sub>O<sub>5</sub> (ferric M<sub>3</sub>O<sub>5</sub>). During reduction the M<sub>2</sub>O<sub>3</sub> and ferric M<sub>3</sub>O<sub>5</sub> phases are converted to ilmenite. The ilmenite phase as well as the residual M<sub>3</sub>O<sub>5</sub> phase in the slag that contains most of the impurities is removed during the leaching stage. The highest TiO<sub>2</sub> content that was achieved was 97.5%.

An important additional benefit that occurs during oxidation is the migration of iron to the outsides of the slag particles where it is easily accessible during leaching. The mechanism whereby this occurs was investigated and it was postulated that the iron migration is linked to high nucleation energy that is needed for the formation of the separate M<sub>2</sub>O<sub>3</sub> crystals. The iron seems to preferentially precipitate at the free surface that is created at the iron-rich rim. This hypothesis was tentatively confirmed through selected experiments.

The following issues remain unresolved:

- The effect of impurities such as Ca and Mg on the mechanism of oxidation;
- A detailed investigation of the crystal changes that occur during oxidation;
- An in depth investigation into the leach behaviour of roasted slag and;
- A larger scale demonstration of the BTS process.

## REFERENCES

- Abuluwefa, H.T., Guthrie, R. and Ajersch, F. 1997. Oxidation of low carbon steel in multicomponent gases: Part I. Reaction mechanism during isothermal oxidation. *Met. Trans. A*, 28A, pp. 1633-1641.
- Akimoto, T., Kinoshita, H. and Furuta, T. 1984. Electron probe microanalysis study on processes of low-temperature oxidation of titanomagnetite. *Earth and Planetary science Letter*, 71, pp. 263-278.
- Amorelli, A., Evans, J.C. and Rowlands, C.C. 1987. An electron spin resonance study of rutile and anatase titanium dioxide polycrystalline powders treated with transition metals ions. *J. Chem. Soc., Faraday Trans.*, 12, pp. 3541-3548.
- Bickley, R.I., Gonzalez-Carreño, T., Gonzalez-Elipé, A.R., Munuera, G. and Palmisano, L. 1994. Characterisation of iron/titanium oxide photocatalysts. *J. Chem. Soc., Faraday Trans.*, 90(15), pp. 2257-2264.
- Bickely, R.I., Gonzalez-Carreño, T. and Palmisano, L. 1991. A study of the interaction between iron(III)oxide and titanium(IV)oxide at elevated temperatures. *Materials Chemistry and Physics*, 29, pp. 475-487.
- Briggs, R.A. and Sacco, A. 1993. The oxidation of ilmenite and its relationship to the FeO-Fe<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> phase diagram at 1073 and 1140 K. *Met. Trans. A*, 24A, June, pp. 1257-1264.
- Borowiec, K., Grau, A.E., Gueguin, M. and Turgeon, J.F. 1996. Method to upgrade titania slag and resulting product. *South African Pat.* 96/9772.
- Borowiec, K., Grau, A.E., Gueguin, M. and Turgeon, J.F. 1998. Method to upgrade titania slag and resulting product. *US Pat.* 5,830,420, 3 Nov.
- Borowiec, K. and Rosenqvist, T. 1981. Phase relations and oxidation studies in the system Fe-Fe<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> at 700-1100°C. *Scand. J. Metall.*, 20, pp. 91-119.
- Borowiec, K., Rosenqvist, T., Tuset, J.Kr. and Ulvensøen, J.H. 1987. Synthetic rutile from titaniferous slags by a pyrometallurgical route. Pyrometallurgy '87, IMM, London (UK), pp.91-119.
- Bull, D.S. 1992. Titanium dioxide pigments by the chloride process. *Paint and Resin*, January/February, pp. 15-19.
- Chiang, Y.M., Henriksen, A.F. and Kingery, W.D. 1981. Characterisation of grain-boundary segregation in MgO. *J. Am. Ceram. Soc.*, 64(7), pp. 385-389.
- Cook, R.F. and Schrott, A.G. 1988. Calcium segregation to grain boundaries in alumina. *J. Am. Ceram. Soc.*, 71(1), pp. 50-58.
- Doan, P.H. 1996. Upgraded slag (UGS): implications for TiO<sub>2</sub> feedstock supply. Proceedings of 12<sup>th</sup> Industrial Minerals Int. Congress, pp. 71-76.
- Elger G.W. and Holmes R.A. 1982. Purifying titanium-bearing slag by promoted sulfation. *U.S. Pat* 4,362,557, 7 Dec.
- Elger, G.W., Kirby, D.E., Rhoads, S.C. and Stickney, W.A. 1974. *Synthesis of rutile from domestic ilmenites*. US Bureau of Mines Report of Investigation 7985, pp. 19.
- Ericksson, G. and Pelton, A.D. 1996. Measurement and thermodynamic evaluation of phase equilibria in the Fe-Ti-O system. *Ber. Bunsenges. Phys. Chem.*, 100(11), pp. 1839-1849.
- FACT thermodynamic database, <http://www.crct.polymtl.ca/fact/fact.htm>
- Fisher, J.R. 1997. Developments in the TiO<sub>2</sub> pigment industry which will drive demand for TiO<sub>2</sub> mineral feedstocks. In: *Heavy Minerals 1997*. Ed. R.E. Robinson, SAIMM, Johannesburg (South Africa), pp. 207-218.
- Gambogi, J. 1991. Minerals Yearbook: Titanium. *Annual Report: United States Department of the Interior, Bureau of Mines*.
- Gambogi, J. 1998. Minerals Yearbook: Titanium. *Annual Report: United States Geological Survey*.
- Gueguin, M. 1986. Process of producing synthetic rutile from titaniferous product having a high reduced titanium oxide content. *US Pat.* 4,629,607, 16 Dec.
- Gueguin, M. 1990. Method of preparing a synthetic rutile from a titaniferous slag containing magnesium values. *US Pat.* 4,933,153, 12 Jun.
- Gueguin, M. 1991. Method of preparing a synthetic rutile from a titaniferous slag containing alkaline earth metals. *US Pat.* 5,389,355, 14 Feb.
- Gueguin, M. 1995. Method of preparing a synthetic rutile from a titaniferous slag containing magnesium values. *US Pat.* 5,063,032, 5 Nov.
- Gueguin, M. and Grau, A.E. 1989. Upgrading of titania slags by fluidized-bed selective





- Hollitt, M.J., O'Brien, B.A. and Grey, T.C. 1993. Production of synthetic rutile. *US Pat. 5,427,749*, 27 Jun.
- Holman, J.P. 1976. *Heat Transfer*. McGraw-Hill Kogakusha Ltd., Tokyo, pp. 503.
- Iscor Heavy Minerals Brochure. 1997. Distributed during the Heavy Minerals Congress.
- Jarish, B. 1977. Upgrading Sorelslag for production of synthetic rutile. *US Pat. 4,038,363*, 26 Jul.
- Kingery, W.D. (1984) Segregation phenomena at surface and at grain boundaries in oxides and carbides. *Solid state ionics*, 12, pp. 299-307.
- Kubaschewski, O., Alcock, C.B. and Spencer, P.J. 1993. *Materials Thermochemistry*, 6<sup>th</sup> edition. Pergamon, Oxford.
- Kunii, D. and Levenspiel, O. 1977. *Fluidization Engineering*. Robert E. Krieger Publishing Co., Huntington, N.Y.
- Leddy, J.J. and Schechter, D.L. 1962. Pressure leaching of titaniferous material. *US Pat. 3,060,002*, 23 Oct.
- Lindsley, D.H. 1976. Experimental studies of oxide minerals. In *Reviews in Mineralogy*, vol.3, Editor D. Rumble III, Mineralogical Society of America, Chelsea, Michigan, pp. L61-L84.
- Lurie, J. 1987. *South African geology*, Lexicon.
- Lynd, L.E., Sigurdson, H., North, C.H. and Anderson, W.W. 1954. Characteristics of titaniferous concentrates. *Trans. AIME*, Aug, pp. 817-824.
- Minkler, W.W. and Baroch, E.F. 1981. The production of titanium, zirconium and hafnium. Proceedings of TMS-AIME USA-China Bilateral Conference: *Metallurgical Treatises* (edited by J.K. Tien and J.F. Elliot), pp. 171-185.
- Nafziger, R.H. and Elger, G.W. 1987. Preparation of titanium feedstock from Minnesota ilmenite by smelting and sulfation-leaching. *US Bureau of Mines RI9065*.
- Nicol, M.J. 1983. The non-oxidative leaching of oxides and sulphides: an electrochemical approach. In *Hydrometallurgy: Research, Development and Plant Practice*, Editors K. Osseo-Asare and J.D. Miller, SME-AIME, Warrendale PA (USA), pp. 177-195.
- Nobile, A. and Davis, M.W. 1989. Importance of the anatase-rutile phase transition and titania grain enlargement in the strong metal-support interaction phenomenon in Fe/TiO<sub>2</sub> catalysts. *J. Catalysis*, 116, pp. 383-398.
- Oden, L.L., Sumner, D.H., Howe, J. 1973. Studies on recovering rutile from titanium-enriched-high iron-smelter slag. *U.S. Bureau of Mines Report of Investigation 7742*, p. 10.
- O'Reilly, W. and Banerjee, S.K. 1966. Oxidation of titanomagnetites and self reversal. *Nature*, 211, pp. 26-28.
- Perry, R.H. and Green, D. (eds.). 1984. *Perry's chemical engineers' handbook*. Sixth edition. McGraw-Hill Book Company, Tokyo.
- Pint, B.A., Garratt-Reed, A.J. and Hobbs, L.W. 1998. Possible role of oxygen potential gradient in enhancing diffusion of foreign ions on  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> grain boundaries. *J. Am. Ceram. Soc.*, 81(2), pp. 305-314.
- Rao, D.B. and Rigaud, M. 1974. Oxidation of ilmenite and the product morphology. *High temperature science*, 6, pp. 323-341.
- Ryall, P.J.C. and Hall, J.M. 1980. Iron loss in titanomagnetites during low temperature oxidation, *J. Geomag. Geoelectr.*, 32, pp. 661-669.
- Readman, P.W. and O'Reilly, W. 1972. Magnetic properties of oxidised (cation-deficient) titanomagnetites (Fe, Ti, )<sub>3</sub>O<sub>4</sub>, *J. Geomag. Geoelectr.*, 24, pp. 69-90.
- Sinha, H.N. 1984. Hydrochloric acid leaching of ilmenite, Proc. Symp. on *Extractive Metallurgy*, AusIMM, Melbourne (Australia), pp. 163-168.
- Tikkanen, M.H.A. and Tholand, N.K.G. 1960. Extraction of iron from iron-bearing titaniferous raw materials. *US Pat. 2,961,298*, 22 Nov.
- Tikkanen, M.H., Tyynälä, T. and Vuoristo, E. 1964. Reducing pressure leaching of an ilmenite concentrate. In: *Unit processes in hydrometallurgy*. Ed. M.E. Wadsworth and Davis, Gordon and Breach Science Publ., New York (USA), pp. 269-283.
- Van Dyk, J.P. 1996. Evaluation of processes that upgrade titanium bearing slags by utilising phosphate additions. *MEng dissertation*, University of Pretoria (South Africa).
- Walpole, E.A. (1993). Acid Regeneration, *World patent WO 9316000*, Aug 19.
- Webster, A.H. and Bright, N.F.H. 1961. The system iron-titanium oxygen at 1200°C and oxygen partial pressures between 1 atm and 2x10<sup>-14</sup> atm. *J. Am. Ceram. Soc.*, 44, pp. 110-116.

## LIST OF APPENDICES

<b>Appendix I</b>	<b>Chemical analysis of the feed slags used for the preliminary investigation</b>
<b>Appendix II</b>	<b>Log sheets for the preliminary investigation experiments</b>
<b>Appendix III</b>	<b>Chemical analyses of the feed slags used for process development phase 1</b>
<b>Appendix IV</b>	<b>Results of the process development phase 1 roast investigation</b>
<b>Appendix V</b>	<b>Results of the process development phase 1 leach investigation</b>
<b>Appendix VI</b>	<b>Titration procedure used to determine the Fe(II), Fe(III) and HCl concentrations of the leach liquors</b>
<b>Appendix VII</b>	<b>Chemical analyses of the feed slags used for process development phase 2</b>
<b>Appendix VIII</b>	<b>List of experiments conducted for process development phase 2</b>
<b>Appendix IX</b>	<b>Phase 2, Series 1 – Logsheets</b>
<b>Appendix X</b>	<b>Phase 2, Series 2 – Logsheets</b>
<b>Appendix XI</b>	<b>Phase 2, Series 3 – Logsheets</b>
<b>Appendix XII</b>	<b>Phase 2, Series 4 – Logsheets</b>
<b>Appendix XIII</b>	<b>Calculation of the gas flow rate necessary for fluidisation</b>
<b>Appendix XIV</b>	<b>Chemical composition profile data</b>
<b>Appendix XV</b>	<b>Reduction leach logsheets</b>
<b>Appendix XVI</b>	<b>Estimation of the oxygen isobars for oxidation and reduction at 850°C</b>
<b>Appendix XVII</b>	<b>Formation of hematite ore ferric pseudobrookite during oxidation</b>
<b>Appendix XVIII</b>	<b>Mössbauer data</b>



## APPENDIX I: Chemical analysis of the feed slags used for the preliminary investigation (wt%)

Reference no.	Sample	TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>
YS3812	Standard slag: As cast	87.7	10.04	0.93	1.16	0.13	0.45	0.13	1.57	-	1.47
YS17	Standard slag: Granulated	72.5	18.27	1.84	0.93	0.99	0.40	0.15	1.16	-	2.11
YS16	High iron slag: Granulated	83.0	10.66	1.90	0.79	0.34	0.46	0.10	1.20	-	1.73
YS3813	Standard slag: Oxidised in the solid state	85.1	10.66	0.91	1.18	0.14	0.45	0.16	1.53	-	1.50
YS13	High iron slag: Oxidised in the solid state	78.3	16.60	1.29	0.67	0.20	0.47	0.11	1.07	-	1.20
-	Standard slag: Oxidised in the molten state	80.1	15.31	1.66	0.82	0.33	0.47	0.11	1.15	-	1.62
-	High iron slag: Oxidised in the molten state	74.6	21.61	1.74	0.69	0.23	0.45	0.10	1.05	-	1.37
-	Standard slag: Phosphate treated	69.5	8.83	0.80	1.64	2.07	0.36	0.12	1.20	7.74	6.62
-	Standard slag: Oxidised and reduced 1	84.3	9.08	0.99	1.04	0.30	0.42	0.13	1.48	-	1.22
-	Standard slag: Oxidised and reduced 2	83.5	9.40	0.95	1.04	0.18	0.40	0.14	1.49	-	1.34

## APPENDIX II: Log sheets for the preliminary investigation experiments

Date:	97/06/10
Test no.	T 01
Temperature (°C)	95
R.p.m.	500
Solution volume (L)	1.0
Solution description	20% HCl
Solid mass (g)	500
Solid description	High iron slag: Granulated
Final volume (L)	0.86
Dry residue mass (g)	467.3
Residue sample no.	PFE 286

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
72.50	18.27	1.84	0.93	0.99	0.40	0.15	1.16	2.11
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
43.41	14.20	1.11	0.49	0.71	0.22	0.10	0.90	0.99

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
79.50	14.66	1.91	0.73	0.13	0.43	0.10	1.06	1.81
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.60	11.40	1.15	0.39	0.09	0.24	0.07	0.82	0.85

### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 281	0.001	0.002	0.001	0.001	0.004	0.001	0.001	0.001	0.001	206
0.5	PFE 282	0.390	13.200	0.340	0.350	2.140	0.017	0.010	0.420	0.290	202
1.0	PFE 283	0.640	14.100	0.360	0.380	2.240	0.022	0.011	0.520	0.280	196
2.0	PFE 284	0.920	14.500	0.370	0.410	2.290	0.026	0.012	0.630	0.260	196
4.0	PFE 285	1.380	15.700	0.420	0.470	2.510	0.034	0.013	0.810	0.240	197
	<b>Accountability</b>	1.030	0.952	1.039	0.907	0.770	1.031	0.645	1.016	0.847	

<b>Date:</b>	97/06/09
<b>Test no.</b>	T 02
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	26% H2SO4
<b>Solid mass (g)</b>	500
<b>Solid description</b>	High iron slag: Granulated
<b>Final volume (L)</b>	0.85
<b>Dry residue mass (g)</b>	463.9
<b>Residue sample no.</b>	PFE 274

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
72.50	18.27	1.84	0.93	0.99	0.40	0.15	1.16	2.11
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
43.41	14.20	1.11	0.49	0.71	0.22	0.10	0.90	0.99

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
79.90	14.41	1.88	0.75	0.13	0.43	0.11	1.07	1.83
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.84	11.20	1.13	0.40	0.09	0.24	0.08	0.83	0.86

**Experimental results**

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	H2SO4
0.0	PFE 269	0.001	0.006	0.001	0.001	0.001	0.001	0.001	0.001	0.001	305
0.5	PFE 270	0.340	7.920	0.300	0.300	0.310	0.013	0.006	0.380	0.280	293
1.0	PFE 271	0.750	10.700	0.350	0.360	0.310	0.020	0.009	0.520	0.280	290
2.0	PFE 272	1.450	13.400	0.400	0.420	0.530	0.029	0.011	0.690	0.260	288
4.0	PFE 273	2.580	16.700	0.490	0.520	0.390	0.044	0.014	0.950	0.240	290
	<b>Accountability</b>	1.033	0.941	1.027	0.936	0.222	1.031	0.703	1.042	0.849	



<b>Date:</b>	97/06/10
<b>Test no.</b>	T 03
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L) *</b>	1.0
<b>Solution description</b>	20% HCl
<b>Solid mass (g)</b>	500
<b>Solid description</b>	Standard slag: Granulated
<b>Final volume (L)</b>	0.87
<b>Dry residue mass (g)</b>	488.4
<b>Residue sample no.</b>	PFE 292

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
83.00	10.66	1.90	0.79	0.34	0.46	0.10	1.20	1.73
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
49.70	8.29	1.14	0.42	0.24	0.26	0.07	0.93	0.81

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
84.90	9.85	1.83	0.75	0.17	0.45	0.08	1.08	1.61
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
50.84	7.66	1.10	0.40	0.12	0.25	0.05	0.84	0.75

**Experimental results**

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 287	0.001	0.004	0.001	0.001	0.003	0.001	0.001	0.001	0.001	207
0.5	PFE 288	0.410	2.160	0.120	0.091	0.440	0.015	0.001	0.250	0.043	208
1.0	PFE 289	0.760	2.540	0.150	0.120	0.480	0.021	0.001	0.340	0.033	207
2.0	PFE 290	1.290	3.130	0.210	0.150	0.560	0.031	0.001	0.490	0.027	204
4.0	PFE 291	1.900	3.650	0.260	0.190	0.640	0.043	0.002	0.660	0.019	206
	<b>Accountability</b>	1.006	0.983	0.980	1.017	0.962	0.974	0.717	1.010	0.910	

Date:	97/06/09
Test no.	T 04
Temperature (°C)	95
R.p.m.	500
Solution volume (L)	1.0
Solution description	26% H2SO4
Solid mass (g)	500
Solid description	Standard slag: Granulated
Final volume (L)	0.85
Dry residue mass (g)	491.7
Residue sample no.	PFE 280

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
83.00	10.66	1.90	0.79	0.34	0.46	0.10	1.20	1.73
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
49.70	8.29	1.14	0.42	0.24	0.26	0.07	0.93	0.81

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
84.10	10.05	1.90	0.73	0.19	0.45	0.08	1.09	1.60
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
50.36	7.81	1.14	0.39	0.14	0.25	0.05	0.84	0.75

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	H2SO4
0.0	PFE 275	0.001	0.013	0.001	0.001	0.001	0.001	0.001	0.001	0.001	341
0.5	PFE 276	0.015	0.250	0.036	0.024	0.200	0.001	0.001	0.025	0.034	340
1.0	PFE 277	0.032	0.300	0.041	0.028	0.200	0.001	0.001	0.033	0.033	336
2.0	PFE 278	0.069	0.410	0.046	0.031	0.220	0.002	0.001	0.051	0.034	337
4.0	PFE 279	0.170	0.620	0.061	0.044	0.270	0.004	0.001	0.089	0.040	337
	<b>Accountability</b>	0.997	0.939	0.993	0.927	0.748	0.964	0.787	0.910	0.918	

Date:	97/06/11
Test no.	T 05
Temperature (°C)	95
R.p.m.	500
Solution volume (L)	1.0
Solution description	20% HCl
Solid mass (g)	500
Solid description	Standard slag: As-cast
Final volume (L)	0.88
Dry residue mass (g)	494.9
Residue sample no.	PFE 310

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.70	10.05	0.93	1.16	0.13	0.45	0.13	1.57	1.47
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.51	7.81	0.56	0.61	0.09	0.25	0.09	1.22	0.69

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
88.40	9.08	0.98	1.08	0.10	0.44	0.11	1.56	1.26
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.93	7.06	0.59	0.57	0.07	0.25	0.08	1.21	0.59

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 305	0.001	0.004	0.001	0.001	0.003	0.001	0.001	0.001	0.001	199
0.5	PFE 306	0.400	1.860	0.008	0.031	0.022	0.001	0.001	0.070	0.014	197
1.0	PFE 307	0.520	2.000	0.010	0.044	0.028	0.002	0.001	0.080	0.018	196
2.0	PFE 308	0.690	2.140	0.013	0.055	0.037	0.002	0.001	0.095	0.023	197
4.0	PFE 309	0.920	2.360	0.017	0.068	0.051	0.004	0.001	0.118	0.026	198
	<b>Accountability</b>	1.001	0.951	1.048	0.942	0.857	0.970	0.838	1.001	0.855	



<b>Date:</b>	97/06/11
<b>Test no.</b>	T 06
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	26% H2SO4
<b>Solid mass (g)</b>	500
<b>Solid description</b>	Standard slag: As-cast
<b>Final volume (L)</b>	0.87
<b>Dry residue mass (g)</b>	492.3
<b>Residue sample no.</b>	PFE 322

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
87.70	10.05	0.93	1.16	0.13	0.45	0.13	1.57	1.47
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.51	7.81	0.56	0.61	0.09	0.25	0.09	1.22	0.69

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
88.00	8.95	0.97	1.09	0.10	0.44	0.11	1.55	1.26
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.69	6.96	0.58	0.58	0.07	0.25	0.08	1.20	0.59

**Experimental results**

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	H2SO4
0.0	PFE 317	0.001	0.016	0.001	0.001	0.001	0.001	0.001	0.001	0.001	305
0.5	PFE 318	0.850	1.780	0.012	0.029	0.021	0.004	0.001	0.087	0.017	302
1.0	PFE 319	1.090	1.930	0.015	0.042	0.028	0.005	0.001	0.100	0.023	302
2.0	PFE 320	1.420	2.140	0.020	0.060	0.040	0.007	0.001	0.120	0.034	303
4.0	PFE 321	1.790	2.460	0.026	0.076	0.053	0.009	0.002	0.150	0.045	302
	<b>Accountability</b>	0.994	0.935	1.035	0.947	0.859	0.968	0.834	0.994	0.856	

Date:	97/06/11
Test no.	T 07
Temperature (°C)	95
R.p.m.	500
Solution volume (L)	1.0
Solution description	20% HCl
Solid mass (g)	500
Solid description	Standard slag: Oxidised in the solid state
Final volume (L)	0.87
Dry residue mass (g)	487.7
Residue sample no.	PFE 316

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.10	10.66	0.91	1.18	0.14	0.45	0.16	1.53	1.50
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
50.96	8.29	0.55	0.62	0.10	0.25	0.11	1.19	0.70

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.50	8.27	0.97	1.01	0.09	0.43	0.12	1.43	1.26
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.40	6.43	0.58	0.53	0.06	0.24	0.08	1.11	0.59

**Experimental results**

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 311	0.001	0.001	0.001	0.001	0.004	0.001	0.001	0.001	0.001	222
0.5	PFE 312	0.310	4.240	0.028	0.085	0.100	0.007	0.003	0.260	0.019	218
1.0	PFE 313	0.540	4.800	0.039	0.120	0.110	0.009	0.005	0.320	0.023	218
2.0	PFE 314	0.940	5.400	0.058	0.160	0.130	0.012	0.008	0.410	0.027	218
4.0	PFE 315	1.510	6.230	0.085	0.210	0.150	0.016	0.011	0.510	0.034	216
	<b>Accountability</b>	1.008	0.894	1.067	0.895	0.895	0.943	0.748	0.990	0.828	

Date:	97/06/11
Test no.	T 08
Temperature (°C)	95
R.p.m.	500
Solution volume (L)	1.0
Solution description	26%H <sub>2</sub> SO <sub>4</sub>
Solid mass (g)	500
Solid description	Standard slag: Oxidised in the solid state
Final volume (L)	0.87
Dry residue mass (g)	491.5
Residue sample no.	PFE 328

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.10	13.71	0.91	1.18	0.14	0.45	0.16	1.53	1.50
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
50.96	10.66	0.55	0.62	0.10	0.25	0.11	1.19	0.70

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.70	8.84	0.94	1.06	0.12	0.42	0.14	1.48	1.30
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.92	6.87	0.57	0.56	0.09	0.24	0.10	1.15	0.61

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	H <sub>2</sub> SO <sub>4</sub>
0.0	PFE 323	0.001	0.004	0.001	0.001	0.001	0.001	0.001	0.001	0.001	344
0.5	PFE 324	0.250	2.890	0.020	0.067	0.094	0.006	0.001	0.190	0.029	339
1.0	PFE 325	0.340	3.250	0.025	0.085	0.110	0.007	0.002	0.220	0.034	342
2.0	PFE 326	0.460	3.510	0.031	0.100	0.120	0.008	0.003	0.250	0.038	341
4.0	PFE 327	0.670	4.010	0.042	0.130	0.140	0.010	0.004	0.300	0.047	339
	<b>Accountability</b>	1.004	0.703	1.036	0.919	1.138	0.940	0.903	0.999	0.867	



<b>Date:</b>	97/06/24
<b>Test no.</b>	T 09
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	20%HCl
<b>Solid mass (g)</b>	500
<b>Solid description</b>	High iron slag Oxidised in the solid state
<b>Final volume (L)</b>	0.86
<b>Dry residue mass (g)</b>	485.2
<b>Residue sample no.</b>	PFE 338

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
78.30	16.59	1.29	0.67	0.20	0.47	0.11	1.07	1.20
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
46.89	12.90	0.78	0.35	0.14	0.26	0.08	0.83	0.56

%								
TiO <sub>2</sub>	Feo	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
80.10	15.57	1.32	0.67	0.19	0.48	0.10	1.04	1.32
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.96	12.10	0.80	0.35	0.14	0.27	0.07	0.81	0.62

**Experimental results**

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 333	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	217
0.5	PFE 334	0.500	5.130	0.030	0.050	0.050	0.010	0.001	0.110	0.014	217
1.0	PFE 335	0.850	6.070	0.050	0.070	0.080	0.014	0.001	0.160	0.028	217
2.0	PFE 336	1.280	6.640	0.060	0.080	0.110	0.017	0.003	0.200	0.041	217
4.0	PFE 337	2.030	7.900	0.090	0.110	0.180	0.023	0.005	0.280	0.046	217
	<b>Accountability</b>	1.000	1.021	1.013	1.025	1.144	1.006	0.891	1.003	1.082	

<b>Date:</b>	97/06/24
<b>Test no.</b>	T 10
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	26% H2SO4
<b>Solid mass (g)</b>	500
<b>Solid description</b>	High iron slag Oxidised in the solid state
<b>Final volume (L)</b>	0.87
<b>Dry residue mass (g)</b>	489.7
<b>Residue sample no.</b>	PFE 344

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
78.30	16.59	1.29	0.67	0.20	0.47	0.11	1.07	1.20
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
46.89	12.90	0.78	0.35	0.14	0.26	0.08	0.83	0.56

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
79.40	15.69	1.30	0.66	0.18	0.48	0.10	1.05	1.22
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.54	12.20	0.78	0.35	0.13	0.27	0.07	0.81	0.57

**Experimental results**

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	H2SO4
0.0	PFE 339	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	502
0.5	PFE 340	0.350	3.450	0.018	0.033	0.035	0.009	0.001	0.060	0.003	502
1.0	PFE 341	0.520	4.030	0.026	0.043	0.047	0.011	0.001	0.079	0.008	502
2.0	PFE 342	0.790	4.610	0.037	0.056	0.066	0.014	0.001	0.110	0.015	502
4.0	PFE 343	1.330	5.580	0.062	0.077	0.120	0.019	0.003	0.160	0.019	501
	<b>Accountability</b>	0.998	1.005	1.001	1.004	1.030	1.013	0.894	0.996	1.001	

<b>Date:</b>	97/06/25
<b>Test no.</b>	T 11
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	20%HCl
<b>Solid mass (g)</b>	500
<b>Solid description</b>	Standard slag: Oxidised in the molten state
<b>Final volume (L)</b>	0.87
<b>Dry residue mass (g)</b>	489.7
<b>Residue sample no.</b>	PFE 344

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
80.10	15.31	1.66	0.82	0.33	0.47	0.11	1.15	1.62
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.96	11.90	1.00	0.43	0.24	0.26	0.08	0.89	0.76

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
79.40	15.69	1.30	0.66	0.18	0.48	0.10	1.05	1.22
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.54	12.20	0.78	0.35	0.13	0.27	0.07	0.81	0.57

**Experimental results**

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 339	0.003	0.001	0.001	0.001	0.007	0.001	0.001	0.001	0.001	502
0.5	PFE 340	0.450	2.470	0.047	0.024	0.220	0.002	0.002	0.060	0.034	502
1.0	PFE 341	0.660	2.650	0.051	0.027	0.240	0.003	0.002	0.080	0.035	502
2.0	PFE 342	1.050	2.920	0.060	0.031	0.260	0.005	0.002	0.120	0.036	502
4.0	PFE 343	1.680	3.550	0.075	0.037	0.320	0.008	0.003	0.190	0.034	501
	<b>Accountability</b>	0.977	1.059	0.781	0.804	0.778	1.005	0.895	0.932	0.746	



<b>Date:</b>	97/06/25
<b>Test no.</b>	T 12
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	26% H2SO4
<b>Solid mass (g)</b>	500
<b>Solid description</b>	Standard slag: Oxidised in the molten state
<b>Final volume (L)</b>	0.86
<b>Dry residue mass (g)</b>	488.2
<b>Residue sample no.</b>	PFE 365

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
80.10	15.31	1.66	0.82	0.33	0.47	0.11	1.15	1.62
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.96	11.90	1.00	0.43	0.24	0.26	0.08	0.89	0.76

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
79.90	14.15	1.64	0.79	0.26	0.46	0.10	1.12	1.67
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
47.84	11.00	0.99	0.42	0.19	0.26	0.07	0.87	0.78

**Experimental results**

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	H2SO4
0.0	PFE 360	0.002	0.002	0.001	0.001	0.001	0.001	0.001	0.001	0.001	515
0.5	PFE 361	0.940	2.500	0.062	0.026	0.120	0.007	0.003	0.071	0.006	516
1.0	PFE 362	1.250	2.720	0.074	0.032	0.130	0.009	0.004	0.090	0.009	516
2.0	PFE 363	1.970	3.370	0.100	0.044	0.140	0.014	0.006	0.140	0.013	515
4.0	PFE 364	2.380	3.400	0.110	0.047	0.150	0.016	0.006	0.160	0.011	515
	<b>Accountability</b>	0.983	0.955	0.984	0.960	0.885	0.966	0.900	0.983	1.009	

<b>Date:</b>	97/06/25
<b>Test no.</b>	T 13
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	20%HCl
<b>Solid mass (g)</b>	500
<b>Solid description</b>	High iron slag: Oxidised in the molten state
<b>Final volume (L)</b>	0.88
<b>Dry residue mass (g)</b>	487.9
<b>Residue sample no.</b>	PFE 359

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.60	21.61	1.74	0.69	0.23	0.45	0.10	1.05	1.37
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
44.67	16.80	1.05	0.37	0.16	0.25	0.07	0.81	0.64

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.30	20.84	1.75	0.70	0.21	0.45	0.10	1.03	1.48
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
44.49	16.20	1.05	0.37	0.15	0.25	0.07	0.80	0.69

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 354	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	204
0.5	PFE 355	0.630	1.950	0.022	0.013	0.079	0.002	0.001	0.041	0.013	204
1.0	PFE 356	1.060	2.490	0.029	0.017	0.092	0.003	0.001	0.066	0.020	204
2.0	PFE 357	1.810	3.330	0.041	0.019	0.110	0.006	0.002	0.110	0.025	204
4.0	PFE 358	2.900	4.620	0.061	0.025	0.160	0.010	0.003	0.170	0.039	203
	<b>Accountability</b>	0.984	0.991	0.992	1.002	1.068	0.982	0.980	0.995	1.065	

<b>Date:</b>	97/06/25
<b>Test no.</b>	T 14
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	26% H2SO4
<b>Solid mass (g)</b>	500
<b>Solid description</b>	High iron slag: Oxidised in the molten state
<b>Final volume (L)</b>	0.86
<b>Dry residue mass (g)</b>	487.9
<b>Residue sample no.</b>	PFE 371

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
74.60	21.61	1.74	0.69	0.23	0.45	0.10	1.05	1.37
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
44.67	16.80	1.05	0.37	0.16	0.25	0.07	0.81	0.64

%								
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2
74.40	20.97	1.75	0.71	0.20	0.45	0.10	1.03	1.42
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
44.55	16.30	1.05	0.38	0.14	0.25	0.07	0.80	0.66

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	H2SO4
0.0	PFE 366	0.001	0.002	0.001	0.001	0.001	0.001	0.0010	0.001	0.001	491
0.5	PFE 367	0.770	1.710	0.026	0.015	0.060	0.003	0.0004	0.040	0.002	483
1.0	PFE 368	1.260	2.340	0.038	0.016	0.072	0.006	0.0009	0.066	0.005	483
2.0	PFE 369	2.000	3.030	0.054	0.021	0.083	0.009	0.0020	0.100	0.009	482
4.0	PFE 370	3.180	4.270	0.078	0.027	0.100	0.014	0.0029	0.160	0.013	479
	<b>Accountability</b>	0.986	0.992	0.994	1.017	0.957	0.985	0.980	0.992	1.015	



Date:	97/06/26
Test no.	T 15
Temperature (°C)	95
R.p.m.	500
Solution volume (L)	1.0
Solution description	20% HCl
Solid mass (g)	500
Solid description	Phosphate slag 10% FeO, 5% CaO and 10% P2O5
Final volume (L)	0.82
Dry residue mass (g)	416.5
Residue sample no.	PFE 377

%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	P2O5
69.50	8.82	0.80	1.64	2.07	0.36	0.12	1.20	6.62	7.74
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P
41.62	6.86	0.48	0.87	1.48	0.20	0.08	0.93	3.09	3.38

%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	P2O5
83.40	6.03	0.09	0.45	0.04	0.38	0.11	0.08	7.54	0.71
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P
49.94	4.69	0.05	0.24	0.03	0.21	0.08	0.06	3.53	0.31

#### Experimental results

Time(h)	Sample no.	g/l										
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P	HCl
0.0	PFE 372	0.005	0.008	0.002	0.001	0.018	0.001	0.001	0.001	0.001	0.000	200
0.5	PFE 373	0.150	9.880	2.160	3.180	6.940	0.083	0.006	4.380	0.190	14.497	175
1.0	PFE 374	0.073	10.780	2.260	3.370	7.280	0.089	0.007	4.590	0.180	15.362	172
2.0	PFE 375	0.057	11.510	2.230	3.340	7.180	0.091	0.008	4.530	0.150	15.068	171
4.0	PFE 376	0.057	14.090	2.430	3.660	7.810	0.100	0.012	4.930	0.110	14.948	170
	<b>Accountability</b>	1.000	0.925	0.975	0.965	0.937	0.965	0.787	0.983	0.955	0.855	

<b>Date:</b>	97/06/26
<b>Test no.</b>	T 16
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	26% H2SO4
<b>Solid mass (g)</b>	500
<b>Solid description</b>	Phosphate slag 10% FeO, 5% CaO and 10% P2O5
<b>Final volume (L)</b>	0.87
<b>Dry residue mass (g)</b>	431.3
<b>Residue sample no.</b>	PFE 383

%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	P2O5
69.50	8.82	0.80	1.64	2.07	0.36	0.12	1.20	6.62	7.74
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P
41.62	6.86	0.48	0.87	1.48	0.20	0.08	0.93	3.09	3.38

%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	P2O5
82.40	6.15	0.07	0.44	1.16	0.37	0.11	0.08	7.34	0.62
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P
49.34	4.78	0.04	0.23	0.83	0.21	0.08	0.06	3.43	0.27

**Experimental results**

		g/l										
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P	H2SO4
0.0	PFE 378	0.001	0.016	0.002	0.0005	0.010	0.001	0.001	0.004	0.001	0.000	482
0.5	PFE 379	0.370	10.970	2.440	3.670	0.220	0.098	0.007	4.880	0.069	17.103	443
1.0	PFE 380	0.430	11.330	2.440	3.660	0.270	0.098	0.008	4.890	0.051	16.991	440
2.0	PFE 381	0.370	9.290	1.940	2.900	0.420	0.078	0.006	3.880	0.015	13.248	437
4.0	PFE 382	0.320	12.350	2.430	3.650	0.210	0.100	0.010	4.900	0.006	16.554	436
	<b>Accountability</b>	1.024	0.932	1.009	1.010	0.510	0.977	0.811	1.032	0.957	0.977	

<b>Date:</b>	97/06/26
<b>Test no.</b>	T 17
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	500
<b>Solution volume (L)</b>	1.0
<b>Solution description</b>	18% H3PO4
<b>Solid mass (g)</b>	500
<b>Solid description</b>	Phosphate slag 10% FeO, 5% CaO and 10% P2O5
<b>Final volume (L)</b>	0.84
<b>Dry residue mass (g)</b>	423.9
<b>Residue sample no.</b>	PFE 389

%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	P2O5
69.50	8.82	0.80	1.64	2.07	0.36	0.12	1.20	6.62	7.74
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P
41.62	6.86	0.48	0.87	1.48	0.20	0.08	0.93	3.09	3.38

%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	P2O5
82.30	6.51	0.09	0.55	0.13	0.38	0.11	0.14	7.21	1.51
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P
49.28	5.06	0.05	0.29	0.09	0.21	0.08	0.11	3.37	0.66

**Experimental results**

		g/l										
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	P	H3PO4
0.0	PFE 384	0.001	0.001	0.001	0.001	0.009	0.001	0.001	0.001	0.001	99.844	322
0.5	PFE 385	0.320	8.790	1.980	2.830	6.340	0.073	0.008	4.010	0.270	116.050	262
1.0	PFE 386	0.280	9.780	2.080	2.940	6.650	0.078	0.009	4.190	0.250	115.200	260
2.0	PFE 387	0.250	8.640	2.170	3.040	6.930	0.082	0.009	4.350	0.240	115.240	256
4.0	PFE 388	0.220	9.420	2.510	3.460	8.000	0.096	0.012	5.010	0.230	128.050	254
	<b>Accountability</b>	1.005	0.872	1.022	0.994	1.014	0.978	0.801	1.057	0.937	1.022	



<b>Date:</b>	97/07/04
<b>Test no.</b>	T 19
<b>Temperature (°C)</b>	107
<b>R.p.m.</b>	
<b>Solution volume (L)</b>	0.5
<b>Solution description</b>	20% HCl
<b>Solid mass (g)</b>	250
<b>Solid description</b>	Standard slag - 850°C, 2h air oxidation, reduction in CO/(CO+CO <sub>2</sub> ) = 1 for 20 minutes
<b>Final volume (L)</b>	0.42
<b>Dry residue mass (g)</b>	221.9
<b>Residue sample no.</b>	PFE 403

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
84.30	9.08	0.99	1.04	0.30	0.42	0.13	1.48	1.22
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
50.48	7.06	0.60	0.55	0.21	0.24	0.09	1.15	0.57

%								
	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
91.60	2.84	0.43	0.70	0.10	0.34	0.10	0.48	1.31
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
54.85	2.21	0.26	0.37	0.07	0.19	0.07	0.37	0.61

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 397	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.0	PFE 398	4.065	9.000	0.660	0.209	0.770	0.097	0.041	1.365	0.037	
2.0	PFE 399	2.290	14.800	1.035	0.369	0.810	0.164	0.050	2.355	0.035	
4.0	PFE 400	1.325	22.400	1.595	0.775	0.865	0.264	0.090	3.680	0.029	
6.0	PFE 401	0.995	26.750	1.965	1.140	0.875	0.333	0.124	4.445	0.027	212.5
	<b>Accountability</b>	0.968	0.941	0.961	0.955	1.027	0.965	0.925	0.965	0.962	

<b>Date:</b>	97/07/07
<b>Test no.</b>	T 20
<b>Temperature (°C)</b>	107
<b>R.p.m.</b>	
<b>Solution volume (L)</b>	0.5
<b>Solution description</b>	20% HCl
<b>Solid mass (g)</b>	250
<b>Solid description</b>	
Standard slag - 850°C, 2h air oxidation, reduction in CO/(CO+CO <sub>2</sub> ) = 0.67 for 20 minutes	
<b>Final volume (L)</b>	0.46
<b>Dry residue mass (g)</b>	226.5
<b>Residue sample no.</b>	PFE 410

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
83.50	9.40	0.95	1.04	0.18	0.40	0.14	1.49	1.34
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
50.00	7.31	0.57	0.55	0.13	0.22	0.10	1.16	0.63

%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
92.10	2.86	0.41	0.75	0.08	0.35	0.12	0.46	1.35
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
55.15	2.22	0.25	0.40	0.06	0.20	0.08	0.36	0.63

**Experimental results**

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0	PFE 405	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.0	PFE 406	3.772	10.377	0.633	0.186	0.395	0.080	0.025	1.577	0.036	
2.0	PFE 407	2.133	16.590	1.044	0.334	0.437	0.141	0.047	2.658	0.034	
4.0	PFE 408	1.289	23.598	1.559	0.696	0.479	0.229	0.087	3.916	0.033	
6.0	PFE 409	0.957	28.073	1.938	1.047	0.508	0.295	0.119	4.707	0.032	173.2
	<b>Accountability</b>	1.003	1.009	1.037	1.012	1.170	1.042	1.012	1.058	0.923	

### APPENDIX III: Chemical composition of the feed slags used for process development phase 1

Cast	PFE	FeO(tot)	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
K6-S-26	418	8.58	83.00	1.42	2.42	0.35	2.65	1.49	0.30	0.46
K6-S-02	436	21.87	71.50	0.76	1.87	0.23	1.52	1.17	0.11	0.43
K6-S-29	437	7.69	86.40	1.12	2.06	0.46	1.60	1.33	0.17	0.45
K6-S-08	467	15.31	79.50	0.71	1.45	0.23	1.85	1.17	0.11	0.46
K6-S-35	469	9.38	87.96	0.93	1.59	0.32	1.17	1.30	0.12	0.45



## APPENDIX IV: Results of the process development phase 1 roast investigation

Chemical composition of slag K6-S-29 (PFE437) which had been oxidised at different temperatures and times

K6-S-29 (PFE437)	PFE	Roast conditions		FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R1/1R2-0	696		1 h	8.86	84.00	1.05	1.93	0.32	1.50	1.29	0.16	0.44
R2/1R2-0	704	950°C	2 h									
R3/1R2-0	712		4 h									
R4/1R2-0	720		1 h	8.47	85.40	1.12	1.90	0.37	1.56	1.30	0.18	0.43
R5/1R2-0	728	850°C	2 h									
R6/1R2-0	736		4 h	8.62	83.10	1.17	2.14	0.40	1.49	1.26	0.17	0.43
R7/1R2-0	744	850°C	3 h	8.39	83.80	1.12	1.97	0.36	1.52	1.31	0.19	0.42
R8/1R2-0	752		4 h	8.07	83.90	1.11	1.98	0.38	1.51	1.30	0.18	0.42
R9/1R2-0	760	750°C	4 h									

Chemical composition of slag K6-S-29 (PFE437) which had been oxidised and reduced at different temperatures and times

K6-S-29 (PFE437)	PFE	Roast conditions		FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R1/1R2-0	696	950°C	0 min	8.86	84.00	1.05	1.93	0.32	1.50	1.29	0.16	0.44
R1/1R2-10	698	ox. 1 h,	10 min	8.90	82.80	1.06	1.97	0.31	1.49	1.26	0.17	0.43
R1/1R2-20	700	900°C	20 min	8.63	83.50	1.07	2.03	0.32	1.47	1.25	0.17	0.43
R1/1R2-40	702	Red. for:	40 min	8.50	83.95	1.19	2.23	0.38	1.47	1.25	0.17	0.43
R2/1R2-0	704	950°C	0 min									
R2/1R2-10	706	ox. 2 h,	10 min									
R2/1R2-20	708	900°C	20 min									
R2/1R2-40	710	Red. for:	40 min									
R3/1R2-0	712	950°C	0 min									
R3/1R2-10	714	ox. 4 h,	10 min									
R3/1R2-20	716	900°C	20 min									
R3/1R2-40	718	Red. for:	40 min									
R4/1R2-0	720	850°C	0 min	8.47	85.40	1.12	1.90	0.37	1.56	1.30	0.18	0.43
R4/1R2-10	722	ox. 1 h,	10 min	9.57	78.80	1.20	2.09	0.40	1.52	1.29	0.21	0.42
R4/1R2-20	724	800°C	20 min	8.88	79.90	1.10	1.91	0.36	1.54	1.30	0.20	0.43
R4/1R2-40	726	Red. for:	40 min	9.05	79.80	1.15	1.89	0.38	1.52	1.27	0.20	0.42
R5/1R2-0	728	850°C	0 min									
R5/1R2-10	730	ox. 2 h,	10 min									
R5/1R2-20	732	800°C	20 min									
R5/1R2-40	734	Red. for:	40 min									
R6/1R2-0	736	850°C	0 min	8.62	83.10	1.17	2.14	0.40	1.49	1.26	0.17	0.43
R6/1R2-10	738	ox. 4 h,	10 min	8.65	83.50	1.17	2.16	0.42	1.50	1.26	0.16	0.43
R6/1R2-20	740	800°C	20 min	8.71	82.70	1.14	2.32	0.45	1.48	1.25	0.16	0.41
R6/1R2-40	742	Red. for:	40 min	7.23	85.30	0.80	2.71	0.21	1.08	0.83	0.17	0.39
R7/1R2-0	744	800°C	0 min	8.39	83.80	1.12	1.97	0.36	1.52	1.31	0.19	0.42
R7/1R2-10	746	ox. 3 h,	10 min	8.31	84.30	1.11	1.94	0.39	1.52	1.31	0.18	0.42
R7/1R2-20	748	750°C	20 min	8.18	84.72	1.13	1.99	0.41	1.52	1.30	0.18	0.42
R7/1R2-40	750	Red. for:	40 min	8.67	83.20	1.12	1.97	0.41	1.51	1.30	0.19	0.42
R8/1R2-0	752	800°C	0 min	8.07	83.90	1.11	1.98	0.38	1.51	1.30	0.18	0.42
R8/1R2-10	754	ox. 4 h,	10 min	8.35	83.90	1.11	1.96	0.39	1.52	1.31	0.19	0.42
R8/1R2-20	756	750°C	20 min	9.31	81.80	1.19	2.35	0.43	1.48	1.29	0.25	0.31
R8/1R2-40	758	Red. for:	40 min	8.40	82.40	1.22	2.33	0.42	1.50	1.28	0.19	0.42
R9/1R2-0	760	750°C	0 min									
R9/1R2-10	762	ox. 3 h, 700°C red. for:	10 min									

**Chemical composition of slag K6-S-29 (PFE437) had been roasted at different temperatures and times and then leached for 5 h**

Roast details				Leach residue											Leach solution										
K6-S-29 (PFE437)	PFE	Roast conditions		FeO (tot)	TiO <sub>2</sub> %	Al <sub>2</sub> O <sub>3</sub> %	SiO <sub>2</sub> %	CaO %	MgO %	MnO %	Cr <sub>2</sub> O <sub>3</sub> %	V <sub>2</sub> O <sub>5</sub> %	Sand g	Fines g	Fines %	Fe g/L	Fe g/L	Ti mg/L	Al Mg/L	Si mg/L	Ca mg/L	Mg mg/L	Mn mg/L	Cr mg/L	V mg/L
R1/1R2-0/1L	697	950°C	0 min	8.03	85.50	0.97	1.86	0.17	1.50	1.20	0.19	0.43	195.1	0.1	0.1	4	5								
R1/1R2-10/1L	699	ox. 1 h,	10 min	6.70	86.60	0.92	1.96	0.19	1.36	0.99	0.19	0.42	188.8	2.2	1.2	12	1								
R1/1R2-20/1L	701	900°C	20 min	6.03	87.10	0.99	2.03	0.19	1.35	0.93	0.18	0.41	190.1	0.2	0.1	17	1								
R1/1R2-40/1L	703	Red. for:	40 min	6.41	87.00	1.03	2.34	0.21	1.32	0.86	0.19	0.41	186.4	1.0	0.5	19	1								
R2/1R2-0/1L	705	950°C	0 min	7.89	85.40	1.02	1.85	0.14	1.53	1.22	0.19	0.43	195.1	0.0	0.0	0	3	1540	520	93	1220	112	340	12	13
R2/1R2-10/1L	707	ox. 2 h,	10 min	6.33	87.90	0.98	2.09	0.15	1.28	0.88	0.19	0.42	190.0	0.0	0.0	14	1	2520	860	16	1160	1180	2230	88	120
R2/1R2-20/1L	709	900°C	20 min	5.79	88.00	1.03	2.00	0.18	1.30	0.85	0.18	0.41	187.4	0.0	0.0	18	0	3910	690	56	1210	1280	2630	77	140
R2/1R2-40/1L	711	Red. for:	40 min	5.67	87.60	1.04	1.99	0.17	1.28	0.82	0.18	0.41	187.3	0.0	0.0	18	0	3400	580	65	1130	1270	2640	72	145
R3/1R2-0/1L	713	950°C	0 min	8.31	84.60	1.05	2.07	0.16	1.52	1.21	0.21	0.42	194.5	0.0	0.0	0	4								
R3/1R2-10/1L	715	ox. 4 h,	10 min	6.10	87.00	0.99	2.14	0.17	1.28	0.86	0.18	0.40	188.4	0.5	0.3	17	0								
R3/1R2-20/1L	717	900°C	20 min	6.46	87.40	1.05	2.10	0.17	1.34	0.90	0.18	0.41	188.3	0.1	0.1	17	0								
R3/1R2-40/1L	719	Red. for:	40 min	6.78	87.30	1.09	2.24	0.18	1.35	0.91	0.20	0.41	184.7	0.0	0.0	18	0								
R4/1R2-0/1L	721	850°C	0 min	7.09	86.80	0.97	1.92	0.21	1.47	1.19	0.19	0.42	192.2	0.0	0.0	5	10								
R4/1R2-10/1L	723	ox. 1 h,	10 min	4.01	90.10	0.74	1.99	0.21	1.13	0.87	0.16	0.38	185.5	0.5	0.3	21	6								
R4/1R2-20/1L	725	800°C	20 min	3.74	90.70	0.75	1.93	0.22	1.07	0.80	0.16	0.38	184.2	0.5	0.3	26	1								
R4/1R2-40/1L	727	Red. for:	40 min	4.00	90.20	0.87	2.01	0.22	1.09	0.77	0.18	0.39	183.8	0.2	0.1	27	0								
R5/1R2-0/1L	729	850°C	0 min	6.96	86.70	0.98	1.89	0.18	1.50	1.20	0.16	0.43	192.5	0.0	0.0	1	10	3610	750	42	965	295	600	28	40
R5/1R2-10/1L	731	ox. 2 h,	10 min	3.69	91.10	0.68	2.12	0.19	0.96	0.70	0.16	0.37	181.8	0.7	0.4	23	5	635	1900	55	1030	2370	3260	180	290
R5/1R2-20/1L	733	800°C	20 min	2.96	92.40	0.70	2.06	0.19	0.91	0.63	0.14	0.36	182.8	0.2	0.1	28	2	665	1880	27	1030	2680	3720	195	325
R5/1R2-40/1L	735	Red. for:	40 min	3.15	91.50	0.74	2.09	0.19	0.93	0.61	0.15	0.36	180.6	0.5	0.3	30	1	650	1770	63	1100	2470	3640	175	305
R6/1R2-0/1L	737	850°C	0 min	7.87	85.70	1.00	2.04	0.19	1.51	1.21	0.20	0.43	191.0	0.0	0.0	1	7	2900	940	45	1260	310	540	22	31
R6/1R2-10/1L	739	ox. 4 h,	10 min	4.66	89.00	0.75	2.25	0.18	1.11	0.81	0.18	0.40	182.4	1.5	0.8	17	7	900	1900	18	1310	1940	2640	140	220
R6/1R2-20/1L	741	800°C	20 min	3.45	91.00	0.66	2.15	0.19	0.96	0.66	0.14	0.36	178.0	1.0	0.6	23	7	815	2240	19	1300	2490	3390	190	290
R6/1R2-40/1L	743	Red. for:	40 min	8.66	85.40	2.23	2.95	0.22	0.76	1.44	0.10	0.42	178.8	1.3	0.7	25	3	750	1940	16	1290	2420	3400	180	285
R7/1R2-0/1L	745	800°C	0 min	6.47	86.60	0.94	2.03	0.21	1.40	1.18	0.18	0.42	190.2	0.0	0.0	3	12								
R7/1R2-10/1L	747	ox. 3 h,	10 min	3.71	89.70	0.76	2.09	0.20	1.10	0.90	0.16	0.39	189.5	0.1	0.1	17	6								
R7/1R2-20/1L	749	750°C	20 min	4.08	88.60	0.80	2.06	0.22	1.17	0.96	0.17	0.40	183.0	0.6	0.3	19	6								
R7/1R2-40/1L	751	Red. for:	40 min	3.77	89.40	0.77	2.11	0.20	1.10	0.88	0.16	0.39	183.5	0.3	0.2	28	5								
R8/1R2-0/1L	753	800°C	0 min	6.07	86.70	0.92	1.96	0.20	1.40	1.19	0.18	0.43	190.8	0.0	0.0	6	11	3420	950	42	970	530	760	24	56
R8/1R2-10/1L	755	ox. 4 h,	10 min	4.61	89.50	0.81	2.04	0.21	1.27	1.07	0.18	0.41	186.8	0.4	0.2	15	2	1230	1350	57	960	1130	1390	61	130
R8/1R2-20/1L	757	750°C	20 min	4.62	89.20	0.78	2.45	0.21	1.20	1.01	0.19	0.39	137.2	0.5	0.4	17	4	1130	1870	14	1170	1420	1700	77	150
R8/1R2-40/1L	759	Red. for:	40 min	4.35	88.90	0.82	2.20	0.21	1.26	1.03	0.18	0.40	184.8	0.2	0.1	17	6	1240	1420	110	1040	1170	1480	60	135
R9/1R2-0/1L	761	750°C	0 min	5.61	87.90	0.93	1.82	0.20	1.39	1.19	0.20	0.42	190.2	0.0	0.0	6	12								
R9/1R2-10/1L	763	ox. 3 h, 700°C red. for:	10 min	5.52	87.40	0.95	2.26	0.22	1.37	1.17	0.20	0.42	189.5	0.1	0.1	11	6								

Leach conditions employed: 200 g roasted slag contacted with 270 mL 20% HCl solution at 106°C.



**Extractions and elemental balances obtained from roast and leach experiments performed on slag K6-S-29 (PFE437) which had been roasted at different temperatures and times and then leached for 5 h**

K6-S-29	Roast conditions			FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R6/1R2-0	850°C ox.	0 min	Extraction (%)	12.8	1.5	18.4	9.0	54.6	3.2	8.3	-12.4	4.5
	4 h.		Balance	103	99	102	92	105	101	99	115	97
R6/1R2-10	800°C.	10 min	Extraction (%)	50.5	2.0	41.1	4.2	60.6	32.0	40.9	-3.4	14.5
	red. for:		Balance	98	98	100	96	98	97	96	121	98
R6/1R2-20		20 min	Extraction (%)	64.6	1.5	48.2	17.1	62.2	42.0	52.7	21.7	21.4
			Balance	95	99	102	83	92	96	95	102	96
R6/1R2-40		40 min	Extraction (%)	-7.9	9.8	-151.0	2.0	5.7	36.6	-56.2	47.0	3.0
			Balance	175	90	313	98	210	114	228	74	115
R8/1R2-0	800°C ox.	0 min	Extraction (%)	28.2	1.4	20.9	5.6	49.8	11.6	12.7	4.60	2.3
	4 h.		Balance	108	100	101	95	98	96	98	98	101
R8/1R2-10	750°C	10 min	Extraction (%)	48.3	0.15	31.7	2.6	49.6	21.8	23.6	11.3	8.6
	red for:		Balance	87	100	99	98	97	95	95	95	99
R8/1R2-20		20 min	Extraction (%)	65.8	24.9	54.9	28.2	66.4	44.2	46.1	47.7	13.4
			Balance	73	75	85	72	85	77	77	58	98
R8/1R2-40		40 min	Extraction (%)	52.1	0.2	37.8	12.7	53.8	22.3	25.6	12.4	11.9
			Balance	96	100	92	89	93	95	95	94	96



## APPENDIX V: Results of the process development phase 1 leach investigation

### A. Details of experiments conducted on PFE418

Chemical composition of the high magnesium slag K6-S-26 (PFE418) which had been oxidised at 850°C for different times

K6-S-26 (PFE418)	PFE	Time (h)	FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R3/0.5h/ox	770	0.5									
R3/1.0h/ox	771	1.0	10.38	81.30	1.34	2.26	0.35	2.62	1.45	0.28	0.46
R3/1.5h/ox	772	1.5									
R3/2.0h/ox	773	2.0	10.27	81.00	1.33	2.23	0.36	2.56	1.41	0.29	0.45
R3/2.5h/ox	774	2.5									
R3/3.0h/ox	775	3.0	10.01	80.00	1.38	2.32	0.37	2.59	1.41	0.28	0.46

Chemical composition of slag K6-S-26 (PFE418) which was oxidised at 850°C for 3 h and reduced at 800°C for 30 min and leached for 5 h

K6-S-26 (PFE418)	PFE	Time (h)	FeO (tot)	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R1/1R2-30	830	0									
R2/1R2-30	831	0									
R3/1R2-30	832	0									
R1/1R2-30/1L-5.0h	809	5	5.51	86.50	0.96	2.43	0.28	2.00	0.98	0.24	0.41
R2/1R2-30/1L-5.0h	810	5	5.49	86.70	0.92	2.44	0.28	1.93	0.95	0.24	0.40
R3/1R2-30/1L-5.0h	811	5	5.26	87.10	0.92	2.46	0.28	1.92	0.93	0.24	0.40

**Chemical composition of slag K6-S-26 (PFE418) which was oxidised at 850°C for 3 h and reduced at 800°C for 30 min and leached for 12 h**

Roast and leach details			Leach residue (%)																						
K6-S-26 (PFE418)	PFE	Time (h)	FeO (tot)	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>	Sand g	Fines g	Fines %	Fe <sup>2+</sup> g/L	Fe <sup>3+</sup> g/L	Ti mg/L	Al mg/L	Si mg/L	Ca mg/L	Mg mg/L	Mn mg/L	Cr mg/L	V mg/L	
R1/1R2-30	830	0																							
R2/1R2-30	831	0																							
R3/1R2-30	832	0																							
R1/1R2-30/1L-0.5h	795	0.5	9.29	81.20	1.29	2.30	0.30	2.50	1.33	0.28	0.44	193.1	0.0	0.0	6	0	2280	210	32	365	180	320	13	20	
R1/1R2-30/1L-1.0h	796	1	8.58	82.40	1.27	2.31	0.30	2.49	1.29	0.29	0.44	191.9	0.0	0.0	7	1	3620	345	35	380	330	500	23	38	
R1/1R2-30/1L-2.0h	797	2	7.64	83.90	1.18	2.37	0.27	2.35	1.19	0.28	0.43	190.0	0.0	0.0	11	2	3260	680	26	450	860	1000	64	89	
R1/1R2-30/1L-4.0h	798	4	6.37	86.00	1.05	2.47	0.28	2.15	1.06	0.26	0.42	184.1	1.0	0.5	16	5	1080	1240	26	460	1790	1760	150	165	
R1/1R2-30/1L-6.0h	799	6	5.22	88.20	0.90	2.56	0.27	1.89	0.92	0.23	0.39	181.1	1.6	0.9	17	6	670	1580	37	520	2410	2190	220	220	
R1/1R2-30/1L-8.0h	800	8	4.76	88.50	0.88	2.56	0.28	1.80	0.87	0.23	0.39	178.6	2.1	1.2	19	6	630	1700	41	530	2900	2500	250	250	
R1/1R2-30/1L-12.0h	801	12	3.83	89.10	0.73	2.55	0.25	1.46	0.71	0.20	0.36	173.1	3.6	2.0	22	10	420	2300	52	610	4160	3360	350	350	

Experimental conditions: 200 g roasted slag contacted with 329 mL 20% HCl solution at 106°C.

## B. Details of experiments conducted on PFE436

Chemical composition of the high iron-containing slag K6-S-02 (PFE436) which had been oxidised at 850°C for different times

K6-S-02 (PFE436)	PFE	Time (h)	FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R3/0.5h/ox	764	0.5									
R3/1.0h/ox	765	1.0	22.51	70.70	0.72	1.46	0.20	1.59	1.10	0.11	0.45
R3/1.5h/ox	766	1.5									
R3/2.0h/ox	767	2.0	22.13	70.70	0.76	1.57	0.22	1.61	1.11	0.12	0.44
R3/2.5h/ox	768	2.5									
R3/3.0h/ox	769	3.0	22.00	70.60	0.77	1.53	0.26	1.59	1.10	0.11	0.44

Chemical composition of slag K6-S-02 (PFE436) which had been oxidised at 850°C for 3 h and reduced at 800°C for 30 min and then leached for different times

K6-S-02 (PFE436)	PFE	Time (h)	FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R2/1R2-30	833	0									
R3/1R2-30	834	0									
R4/1R2-30	835	0									
R1/1R2-30/1L-5.0h	815	5									
R2/1R2-30/1L-5.0h	816	5	9.01	85.80	0.47	1.93	0.14	0.86	0.44	0.09	0.34
R3/1R2-30/1L-5.0h	817A	5	8.95	86.00	0.47	1.88	0.14	0.86	0.43	0.08	0.34
R4/1R2-30/1L-5.0h	817B	5	8.50	86.50	0.46	1.90	0.14	0.82	0.42	0.09	0.33



**Chemical composition of slag K6-S-02 (PFE436) which had been oxidised at 850°C for 3 h and reduced at 800°C for 30 min and then leached for different times**

Roast and leach details	Leach residue																						
	PFE	Time (h)	FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>	Sand g	Fines g	Fines %	Fe <sup>+</sup> g/L	Fe <sup>2+</sup> g/L	Ti mg/L	Al mg/L	Si mg/L	Ca mg/L	Mn mg/L	Cr mg/L	V mg/L
R2/1R2-30/1L-0.5h	788	0.5	19.56	73.70	0.68	1.59	0.18	1.61	1.03	0.13	0.46	187.6	0.0	0.0	9	2	3500	145	40	217	350	19	78
R2/1R2-30/1L-1.0h	789	1	16.85	78.30	0.65	1.73	0.17	1.46	0.87	0.12	0.44	180.5	0.0	0.0	16	3	4880	270	42	250	795	38	155
R2/1R2-30/1L-2.0h	790	2	14.41	81.20	0.67	1.86	0.17	1.29	0.70	0.12	0.42	173.5	0.3	0.2	25	5	2400	450	43	290	1350	65	155
R2/1R2-30/1L-4.0h	791	4	9.53	85.80	0.52	1.97	0.16	0.94	0.47	0.10	0.35	161.8	1.7	1.0	33	8	690	730	43	330	1960	105	275
R2/1R2-30/1L-6.0h	792	6	7.84	87.70	0.43	1.92	0.14	0.76	0.37	0.09	0.33	158.1	2.2	1.4	34	9	455	830	43	340	2110	120	325
R2/1R2-30/1L-8.0h	793	8	7.58	88.10	0.43	1.99	0.14	0.76	0.38	0.09	0.32	157.2	2.2	1.4	35	7	380	890	47	360	2120	125	340
R2/1R2-30/1L-12.0h	794	12	7.32	88.30	0.40	2.05	0.14	0.71	0.36	0.09	0.31	155.2	4.2	2.6	38	13	305	890	42	345	2210	135	365

Leach conditions employed: 200 g roasted slag contacted with 510 ml 20% HCl solution at 106°C.

### C. Details of experiments conducted on PFE467

Chemical composition of slag K6-S-08 (PFE467) which had been oxidised at 850°C for different times

K6-S-08 (PFE467)	PFE	Time (h)	FeO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R3/0.5h/ox	776	0.5								
R3/1.0h/ox	777	1.0	15.44	0.69	1.15	0.24	1.85	1.16	0.10	0.46
R3/1.5h/ox	778	1.5								
R3/2.0h/ox	779	2.0	15.44	0.68	1.21	0.23	1.82	1.13	0.10	0.45
R3/2.5h/ox	780	2.5								
R3/3.0h/ox	781	3.0	15.44	0.63	1.23	0.26	1.61	1.13	0.11	0.45

Chemical composition of slag K6-S-08 (PFE467) which had been oxidised at 850°C for 3 h and reduced for different times and then leached for 5 h

K6-S-08 (PFE467)	PFE	Time (min)	FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R1/1R2-0/1L-5.0h	818	0	14.02	79.90	0.60	1.16	0.09	1.79	1.04	0.10	0.46
R1/1R2-10/1L-5.0h	819	10	9.19	85.50	0.44	1.29	0.11	1.39	0.73	0.09	0.41
R1/1R2-20/1L-5.0h	820	20	7.77	86.80	0.42	1.46	0.10	1.46	0.77	0.09	0.43
R1/1R2-30/1L-5.0h	812	30									
R2/1R2-10/1L-5.0h	822	10	9.91	84.60	0.48	1.33	0.10	1.46	0.77	0.09	0.43
R2/1R2-20/1L-5.0h	821	20									
R2/1R2-20/1L-5.0h	823	20	7.90	87.40	0.42	1.36	0.10	1.23	0.62	0.08	0.40
R2/1R2-30/1L-5.0h	813	30	6.83	88.20	0.40	1.47	0.09	1.10	0.54	0.08	0.38
R3/1R2-0/1L-5.0h	824	0	13.77	80.10	0.60	1.22	0.10	1.76	1.04	0.10	0.45
R3/1R2-10/1L-5.0h	825	10	9.67	85.20	0.47	1.45	0.11	1.45	0.77	0.09	0.41
R3/1R2-20/1L-5.0h	826	20	8.41	86.50	0.44	1.39	0.10	1.29	0.67	0.08	0.39
R3/1R2-30/1L-5.0h	814	30	6.88	88.10	0.42	1.44	0.10	1.12	0.56	0.08	0.38

**Chemical composition of slag K6-S-08 (PFE467) which had been oxidised at 850°C for 3 h and reduced for 30 min at 800°C and then leached for different times**

Roast and leach details			Leach residue																						
K6-S-08 (PFE467)	PFE	Time (h)	FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>	Sand g	Fines g	Fines %	Fe <sup>++</sup> g/L	Fe <sup>+++</sup> g/L	Ti mg/L	Al mg/L	Si mg/L	Ca mg/L	Mg mg/L	Mn mg/L	Cr mg/L	V mg/L	
R1/1R2-30	836	0																							
R2/1R2-30	837	0																							
R3/1R2-30	838	0																							
R1/1R2-30/1L-0.5h	802	0.5	14.02	79.70	0.69	1.34	0.19	1.78	1.04	0.11	0.46	189.8	0.0	0.0	10	0.0	4030	216	46	290	305	460	17	46	
R1/1R2-30/1L-1.0h	803	1	11.75	82.90	0.64	1.34	0.18	1.63	0.91	0.10	0.44	184.7	0.0	0.0	17	2	4700	360	47	360	800	980	36	105	
R1/1R2-30/1L-2.0h	804	2	9.17	85.30	0.53	1.38	0.15	1.39	0.73	0.09	0.41	179.3	0.2	0.1	25	3	1860	620	59	470	1580	1700	66	200	
R1/1R2-30/1L-4.0h	805	4	7.40	87.50	0.44	1.44	0.14	1.17	0.58	0.09	0.38	173.4	0.9	0.5	29	4	720	830	66	530	2080	2130	90	270	
R1/1R2-30/1L-6.0h	806	6	5.96	89.60	0.40	1.43	0.09	1.04	0.52	0.07	0.36	169.9	2.0	1.2	30	7	450	920	66	550	2330	2240	98	300	
R1/1R2-30/1L-8.0h	807	8	5.37	90.10	0.36	1.50	0.08	0.93	0.46	0.07	0.34	167.4	1.9	1.1	31	11	385	1070	70	625	2740	2540	115	355	
R1/1R2-30/1L-12.0h	808	12	4.85	90.80	0.34	1.51	0.09	0.87	0.44	0.07	0.33	165.6	2.1	1.3	32	11	280	1110	74	605	2940	2640	125	390	

Leach conditions employed: 200 g roasted slag contacted with 390 mL 20% HCl solution at 106°C.



Chemical composition of slag No-3-00 (PFC40/) which had been oxidised at 850°C for 3 h and reduced for 30 min at 800°C and then leached for different times in 18, 20 and 33 % HCl solution at stoichiometric excesses of 20, 40 and 70 per cent respectively

No	HCl concentration (%)	Excess HCl (%)	Solution volume (mL)	Time (h)	Fe <sup>2+</sup> (g/L)	Fe <sup>3+</sup> (g/L)	HCl (g/L)	Sand (g)	Fines (g)	Fines (%)	FeO (%)	TiO <sub>2</sub> (%)
1	18	20	693	1	12	2	180	417.8	2.6	0.6	6.2	89.0
				2	29	3	165					
				4	34	8	130					
				6	38	9						
				8	40	10						
				12	45	14	66					
2	18	40	808	1	9	0.5	185	425.7	2.8	0.7	5.8	89.5
				2	18	3	170					
				4	30	7	145					
				6	33	8						
				8	34	10						
				12	37	13	76					
3	18	70	983	1	6	1	190	423.8	3.6	0.8	5.6	89.9
				2	16	1	170					
				4	25	5	150					
				6	28	8						
				8	28	11						
				12	32	11	91					
4	20	20	618	1	13	1	190	426.9	3.0	0.7	6.0	89.2
				2	26	3	175					
				4	41	8	140					
				6	44	9						
				8	47	12						
				12	50	14	50					
5	20	40	720	1	15	1	190	426.2	1.8	0.4	5.6	89.8
				2	26	4	180					
				4	35	8	145					
				6	38	10						
				8	40	12						
				12	42	12	70					
6	20	70	875	1	14	1	190	423.6	2.9	0.7	5.4	90.0
				2	22	3	180					
				4	32	7	165					
				6	32	11						
				8	34	11						
				12	35	12	86					
7	33	20	355	1	38	10	240	421.0	1.1	0.3	4.7	91.0
				2	63	17	205					
				4	81	27	135					
				6	87	34						
				8	88	34						
				12	87	35	31					
8	33	40	413	1	25	6	295	416.9	1.8	0.4	4.6	91.0
				2	50	13	240					
				4	70	23	140					
				6	75	25						
				8	73	28						
				12	74	31	40					
9	33	70	503	1	29	5	295	414.7	3.0	0.7	4.0	91.8
				2	39	11	255					
				4	57	22	155					
				6	60	25						
				8	61	29						
				12	59	28	77					

Leach conditions employed: 500 g roasted slag contacted with hydrochloric acid solutions of different strengths and different stoichiometric excesses.  
FeO and TiO<sub>2</sub> analyses performed by ALS.

**D. Details of experiments conducted on PFE657**

**Chemical composition of slag K6-S-30 (PFE657) which had been oxidised at 850°C for different times**

K6-S-30 (PFE657)	PFE	Time (h)	FeO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R12/0.5h/ox	782	0.5								
R12/1.0h/ox	783	1.0	7.62	0.90	1.57	0.31	1.44	1.26	0.15	0.43
R12/1.5h/ox	784	1.5								
R12/2.0h/ox	785	2.0	7.57	0.91	1.62	0.33	1.46	1.27	0.15	0.43
R12/2.5h/ox	786	2.5								
R12/3.0h/ox	787	3.0	7.57	0.95	1.71	0.35	1.46	1.27	0.15	0.43

**Chemical composition of slag K6-S-30 (PFE657) which had been oxidised at 850°C for 3 h and reduced at 800°C for 30 min and then leached for different times**

K6-S-30 (PFE657)	PFE	Time (h)	FeO (tot)	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
R1/1R2-30	827	0									
R2/1R2-30	828	0									
R3/1R2-30	829	0									
R1/1R2-30/1L-5.0h	846	5	3.19	91.70	0.62	1.85	0.15	0.82	0.58	0.12	0.36
R2/1R2-30/1L-5.0h	847	5	2.48	93.00	0.59	1.86	0.15	0.80	0.57	0.12	0.36
R3/1R2-30/1L-5.0h	848	5	2.65	92.50	0.60	1.80	0.14	0.79	0.54	0.11	0.35

**Chemical composition of slag K6-S-30 (PFE657) which had been oxidised at 850°C for 3 h and reduced at 800°C for 30 min and then leached for different times**

Roast and leach details			Leach residue													
K6-S-30 (PFE657)	PFE	Time (h)	FeO	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>	Sand g	Fines g	Fines %	Fe <sup>2+</sup> g/L	Fe <sup>3+</sup> g/L
R1/1R2-30	827	0														
R2/1R2-30	828	0														
R3/1R2-30	829	0														
R1/1R2-30/1L-0.5h	839	0.5	6.82	86.80	0.95	1.75	0.26	1.36	1.15	0.15	0.43	192.3	0.0	0.0	11	1
R1/1R2-30/1L-1.0h	840	1	6.21	88.20	0.91	1.66	0.25	1.27	1.04	0.15	0.41	190.3	0.0	0.0	14	1
R1/1R2-30/1L-2.0h	841	2	4.16	89.90	0.80	1.65	0.20	1.05	0.82	0.13	0.37	187.4	0.0	0.0	21	2
R1/1R2-30/1L-4.0h	842	4	2.87	92.10	0.65	1.80	0.16	0.87	0.64	0.12	0.37	182.7	0.5	0.3	28	4
R1/1R2-30/1L-6.0h	843	6	2.35	92.70	0.56	1.84	0.14	0.75	0.52	0.11	0.35	179.9	0.9	0.5	28	4
R1/1R2-30/1L-8.0h	844	8	1.79	93.70	0.53	1.85	0.17	0.64	0.44	0.10	0.34	177.4	1.3	0.7	31	6
R1/1R2-30/1L-12.0h	845	12	1.54	94.10	0.47	1.84	0.11	0.55	0.37	0.10	0.33	175.5	1.4	0.8	31	5

Leach conditions employed: 200 g roasted slag contacted with 235 mL 20% HCl solution at 106°C.



## **APPENDIX VI: Titration procedure used to determine the Fe(II), Fe(III) and HCl concentrations of the leach liquors**

### **Method for the determination of the Fe(II) content**

Add 1 mL of sample to approximately 50 mL of water. Acidify the solution with 10 mL of 50/50 H<sub>2</sub>SO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub> then add 5 drops of diphenylamine sulphonic acid (indicator).

Titrate with 0.1N potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>3</sub>) until the solution goes purple.

$$\text{g/L Fe(II)} = \text{titre} \times 5.585$$

- The indicator can be prepared by dissolving 0.4 g of barium diphenylamine sulphonic acid in 100 mL of water.
- The acidifying reagent can be prepared by slowly adding H<sub>3</sub>PO<sub>4</sub> to H<sub>2</sub>SO<sub>4</sub>. The reaction is extremely exothermic and the solutions are very corrosive. Care must be taken to avoid burns and splashes.
- A standard solution of potassium dichromate can be prepared by dissolving 4.9 g of solid potassium dichromate in 1L of distilled water. The K<sub>2</sub>Cr<sub>2</sub>O<sub>3</sub> must be stored in a desiccator for 1 week to ensure dehydration before it is used as a standard.

### **Method for the determination of the Fe(III) content**

Dissolve 5 g of KI in 50 mL of water and add 1 mL of sample. Add starch and wait 5 min. before titrating till clear with 0.1 N sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>).

$$\text{g/L Fe(III)} = \text{titre} \times 5.585$$

Hazards:

50/50 H<sub>2</sub>SO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub> should be used to acidify the titrant liquor for measurement of Fe(II). This aggressive acid should be used with care. Aside from being very acidic the solution reacts with water in an exothermic hydrolysis reaction and will cause severe burns on contact with skin.

### **Method for the determination of the HCl content**

Add 2 mL of sample to 50 mL of water and add 2 drops of bromophenol blue indicator.

Titrate with 1 M NaOH until blue end point is detected (at higher concentrations the precipitation of FeCl inhibits easy detection of the end point).

$$\text{g/L HCl} = \text{titre} \times 18.25$$

## APPENDIX VII: Chemical composition of the feed material

PFE no.	Cast no.	Fe <sup>0</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	Ti <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	CaO	MgO	MnO	Cr <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>
469	K6-S-35	0.03	9.3	-	32.1	52.3	0.93	1.59	0.32	1.17	1.30	0.12	0.45
436	K6-S-2	-	21.9	-	13.6	58.4	0.76	1.87	0.23	1.52	1.17	0.11	0.43

## APPENDIX VIII: List of experiments conducted for process development phase 2

### Series 1: Standard feed slag

Test no.	Roast temperature	Oxidation time	Oxidation atmosphere	Reduction time	Reduction atmosphere	Particle size range	Sample PFE numbers			
							Feed	Oxidation	Reduction	Leach residue
1	750°C	2 h	Air	20 min.	100% CO	+300-500µm	1013	979	-	1018
2	750°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	980	-	1022
3	750°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	981	-	1026
4	750°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	982	-	1030
5	750°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	983	-	1034
6	850°C	2 h	Air	20 min.	100% CO	+300-500µm	1013	984	-	1038
7	850°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	985	-	1042
8	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	986	-	1046
9	850°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	987	1047	1051
10	850°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	988	1052	1056
11	950°C	2 h	Air	20 min.	100% CO	+300-500µm	1013	989	1057	1061
12	950°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	990	1062	1066
13	950°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	991	1067	1071
14	950°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	992	1072	1076
15	950°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	993	1077	1081
16	850°C	½ h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1172	-	1180
17	850°C	1 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1173	-	1184
18	850°C	4 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1174	-	1188
19	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1012	1175	-	1192
20	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1014	1176	-	1196



### Series 2: High iron feed slag

Test no.	Roast temperature	Oxidation time	Oxidation atmosphere	Reduction time	Reduction atmosphere	Particle size range	Sample PFE numbers			
							Feed	Oxidation	Reduction	Leach residue
1	750°C	2 h	Air	20 min.	100% CO	+300-500µm	1010	994	1082	1086
2	750°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	995	1089	1093
3	750°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	996	1094	1098
4	750°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	997	1129	1133
5	750°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	998	1134	1138
6	850°C	2 h	Air	20 min.	100% CO	+300-500µm	1010	999	1099	1103
7	850°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1000	1104	1108
8	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1001	1109	1113
9	850°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1002	1114	1118
10	850°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1003	1119	1123
11	950°C	2 h	Air	20 min.	100% CO	+300-500µm	1010	1004	1124	1128
12	950°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1005	1139	1143
13	950°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1006	1144	1148
14	950°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1007	1149	1153
15	950°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1166	1154	1158
16	850°C	½ h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1167	1197	1201
17	850°C	1 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1168	1202	1206
18	850°C	4 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1169	1207	1211
19	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1009	1170	1212	1216
20	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1011	1171	1217	1221

### Series 3: Standard feed slag

Test no	Roast temperature	Oxidation time	Oxidation atmosphere	Reduction time	Reduction atmosphere	Particle size range	Sample PFE numbers			
							Feed	Oxidation	Reduction	Leach residue
1	850°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1314	1319
2	950°C	2 h	Air	20 min.	100% CO	+300-500µm	1013	-	1320	1325
3	950°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1326	1331
4	950°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1332	1337
5	950°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1338	1343
6	950°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1344	1349
7	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1012	1374	1350	1355
8	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1375	1356	1361
9	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1014	1376	1385	1390
10	800°C	2 h	Air	20 min.	100% CO	+300-500µm	1013	-	1391	1396
11	800°C	2 h	100% CO <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1397	1402
12	800°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1403	1408
13	800°C	2 h	12% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1409	1414
14	800°C	2 h	4% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	-	1415	1420
15	850°C	½ h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1382	1421	1426
16	850°C	1 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1383	1427	1432
17	850°C	3 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1384	1433	1438
18	850°C	4 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1377	1362	1367
19	850°C	8 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1378	1368	1373
20	850°C	2 h	100% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1013	1499	1469	1474
21	850°C	2 h	8% O <sub>2</sub>	5min.	100% CO	+300-500µm	1013	-	1439	1444
22	850°C	2 h	8% O <sub>2</sub>	10 min.	100% CO	+300-500µm	1013	-	1475	1480
23	850°C	2 h	8% O <sub>2</sub>	40 min.	100% CO	+300-500µm	1013	-	1481	1486
24	850°C	2 h	8% O <sub>2</sub>	80 min.	100% CO	+300-500µm	1013	-	1487	1492
25	850°C	½ h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1012	-	1493	1498
26	850°C	1 h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1012	-	1507	1512
27	850°C	3 h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1012	-	1513	1518
28	850°C	4 h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1012	-	1519	1524
29	850°C	8 h	8% O <sub>2</sub>	20 min.	100% CO	+150-300µm	1012	-	1525	1530
30	850°C	½ h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1014	-	1531	1536
31	850°C	1 h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1014	-	1540	1545
32	850°C	3 h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1014	-	1546	1551
33	850°C	4 h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1014	-	1552	1557
34	850°C	8 h	8% O <sub>2</sub>	20 min.	100% CO	+500-700µm	1014	-	1558	1563

**Series 4: High iron feed slag**

Test no.	Roast temperature	Oxidation time	Oxidation atmosphere	Reduction time	Reduction atmosphere	Particle size range	Sample PFE numbers			
							Feed	Oxidation	Reduction	Leach residue
1	800°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010		1642	1647
2	850°C	½ h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1500	1564	1569
3	850°C	1 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1501	1606	1611
4	850°C	2 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1502	1612	1617
5	850°C	3 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1503	1618	1623
6	850°C	4 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1504	1624	1628
7	850°C	8 h	8% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1505	1630	1635
8	850°C	2 h	100% O <sub>2</sub>	20 min.	100% CO	+300-500µm	1010	1506	1636	1641



## APPENDIX IX: Phase 2, Series 1 - Log sheets

Date	18/3/98
Test no.	Series 1: Test 1
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.33	14.84
CO <sub>2</sub>		
Air	11 l/min	
CO		11 l/min
Final sample no.	PFE979	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	14.84
Final volume	
Residue mass	13.4
Residue sample no.	PFE1018

PFE 1013		Feed Slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65		

		Reduced slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
51.43	7.12	0.87	0.43	0.23	0.26	0.09	0.88	0.65		

PFE1018		Leach residue								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
92.40	3.83	0.94	0.86	0.11	0.45	0.11	0.81	1.16		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
55.44	2.99	0.56	0.46	0.08	0.25	0.07	0.62	0.53		

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1015	397.000	1030.000	65.100	16.500	51.400	6.190	21.900	81.900	26.300
6.0	PFE1016	494.000	1090.000	71.900	23.000	55.000	7.840	21.900	94.600	24.300
12.0	PFE1017	618.000	1150.000	80.300	32.400	56.800	9.890	22.500	107.000	34.200
<b>Accountability</b>		0.986	1.083	1.117	0.830	0.890	0.982	0.660	0.953	1.088

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		2.601	48.740	25.180	12.864	74.332	17.931	77.946	31.339	13.643
6.0		3.236	51.579	27.811	17.932	175.164	10.312	77.946	36.198	12.605
12.0		4.049	54.419	31.060	25.260	82.141	13.009	80.081	40.943	17.741

Date	18/3/98
Test no.	Series 1: Test 2
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.2	14.93
CO2	11 l/min	
Air		
CO		11 l/min
Final sample no.	PFE980	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	14.93
Final volume	
Residue mass	14.1
Residue sample no.	PFE1022

PFE 1013		Feed Slag							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

		Reduced slag							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.45	7.12	0.87	0.43	0.23	0.26	0.09	0.88	0.65	

PFE1022		Leach residue							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
89.00	5.85	1.23	0.96	0.11	0.49	0.12	1.06	0.93	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
53.40	4.57	0.74	0.51	0.08	0.27	0.08	0.82	0.43	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1019	162.000	600.000	31.200	8.810	46.500	0.810	19.200	22.500	25.900
6.0	PFE1020	251.000	620.000	34.600	12.600	56.800	2.040	19.400	29.000	24.300
12.0	PFE1021	463.000	678.000	40.300	18.800	60.900	4.150	19.700	41.900	30.400
	Accountability	0.990	1.081	1.047	0.795	0.839	0.938	0.662	0.967	1.285

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		1.055	28.211	11.991	6.825	66.816	2.360	67.899	8.554	13.349
6.0		1.634	29.151	13.298	9.761	181.926	2.666	68.606	11.026	12.524
12.0		3.014	31.878	15.488	14.564	87.507	5.424	69.667	15.930	15.669

Date	18/3/98
Test no.	Series 1: Test 3
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.37	14.84
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		11 l/min
Final sample no.	PFE981	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	14.84
Final volume	
Residue mass	13.6
Residue sample no.	PFE1026

PFE 1013		Feed Slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65		

		Reduced slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
51.33	7.11	0.87	0.43	0.23	0.26	0.09	0.88	0.65		

PFE1026		Leach residue								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
92.50	4.36	1.07	0.90	0.10	0.48	0.11	0.92	1.20		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
55.50	3.41	0.64	0.48	0.07	0.27	0.07	0.71	0.55		

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1023	353.000	1040.000	79.600	19.900	71.400	5.220	21.400	71.800	34.500
6.0	PFE1024	439.000	1060.000	85.300	24.900	77.900	2.980	20.800	81.100	38.400
12.0	PFE1025	591.000	1110.000	91.800	34.000	80.000	8.340	21.600	94.100	38.100
	Accountability	0.971	1.035	0.968	0.782	0.695	0.932	0.669	0.909	1.022

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		2.317	49.310	30.850	15.545	103.458	15.151	76.316	27.528	17.931
6.0		2.882	50.258	33.059	19.451	248.584	3.927	74.176	31.094	19.958
12.0		3.879	52.629	35.578	26.560	115.920	10.992	77.029	36.078	19.802



Date	18/3/98
Test no.	Series 1: Test 4
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.443	14.84
CO <sub>2</sub>	4.7 l/min	
Air	6.3 l/min	
CO		11 l/min
Final sample no.	PFE982	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	14.84
Final volume	
Residue mass	13.5
Residue sample no.	PFE1030

PFE 1013		Feed Slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.14	7.08	0.87	0.43	0.23	0.25	0.09	0.88	0.65	

PFE1030		Leach residue							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
92.10	4.04	1.04	0.89	0.11	0.47	0.11	0.89	1.20	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
55.26	3.16	0.62	0.47	0.08	0.26	0.07	0.69	0.55	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1027	403.000	1100.000	73.000	19.200	59.300	6.130	21.100	81.600	23.700
6.0	PFE1028	493.000	1150.000	79.500	24.900	63.800	7.530	21.900	93.000	31.600
12.0	PFE1029	599.000	1180.000	86.600	33.400	72.500	9.690	21.900	102.000	39.700
	<b>Accountability</b>	0.978	1.034	1.008	0.793	0.735	0.936	0.664	0.905	1.016

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		2.655	52.342	28.393	15.052	86.233	17.856	75.516	31.397	12.362
6.0		3.248	54.721	30.921	19.521	204.319	9.960	78.379	35.784	16.483
12.0		3.946	56.148	33.683	26.185	105.429	12.817	78.379	39.247	20.708

Date	18/3/98
Test no.	Series 1: Test 5
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.31	14.84
CO <sub>2</sub>	8.9 l/min	
Air	2.1 l/min	
CO		11 l/min
Final sample no.	PFE983	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	14.84
Final volume	
Residue mass	13.5
Residue sample no.	PFE1034

PFE 1013 Feed Slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65

Reduced slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.48	7.13	0.87	0.43	0.23	0.26	0.09	0.88	0.65

PFE1034 Leach residue								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
91.70	4.17	1.05	0.84	0.09	0.46	0.11	0.89	1.31
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
55.02	3.26	0.63	0.45	0.06	0.26	0.07	0.69	0.60

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1031	402.000	1040.000	64.800	18.600	53.000	5.210	20.000	76.000	24.500
6.0	PFE1032	503.000	1110.000	74.600	25.600	65.000	7.610	20.800	88.900	28.400
12.0	PFE1033	650.000	1210.000	83.000	35.400	66.200	10.300	21.900	104.000	37.400
	Accountability	0.985	1.012	1.022	0.825	0.829	0.953	0.668	0.905	0.964

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		2.631	49.165	25.040	14.487	76.571	15.077	71.113	29.053	12.696
6.0		3.292	52.474	28.827	19.939	206.808	10.000	73.958	33.984	14.717
12.0		4.254	57.201	32.072	27.572	95.641	13.535	77.869	39.756	19.381

Date	16/3/98
Test no.	Series 1: Test 6
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	20
Solid mass after	25.16	19.12
CO <sub>2</sub>		
Air	11 l/min	
CO		11 l/min
Final sample no.	PFE984	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	19.12
Final volume	
Residue mass	16.8
Residue sample no.	PFE1038

PFE 1013 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
43.01	5.95	0.73	0.36	0.19	0.21	0.08	0.74	0.54	

PFE1038 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
95.00	1.75	0.64	0.62	0.07	0.43	0.11	0.39	1.46	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
57.00	1.37	0.38	0.33	0.05	0.24	0.07	0.30	0.67	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1035	1360.000	2130.000	161.000	63.000	71.600	22.600	25.100	278.000	24.400
6.0	PFE1036	1460.000	2250.000	186.000	84.400	76.300	28.200	26.800	304.000	31.500
12.0	PFE1037	1315.000	2420.000	218.000	105.000	85.500	34.500	29.100	334.000	39.100
	Accountability	0.966	0.951	0.965	0.772	0.877	0.854	0.671	0.779	0.943

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	8.270	77.790	57.802	45.590	96.108	100.869	82.920	98.736	11.748
6.0	8.878	82.173	66.777	61.076	374.411	34.429	88.536	107.971	15.166
12.0	7.996	88.381	78.266	75.984	114.766	42.121	96.134	118.626	18.826



Date	17/3/98
Test no.	Series 1: Test 7
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.279	15.29
CO2	11 l/min	
Air		
CO		11 l/min
Final sample no.	PFE985	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	15.29
Final volume	
Residue mass	12.3
Residue sample no.	PFE1042

PFE 1013		Feed Slag							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

		Reduced slag							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
50.04	6.93	0.85	0.42	0.23	0.25	0.09	0.86	0.63	

PFE1042		Leach residue							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
92.70	3.65	1.05	0.88	0.09	0.47	0.12	0.79	1.31	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
55.62	2.85	0.63	0.47	0.06	0.26	0.08	0.61	0.60	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1039	505.000	1110.000	58.000	11.700	44.600	7.640	21.200	92.300	24.300
6.0	PFE1040	671.000	1240.000	67.900	17.300	50.600	10.400	21.300	120.000	26.600
12.0	PFE1041	794.000	1280.000	76.700	25.400	54.700	13.700	22.200	137.000	23.700
	Accountability	1.057	1.069	1.119	0.918	0.984	0.972	0.666	0.914	1.124

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		3.300	52.394	22.378	9.099	64.337	23.435	75.265	35.230	12.573
6.0		4.385	58.530	26.198	13.454	170.643	13.645	75.620	45.802	13.764
12.0		5.189	60.418	29.593	19.753	78.906	17.975	78.815	52.291	12.263

Date	17/3/98
Test no.	Series 1: Test 8
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.571	15.452
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		11 l/min
Final sample no.	PFE986	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	15.452
Final volume	
Residue mass	12.8
Residue sample no.	PFE1046

PFE 1013 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
48.81	6.76	0.83	0.41	0.22	0.24	0.09	0.84	0.62	

PFE1046 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
96.40	0.99	0.49	0.66	0.08	0.38	0.10	0.26	1.36	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
57.84	0.77	0.29	0.35	0.06	0.21	0.07	0.20	0.63	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1043	1240.000	1720.000	135.000	52.600	52.600	21.400	23.000	228.000	26.200
6.0	PFE1044	1435.000	1890.000	161.000	59.900	59.900	27.700	24.600	264.000	25.500
12.0	PFE1045	1355.000	2010.000	186.000	64.700	64.700	32.800	26.000	287.000	27.100
	Accountability	0.933	0.946	0.978	0.822	0.862	0.861	0.640	0.764	1.017

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		8.220	82.356	52.836	41.495	76.969	68.005	82.831	88.278	13.752
6.0		9.513	90.496	63.012	47.254	209.280	36.867	88.594	102.216	13.384
12.0		8.982	96.242	72.797	51.041	94.675	43.655	93.636	111.121	14.224

Date	17/3/98
Test no.	Series 1: Test 9
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.589	14.72
CO <sub>2</sub>	4.7 l/min	
Air	6.3 l/min	
CO		11 l/min
Final sample no.	PFE987	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.72
Final volume	
Residue mass	11.3
Residue sample no.	PFE1051

PFE 1013 Feed Slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65

PFE1047 Reduced slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.10	8.85	1.47	0.78	0.35	0.48	0.13	1.20	1.33
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.26	6.90	0.88	0.41	0.25	0.27	0.09	0.92	0.61

PFE1051 Leach residue								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
95.60	2.10	0.67	0.68	0.06	0.37	0.10	0.34	1.25
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
57.36	1.64	0.40	0.36	0.04	0.21	0.07	0.26	0.58

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1048	1200.000	1650.000	138.000	28.400	60.700	21.900	23.000	225.000	16.000
6.0	PFE1049	1325.000	1800.000	161.000	42.800	61.300	28.000	25.400	254.000	26.400
12.0	PFE1050	1145.000	1840.000	176.000	62.600	57.900	32.100	27.500	264.000	33.800
	Accountability	1.016	0.857	0.907	0.787	1.010	0.935	0.566	0.785	1.025

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		8.368	87.109	57.020	25.036	89.018	55.891	94.818	88.741	9.531
6.0		9.240	95.028	66.523	37.730	169.223	37.962	104.712	100.179	15.726
12.0		7.985	97.139	72.721	55.185	84.912	43.520	113.370	104.123	20.134



Date	17/3/98
Test no.	Series 1: Test 10
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.835	14.65
CO <sub>2</sub>	8.9 l/min	
Air	2.1 l/min	
CO		11 l/min
Final sample no.	PFE988	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.65
Final volume	
Residue mass	11.3
Residue sample no.	PFE1056

PFE 1013 Feed Slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65

PFE1052 Reduced slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.40	8.83	1.43	0.80	0.30	0.48	0.12	1.19	1.32
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.44	6.89	0.86	0.42	0.21	0.27	0.08	0.92	0.61
50.83	7.04	0.86	0.43	0.22	0.25	0.09	0.87	0.64

PFE1056 Leach residue								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.10	0.95	0.45	0.64	0.08	0.37	0.10	0.24	1.49
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
57.66	0.74	0.27	0.34	0.06	0.21	0.07	0.18	0.69

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1053	1002.000	1600.000	120.000	24.200	51.400	18.900	22.700	206.000	23.700
6.0	PFE1054	1110.000	1670.000	135.000	35.100	49.400	23.400	24.400	224.000	26.500
12.0	PFE1055	1170.000	1890.000	168.000	55.300	54.300	31.300	26.600	262.000	36.000
	Accountability	1.008	0.914	1.023	0.877	0.866	0.939	0.531	0.823	0.868

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	6.999	83.278	51.231	20.907	88.394	47.988	101.900	82.351	14.297
6.0	7.753	86.921	57.635	30.323	158.289	31.888	109.531	89.546	15.986
12.0	8.173	98.372	71.723	47.775	93.381	42.653	119.407	104.737	21.717

Date	19/3/98
Test no.	eries 1: Test 11
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.781	14.65
CO <sub>2</sub>		
Air	11 l/min	
CO		11 l/min
Final sample no.	PFE989	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.65
Final volume	
Residue mass	11.4
Residue sample no.	PFE1061

PFE 1013 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

PFE1057 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.90	9.01	1.45	0.82	0.36	0.50	0.12	1.23	1.44	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
52.14	7.03	0.87	0.43	0.26	0.28	0.08	0.95	0.66	
50.97	7.06	0.86	0.43	0.23	0.25	0.09	0.87	0.64	

PFE1061 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
95.80	1.23	0.56	0.73	0.09	0.39	0.10	0.32	1.59	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
57.48	0.96	0.34	0.39	0.06	0.22	0.07	0.25	0.73	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1058	898.000	1310.000	86.000	25.000	57.900	12.800	22.500	186.000	17.800
6.0	PFE1059	1240.000	1510.000	114.000	39.600	62.400	21.000	24.400	223.000	30.000
12.0	PFE1060	1515.000	1700.000	152.000	61.300	68.500	33.100	25.900	256.000	39.000
Accountability		0.974	1.000	1.039	0.794	0.840	0.922	0.538	0.828	0.879

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	6.309	68.279	36.209	21.071	82.976	31.200	101.002	71.937	9.843
6.0	8.711	78.704	47.998	33.377	166.620	27.473	109.531	86.247	16.590
12.0	10.643	88.253	63.997	51.666	98.167	43.302	116.264	99.011	21.567

Date	19/3/98
Test no.	Series 1: Test 12
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.566	14.72
CO <sub>2</sub>	11 l/min	
Air		
CO		11 l/min
Final sample no.	PFE990	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.72
Final volume	
Residue mass	12.1
Residue sample no.	PFE1066

PFE 1013 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

PFE1062 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.30	9.18	1.57	0.88	0.35	0.50	0.13	1.25	1.44	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.78	7.16	0.94	0.47	0.25	0.28	0.09	0.96	0.66	

PFE1066 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
93.70	3.20	0.80	0.70	0.05	0.39	0.12	0.51	1.49	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
56.22	2.50	0.48	0.37	0.04	0.22	0.08	0.39	0.69	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1063	687.000	748.000	59.700	17.000	51.100	8.730	20.900	121.000	22.400
6.0	PFE1064	912.000	970.000	74.800	23.700	56.400	12.500	21.700	165.000	25.400
12.0	PFE1065	1115.000	1210.000	94.700	33.300	62.000	17.300	23.000	207.000	31.800
	Accountability	0.965	1.083	1.226	1.040	0.966	1.095	0.567	0.874	0.920

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	4.835	38.070	23.096	13.283	74.939	21.389	86.161	45.814	12.324
6.0	6.419	49.369	28.938	18.518	155.696	16.269	89.459	62.474	13.974
12.0	7.847	61.583	36.637	26.020	90.925	22.517	94.818	78.376	17.495



Date	20/3/98
Test no.	Series 1: Test 13
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.648	14.43
CO2	6.8 l/min	
Air	4.2 l/min	
CO		11 l/min
Final sample no.	PFE991	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.43
Final volume	
Residue mass	11.4
Residue sample no.	PFE1071

PFE 1013 Feed Slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

PFE1067 Reduced slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
85.10	9.55	1.62	0.94	0.35	0.53	0.13	1.28	1.48	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.06	7.45	0.97	0.50	0.25	0.30	0.09	0.99	0.68	

PFE1071 Leach residue									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
92.10	3.99	0.96	0.77	0.32	0.43	0.12	0.50	1.26	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
55.26	3.12	0.58	0.41	0.23	0.24	0.08	0.39	0.58	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1068	751.000	1040.000	70.000	26.300	751.000	11.900	21.100	132.000	23.000
6.0	PFE1069	1100.000	1230.000	97.600	41.600	1100.000	19.800	23.100	170.000	22.700
12.0	PFE1070	1625.000	1520.000	143.000	65.100	1625.000	33.100	25.700	226.000	36.800
	Accountability	0.964	0.897	0.952	0.846	0.040	0.906	0.536	0.844	1.082

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	5.476	51.979	26.812	19.654	1125.143	26.923	88.864	49.862	12.578
6.0	8.021	61.475	37.383	31.087	2972.435	24.837	97.287	64.216	12.414
12.0	11.849	75.970	54.773	48.649	2434.563	41.520	108.237	85.369	20.124

Date	20/3/98
Test no.	Series 1: Test 14
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.766	14.63
CO2	4.7 l/min	
Air	6.3 l/min	
CO		11 l/min
Final sample no.	PFE992	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.63
Final volume	
Residue mass	11.5
Residue sample no.	PFE1076

PFE 1013 Feed Slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

PFE1072 Reduced slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
86.10	9.20	1.55	0.93	0.47	0.49	0.14	1.24	1.64	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.66	7.18	0.93	0.49	0.33	0.27	0.10	0.95	0.75	

PFE1076 Leach residue									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
93.90	3.16	0.78	0.69	0.05	0.38	0.12	0.48	1.38	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
56.34	2.47	0.47	0.37	0.04	0.21	0.08	0.37	0.63	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1073	732.00	980.000	67.100	25.600	55.600	11.300	20.800	131.000	23.300
6.0	PFE1074	1090.00	1180.000	93.400	38.600	55.500	18.900	23.000	170.000	24.900
12.0	PFE1075	1575.00	1450.000	134.000	61.400	59.200	31.200	25.700	219.000	35.200
Accountability		0.969	0.969	1.049	0.923	1.350	0.933	0.584	0.856	1.135

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	5.198	50.098	26.468	19.053	61.121	28.065	80.149	50.331	11.330
6.0	7.740	60.322	36.842	28.728	113.345	25.267	88.627	65.315	12.108
12.0	11.184	74.124	52.856	45.697	65.079	41.710	99.031	84.141	17.117

Date	20/3/98
Test no.	eries 1: Test 15
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.6	14.43
CO2	8.9 l/min	
Air	2.1 l/min	
CO		11 l/min
Final sample no.	PFE993	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.43
Final volume	
Residue mass	11.1
Residue sample no.	PFE1081

PFE 1013		Feed Slag							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

PFE1077		Reduced slag							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
87.40	8.02	1.46	0.81	0.31	0.45	0.11	1.10	1.15	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
52.44	6.26	0.88	0.43	0.22	0.25	0.07	0.85	0.53	
52.20	7.23	0.88	0.44	0.23	0.26	0.10	0.89	0.66	

PFE1081		Leach residue							
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
93.10	3.81	0.91	0.72	0.07	0.42	0.12	0.62	1.27	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
55.86	2.98	0.55	0.38	0.05	0.24	0.08	0.48	0.58	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1078	647.00	930.000	58.000	22.900	51.600	9.350	21.200	110.000	22.700
6.0	PFE1079	942.00	1080.000	79.800	33.900	55.400	16.500	22.500	144.000	27.300
12.0	PFE1080	959.00	1100.000	81.200	34.900	56.500	16.800	22.500	146.000	28.500
	Accountability	1.054	0.954	1.162	0.964	0.875	0.981	0.495	0.903	0.898

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		4.593	55.349	24.650	19.860	87.282	24.915	105.518	48.351	15.976
6.0		6.688	64.276	33.915	29.399	169.019	24.377	111.989	63.296	19.213
12.0		6.808	65.466	34.510	30.266	95.570	24.820	111.989	64.175	20.058



Date	1/4/98
Test no.	Series 1: Test 16
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850°C	850°C
Time	30 min	30 min
Solid mass before	15	12
Solid mass after	15.453	11.8
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1172	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	11.8
Final volume	
Residue mass	10.3
Residue sample no.	PFE1180

PFE 1013 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

PFE1077 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.05	7.07	0.86	0.43	0.23	0.25	0.09	0.87	0.64	

PFE1180 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
94.80	2.60	0.73	0.72	0.06	0.73	0.09	0.62	1.22	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
56.88	2.03	0.44	0.38	0.04	0.41	0.06	0.48	0.56	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1177	500.00	1040.000	65.500	14.900	34.100	9.760	1.470	112.000	16.200
6.0	PFE1178	565.00	1010.000	69.000	19.300	35.800	10.600	2.580	115.000	17.600
12.0	PFE1179	766.00	1170.000	88.000	31.900	42.700	14.900	3.110	141.000	28.800
	Accountability	0.965	1.050	1.145	0.916	1.043	0.605	1.411	0.862	1.054

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		4.150	62.346	32.096	14.717	63.532	22.645	6.628	54.292	10.646
6.0		4.689	60.547	33.811	19.062	92.872	17.663	11.633	55.747	11.566
12.0		6.357	70.139	43.121	31.507	79.555	24.829	14.023	68.350	18.926

Date	1/4/98
Test no.	eries 1: Test 17
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850°C	850°C
Time	1 h	30 min
Solid mass before	15	12
Solid mass after	15.579	11.77
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1173	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	11.77
Final volume	
Residue mass	10
Residue sample no.	PFE1184

PFE 1013 Feed Slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65

PFE Reduced slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
50.77	7.03	0.86	0.43	0.23	0.25	0.09	0.87	0.64

PFE1184 Leach residue								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
95.50	1.51	0.54	0.76	0.05	0.35	0.09	0.41	1.40
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
57.30	1.18	0.32	0.40	0.04	0.20	0.06	0.32	0.64

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1181	720.00	1180.000	85.100	17.800	41.000	12.900	1.790	143.000	15.000
6.0	PFE1182	879.00	1290.000	100.000	26.700	44.900	16.600	3.470	165.000	21.700
12.0	PFE1183	1045.00	1410.000	119.000	42.700	47.700	21.600	4.900	187.000	29.600
Accountability		0.956	1.005	1.101	0.815	0.969	0.979	1.283	0.818	0.953

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		6.024	71.315	42.040	17.724	77.206	30.022	8.137	69.885	9.938
6.0		7.355	77.963	49.401	26.586	117.130	27.887	15.774	80.637	14.376
12.0		8.744	85.216	58.787	42.518	89.823	36.286	22.274	91.388	19.610

Date	1/4/98
Test no.	Series 1: Test 18
Solid description	PFE657
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850°C	850°C
Time	4 h	30 min
Solid mass before	15	12
Solid mass after	15.694	11.751
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1174	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	11.751
Final volume	
Residue mass	9.6
Residue sample no.	PFE1188

PFE 1013 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.20	9.18	1.46	0.82	0.33	0.46	0.14	1.15	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.72	7.16	0.88	0.43	0.23	0.26	0.10	0.89	0.65	

PFE Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
50.48	6.99	0.86	0.42	0.22	0.25	0.09	0.86	0.64	

PFE1188 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
97.40	0.60	0.30	0.54	0.06	0.31	0.08	0.18	1.30	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
58.44	0.47	0.18	0.29	0.04	0.17	0.05	0.14	0.60	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1185	985.00	1280.000	103.000	21.000	35.500	18.600	2.540	176.000	16.700
6.0	PFE1186	1135.00	1380.000	123.000	31.600	37.800	23.200	4.560	198.000	20.700
12.0	PFE1187	1115.00	1530.000	148.000	52.600	40.900	30.500	6.310	225.000	31.600
Accountability		0.962	1.014	1.101	0.927	1.072	0.926	1.303	0.807	1.023

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		8.302	77.930	51.258	21.065	67.452	43.466	11.631	86.647	11.146
6.0		9.567	84.018	61.211	31.698	99.176	39.262	20.881	97.478	13.815
12.0		9.398	93.151	73.653	52.763	77.712	51.616	28.895	110.771	21.090



Date	1/4/98
Test no.	eries 1: Test 19
Solid description	PFE657
Particle size fraction	+150-300µm

Roast		
	Oxidation	Reduction
Temperature	850°C	850°C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.632	11.788
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1175	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	11.788
Final volume	
Residue mass	9.7
Residue sample no.	PFE1192

PFE 1013		Feed Slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
85.20	10.39	1.46	0.82	0.29	0.46	0.15	1.15	1.60	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
51.12	8.12	0.88	0.43	0.21	0.26	0.10	0.89	0.74	

PFE		Reduced slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
49.94	7.93	0.86	0.42	0.20	0.25	0.10	0.86	0.72	

PFE1192		Leach residue							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
97.10	0.58	0.28	0.50	0.05	0.32	0.08	0.18	1.42	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
58.26	0.45	0.17	0.27	0.04	0.18	0.05	0.14	0.65	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1189	1180.00	1380.000	112.000	27.700	32.700	21.400	4.430	193.000	17.800
6.0	PFE1190	1200.00	1430.000	125.000	39.300	33.800	25.200	4.960	205.000	20.700
12.0	PFE1191	1105.00	1580.000	150.000	62.500	38.000	31.100	7.290	232.000	27.200
Accountability		0.949	1.122	1.105	0.879	1.038	0.901	1.316	0.788	1.101

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		10.023	73.796	55.517	27.676	70.201	50.126	18.859	94.641	10.502
6.0		10.193	76.470	61.961	39.266	100.831	42.478	21.115	100.526	12.213
12.0		9.386	84.492	74.353	62.446	81.579	52.424	31.034	113.766	16.047

Date	1/4/98
Test no.	Series 1: Test 20
Solid description	PFE657
Particle size fraction	+500-700µm

Roast		
	Oxidation	Reduction
Temperature	850°C	850°C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.604	11.84
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1176	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	11.84
Final volume	
Residue mass	9.8
Residue sample no.	PFE1196

PFE 1013		Feed Slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
89.40	7.05	1.79	0.96	0.65	0.50	0.15	1.31	2.02	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
53.64	5.51	1.07	0.51	0.46	0.28	0.10	1.01	0.93	

PFE		Reduced slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
52.26	5.37	1.05	0.50	0.44	0.27	0.10	0.98	0.91	

PFE1196		Leach residue							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
96.60	1.02	0.36	0.55	0.07	0.30	0.07	0.32	1.59	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
57.96	0.80	0.22	0.29	0.05	0.17	0.05	0.25	0.73	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1193	1130.00	1300.000	116.000	31.800	49.800	21.200	4.270	177.000	18.900
6.0	PFE1194	1240.00	1400.000	132.000	47.100	54.500	25.400	5.980	197.000	23.800
12.0	PFE1195	1180.00	1530.000	154.000	68.500	61.100	30.300	7.500	222.000	33.500
Accountability		0.987	0.754	1.262	0.934	1.483	1.022	1.398	0.861	1.212

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		9.131	102.265	46.815	27.090	47.405	46.006	18.145	76.058	8.816
6.0		10.020	110.131	53.273	40.124	72.726	39.320	25.412	84.652	11.102
12.0		9.535	120.358	62.151	58.355	58.161	46.905	31.871	95.395	15.627

## APPENDIX X: Phase2, Series 2 - Log sheets

Date	24/3/98
Test no.	Series 2: Test 1
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.072	11.9
CO <sub>2</sub>		
Air	11 l/min	
CO		11 l/min
Final sample no.	PFE994	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.9
Final volume	
Residue mass	8.5
Residue sample no.	PFE1086

PFE1010 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1082 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
73.10	21.89	1.88	0.65	0.24	0.45	0.12	1.04	1.47	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
43.86	17.10	1.13	0.34	0.17	0.25	0.08	0.80	0.68	

PFE1086 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
78.20	17.02	1.31	0.75	0.17	0.39	0.28	0.59	2.82	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
46.92	13.30	0.79	0.40	0.12	0.22	0.19	0.45	1.30	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1083	1020.000	1630.000	57.800	13.900	20.600	10.100	21.600	89.100	19.500
6.0	PFE1084	1200.000	1860.000	69.300	19.900	23.800	13.200	22.300	105.000	22.400
12.0	PFE1085	1080.000	1960.000	75.800	24.100	23.500	14.800	23.200	111.000	26.000
Accountability		1.056	0.883	1.174	0.819	0.844	1.058	0.320	0.927	0.598

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	10.668	43.726	23.505	18.508	55.455	21.843	121.425	51.038	13.228
6.0	12.550	49.895	28.182	26.498	76.121	24.028	125.360	60.146	15.196
12.0	11.295	52.578	30.825	32.090	63.262	26.940	130.419	63.583	17.638



Date	24/3/98
Test no.	Series 2: Test 2
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.04	11.95
CO <sub>2</sub>	11 l/min	
Air		
CO		11 l/min
Final sample no.	PFE995	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.95
Final volume	
Residue mass	10
Residue sample no.	PFE1093

PFE1010 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1089 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
76.40	19.46	1.57	0.64	0.20	0.44	0.11	0.99	1.31	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
45.84	15.20	0.94	0.34	0.14	0.25	0.07	0.76	0.60	

PFE1093 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
78.70	17.15	1.53	0.65	0.13	0.45	0.17	0.74	1.40	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
47.22	13.40	0.92	0.34	0.09	0.25	0.12	0.57	0.64	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1090	429.000	707.000	12.300	3.130	16.000	1.450	13.800	48.500	16.500
6.0	PFE1091	500.000	772.000	14.500	6.110	18.900	1.670	14.300	54.200	13.500
12.0	PFE1092	566.000	801.000	16.500	5.100	21.000	2.360	13.300	56.600	21.400
Accountability		1.003	0.956	1.031	1.004	0.788	1.023	0.450	0.979	0.879

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	4.273	21.239	5.962	4.214	51.450	3.222	84.243	29.052	12.503
6.0	4.981	23.192	7.029	8.225	72.871	3.095	87.295	32.466	10.230
12.0	5.638	24.063	7.998	6.865	67.528	4.373	81.191	33.904	16.216

Date	25/3/98
Test no.	Series 2: Test 3
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.189	11.86
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		11 l/min
Final sample no.	PFE996	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.86
Final volume	
Residue mass	8.9
Residue sample no.	PFE1098

PFE1010		Feed Slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1094		Reduced slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
75.60	20.35	1.61	0.66	0.21	0.46	0.11	1.03	1.41	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
45.36	15.90	0.97	0.35	0.15	0.26	0.07	0.79	0.65	

PFE1098		Leach residue							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
81.00	14.21	1.19	0.68	0.15	0.43	0.19	0.59	1.90	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
48.60	11.10	0.71	0.36	0.11	0.24	0.13	0.45	0.87	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1095	975.000	1510.000	52.800	13.600	17.900	9.790	16.300	83.100	13.200
6.0	PFE1096	1045.000	1700.000	63.500	18.800	22.800	11.800	16.300	95.900	13.700
12.0	PFE1097	915.000	1790.000	68.400	20.700	20.000	13.300	17.100	102.000	20.500
	Accountability	1.030	0.917	1.073	0.895	0.831	0.996	0.405	0.942	0.800

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		9.896	43.724	25.165	17.900	55.273	20.637	100.329	48.241	9.370
6.0		10.607	49.226	30.265	24.744	83.034	21.090	100.329	55.671	9.725
12.0		9.287	51.832	32.600	27.245	61.758	23.771	105.253	59.212	14.552

Date	25/3/98
Test no.	Series 2: Test 4
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.224	11.8
CO <sub>2</sub>	4.7 l/min	
Air	6.3 l/min	
CO		11 l/min
Final sample no.	PFE997	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.8
Final volume	
Residue mass	8.8
Residue sample no.	PFE1133

PFE1010 Feed Slag		%							
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1129 Reduced slag		%							
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
75.20	20.86	1.58	0.67	0.19	0.46	0.13	1.03	1.29	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
45.12	16.30	0.95	0.36	0.13	0.26	0.09	0.79	0.59	

PFE1133 Leach residue		%							
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
80.20	15.36	1.37	0.71	0.28	0.44	0.14	0.65	2.10	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
48.12	12.00	0.82	0.38	0.20	0.25	0.10	0.50	0.97	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1130	961.000	1440.000	49.900	13.400	18.100	8.930	15.100	78.800	17.800
6.0	PFE1131	1025.000	1620.000	60.000	17.600	18.400	11.900	16.500	92.900	17.800
12.0	PFE1132	913.000	1640.000	64.200	20.100	22.600	12.200	16.200	93.800	20.700
Accountability		1.039	0.938	0.980	0.888	0.506	1.001	0.579	0.942	0.672

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		9.861	40.900	24.369	17.470	62.117	18.720	79.081	45.999	13.887
6.0		10.517	46.012	29.301	22.946	73.655	21.387	86.413	54.229	13.887
12.0		9.368	46.580	31.353	26.205	77.561	21.926	84.842	54.755	16.150



Date	25/3/98
Test no.	Series 2: Test 5
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	750 °C	750 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.179	11.8
CO <sub>2</sub>	8.9 l/min	
Air	2.1 l/min	
CO		11 l/min
Final sample no.	PFE998	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.8
Final volume	
Residue mass	8.9
Residue sample no.	PFE1138

PFE1010		Feed Slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61		

PFE1134		Reduced slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
75.50	20.48	1.60	0.67	0.18	0.47	0.11	1.03	1.23		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
45.30	16.00	0.96	0.36	0.13	0.26	0.07	0.79	0.57		

PFE1138		Leach residue								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
81.00	14.85	1.36	0.64	0.12	0.44	0.13	0.65	1.49		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
48.60	11.60	0.82	0.34	0.09	0.25	0.09	0.50	0.69		

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1135	859.000	1360.000	45.100	10.700	17.200	8.110	14.700	75.400	17.900
6.0	PFE1136	946.000	1500.000	53.500	15.100	18.700	10.200	15.500	86.500	15.600
12.0	PFE1137	808.000	1440.000	54.900	16.800	23.000	9.900	15.700	83.500	17.300
	Accountability	1.034	0.986	1.036	0.994	0.723	1.058	0.514	0.993	0.877

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		8.779	39.352	21.750	13.950	62.308	16.639	90.983	44.014	14.647
6.0		9.668	43.403	25.801	19.687	79.014	17.942	95.935	50.493	12.765
12.0		8.258	41.667	26.476	21.903	83.319	17.414	97.173	48.742	14.156

Date	23/3/98
Test no.	Series 2: Test 6
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.48	14.58
CO <sub>2</sub>		
Air	11 l/min	
CO		11 l/min
Final sample no.	PFE999	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.58
Final volume	
Residue mass	9.3
Residue sample no.	PFE1103

PFE1010		Feed Slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1099		Reduced slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
74.90	20.35	1.59	0.69	0.20	0.46	0.11	1.03	1.36	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
44.94	15.90	0.95	0.37	0.14	0.26	0.07	0.79	0.63	

PFE1103		Leach residue							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
89.70	7.18	0.71	0.58	0.15	0.33	0.15	0.29	1.94	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
53.82	5.61	0.43	0.31	0.11	0.18	0.10	0.22	0.89	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1100	1945.000	3490.000	156.000	21.700	21.800	28.300	18.700	179.000	14.700
6.0	PFE1101	1725.000	3840.000	181.000	30.300	24.700	32.600	19.900	204.000	18.000
12.0	PFE1102	1360.000	4030.000	203.000	42.800	22.700	38.400	22.400	215.000	21.700
	Accountability	1.073	0.851	0.918	0.993	0.907	0.961	0.491	0.840	0.905

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		15.935	80.816	60.207	21.848	56.525	74.595	92.047	83.099	8.651
6.0		14.133	88.921	69.855	30.506	118.108	46.595	97.954	94.705	10.594
12.0		11.142	93.321	78.346	43.091	58.858	54.885	110.260	99.812	12.771

Date	23/3/98
Test no.	Series 2: Test 7
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.08	14.93
CO2	11 l/min	
Air		
CO		11 l/min
Final sample no.	PFE1000	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	14.93
Final volume	
Residue mass	11.8
Residue sample no.	PFE1108

PFE1010 Feed Slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1104 Reduced slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
75.50	20.10	1.65	0.66	0.20	0.45	0.11	1.00	1.35	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
45.30	15.70	0.99	0.35	0.14	0.25	0.07	0.77	0.62	

PFE1108 Leach residue									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
81.30	14.46	1.34	0.67	0.14	0.43	0.17	0.64	1.64	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
48.78	11.30	0.80	0.36	0.10	0.24	0.12	0.49	0.75	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1105	851.000	1510.000	35.100	8.940	22.700	6.890	19.800	78.400	12.700
6.0	PFE1106	1035.000	1670.000	42.200	10.500	21.900	8.810	15.400	93.400	16.100
12.0	PFE1107	1145.000	1780.000	48.400	14.900	22.300	10.600	16.300	101.000	21.400
Accountability		1.069	1.054	1.241	1.058	0.927	1.116	0.512	1.058	0.930

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		6.291	32.210	11.874	8.559	53.536	20.410	88.649	34.099	6.849
6.0		7.652	35.623	14.275	10.053	115.129	11.708	68.949	40.622	8.682
12.0		8.465	37.969	16.373	14.265	52.593	14.087	72.979	43.928	11.541



Date	25/3/98
Test no.	Series 2: Test 8
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.548	14.56
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		11 l/min
Final sample no.	PFE1001	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.56
Final volume	
Residue mass	9.6
Residue sample no.	PFE1113

PFE1010 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1109 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
75.40	20.74	1.59	0.67	0.20	0.46	0.11	1.03	1.32	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
45.24	16.20	0.95	0.36	0.14	0.26	0.07	0.79	0.61	

PFE1113 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
90.60	6.35	0.63	0.60	0.18	0.32	0.16	0.24	2.07	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
54.36	4.96	0.38	0.32	0.13	0.18	0.11	0.18	0.95	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1110	1890.000	3380.000	151.000	21.800	24.200	27.300	18.900	174.000	13.900
6.0	PFE1111	1750.000	3770.000	178.000	30.800	25.800	32.700	21.100	200.000	10.300
12.0	PFE1112	1420.000	3980.000	200.000	41.700	22.500	37.900	22.400	213.000	23.000
Accountability		1.035	0.891	0.949	0.937	0.819	0.966	0.469	0.866	0.800

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		15.405	76.933	58.363	22.637	62.840	71.853	93.169	80.897	8.441
6.0		14.263	85.810	68.799	31.982	123.186	46.807	104.014	92.985	6.255
12.0		11.574	90.590	77.302	43.301	58.426	54.250	110.422	99.029	13.967

Date	24/3/98
Test no.	Series 2: Test 9
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.451	14.42
CO <sub>2</sub>	4.7 l/min	
Air	4.3 l/min	
CO		11 l/min
Final sample no.	PFE1002	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.42
Final volume	
Residue mass	9.5
Residue sample no.	PFE1118

PFE1010 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1114 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
74.90	20.48	1.60	0.66	0.20	0.46	0.11	1.02	1.30	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
44.94	16.00	0.96	0.35	0.14	0.26	0.07	0.79	0.60	

PFE1118 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
90.30	6.49	0.60	0.58	0.15	0.31	0.18	0.22	2.02	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
54.18	5.07	0.36	0.31	0.11	0.17	0.12	0.17	0.93	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1115	2045.000	3530.000	162.000	22.300	22.800	30.800	19.500	184.000	15.600
6.0	PFE1116	1810.000	3840.000	186.000	30.600	22.700	34.700	20.700	205.000	14.700
12.0	PFE1117	1440.000	4020.000	209.000	41.900	30.600	40.000	22.900	216.000	22.200
	Accountability	1.028	0.862	0.929	0.936	0.750	0.947	0.435	0.849	0.808

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		16.954	82.200	62.873	23.752	59.822	80.228	97.129	87.286	9.719
6.0		15.006	89.419	72.187	32.593	107.265	50.188	103.107	97.248	9.159
12.0		11.938	93.610	81.113	44.628	80.288	57.854	114.065	102.466	13.831

Date	24/3/98
Test no.	Series 2: Test 10
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	30 min
Solid mass before	20	15
Solid mass after	20.125	14.5
CO <sub>2</sub>	8.9 l/min	
Air	2.1 l/min	
CO		11 l/min
Final sample no.	PFE1003	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	13.5
Final volume	
Residue mass	8.9
Residue sample no.	PFE1123

PFE1010 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1119 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
74.70	20.74	1.61	0.67	0.21	0.46	0.11	1.04	1.39	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
44.82	16.20	0.97	0.36	0.15	0.26	0.07	0.80	0.64	

PFE1123 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
86.90	9.46	0.80	0.65	0.18	0.35	0.22	0.30	2.35	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
52.14	7.39	0.48	0.34	0.13	0.20	0.15	0.23	1.08	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1120	1830.000	3220.000	140.000	19.000	23.700	25.800	17.800	165.000	14.500
6.0	PFE1121	1770.000	3510.000	159.000	24.700	23.200	29.400	19.400	186.000	14.800
12.0	PFE1122	1480.000	3750.000	181.000	35.200	23.500	33.500	20.000	200.000	14.300
Accountability		1.125	0.864	0.979	0.993	0.870	1.017	0.433	0.897	0.835

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	15.122	73.617	53.677	19.817	58.872	67.605	88.136	76.313	8.399
6.0	14.626	80.247	60.962	25.762	105.030	42.271	96.059	86.025	8.573
12.0	12.230	85.734	69.397	36.714	58.375	48.165	99.030	92.500	8.283



Date	24/3/98
Test no.	Series 2: Test 11
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.549	11.36
CO <sub>2</sub>		
Air	11 l/min	
CO		11 l/min
Final sample no.	PFE1004	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.36
Final volume	
Residue mass	6.9
Residue sample no.	PFE1128

PFE1010 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1124 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
75.70	20.22	1.54	0.68	0.20	0.46	0.11	1.02	1.26	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
45.42	15.80	0.92	0.36	0.14	0.26	0.07	0.79	0.58	

PFE1128 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
90.50	6.30	1.23	0.79	0.07	0.46	0.10	0.41	1.89	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
54.30	4.87	0.74	0.42	0.05	0.26	0.07	0.32	0.87	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1125	1565.000	2800.000	91.200	20.700	20.400	17.500	17.800	136.000	12.400
6.0	PFE1126	1760.000	2980.000	106.000	25.300	22.500	20.500	18.000	149.000	15.200
12.0	PFE1127	1830.000	3100.000	122.000	32.300	20.900	25.400	18.700	158.000	18.300
Accountability		1.009	0.868	0.855	0.829	1.060	0.876	0.552	0.807	0.868

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	16.629	85.529	47.636	27.720	69.335	35.190	114.849	83.572	10.325
6.0	18.701	91.027	55.366	33.880	82.077	38.408	116.140	91.560	12.657
12.0	19.445	94.692	63.723	43.254	71.034	47.588	120.656	97.090	15.238

Date	26/3/98
Test no.	Series 2: Test 12
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.204	11.82
CO <sub>2</sub>	11 l/min	
Air		
CO		11 l/min
Final sample no.	PFE1005	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.82
Final volume	
Residue mass	7.7
Residue sample no.	PFE1143

PFE1010		Feed Slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61		

PFE1139		Reduced slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
75.00	20.86	1.66	0.69	0.21	0.47	0.12	1.06	1.40		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
45.00	16.30	1.00	0.37	0.15	0.26	0.08	0.82	0.64		

PFE1143		Leach residue								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
83.60	11.89	1.34	0.70	0.17	0.45	0.13	0.46	2.03		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
50.16	9.29	0.80	0.37	0.12	0.25	0.09	0.35	0.93		

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1140	1130.000	1520.000	54.000	10.800	20.600	11.200	15.400	79.000	15.800
6.0	PFE1141	1645.000	2120.000	77.000	17.200	18.500	17.400	16.900	122.000	17.400
12.0	PFE1142	1545.000	2230.000	83.900	20.000	18.300	18.300	17.300	131.000	18.900
Accountability		1.051	0.964	1.038	1.026	0.875	0.997	0.571	0.952	0.857

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		11.604	43.092	25.054	13.647	63.846	23.021	87.211	44.727	11.337
6.0		16.893	60.102	35.725	21.734	67.126	30.550	95.706	69.073	12.486
12.0		15.866	63.221	38.927	25.272	56.717	32.130	97.971	74.168	13.562

Date	26/3/98
Test no.	Series 2: Test 13
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.587	11.35
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		11 l/min
Final sample no.	PFE1006	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.35
Final volume	
Residue mass	7.1
Residue sample no.	PFE1148

PFE1010 Feed Slag		%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61		

PFE1139 Reduced slag		%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
74.60	20.74	1.65	0.70	0.20	0.49	0.12	1.05	1.35		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
44.76	16.20	0.99	0.37	0.14	0.27	0.08	0.81	0.62		

PFE1143 Leach residue		%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
89.20	7.08	1.08	0.64	0.16	0.41	0.12	0.38	2.09		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
53.52	5.53	0.65	0.34	0.11	0.23	0.08	0.29	0.96		

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1145	1410.000	2680.000	81.700	18.400	19.700	15.200	16.500	125.000	17.700
6.0	PFE1146	1610.000	2870.000	104.000	24.800	23.600	20.500	18.000	142.000	17.200
12.0	PFE1147	1610.000	2920.000	116.000	31.200	22.600	23.800	18.600	146.000	14.300
Accountability		1.006	0.905	0.985	0.968	0.759	1.007	0.560	0.892	0.852

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		15.218	79.919	39.867	23.959	67.020	28.666	97.684	74.690	13.769
6.0		17.377	85.585	50.749	32.293	86.007	36.091	106.564	84.847	13.380
12.0		17.377	87.076	56.605	40.627	76.886	41.901	110.117	87.237	11.124



Date	26/3/98
Test no.	eries 2: Test 14
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.55	11.4
CO <sub>2</sub>	4.7 l/min	
Air	6.3 l/min	
CO		11 l/min
Final sample no.	PFE1007	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.4
Final volume	
Residue mass	7.2
Residue sample no.	PFE1153

PFE1010		Feed Slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61		

PFE1149		Reduced slag								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
74.50	20.22	1.65	0.70	0.24	0.47	0.12	1.01	1.73		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
44.70	15.80	0.99	0.37	0.17	0.26	0.08	0.78	0.80		

PFE1153		Leach residue								
%										
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
89.70	6.75	1.00	0.61	0.15	0.39	0.12	0.33	2.06		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
53.82	5.27	0.60	0.32	0.11	0.22	0.08	0.25	0.95		

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1150	1480.000	2670.000	86.300	18.500	20.700	16.300	17.300	128.000	17.400
6.0	PFE1151	1670.000	2890.000	108.000	26.100	22.600	21.200	18.000	145.000	17.500
12.0	PFE1152	1650.000	2910.000	120.000	32.200	20.400	24.800	19.300	148.000	21.700
Accountability		0.989	0.896	0.998	0.980	0.992	0.973	0.547	0.876	1.047

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		15.918	81.244	41.909	23.974	58.403	32.204	101.928	79.129	10.512
6.0		17.962	87.938	52.448	33.822	68.967	38.725	106.052	89.638	10.572
12.0		17.747	88.547	58.275	41.727	57.557	45.300	113.711	91.493	13.110

Date	30/3/98
Test no.	Series 2: Test 15
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	950 °C	950 °C
Time	2 h	30 min
Solid mass before	15	12
Solid mass after	15.579	11.41
CO2	8.9 l/min	
Air	2.1 l/min	
CO		11 l/min
Final sample no.	PFE1008	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.41
Final volume	
Residue mass	7.2
Residue sample no.	PFE1158

PFE1010 Feed Slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1154 Reduced slag									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
77.20	19.07	1.52	0.65	0.18	0.44	0.11	0.96	1.23	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
46.32	14.90	0.91	0.34	0.13	0.25	0.07	0.74	0.57	

PFE1158 Leach residue									
%									
TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	
90.00	5.44	0.77	0.70	0.16	0.36	0.13	0.19	2.93	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
54.00	4.25	0.46	0.37	0.11	0.20	0.09	0.15	1.35	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1155	1620.000	2870.000	110.000	18.300	19.800	19.300	18.200	149.000	17.100
6.0	PFE1156	1520.000	3000.000	126.000	25.200	20.100	22.400	18.900	159.000	18.300
12.0	PFE1157	1420.000	3140.000	143.000	34.400	23.300	26.900	20.000	167.000	16.500
Accountability		1.049	0.827	0.906	0.817	0.671	0.917	0.476	0.818	0.559

Extraction									
%									
Time(h)	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	16.798	92.516	57.932	25.514	74.414	40.770	116.866	96.815	14.516
6.0	15.761	96.706	66.358	35.134	81.863	43.664	121.361	103.313	15.535
12.0	14.724	101.219	75.311	47.961	87.568	52.436	128.424	108.511	14.007

Date	30/3/98
Test no.	Series 2: Test 16
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	½ h	30 min
Solid mass before	15	12
Solid mass after	15.27	11.6
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1167	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.6
Final volume	
Residue mass	8.3
Residue sample no.	PFE1201

PFE1010		Feed Slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1197		Reduced slag							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
72.60	21.89	1.61	0.83	0.18	0.47	0.11	1.09	1.52	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
43.56	17.10	0.97	0.44	0.13	0.26	0.07	0.84	0.70	

PFE1201		Leach residue							
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
82.80	12.40	1.16	0.68	0.12	0.42	0.09	0.59	1.67	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
49.68	9.69	0.70	0.36	0.09	0.24	0.06	0.45	0.77	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1198	1350.000	1890.000	75.000	14.800	14.600	17.200	2.960	101.000	21.000
6.0	PFE1199	1470.000	2180.000	90.000	20.500	16.900	20.000	3.970	119.000	22.800
12.0	PFE1200	1220.000	2200.000	97.000	27.300	16.600	21.500	4.210	120.000	25.900
Accountability		0.975	0.952	0.964	1.070	0.881	0.922	1.104	0.911	0.966

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		14.619	52.135	36.623	15.870	53.887	34.635	18.666	56.763	14.167
6.0		15.918	60.135	43.947	21.982	70.086	35.843	25.035	66.880	15.381
12.0		13.211	60.686	47.365	29.273	61.269	38.532	26.549	67.442	17.473



Date	30/3/98
Test no.	Series 2: Test 17
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	1 h	30 min
Solid mass before	15	12
Solid mass after	15.398	11.63
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1168	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.63
Final volume	
Residue mass	7.8
Residue sample no.	PFE1206

PFE1010 Feed Slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61

PFE1202 Reduced slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
72.00	22.02	1.59	0.77	0.19	0.48	0.11	1.14	1.44
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
43.20	17.20	0.95	0.41	0.13	0.27	0.07	0.88	0.66

PFE1206 Leach residue								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
89.30	8.09	0.83	0.54	0.11	0.33	0.07	0.39	1.60
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
53.58	6.32	0.50	0.29	0.08	0.18	0.05	0.30	0.74

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1203	1655.000	2240.000	96.000	18.300	19.300	21.000	3.290	117.000	19.500
6.0	PFE1204	1795.000	2800.000	125.000	28.500	17.600	28.200	6.010	149.000	23.600
12.0	PFE1205	1420.000	2900.000	137.000	39.100	18.900	31.900	6.540	155.000	26.100
Accountability		0.939	0.941	0.945	1.036	0.923	0.941	1.139	0.925	0.999

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		18.020	61.257	47.333	21.092	67.295	41.523	20.689	62.694	13.847
6.0		19.544	76.571	61.631	32.848	69.343	49.347	37.793	79.841	16.758
12.0		15.461	79.306	67.547	45.066	65.900	55.821	41.126	83.056	18.533

Date	31/3/98
Test no.	eries 2: Test 18
Solid description	PFE436
Particle size fraction	+300-500µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	4 h	30 min
Solid mass before	15	12
Solid mass after	15.538	11.65
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1169	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.65
Final volume	
Residue mass	6.9
Residue sample no.	PFE1211

PFE1010 Feed Slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61	

PFE1207 Reduced slag									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
70.80	23.68	1.48	0.68	0.16	0.44	0.10	1.06	1.39	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
42.48	18.50	0.89	0.36	0.11	0.25	0.07	0.82	0.64	

PFE1211 Leach residue									
%									
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
96.10	2.07	0.29	0.42	0.11	0.23	0.05	0.12	1.84	
%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
57.66	1.62	0.17	0.22	0.08	0.13	0.03	0.09	0.85	

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1208	2260.000	3130.000	149.000	26.700	18.600	33.900	5.060	165.000	20.400
6.0	PFE1209	1965.000	3610.000	13.000	39.500	22.100	40.400	7.870	192.000	25.700
12.0	PFE1210	1460.000	3890.000	209.000	57.500	23.100	46.700	9.820	207.000	27.100
Accountability		0.961	0.958	0.812	0.870	0.714	0.814	0.998	0.791	0.946

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		24.977	79.432	78.776	34.781	76.870	73.262	34.935	94.909	14.979
6.0		21.717	91.613	6.873	51.456	103.594	76.977	54.336	110.440	18.870
12.0		16.136	98.718	110.498	74.904	95.467	88.981	67.799	119.068	19.898

Date	31/3/98
Test no.	Series 2: Test 19
Solid description	PFE436
Particle size fraction	+150-300µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	20 min
Solid mass before	15	12
Solid mass after	15.571	11.55
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1170	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.55
Final volume	
Residue mass	7.4
Residue sample no.	PFE1216

PFE1010 Feed Slag		%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61		

PFE1212 Reduced slag		%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
73.80	20.99	1.60	0.70	0.17	0.46	0.10	1.04	1.20		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
44.28	16.40	0.96	0.37	0.12	0.26	0.07	0.80	0.55		

PFE1216 Leach residue		%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>		
96.50	1.59	0.23	0.42	0.10	0.23	0.05	0.09	1.86		
%										
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si		
57.90	1.24	0.14	0.22	0.07	0.13	0.03	0.07	0.86		

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1213	2020.000	3110.000	155.000	26.400	17.200	33.400	5.560	164.000	22.800
6.0	PFE1214	1815.000	3490.000	183.000	38.900	19.100	39.800	7.970	184.000	24.100
12.0	PFE1215	1430.000	3670.000	203.000	56.200	19.700	45.000	10.300	193.000	29.000
<b>Accountability</b>		0.934	0.898	0.907	0.878	0.843	0.848	0.936	0.831	0.748

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		21.620	89.874	76.521	33.725	67.537	68.395	38.751	97.059	19.576
6.0		19.426	100.855	90.344	49.693	83.473	73.224	55.548	108.896	20.692
12.0		15.305	106.057	100.217	71.793	77.353	82.791	71.787	114.222	24.899



Date	31/3/98
Test no.	Series 2: Test 20
Solid description	PFE436
Particle size fraction	+500-700µm

Roast		
	Oxidation	Reduction
Temperature	850 °C	850 °C
Time	2 h	20 min
Solid mass before	15	12
Solid mass after	15.435	11.7
CO <sub>2</sub>	6.8 l/min	
Air	4.2 l/min	
CO		9 l/min
Final sample no.	PFE1171	-

Leach	
Temperature	105°C
Time	12 h
Solution volume	0.5
Solid mass	10.71
Final volume	
Residue mass	7.6
Residue sample no.	PFE1221

PFE1010 Feed Slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
69.70	24.83	1.88	0.89	0.30	0.47	0.33	1.07	3.50
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
41.82	19.40	1.13	0.47	0.21	0.26	0.22	0.82	1.61

PFE1217 Reduced slag								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
71.40	22.78	1.50	0.68	0.24	0.42	0.10	1.16	1.64
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
42.84	17.80	0.90	0.36	0.17	0.24	0.07	0.89	0.75

PFE1221 Leach residue								
%								
TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
88.40	8.12	0.84	0.63	0.16	0.33	0.08	0.43	1.87
%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
53.04	6.34	0.50	0.33	0.11	0.18	0.05	0.33	0.86

Leach results		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0	-	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0	PFE1218	1915.000	2650.000	106.000	18.300	17.200	24.400	3.960	140.000	22.400
6.0	PFE1219	1925.000	2970.000	125.000	27.100	21.200	28.100	4.930	161.000	23.600
12.0	PFE1220	1625.000	3210.000	141.000	38.500	20.000	33.200	6.590	176.000	28.000
Accountability		0.947	0.914	0.886	0.865	0.979	0.822	0.980	0.845	1.018

Extraction		%								
Time(h)		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
3.0		20.869	69.503	54.985	23.705	47.124	55.554	27.187	73.175	13.862
6.0		20.978	77.896	64.841	35.105	66.623	55.776	33.847	84.151	14.605
12.0		17.709	84.191	73.140	49.872	54.795	65.899	45.244	91.991	17.328

## APPENDIX XI: Phase 2, Series 3 - Log sheets

Date:	98/07/13
Test no.	Series 3: Test 1
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1314
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	13.1
Solid description	Oxidation 2h, 12% O <sub>2</sub> , 850°C Reduction 20 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	10.86
Residue sample no.	PFE 1319

%												
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
85.60			8.91		1.27	0.91	0.30	0.25	0.08	1.27	1.52	
%												
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si				
51.26	6.93	0.77	0.48	0.21	0.14	0.05	0.80	0.71				

%												
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	
95.50			1.69		0.25	0.50	0.08	0.17	0.05	0.16	1.61	
%												
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si				
57.19	1.31	0.15	0.26	0.06	0.10	0.03	0.10	0.75				

### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1315	0.758	1.070	0.088	0.016	0.042	0.015	0.002	0.145	0.014	
3.0	PFE 1316	1.110	1.370	0.122	0.028	0.045	0.022	0.005	0.200	0.019	
6.0	PFE 1317	1.235	1.510	0.150	0.048	0.049	0.029	0.006	0.227	0.025	
12.0	PFE 1318	1.115	1.670	0.180	0.074	0.056	0.037	0.009	0.252	0.033	
	Accountability	1.003	1.002	0.971	0.961	1.139	1.458	1.078	1.198	1.035	

### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1315	5.644	58.963	43.902	12.680	74.756	40.763	15.345	68.853	7.520
3.0	PFE 1316	8.265	75.495	60.864	22.190	80.095	59.786	33.481	94.969	10.205
6.0	PFE 1317	9.196	83.210	74.833	38.040	87.215	78.809	44.641	107.790	13.428
12.0	PFE 1318	8.303	92.027	89.800	58.646	99.674	100.550	64.172	119.661	17.725

Date:	98/07/13
Test no.	Series 3: Test 2
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1320
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	13.9
Solid description	Oxidation 2h, 21% O <sub>2</sub> , 950°C Reduction 20 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	12.07
Residue sample no.	PFE 1325

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.20			8.49		1.28	0.93	0.32	0.26	0.08	1.27	1.50
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.62	6.60	0.77	0.49	0.23	0.15	0.05	0.80	0.70			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
92.20			3.64		0.63	0.61	0.07	0.20	0.06	0.39	1.57
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.21	2.83	0.38	0.32	0.05	0.11	0.04	0.25	0.73			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1321	0.421	0.717	0.047	0.013	0.039	0.007	0.001	0.094	0.013	
3.0	PFE 1322	0.763	0.979	0.068	0.022	0.049	0.012	0.002	0.148	0.019	
6.0	PFE 1323	1.235	1.280	0.102	0.040	0.056	0.020	0.004	0.200	0.027	
12.0	PFE 1324	1.615	1.570	0.146	0.068	0.058	0.034	0.007	0.247	0.029	
	Accountability	1.027	1.138	1.023	0.995	1.033	1.386	1.028	1.250	1.043	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1321	2.934	39.079	21.926	9.501	61.332	17.239	9.203	42.067	6.668	
3.0	PFE 1322	5.317	53.359	31.722	16.078	77.058	29.552	13.805	66.232	9.746	
6.0	PFE 1323	8.607	69.764	47.583	29.233	88.067	49.253	25.638	89.503	13.850	
12.0	PFE 1324	11.255	85.570	68.109	49.697	91.212	83.730	44.044	110.536	14.876	

Date:	98/07/13
Test no.	Series 3: Test 3
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1326
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	14.3
Solid description	Oxidation 2h, CO <sub>2</sub> , 950°C Reduction 20 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	12.65
Residue sample no.	PFE 1331

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.70			8.94		1.26	0.93	0.29	2.50	0.08	1.27	1.45
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.32	6.95	0.76	0.49	0.21	1.40	0.05	0.80	0.68			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
92.80			3.42		0.88	0.70	0.07	0.24	0.06	0.53	1.41
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.57	2.66	0.53	0.37	0.05	0.13	0.04	0.34	0.66			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1327	0.412	0.468	0.045	0.013	0.041	0.007	0.001	0.073	0.015	
3.0	PFE 1328	0.654	0.717	0.060	0.020	0.046	0.010	0.001	0.122	0.021	
6.0	PFE 1329	0.897	0.970	0.079	0.028	0.051	0.015	0.002	0.171	0.026	
12.0	PFE 1330	1.025	1.140	0.097	0.042	0.055	0.018	0.003	0.201	0.030	
	Accountability	1.020	0.851	1.018	0.926	1.072	0.125	0.808	1.148	1.000	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1327	2.807	23.546	20.729	9.235	69.157	1.718	5.751	31.755	7.737
3.0	PFE 1328	4.456	36.074	27.639	14.208	77.591	2.490	3.834	53.070	10.832
6.0	PFE 1329	6.112	48.803	36.391	19.891	86.025	3.734	14.058	74.385	13.411
12.0	PFE 1330	6.984	57.356	44.683	29.836	92.772	4.481	16.614	87.435	15.474



Date:	98/07/13
Test no.	Series 3: Test 4
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1332
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	15.4
<b>Solid description</b>	
Oxidation 2h, 8% O <sub>2</sub> , 950°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	13.1
Residue sample no.	PFE 1337

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.60			7.24		1.27	0.93	0.30	0.26	0.07	1.28	1.50

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.46	5.63	0.77	0.49	0.21	0.15	0.05	0.81	0.70

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
93.50			2.97		0.65	0.62	0.07	0.20	0.06	0.41	1.53

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
55.99	2.31	0.39	0.33	0.05	0.11	0.04	0.26	0.72

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1333	0.496	0.810	0.051	0.016	0.047	0.008	0.002	0.111	0.014	
3.0	PFE 1334	0.865	1.090	0.076	0.027	0.056	0.014	0.003	0.166	0.022	
6.0	PFE 1335	1.305	1.340	0.108	0.045	0.064	0.022	0.005	0.212	0.028	
12.0	PFE 1336	1.640	1.670	0.157	0.074	0.075	0.036	0.007	0.265	0.033	
	<b>Accountability</b>	0.997	1.213	1.018	0.987	1.233	1.344	1.152	1.220	1.005	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1333	3.070	46.727	21.643	10.554	71.161	18.449	12.206	44.486	6.482
3.0	PFE 1334	5.354	62.880	32.253	17.811	84.788	31.119	18.987	66.528	10.186
6.0	PFE 1335	8.077	77.302	45.833	29.684	96.900	48.901	33.905	84.963	12.964
12.0	PFE 1336	10.151	96.339	66.627	48.814	113.555	80.020	48.824	106.204	15.279

Date:	98/07/13
Test no.	Series 3: Test 5
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1338
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	14.3
<b>Solid description</b>	
Oxidation 2h, 12% O <sub>2</sub> , 950°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	12.29
Residue sample no.	PFE 1343

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.30			7.69		1.28	0.91	0.29	0.25	0.07	1.28	1.47
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
52.28	5.98	0.77	0.48	0.21	0.14	0.05	0.81	0.69			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
93.40			3.42		0.75	0.69	0.12	0.22	0.07	0.51	1.53
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.93	2.66	0.45	0.37	0.09	0.12	0.05	0.32	0.72			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1339	0.386	0.685	0.041	0.013	0.037	0.006	0.000	0.088	0.014	
3.0	PFE 1340	0.690	0.933	0.062	0.022	0.049	0.011	0.002	0.141	0.018	
6.0	PFE 1341	1.115	1.240	0.096	0.037	0.066	0.018	0.004	0.195	0.027	
12.0	PFE 1342	1.470	1.440	0.126	0.058	0.058	0.028	0.006	0.228	0.035	
	<b>Accountability</b>	1.005	1.140	1.006	1.015	1.266	1.358	1.210	1.221	1.052	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1339	2.582	40.066	18.592	9.438	62.410	14.937	2.921	37.981	7.123	
3.0	PFE 1340	4.615	54.571	28.114	15.972	82.651	27.385	13.145	60.856	9.158	
6.0	PFE 1341	7.458	72.528	43.531	26.862	111.326	44.811	30.671	84.162	13.737	
12.0	PFE 1342	9.832	84.226	57.135	42.108	97.832	69.706	40.895	98.405	17.807	

Date:	98/07/13
Test no.	Series 3: Test 6
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1344
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.77
<b>Solid description</b>	
Oxidation 2h, 4% O <sub>2</sub> , 950°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.53
Residue sample no.	PFE 1349

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.90			6.84		1.26	0.92	0.30	0.26	0.08	1.27	1.45

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.63	5.32	0.76	0.49	0.21	0.15	0.05	0.80	0.68

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
94.00			2.11		0.77	0.72	0.08	0.22	0.06	0.50	1.53

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
56.29	1.64	0.46	0.38	0.06	0.12	0.04	0.32	0.72

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1345	0.250	0.384	0.024	0.008	0.025	0.005	0.001	0.054	0.014	
3.0	PFE 1346	0.419	0.526	0.035	0.011	0.030	0.007	0.000	0.082	0.017	
6.0	PFE 1347	0.660	0.690	0.050	0.020	0.031	0.011	0.002	0.110	0.020	
12.0	PFE 1348	0.971	0.867	0.075	0.035	0.038	0.019	0.004	0.138	0.017	
	<b>Accountability</b>	1.010	1.094	1.016	1.023	1.152	1.365	0.961	1.208	1.042	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1345	2.708	41.174	18.027	9.250	66.467	18.735	11.461	38.302	11.774
3.0	PFE 1346	4.538	56.400	26.289	12.880	79.761	28.103	4.168	58.162	14.298
6.0	PFE 1347	7.149	73.984	37.556	23.419	82.419	42.935	18.754	78.022	16.821
12.0	PFE 1348	10.518	92.963	56.334	40.982	101.030	74.160	37.509	97.882	14.298

Date:	98/07/13
Test no.	Series 3: Test 7
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1350
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	7.57
Solid description	+150-300µm
Oxidation 2h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	6.16
Residue sample no.	PFE 1355

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.80			6.82		1.24	0.91	0.25	0.26	0.07	1.28	1.47
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
52.57	5.30	0.75	0.48	0.18	0.15	0.05	0.81	0.69			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
97.30			1.16		0.33	0.55	0.07	0.20	0.06	0.18	1.46
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
58.26	0.90	0.20	0.29	0.05	0.11	0.04	0.11	0.68			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1351	0.563	0.800	0.061	0.011	0.030	0.010	0.003	0.110	0.015	
3.0	PFE 1352	0.699	0.895	0.075	0.016	0.029	0.013	0.002	0.130	0.017	
6.0	PFE 1353	0.791	0.963	0.087	0.026	0.025	0.017	0.003	0.144	0.020	
12.0	PFE 1354	0.793	1.030	0.099	0.037	0.027	0.019	0.003	0.154	0.028	
	<b>Accountability</b>	0.994	1.331	1.015	0.934	1.181	1.404	1.125	1.275	1.048	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1351	7.073	99.668	53.937	15.086	110.885	45.219	35.867	89.684	14.416	
3.0	PFE 1352	8.782	111.504	66.317	21.943	107.189	58.785	24.831	105.990	16.339	
6.0	PFE 1353	9.937	119.975	76.927	35.658	92.404	76.872	44.144	117.404	19.222	
12.0	PFE 1354	9.963	128.323	87.538	50.744	99.797	85.916	46.903	125.557	26.911	



Date:	98/07/13
Test no.	Series 3: Test 8
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1356
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.37
Solid description	+150-500µm
Oxidation 2h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	6.78
Residue sample no.	PFE 1361

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.90			8.79		1.24	0.91	0.28	0.25	0.07	1.26	1.54

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.44	6.83	0.75	0.48	0.20	0.14	0.05	0.80	0.72

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
97.10			1.13		0.26	0.43	0.07	0.18	0.06	0.16	1.54

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
58.14	0.88	0.16	0.23	0.05	0.10	0.04	0.10	0.72

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1357	0.611	0.868	0.072	0.014	0.027	0.013	0.002	0.118	0.014	
3.0	PFE 1358	0.774	0.987	0.089	0.025	0.031	0.016	0.002	0.141	0.017	
6.0	PFE 1359	0.855	1.050	0.104	0.038	0.033	0.020	0.003	0.155	0.022	
12.0	PFE 1360	0.785	1.110	0.116	0.052	0.031	0.023	0.006	0.164	0.029	
	<b>Accountability</b>	1.002	1.007	1.018	0.947	1.079	1.470	1.367	1.241	1.025	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1357	7.096	75.884	57.579	17.365	80.588	55.293	21.210	88.392	11.616
3.0	PFE 1358	8.989	86.288	71.174	31.009	92.526	68.053	29.944	105.621	14.105
6.0	PFE 1359	9.930	91.795	83.170	47.134	98.496	85.066	36.182	116.108	18.254
12.0	PFE 1360	9.117	97.041	92.766	64.499	92.526	97.826	78.602	122.850	24.062

Date:	98/07/14
Test no.	Series 3: Test 9
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1385
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	10.18
<b>Solid description</b>	
Oxidation 2h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	8.73
Residue sample no.	PFE 1390

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
84.50			7.28		1.22	0.88	0.58	0.43	0.11	1.26	1.51
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
50.60	7.28	0.73	0.47	0.41	0.24	0.08	0.80	0.71			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
92.70			3.20		0.21	0.45	0.04	0.28	0.07	0.15	1.61
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.51	3.20	0.13	0.24	0.03	0.16	0.05	0.09	0.75			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1386	0.617	0.822	0.080	0.018	0.033	0.013	0.003	0.127	0.034	
3.0	PFE 1387	0.835	1.030	0.102	0.031	0.036	0.018	0.005	0.161	0.038	
6.0	PFE 1388	0.999	1.180	0.126	0.048	0.042	0.024	0.006	0.184	0.046	
12.0	PFE 1389	0.924	1.170	0.134	0.056	0.040	0.027	0.006	0.182	0.036	
	<b>Accountability</b>	1.024	1.111	0.967	0.962	0.504	1.053	0.909	1.144	1.156	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1386	5.989	55.458	53.464	18.983	39.095	26.431	21.542	78.219	23.656	
3.0	PFE 1387	8.105	69.491	68.166	32.693	42.650	36.597	30.029	99.160	26.439	
6.0	PFE 1388	9.697	79.611	84.206	50.621	49.758	48.796	38.515	113.325	32.005	
12.0	PFE 1389	8.969	78.936	89.552	59.057	47.388	54.896	39.821	112.093	25.047	

Date:	98/07/14
Test no.	Series 3: Test 10
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1391
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	7.21
<b>Solid description</b>	
Oxidation 2h, 21% O <sub>2</sub> , 800°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	6.28
Residue sample no.	PFE 1396

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.00			7.50		1.19	0.88	0.37	0.45	0.11	1.24	1.31
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
50.90	7.50	0.72	0.47	0.26	0.25	0.08	0.78	0.61			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
92.70			3.16		0.68	0.59	0.03	0.39	0.09	0.61	1.22
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.51	3.16	0.41	0.31	0.02	0.22	0.06	0.39	0.57			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1392	0.394	0.684	0.047	0.014	0.018	0.009	0.002	0.073	0.030	
3.0	PFE 1393	0.441	0.694	0.049	0.017	0.021	0.010	0.003	0.077	0.033	
6.0	PFE 1394	0.515	0.748	0.057	0.024	0.023	0.011	0.003	0.086	0.042	
12.0	PFE 1395	0.591	0.802	0.065	0.030	0.025	0.013	0.004	0.096	0.042	
	<b>Accountability</b>	1.024	1.059	1.075	0.982	0.676	1.081	1.072	1.211	1.251	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1392	5.368	63.245	45.467	20.846	47.198	24.414	20.277	64.505	33.970
3.0	PFE 1393	6.009	64.170	47.401	25.313	55.064	26.608	25.808	68.039	37.367
6.0	PFE 1394	7.017	69.163	55.141	35.736	60.309	30.174	23.964	75.992	47.558
12.0	PFE 1395	8.052	74.156	62.880	44.670	65.553	35.660	40.555	84.828	47.558

Date:	98/07/27
Test no.	Series 3: Test 11
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1397
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	7.81
<b>Solid description</b>	
Oxidation 2h, CO <sub>2</sub> , 800°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.23
Residue sample no.	PFE 1402

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.50			5.36		1.24	0.93	0.54	0.45	0.11	1.26	1.37
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
52.40	5.36	0.75	0.49	0.39	0.25	0.08	0.80	0.64			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
89.60			5.33		1.04	0.80	0.01	0.45	0.11	1.11	1.24
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
53.65	5.33	0.63	0.42	0.01	0.25	0.08	0.70	0.58			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1398	0.129	0.452	0.021	0.007	0.015	0.004	0.001	0.022	0.028	
3.0	PFE 1399	0.138	0.435	0.020	0.008	0.017	0.003	0.001	0.023	0.030	
6.0	PFE 1400	0.175	0.444	0.022	0.010	0.021	0.004	0.001	0.026	0.035	
12.0	PFE 1401	0.279	0.497	0.027	0.016	0.024	0.006	0.002	0.035	0.041	
	<b>Accountability</b>	0.978	1.475	0.989	0.979	0.380	1.053	1.046	1.069	1.212	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1398	1.576	53.987	17.998	9.495	24.879	9.117	7.658	17.662	27.988	
3.0	PFE 1399	1.686	51.957	17.141	10.536	28.196	8.104	6.807	18.464	29.987	
6.0	PFE 1400	2.138	53.032	18.855	13.007	34.831	10.636	7.658	20.873	34.985	
12.0	PFE 1401	3.409	59.362	23.140	20.811	39.807	14.181	13.614	28.098	40.982	



Date:	98/07/27
Test no.	Series 3: Test 12
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1403
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.03
Solid description	Oxidation 2h, 8% O <sub>2</sub> , 800°C Reduction 20 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	6.88
Residue sample no.	PFE 1408

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.40			6.45		1.24	0.92	0.43	0.46	0.12	1.28	1.33
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
52.34	6.45	0.75	0.49	0.31	0.26	0.08	0.81	0.62			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
94.70			2.17		0.60	0.50	0.02	0.36	0.08	0.55	1.23
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
56.71	2.17	0.36	0.26	0.01	0.20	0.05	0.35	0.58			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1404	0.499	0.753	0.053	0.020	0.022	0.010	0.003	0.079	0.031	
3.0	PFE 1405	0.594	0.810	0.062	0.026	0.025	0.012	0.003	0.092	0.035	
6.0	PFE 1406	0.689	0.874	0.071	0.033	0.029	0.015	0.004	0.104	0.040	
12.0	PFE 1407	0.748	0.940	0.082	0.040	0.032	0.018	0.006	0.117	0.042	
	Accountability	1.010	1.132	1.039	0.925	0.635	1.062	0.973	1.193	1.182	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1404	5.937	72.693	44.179	25.577	44.568	24.094	21.241	60.720	31.044	
3.0	PFE 1405	7.067	78.195	51.681	33.250	50.646	28.913	25.034	70.711	35.049	
6.0	PFE 1406	8.197	84.374	59.183	42.201	58.749	36.142	29.586	79.935	40.057	
12.0	PFE 1407	8.899	90.745	68.353	51.153	64.827	43.370	45.517	89.926	42.059	

Date:	98/07/27
Test no.	Series 3: Test 13
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1409
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.13
<b>Solid description</b>	
Oxidation 2h, 12% O <sub>2</sub> , 800°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.06
Residue sample no.	PFE 1414

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.20			6.27		1.22	0.88	0.39	0.44	0.12	1.26	1.29

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
52.22	6.27	0.73	0.47	0.28	0.25	0.08	0.80	0.60

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
92.90			2.65		0.60	0.55	0.01	0.37	0.09	0.55	1.30

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
55.63	2.65	0.36	0.29	0.01	0.21	0.06	0.35	0.61

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1410	0.510	0.787	0.055	0.019	0.023	0.010	0.003	0.083	0.033	
3.0	PFE 1411	0.614	0.852	0.064	0.027	0.026	0.012	0.003	0.097	0.036	
6.0	PFE 1412	0.691	0.901	0.072	0.032	0.030	0.015	0.005	0.107	0.040	
12.0	PFE 1413	0.772	0.980	0.084	0.041	0.033	0.018	0.005	0.121	0.045	
	<b>Accountability</b>	1.009	1.261	1.069	1.027	0.692	1.135	0.994	1.236	1.298	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1410	6.007	77.195	46.025	25.090	50.741	24.880	18.732	64.009	33.652
3.0	PFE 1411	7.232	83.570	53.556	35.654	57.360	29.856	21.729	74.806	36.712
6.0	PFE 1412	8.139	88.376	60.250	42.257	66.184	37.320	33.718	82.518	40.791
12.0	PFE 1413	9.093	96.125	70.292	54.141	72.803	44.784	38.213	93.315	45.889

Date:	98/07/27
Test no.	Series 3: Test 14
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1415
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	9.14
<b>Solid description</b>	
Oxidation 2h, 4% O <sub>2</sub> , 800°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.74
Residue sample no.	PFE 1420

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.10			6.93		1.22	0.93	0.56	0.43	0.20	1.25	1.73
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.56	6.93	0.73	0.49	0.40	0.24	0.14	0.79	0.81			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
94.00			1.97		0.68	0.57	0.01	0.37	0.15	0.64	1.35
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
56.29	1.97	0.41	0.30	0.01	0.21	0.10	0.41	0.63			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1416	0.442	0.696	0.044	0.015	0.016	0.008	0.001	0.068	0.027	
3.0	PFE 1417	0.447	0.644	0.044	0.018	0.014	0.009	0.002	0.068	0.026	
6.0	PFE 1418	0.503	0.678	0.050	0.024	0.017	0.011	0.003	0.075	0.030	
12.0	PFE 1419	0.498	0.655	0.051	0.026	0.017	0.012	0.004	0.076	0.032	
	<b>Accountability</b>	0.974	0.734	0.827	0.782	0.233	0.978	0.769	0.927	0.862	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1416	4.690	54.941	32.751	16.672	21.866	18.795	3.999	47.020	18.262
3.0	PFE 1417	4.743	50.837	32.751	20.006	19.133	20.607	7.998	47.020	17.586
6.0	PFE 1418	5.337	53.521	37.217	26.675	23.233	24.910	11.997	51.860	20.291
12.0	PFE 1419	5.284	51.705	37.961	28.898	23.233	27.174	15.196	52.551	21.644

Date:	98/07/27
Test no.	Series 3: Test 15
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1421
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	6.14
<b>Solid description</b>	
Oxidation 0.5h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	5.07
Residue sample no.	PFE 1426

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.10			7.21		1.21	0.88	0.41	0.42	0.11	1.22	1.36
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
50.96	7.21	0.73	0.47	0.29	0.24	0.08	0.77	0.64			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
94.40			1.83		0.52	0.61	0.01	0.36	0.20	0.41	1.48
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
56.53	1.83	0.31	0.32	0.01	0.20	0.14	0.26	0.69			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1422	0.259	0.376	0.026	0.005	0.005	0.005	0.001	0.047	0.023	
3.0	PFE 1423	0.349	0.454	0.034	0.008	0.008	0.007	0.002	0.059	0.025	
6.0	PFE 1424	0.376	0.445	0.036	0.012	0.008	0.008	0.002	0.060	0.027	
12.0	PFE 1425	0.440	0.496	0.044	0.018	0.010	0.010	0.003	0.068	0.029	
	<b>Accountability</b>	0.980	0.730	0.802	0.846	0.270	1.018	1.788	0.939	1.244	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1422	4.139	42.467	29.047	7.868	13.615	17.256	7.576	49.567	29.458	
3.0	PFE 1423	5.577	51.277	37.984	14.338	20.840	23.123	19.482	62.223	32.019	
6.0	PFE 1424	6.009	50.260	40.219	20.982	21.674	28.300	25.976	63.278	34.581	
12.0	PFE 1425	7.031	56.021	49.156	31.473	27.787	34.512	32.470	71.715	37.143	



Date:	98/07/27
Test no.	Series 3: Test 16
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1427
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	7.03
<b>Solid description</b>	
Oxidation 1h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	5.69
Residue sample no.	PFE 1432

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.50			6.08		1.18	0.88	0.35	0.43	0.12	1.24	1.34
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.80	6.08	0.71	0.47	0.25	0.24	0.08	0.78	0.63			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.20			1.31		0.41	0.51	0.01	0.34	0.16	0.29	1.41
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.60	1.31	0.25	0.27	0.01	0.19	0.11	0.18	0.66			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1428	0.348	0.491	0.039	0.007	0.010	0.007	0.002	0.065	0.023	
3.0	PFE 1429	0.434	0.555	0.045	0.011	0.010	0.009	0.002	0.076	0.025	
6.0	PFE 1430	0.500	0.597	0.053	0.018	0.012	0.012	0.003	0.084	0.027	
12.0	PFE 1431	0.501	0.608	0.058	0.024	0.012	0.013	0.003	0.087	0.031	
	<b>Accountability</b>	0.964	0.839	0.814	0.790	0.342	0.988	1.294	0.922	1.176	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1428	4.779	57.437	39.022	10.843	28.429	20.904	12.998	58.907	26.113
3.0	PFE 1429	5.959	64.924	45.025	16.799	28.429	26.498	19.064	68.875	28.383
6.0	PFE 1430	6.866	69.837	53.029	27.489	34.115	35.330	23.396	76.125	30.654
12.0	PFE 1431	6.879	71.124	58.032	36.651	34.115	38.275	23.396	78.844	35.195

Date:	98/07/27
Test no.	Series 3: Test 17
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1433
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	6.88
<b>Solid description</b>	
Oxidation 3h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	5.44
Residue sample no.	PFE 1438

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
84.70			7.11		1.18	0.94	0.37	0.45	0.23	1.28	1.75
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
50.72	7.11	0.71	0.50	0.26	0.25	0.16	0.81	0.82			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
97.50			0.56		0.10	0.34	0.01	0.26	0.07	0.07	1.39
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
58.38	0.56	0.06	0.18	0.01	0.15	0.05	0.04	0.65			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1434	0.427	0.486	0.045	0.012	0.011	0.010	0.002	0.076	0.025	
3.0	PFE 1435	0.538	0.575	0.057	0.020	0.011	0.013	0.004	0.090	0.026	
6.0	PFE 1436	0.600	0.635	0.068	0.029	0.012	0.015	0.003	0.098	0.027	
12.0	PFE 1437	0.555	0.658	0.074	0.034	0.013	0.016	0.005	0.101	0.032	
	<b>Accountability</b>	0.985	0.687	0.759	0.728	0.354	0.879	0.454	0.886	0.890	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1434	6.118	49.676	46.007	17.530	30.227	28.747	8.777	68.178	22.207	
3.0	PFE 1435	7.709	58.773	58.275	29.217	30.227	37.371	17.092	80.737	23.096	
6.0	PFE 1436	8.597	64.906	69.521	42.364	32.975	43.120	15.707	87.913	23.984	
12.0	PFE 1437	7.953	67.257	75.655	49.668	35.722	45.995	24.022	90.605	28.425	

Date:	98/07/13
Test no.	Series 3: Test 18
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1362
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	9.14
<b>Solid description</b>	
Oxidation 4h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.52
Residue sample no.	PFE 1367

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.60			7.15		1.28	0.91	0.29	0.26	0.07	1.27	1.45
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
52.46	5.56	0.77	0.48	0.21	0.15	0.05	0.80	0.68			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
97.40			0.85		0.22	0.44	0.08	0.18	0.05	0.14	1.61
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
58.32	0.66	0.13	0.23	0.06	0.10	0.03	0.09	0.75			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1363	0.698	0.983	0.082	0.017	0.031	0.014	0.003	0.136	0.015	
3.0	PFE 1364	0.881	1.120	0.100	0.028	0.033	0.018	0.004	0.164	0.018	
6.0	PFE 1365	0.955	1.210	0.122	0.046	0.037	0.023	0.005	0.181	0.022	
12.0	PFE 1366	0.854	1.330	0.150	0.067	0.072	0.028	0.007	0.200	0.028	
	<b>Accountability</b>	0.999	1.308	1.101	1.058	1.880	1.512	1.256	1.343	1.117	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1363	7.279	96.749	58.175	19.310	81.810	52.432	28.564	92.558	12.105	
3.0	PFE 1364	9.188	110.233	70.945	31.804	87.088	67.413	44.559	111.615	14.526	
6.0	PFE 1365	9.960	119.091	86.553	52.250	97.644	86.139	57.127	123.184	17.754	
12.0	PFE 1366	8.906	130.902	106.418	76.103	190.010	104.865	75.408	136.115	22.596	

Date:	98/07/13
Test no.	Series 3: Test 19
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1368
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.98
<b>Solid description</b>	
Oxidation 8h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.44
Residue sample no.	PFE 1373

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.70			8.27		1.27	0.94	0.28	0.26	0.07	1.27	1.43
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.92	6.43	0.77	0.50	0.20	0.15	0.05	0.80	0.67			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	FeO	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.60			1.12		0.20	0.44	0.07	0.18	0.05	0.13	1.55
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.84	0.87	0.12	0.23	0.05	0.10	0.03	0.08	0.72			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1369	0.604	0.873	0.076	0.013	0.028	0.012	0.003	0.121	0.015	
3.0	PFE 1370	0.783	1.020	0.095	0.024	0.033	0.016	0.003	0.149	0.018	
6.0	PFE 1371	0.898	1.140	0.116	0.041	0.036	0.022	0.004	0.169	0.020	
12.0	PFE 1372	0.864	1.220	0.135	0.059	0.038	0.025	0.006	0.183	0.027	
	<b>Accountability</b>	1.009	1.089	1.021	0.960	1.188	1.431	1.197	1.251	1.100	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1369	6.478	75.610	55.311	14.550	77.895	45.743	33.724	83.817	12.493	
3.0	PFE 1370	8.398	88.341	69.139	26.861	91.805	60.990	32.561	103.213	14.991	
6.0	PFE 1371	9.631	98.735	84.422	45.888	100.151	83.862	46.516	117.067	16.657	
12.0	PFE 1372	9.266	105.663	98.250	66.034	105.715	95.297	68.611	126.765	22.487	



<b>Date:</b>	98/07/27
<b>Test no.</b>	Series 3: Test 20
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	
<b>Feed sample no.</b>	PFE 1469
<b>Solution volume (L)</b>	0.5
<b>Solution description</b>	20% HCl
<b>Solid mass (g)</b>	8.39
<b>Solid description</b>	
Oxidation 2h, 100% O <sub>2</sub> , 850°C	
Reduction 20 min.	
<b>Final volume (liter)</b>	0.4
<b>Number of samples</b>	4
<b>Volume of samples (l)</b>	0.025
<b>Dry residue mass (g)</b>	6.77
<b>Residue sample no.</b>	PFE 1474

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.50			6.33		1.21	0.92	0.42	0.44	0.12	1.29	1.32
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.80	6.33	0.73	0.49	0.30	0.25	0.08	0.82	0.62			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
97.70			0.42		0.21	0.41	0.03	0.29	0.07	0.15	1.37
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
58.50	0.42	0.13	0.22	0.02	0.16	0.05	0.09	0.64			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1470	0.514	0.655	0.057	0.012	0.017	0.010	0.001	0.090	0.024	
3.0	PFE 1471	0.703	0.807	0.075	0.023	0.019	0.014	0.003	0.118	0.028	
6.0	PFE 1472	0.816	0.909	0.092	0.036	0.022	0.019	0.003	0.135	0.031	
12.0	PFE 1473	0.756	0.940	0.101	0.046	0.022	0.021	0.005	0.141	0.035	
	<b>Accountability</b>	0.993	0.873	0.892	0.853	0.465	0.989	0.756	1.042	1.148	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1470	5.914	61.666	46.602	14.688	33.746	24.109	7.261	65.693	23.177	
3.0	PFE 1471	8.088	75.976	61.319	28.151	37.716	33.752	18.152	86.131	27.040	
6.0	PFE 1472	9.389	85.579	75.217	44.063	43.672	45.807	21.782	98.539	29.937	
12.0	PFE 1473	8.698	88.498	82.576	56.302	43.672	50.628	32.673	102.919	33.800	

Date:	98/07/27
Test no.	Series 3: Test 21
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1439
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.9
<b>Solid description</b>	
Oxidation 2h, 8% O <sub>2</sub> , 850°C	
Reduction 5 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	5.44
Residue sample no.	PFE 1444

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.60			6.26		1.20	0.87	0.37	0.43	0.11	1.24	1.20

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.86	6.26	0.72	0.46	0.26	0.24	0.08	0.78	0.56

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.70			0.66		0.19	0.32	0.01	0.30	0.06	0.16	1.34

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
57.90	0.66	0.11	0.17	0.01	0.17	0.04	0.10	0.63

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1440	0.595	0.690	0.063	0.028	0.014	0.012	0.003	0.092	0.025	
3.0	PFE 1441	0.685	0.782	0.076	0.036	0.017	0.014	0.005	0.109	0.026	
6.0	PFE 1442	0.642	0.797	0.083	0.040	0.016	0.016	0.005	0.115	0.028	
12.0	PFE 1443	0.589	0.893	0.097	0.048	0.021	0.019	0.005	0.131	0.033	
	<b>Accountability</b>	0.744	0.807	0.786	0.757	0.423	0.829	0.667	0.942	0.986	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1440	6.446	61.923	48.961	34.164	29.739	27.907	23.894	65.857	25.035
3.0	PFE 1441	7.421	70.180	59.064	43.925	36.111	32.558	34.347	78.026	26.037
6.0	PFE 1442	6.955	71.526	64.504	48.805	33.987	37.209	35.094	82.321	28.039
12.0	PFE 1443	6.381	80.141	75.384	58.566	44.608	44.186	35.841	93.775	33.046

Date:	98/07/27
Test no.	Series 3: Test 22
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1475
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.53
Solid description	Oxidation 2h, 8% O <sub>2</sub> , 850°C Reduction 10 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	6.9
Residue sample no.	PFE 1480

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.10			6.38		1.17	0.85	0.33	0.43	0.20	1.23	1.26

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.56	6.38	0.70	0.45	0.24	0.24	0.14	0.78	0.59

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.20			1.61		0.22	0.36	0.09	0.27	0.07	0.17	1.34

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
57.60	1.61	0.13	0.19	0.06	0.15	0.05	0.11	0.63

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1476	0.574	0.700	0.057	0.020	0.014	0.010	0.001	0.091	0.024	
3.0	PFE 1477	0.746	0.829	0.076	0.032	0.017	0.015	0.002	0.116	0.028	
6.0	PFE 1478	0.776	0.877	0.088	0.041	0.020	0.019	0.004	0.127	0.030	
12.0	PFE 1479	0.703	0.945	0.100	0.048	0.021	0.022	0.005	0.140	0.031	
	<b>Accountability</b>	<b>0.980</b>	<b>1.009</b>	<b>0.909</b>	<b>0.904</b>	<b>0.701</b>	<b>0.988</b>	<b>0.456</b>	<b>1.081</b>	<b>1.148</b>	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1476	6.526	64.313	47.404	26.060	34.790	24.265	4.713	68.520	23.882	
3.0	PFE 1477	8.482	76.165	63.206	41.696	42.245	36.397	9.855	87.344	27.862	
6.0	PFE 1478	8.823	80.575	73.186	53.424	49.700	46.103	14.997	95.626	29.853	
12.0	PFE 1479	7.993	86.822	83.165	62.545	52.185	53.382	19.710	105.415	30.848	

Date:	98/07/27
Test no.	Series 3: Test 23
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1481
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	10.44
<b>Solid description</b>	
Oxidation 2h, 8% O <sub>2</sub> , 850°C	
Reduction 40 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	8.58
Residue sample no.	PFE 1486

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.70			6.51		1.22	0.88	0.52	0.44	0.20	1.25	1.39
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.32	6.51	0.73	0.47	0.37	0.25	0.14	0.79	0.65			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.60			0.90		0.41	0.62	0.12	0.34	0.13	0.23	1.46
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.84	0.90	0.25	0.33	0.09	0.19	0.09	0.15	0.68			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1482	0.448	0.821	0.056	0.009	0.018	0.009	0.001	0.100	0.027	
3.0	PFE 1483	0.748	1.120	0.087	0.017	0.027	0.015	0.003	0.154	0.030	
6.0	PFE 1484	0.756	1.010	0.087	0.021	0.025	0.017	0.002	0.146	0.032	
12.0	PFE 1485	0.827	1.130	0.106	0.033	0.027	0.021	0.004	0.166	0.034	
	<b>Accountability</b>	0.997	0.887	0.904	0.875	0.513	1.001	0.659	1.076	1.096	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1482	4.181	60.399	36.493	9.049	23.193	18.019	2.101	60.536	19.899
3.0	PFE 1483	6.981	82.396	56.694	17.482	34.789	29.062	8.752	93.226	22.110
6.0	PFE 1484	7.055	74.304	56.694	21.595	32.212	32.937	8.402	88.383	23.584
12.0	PFE 1485	7.718	83.132	69.075	33.935	34.789	40.687	14.354	100.490	25.058



Date:	98/07/27
Test no.	Series 3: Test 24
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1487
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	6.87
<b>Solid description</b>	
Oxidation 2h, 8% O <sub>2</sub> , 850°C	
Reduction 80 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	5.72
Residue sample no.	PFE 1492

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.00			5.93		1.21	0.87	0.50	0.43	0.12	1.26	1.24
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
52.10	5.93	0.73	0.46	0.36	0.24	0.08	0.80	0.58			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
95.50			1.08		0.48	0.70	0.07	0.35	0.17	0.27	1.32
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.19	1.08	0.29	0.37	0.05	0.20	0.12	0.17	0.62			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1488	0.272	0.641	0.039	0.006	0.013	0.006	0.000	0.071	0.024	
3.0	PFE 1489	0.400	0.730	0.052	0.010	0.016	0.009	0.001	0.096	0.029	
6.0	PFE 1490	0.510	0.778	0.063	0.014	0.018	0.012	0.001	0.111	0.034	
12.0	PFE 1491	0.627	0.854	0.078	0.021	0.022	0.016	0.003	0.127	0.037	
	<b>Accountability</b>	0.992	1.122	1.030	0.959	0.523	1.104	1.371	1.233	1.313	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1488	3.800	78.671	38.940	9.958	26.473	18.077	2.660	64.797	30.131
3.0	PFE 1489	5.588	89.595	51.921	15.174	32.582	27.115	5.320	87.614	36.408
6.0	PFE 1490	7.125	95.486	62.904	22.129	36.655	36.153	7.094	101.303	42.686
12.0	PFE 1491	8.759	104.813	77.881	33.194	44.801	48.204	23.054	115.905	46.452

Date:	98/07/27
Test no.	Series 3: Test 25
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1493
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	9.06
<b>Solid description</b>	
Oxidation 0.5h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.48
Residue sample no.	PFE 1498

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.40			6.65		1.15	0.85	0.30	0.43	0.24	1.24	1.47

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.74	6.65	0.69	0.45	0.21	0.24	0.16	0.78	0.69

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
94.20			0.95		0.39	0.46	0.01	0.33	0.08	0.29	1.26

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
56.41	0.95	0.23	0.24	0.01	0.19	0.05	0.18	0.59

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1494	0.577	0.818	0.057	0.012	0.012	0.011	0.001	0.104	0.026	
3.0	PFE 1495	0.780	0.981	0.076	0.022	0.016	0.015	0.002	0.133	0.031	
6.0	PFE 1496	0.855	1.040	0.089	0.034	0.019	0.019	0.004	0.146	0.033	
12.0	PFE 1497	0.760	1.010	0.093	0.044	0.021	0.020	0.004	0.144	0.038	
	<b>Accountability</b>	0.977	0.906	0.961	0.920	0.520	1.051	0.396	1.138	0.988	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1494	6.155	67.885	45.407	14.721	30.883	25.130	1.681	73.133	20.879
3.0	PFE 1495	8.320	81.412	60.543	26.989	41.177	34.268	7.060	93.525	24.894
6.0	PFE 1496	9.120	86.308	70.899	41.711	48.898	43.406	13.111	102.667	26.500
12.0	PFE 1497	8.107	83.819	74.086	53.979	54.045	45.690	13.784	101.260	30.515

Date:	98/07/27
Test no.	Series 3: Test 26
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1507
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.81
Solid description	Oxidation 1h, 8% O <sub>2</sub> , 850°C Reduction 20 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.16
Residue sample no.	PFE 1512

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.00			7.06		1.15	0.82	0.27	0.41	0.12	1.25	1.21
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.50	7.06	0.69	0.43	0.19	0.23	0.08	0.79	0.57			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
95.30			1.48		0.26	0.38	0.12	0.30	0.12	0.18	1.35
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.07	1.48	0.16	0.20	0.09	0.17	0.08	0.11	0.63			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1508	0.686	0.915	0.070	0.019	0.020	0.011	0.009	0.125	0.027	
3.0	PFE 1509	0.794	0.985	0.084	0.029	0.022	0.014	0.010	0.141	0.030	
6.0	PFE 1510	0.801	0.988	0.091	0.039	0.028	0.016	0.011	0.145	0.033	
12.0	PFE 1511	0.772	1.070	0.105	0.051	0.026	0.020	0.012	0.158	0.041	
	<b>Accountability</b>	0.981	0.975	0.972	0.967	1.076	1.039	1.579	1.171	1.281	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1508	7.560	73.555	57.346	24.847	58.814	27.103	60.156	89.671	27.088	
3.0	PFE 1509	8.750	79.182	68.815	37.925	64.695	34.495	69.145	101.149	30.098	
6.0	PFE 1510	8.828	79.423	74.550	51.003	82.339	39.423	76.059	104.018	33.108	
12.0	PFE 1511	8.508	86.015	86.019	66.696	76.458	49.279	82.974	113.344	41.134	

Date:	98/07/27
Test no.	Series 3: Test 27
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1513
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	7.31
<b>Solid description</b>	
Oxidation 3h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	5.81
Residue sample no.	PFE 1518

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.80			6.20		1.16	0.87	0.44	0.43	0.12	1.26	1.25

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.98	6.20	0.70	0.46	0.31	0.24	0.08	0.80	0.58

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
97.50			0.72		0.20	0.35	0.01	0.29	0.14	0.13	1.38

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
58.38	0.72	0.12	0.19	0.01	0.16	0.10	0.08	0.65

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1514	0.603	0.774	0.061	0.018	0.018	0.010	0.009	0.106	0.024	
3.0	PFE 1515	0.643	0.751	0.066	0.025	0.017	0.011	0.009	0.108	0.025	
6.0	PFE 1516	0.686	0.791	0.075	0.033	0.019	0.014	0.010	0.116	0.028	
12.0	PFE 1517	0.622	0.804	0.081	0.040	0.019	0.016	0.011	0.120	0.032	
	<b>Accountability</b>	0.971	0.930	0.870	0.852	0.407	0.948	1.776	1.047	1.222	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1514	7.935	85.389	59.708	26.739	39.146	28.031	70.833	90.917	28.091
3.0	PFE 1515	8.462	82.852	64.602	37.138	36.971	31.146	76.667	92.632	29.261
6.0	PFE 1516	9.028	87.264	73.411	49.022	41.321	39.640	83.333	99.494	32.773
12.0	PFE 1517	8.185	88.699	79.284	59.421	41.321	45.303	91.667	102.925	37.454



Date:	98/07/27
Test no.	Series 3: Test 28
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1519
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	8.91
Solid description	Oxidation 4h, 8% O <sub>2</sub> , 850°C Reduction 20 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.14
Residue sample no.	PFE 1524

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.60			6.53		1.15	0.85	0.41	0.43	0.22	1.25	1.34
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.26	6.53	0.69	0.45	0.29	0.24	0.15	0.79	0.63			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
95.50			1.31		0.15	0.28	0.04	0.27	0.06	0.11	1.29
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.19	1.31	0.09	0.15	0.03	0.15	0.04	0.07	0.60			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1520	0.897	0.976	0.083	0.028	0.022	0.014	0.010	0.138	0.028	
3.0	PFE 1521	0.961	1.040	0.096	0.041	0.024	0.018	0.011	0.152	0.032	
6.0	PFE 1522	0.923	1.110	0.111	0.053	0.026	0.021	0.012	0.166	0.036	
12.0	PFE 1523	0.737	1.140	0.119	0.061	0.025	0.024	0.013	0.172	0.042	
	<b>Accountability</b>	0.974	1.079	0.993	0.949	0.530	1.011	0.667	1.208	1.115	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1520	9.820	83.874	67.233	34.928	42.126	32.522	35.428	97.886	25.082	
3.0	PFE 1521	10.521	89.374	77.763	51.145	45.955	41.813	41.021	107.816	28.665	
6.0	PFE 1522	10.105	95.390	89.914	66.114	49.785	48.782	44.751	117.746	32.248	
12.0	PFE 1523	8.069	97.968	96.394	76.094	47.870	55.751	48.480	122.002	37.622	

Date:	98/07/27
Test no.	Series 3: Test 29
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1525
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	7.66
<b>Solid description</b>	
Oxidation 8h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	6.16
Residue sample no.	PFE 1530

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.70			7.01		1.15	0.85	0.33	0.42	0.13	1.24	1.26

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
51.32	7.01	0.69	0.45	0.24	0.24	0.09	0.78	0.59

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.40			1.31		0.11	0.29	0.02	0.27	0.07	0.09	1.34

%								
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
57.72	1.31	0.07	0.15	0.01	0.15	0.05	0.06	0.63

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1526	0.811	0.902	0.077	0.025	0.022	0.013	0.009	0.129	0.026	
3.0	PFE 1527	0.875	0.929	0.088	0.035	0.023	0.016	0.010	0.137	0.029	
6.0	PFE 1528	0.921	1.040	0.106	0.050	0.028	0.020	0.012	0.156	0.034	
12.0	PFE 1529	0.652	0.930	0.100	0.050	0.024	0.019	0.012	0.142	0.031	
	<b>Accountability</b>	0.988	0.977	0.958	0.935	0.681	1.005	1.252	1.179	1.179	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1526	10.316	83.990	72.551	36.275	60.879	35.963	67.536	107.292	28.811
3.0	PFE 1527	11.130	86.505	82.915	50.785	63.646	44.262	73.408	113.946	32.135
6.0	PFE 1528	11.715	96.840	99.875	72.550	77.482	55.328	88.090	129.748	37.676
12.0	PFE 1529	8.293	86.598	94.222	72.550	66.413	52.561	88.090	118.104	34.351

Date:	98/07/27
Test no.	Series 3: Test 30
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1531
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	10.91
<b>Solid description</b>	
Oxidation 0.5h, 8% O <sub>2</sub> , 850°C +500-700µm	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	9.35
Residue sample no.	PFE 1536

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.70			6.66		1.29	0.91	0.57	0.42	0.22	1.31	1.63
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.32	6.66	0.78	0.48	0.41	0.24	0.15	0.83	0.76			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
92.10			3.16		0.73	0.74	0.07	0.38	0.15	0.67	1.49
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.15	3.16	0.44	0.39	0.05	0.21	0.10	0.42	0.70			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1532	0.468	0.777	0.065	0.012	0.038	0.007	0.008	0.090	0.027	
3.0	PFE 1533	0.617	0.875	0.075	0.016	0.042	0.010	0.008	0.110	0.030	
6.0	PFE 1534	0.717	0.910	0.082	0.022	0.044	0.012	0.009	0.120	0.026	
12.0	PFE 1535	0.655	0.875	0.082	0.022	0.040	0.011	0.009	0.111	0.026	
	<b>Accountability</b>	0.976	0.976	0.937	0.888	0.535	0.974	0.843	1.018	0.933	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1532	4.180	53.468	38.333	11.419	42.744	13.596	22.842	49.748	16.238	
3.0	PFE 1533	5.510	60.211	44.231	15.225	47.243	19.423	25.278	60.803	18.042	
6.0	PFE 1534	6.403	62.620	48.359	20.935	49.493	23.308	26.497	66.330	15.637	
12.0	PFE 1535	5.850	60.211	48.359	20.935	44.993	21.365	27.715	61.356	15.637	

Date:	98/07/27
Test no.	Series 3: Test 31
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1540
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	9.3
<b>Solid description</b>	
Oxidation 1h, 8% O <sub>2</sub> , 850°C +500-700µm	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.76
Residue sample no.	PFE 1545

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.10			6.57		1.24	0.90	0.59	0.41	0.23	1.29	1.64
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
50.96	6.57	0.75	0.48	0.42	0.23	0.16	0.82	0.77			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
93.70			1.67		0.60	0.68	0.14	0.34	0.10	0.51	1.51
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
56.11	1.67	0.36	0.36	0.10	0.19	0.07	0.32	0.71			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1541	0.419	0.648	0.055	0.009	0.024	0.007	0.009	0.078	0.020	
3.0	PFE 1542	0.600	0.805	0.072	0.015	0.032	0.010	0.010	0.106	0.024	
6.0	PFE 1543	0.655	0.800	0.075	0.021	0.032	0.012	0.012	0.111	0.026	
12.0	PFE 1544	0.851	0.991	0.098	0.035	0.041	0.017	0.014	0.139	0.028	
	<b>Accountability</b>	0.999	0.953	1.041	0.972	0.672	1.043	0.798	1.159	0.950	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1541	4.421	53.027	39.586	9.933	30.596	16.105	30.074	51.363	14.024	
3.0	PFE 1542	6.330	65.875	51.821	16.931	40.795	23.341	34.175	69.801	16.829	
6.0	PFE 1543	6.911	65.465	53.980	23.703	40.795	28.009	41.010	73.093	18.232	
12.0	PFE 1544	8.978	81.095	70.534	39.506	52.268	39.680	47.845	91.531	19.634	



Date:	98/07/27
Test no.	Series 3: Test 32
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1546
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	11.08
<b>Solid description</b>	
Oxidation 3h, 8% O <sub>2</sub> , 850°C +500-700µm	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	8.93
Residue sample no.	PFE 1551

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.40			6.36		1.28	0.95	0.58	0.41	0.18	1.29	1.50
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.14	6.36	0.77	0.50	0.41	0.23	0.12	0.82	0.70			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
95.50			1.41		0.20	0.49	0.05	0.27	0.06	0.15	1.53
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.19	1.41	0.12	0.26	0.04	0.15	0.04	0.09	0.72			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1547	0.705	0.941	0.087	0.023	0.047	0.013	0.009	0.137	0.024	
3.0	PFE 1548	0.937	1.150	0.110	0.036	0.045	0.019	0.011	0.173	0.026	
6.0	PFE 1549	1.170	1.360	0.141	0.057	0.050	0.026	0.013	0.207	0.028	
12.0	PFE 1550	1.120	1.470	0.162	0.072	0.052	0.031	0.015	0.226	0.021	
	<b>Accountability</b>	0.993	1.136	0.983	0.985	0.600	1.073	0.768	1.236	0.955	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1547	6.221	66.767	50.915	20.643	51.159	25.469	31.888	75.721	15.444	
3.0	PFE 1548	8.269	81.596	64.376	32.311	48.982	37.224	40.318	95.619	16.731	
6.0	PFE 1549	10.325	96.497	82.518	51.160	54.424	50.938	47.648	114.411	18.018	
12.0	PFE 1550	9.883	104.301	94.808	64.623	56.601	60.733	54.979	124.913	13.514	

<b>Date:</b>	98/07/27
<b>Test no.</b>	Series 3: Test 33
<b>Temperature (°C)</b>	95
<b>R.p.m.</b>	
<b>Feed sample no.</b>	PFE 1552
<b>Solution volume (L)</b>	0.5
<b>Solution description</b>	20% HCl
<b>Solid mass (g)</b>	9.72
<b>Solid description</b>	
Oxidation 4h, 8% O <sub>2</sub> , 850°C +500-700µm	
Reduction 20 min.	
<b>Final volume (liter)</b>	0.4
<b>Number of samples</b>	4
<b>Volume of samples (l)</b>	0.025
<b>Dry residue mass (g)</b>	7.76
<b>Residue sample no.</b>	PFE 1557

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
84.90			7.45		1.23	0.92	0.57	0.42	0.22	1.28	1.49
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
50.84	7.45	0.74	0.49	0.41	0.24	0.15	0.81	0.70			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
97.00			0.44		0.19	0.41	0.06	0.26	0.08	0.17	1.54
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
58.08	0.44	0.11	0.22	0.04	0.15	0.05	0.11	0.72			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1553	0.713	0.996	0.086	0.022	0.033	0.014	0.009	0.132	0.023	
3.0	PFE 1554	0.908	1.140	0.109	0.034	0.039	0.019	0.011	0.164	0.027	
6.0	PFE 1555	1.030	1.230	0.129	0.051	0.041	0.024	0.012	0.184	0.024	
12.0	PFE 1556	0.919	1.280	0.142	0.062	0.043	0.027	0.014	0.193	0.023	
	<b>Accountability</b>	1.000	0.870	1.024	0.936	0.590	1.027	0.728	1.239	0.988	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1553	7.214	68.771	59.704	23.243	41.664	30.521	30.082	83.816	16.985	
3.0	PFE 1554	9.188	78.714	75.672	35.920	49.239	41.422	37.603	104.135	19.938	
6.0	PFE 1555	10.422	84.928	89.556	53.881	51.764	52.322	41.021	116.834	17.723	
12.0	PFE 1556	9.299	88.381	98.581	65.502	54.289	58.862	47.858	122.549	16.985	

Date:	98/07/27
Test no.	Series 3: Test 34
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1558
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	9.58
<b>Solid description</b>	
Oxidation 8h, 8% O <sub>2</sub> , 850°C +500-700µm	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.74
Residue sample no.	PFE 1563

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
86.00			6.65		1.25	0.91	0.53	0.42	0.20	1.29	1.48
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.50	6.65	0.75	0.48	0.38	0.24	0.14	0.82	0.69			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
96.00			1.27		0.13	0.34	0.01	0.26	0.06	0.13	1.47
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.49	1.27	0.08	0.18	0.01	0.15	0.04	0.08	0.69			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1559	0.701	0.929	0.082	0.022	0.032	0.013	0.009	0.131	0.021	
3.0	PFE 1560	0.880	1.090	0.104	0.036	0.037	0.018	0.011	0.159	0.023	
6.0	PFE 1561	0.999	1.200	0.126	0.054	0.037	0.024	0.012	0.181	0.025	
12.0	PFE 1562	0.871	1.250	0.140	0.065	0.040	0.028	0.014	0.191	0.032	
	<b>Accountability</b>	0.986	1.065	0.968	0.926	0.529	1.056	0.731	1.209	1.022	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1559	7.105	72.912	56.835	23.841	44.086	28.755	33.956	83.742	15.841	
3.0	PFE 1560	8.919	85.548	72.084	39.013	50.974	39.815	41.968	101.641	17.349	
6.0	PFE 1561	10.125	94.181	87.332	58.520	50.974	53.087	45.783	115.705	18.858	
12.0	PFE 1562	8.828	98.105	97.035	70.441	55.107	61.935	53.413	122.097	24.138	

## APPENDIX XII: Phase 2, Series 4 - Log sheets

Date:	98/07/27
Test no.	Series 4: Test 1
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1642
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	11.98
Solid description	Oxidation 2h, 8% O <sub>2</sub> , 800°C Reduction 20 min.
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	9.08
Residue sample no.	PFE 1647

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.40			16.10		1.55	0.64	0.14	0.43	0.15	1.08	1.34
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
44.55	16.10	0.93	0.34	0.10	0.24	0.10	0.68	0.63			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
87.10			7.46		1.00	0.54	0.08	0.36	0.08	0.51	1.58
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
52.16	7.46	0.60	0.29	0.06	0.20	0.05	0.32	0.74			

### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1643	1.400	2.130	0.078	0.015	0.016	0.017	0.003	0.104	0.015	
3.0	PFE 1644	1.620	2.430	0.092	0.019	0.017	0.020	0.004	0.123	0.021	
6.0	PFE 1645	1.510	2.660	0.105	0.028	0.018	0.023	0.005	0.138	0.027	
12.0	PFE 1646	1.240	2.820	0.118	0.037	0.018	0.026	0.007	0.146	0.030	
	Accountability	1.001	1.030	0.972	1.042	1.140	1.046	0.644	1.183	1.075	

### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1643	13.115	55.216	34.865	18.483	66.730	29.371	13.017	63.501	9.993
3.0	PFE 1644	15.177	62.993	41.122	23.412	70.901	34.554	16.678	75.102	13.991
6.0	PFE 1645	14.146	68.956	46.933	34.502	75.072	39.737	20.746	84.261	17.988
12.0	PFE 1646	11.617	73.103	52.744	45.591	75.072	44.920	26.848	89.145	19.987



Date:	98/07/27
Test no.	Series 4: Test 2
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1564
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	11.24
<b>Solid description</b>	
Oxidation 0.5h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	8.6
Residue sample no.	PFE 1569

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.00			16.40		1.57	0.70	0.23	0.44	0.20	1.12	1.47

%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
44.31	16.40	0.95	0.37	0.16	0.25	0.14	0.71	0.69	

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
84.70			8.85		1.07	0.64	0.14	0.38	0.09	0.53	1.55

%									
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
50.72	8.85	0.64	0.34	0.10	0.21	0.06	0.34	0.72	

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1565	1.130	1.860	0.075	0.012	0.013	0.014	0.009	0.094	0.022	
3.0	PFE 1566	1.340	2.110	0.087	0.015	0.016	0.016	0.009	0.111	0.026	
6.0	PFE 1567	1.320	2.280	0.097	0.019	0.017	0.019	0.010	0.124	0.028	
12.0	PFE 1568	1.150	2.390	0.106	0.026	0.017	0.021	0.011	0.130	0.028	
	<b>Accountability</b>	0.987	1.016	0.981	0.977	0.896	1.007	0.675	1.118	0.976	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1565	11.344	50.451	35.276	14.409	35.175	25.194	28.290	58.989	14.240	
3.0	PFE 1566	13.452	57.232	40.920	18.011	43.293	28.793	29.266	69.657	16.829	
6.0	PFE 1567	13.251	61.844	45.623	22.814	45.998	34.192	32.518	77.815	18.124	
12.0	PFE 1568	11.545	64.827	49.856	31.219	45.998	37.791	35.770	81.580	18.124	

Date:	98/07/27
Test no.	Series 4: Test 3
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1606
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	13.13
<b>Solid description</b>	
Oxidation 1h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	9.67
Residue sample no.	PFE 1611

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.00			16.60		1.58	0.66	0.13	0.44	0.11	1.09	1.35
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
44.31	16.60	0.95	0.35	0.09	0.25	0.08	0.69	0.63			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
85.40			6.00		0.83	0.54	0.07	0.35	0.12	0.38	1.75
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
51.14	6.00	0.50	0.29	0.05	0.20	0.08	0.24	0.82			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1607	1.670	2.570	0.099	0.020	0.016	0.021	0.003	0.126	0.015	
3.0	PFE 1608	1.830	3.020	0.121	0.021	0.018	0.026	0.005	0.155	0.020	
6.0	PFE 1609	1.600	3.300	0.137	0.030	0.020	0.029	0.006	0.173	0.023	
12.0	PFE 1610	1.260	3.480	0.153	0.042	0.021	0.033	0.007	0.183	0.026	
	<b>Accountability</b>	0.958	1.007	0.948	1.008	1.196	1.051	1.122	1.190	1.098	

#### Extraction

Time(h)	Sample no.	%									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1607	14.352	58.956	39.609	21.804	65.569	32.351	16.196	69.551	9.051	
3.0	PFE 1608	15.727	69.279	48.411	22.894	73.765	40.054	24.800	85.559	12.067	
6.0	PFE 1609	13.750	75.703	54.812	32.706	81.961	44.676	29.862	95.495	13.877	
12.0	PFE 1610	10.828	79.832	61.214	45.789	86.060	50.838	35.429	101.015	15.688	

Date:	98/07/27
Test no.	Series 4: Test 4
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1612
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	12.54
<b>Solid description</b>	
Oxidation 2h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	8.83
Residue sample no.	PFE 1617

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.30			16.40		1.58	0.65	0.12	0.44	0.12	1.09	1.28
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
44.49	16.40	0.95	0.34	0.09	0.25	0.08	0.69	0.60			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
93.30			3.33		0.51	0.51	0.07	0.31	0.11	0.21	1.78
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.87	3.33	0.31	0.27	0.05	0.17	0.08	0.13	0.83			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1613	1.810	2.830	0.115	0.017	0.014	0.024	0.004	0.140	0.017	
3.0	PFE 1614	1.930	3.320	0.141	0.022	0.018	0.029	0.005	0.170	0.022	
6.0	PFE 1615	1.610	3.640	0.162	0.031	0.018	0.033	0.007	0.189	0.026	
12.0	PFE 1616	1.220	3.860	0.180	0.045	0.018	0.038	0.009	0.201	0.032	
	<b>Accountability</b>	0.996	1.013	0.918	1.010	1.196	1.056	1.015	1.209	1.171	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1613	16.221	68.804	48.175	19.704	65.078	38.712	17.974	80.915	11.327	
3.0	PFE 1614	17.296	80.717	59.067	25.499	83.672	46.778	26.232	98.254	14.659	
6.0	PFE 1615	14.429	88.497	67.864	35.931	83.672	53.230	33.519	109.236	17.324	
12.0	PFE 1616	10.934	93.846	75.404	52.158	83.672	61.295	41.291	116.171	21.322	

Date:	98/07/27
Test no.	Series 4: Test 5
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1618
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	6.65
<b>Solid description</b>	
Oxidation 3h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	3.94
Residue sample no.	PFE 1623

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.40			16.70		1.60	0.67	0.14	0.44	0.11	1.10	1.30
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
44.55	16.70	0.96	0.35	0.10	0.25	0.08	0.70	0.61			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
93.00			2.35		0.39	0.51	0.07	0.28	0.14	0.14	1.93
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
55.69	2.35	0.23	0.27	0.05	0.16	0.10	0.09	0.90			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1619	1.140	1.590	0.066	0.012	0.015	0.015	0.002	0.080	0.013	
3.0	PFE 1620	1.400	1.850	0.081	0.014	0.012	0.019	0.003	0.095	0.018	
6.0	PFE 1621	1.410	2.020	0.093	0.020	0.013	0.022	0.004	0.105	0.023	
12.0	PFE 1622	1.200	2.160	0.105	0.028	0.015	0.025	0.005	0.112	0.029	
	<b>Accountability</b>	0.936	0.984	0.893	0.975	1.348	1.071	1.192	1.194	1.200	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1619	19.240	71.586	51.485	25.445	112.701	45.625	20.986	86.398	16.083
3.0	PFE 1620	23.628	83.292	63.186	29.686	90.161	57.792	30.979	102.597	22.268
6.0	PFE 1621	23.796	90.946	72.547	42.408	97.675	66.917	40.972	113.397	28.454
12.0	PFE 1622	20.252	97.249	81.908	59.372	112.701	76.042	48.967	120.957	35.877



Date:	98/07/27
Test no.	Series 4: Test 6
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1624
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	11.48
<b>Solid description</b>	
Oxidation 4h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.63
Residue sample no.	PFE 1629

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
73.30			17.40		1.55	0.66	0.13	0.43	0.18	1.09	1.29
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
43.89	17.40	0.93	0.35	0.09	0.24	0.12	0.69	0.60			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
95.40			1.48		0.22	0.34	0.05	0.23	0.09	0.10	1.84
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
57.13	1.48	0.13	0.18	0.04	0.13	0.06	0.06	0.86			

#### Experimental results

		g/l									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1625	2.070	2.940	0.125	0.022	0.017	0.028	0.005	0.152	0.016	
3.0	PFE 1626	2.000	3.360	0.151	0.030	0.019	0.032	0.006	0.175	0.019	
6.0	PFE 1627	1.590	3.630	0.172	0.044	0.020	0.038	0.008	0.190	0.023	
12.0	PFE 1628	1.190	3.870	0.193	0.059	0.020	0.043	0.010	0.200	0.025	
	<b>Accountability</b>	0.988	0.956	0.919	0.991	1.137	1.064	0.649	1.234	1.113	

#### Extraction

		%									
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1625	20.540	73.591	58.306	27.432	79.680	50.482	17.334	95.963	11.555	
3.0	PFE 1626	19.846	84.104	70.434	37.407	89.055	57.694	21.225	110.483	13.722	
6.0	PFE 1627	15.777	90.863	80.229	54.864	93.742	68.511	28.654	119.953	16.610	
12.0	PFE 1628	11.808	96.870	90.025	73.567	93.742	77.526	35.376	126.267	18.055	

Date:	98/07/27
Test no.	Series 4: Test 7
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1630
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	10.56
<b>Solid description</b>	
Oxidation 8h, 8% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	7.02
Residue sample no.	PFE 1635

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.40			16.20		1.60	0.67	0.13	0.43	0.14	1.09	1.48
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
44.55	16.20	0.96	0.35	0.09	0.24	0.10	0.69	0.69			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe <sup>o</sup>	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
94.10			2.60		0.26	0.43	0.07	0.25	0.12	0.11	2.09
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
56.35	2.60	0.16	0.23	0.05	0.14	0.08	0.07	0.98			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1631	1.790	2.650	0.112	0.019	0.017	0.025	0.004	0.136	0.017	
3.0	PFE 1632	1.920	3.080	0.137	0.023	0.017	0.030	0.005	0.160	0.021	
6.0	PFE 1633	1.630	3.340	0.156	0.033	0.018	0.034	0.007	0.174	0.026	
12.0	PFE 1634	1.260	3.530	0.174	0.048	0.019	0.039	0.008	0.183	0.031	
	<b>Accountability</b>	0.976	1.065	0.891	0.989	1.265	1.085	0.941	1.233	1.130	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1631	19.024	77.453	55.019	25.371	86.622	49.000	18.789	93.342	11.633
3.0	PFE 1632	20.406	90.021	67.300	30.712	86.622	58.800	25.217	109.814	14.371
6.0	PFE 1633	17.324	97.620	76.634	44.065	91.718	66.640	33.623	119.422	17.792
12.0	PFE 1634	13.391	103.173	85.476	64.094	96.813	76.440	41.534	125.599	21.214

Date:	98/07/27
Test no.	Series 4: Test 8
Temperature (°C)	95
R.p.m.	
Feed sample no.	PFE 1636
Solution volume (L)	0.5
Solution description	20% HCl
Solid mass (g)	15.04
<b>Solid description</b>	
Oxidation 2h, 100% O <sub>2</sub> , 850°C	
Reduction 20 min.	
Final volume (liter)	0.4
Number of samples	4
Volume of samples (l)	0.025
Dry residue mass (g)	10.8
Residue sample no.	PFE 1641

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
74.40			16.00		1.56	0.65	0.12	0.43	0.11	1.09	1.42
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
44.55	16.00	0.94	0.34	0.09	0.24	0.08	0.69	0.66			

%											
TiO <sub>2</sub>	Ti <sub>2</sub> O <sub>3</sub>	Fe°	Fe (tot)	Fe <sub>2</sub> O <sub>3</sub>	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
93.80			2.92		0.44	0.42	0.09	0.27	0.10	0.20	1.84
%											
Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si			
56.17	2.92	0.27	0.22	0.06	0.15	0.07	0.13	0.86			

#### Experimental results

Time(h)	Sample no.	g/l									
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	HCl
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
1.5	PFE 1637	2.190	3.420	0.140	0.025	0.019	0.030	0.005	0.173	0.017	
3.0	PFE 1638	2.090	3.960	0.167	0.031	0.020	0.036	0.007	0.205	0.019	
6.0	PFE 1639	1.680	4.400	0.199	0.047	0.024	0.042	0.009	0.230	0.023	
12.0	PFE 1640	1.260	4.650	0.222	0.065	0.023	0.050	0.012	0.242	0.019	
	<b>Accountability</b>	1.003	1.026	0.920	1.016	1.374	1.076	1.123	1.211	1.021	

#### Extraction

Time(h)	Sample no.	%								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE 1637	16.342	71.061	49.526	24.160	73.640	41.285	23.860	83.368	8.513
3.0	PFE 1638	15.596	82.281	59.078	29.958	77.516	49.542	29.604	98.789	9.515
6.0	PFE 1639	12.536	91.423	70.398	45.421	93.019	57.799	38.883	110.836	11.518
12.0	PFE 1640	9.402	96.617	78.534	62.816	89.143	68.809	53.022	116.619	9.515

## APPENDIX XIII: Calculation of the gas flow rate necessary for fluidisation

There is an upper- and lower limit to the gas flow in a fluid bed. At the minimum velocity the bed of particles is just fluidised, while almost all of the particles are entrained at the maximum velocity. These two gas velocities  $u_{mf}$  and  $u_t$  were calculated for a bed of standard titania slag. The slag was separated into three narrow size fractions that was each roasted separately. This meant that the limiting gas velocities had to be calculated for each size fraction.

### Minimum gas velocity for fluidisation

The minimum superficial gas velocity necessary for fluidisation can be determined with the following empirical relationship (Perry & Green, 1984, p9-47):

$$\frac{1.75}{\phi_s \varepsilon_{mf}^3} \left( \frac{d_p u_{mf} \rho_g}{\mu} \right)^2 + \frac{150(1 - \varepsilon_{mf})}{\phi_s^2 \varepsilon_{mf}^3} \left( \frac{d_p u_{mf} \rho_g}{\mu} \right) = \frac{d_p^3 \rho_g (\rho_s - \rho_g) g}{\mu^2} \quad [1]$$

with

$u_{mf}$ =	Superficial gas velocity at minimum fluidisation conditions (cm.s <sup>-1</sup> )
$\phi_s$ =	Sphericity of the particles (dimensionless)
$\varepsilon_{mf}$ =	Void fraction of the bed at minimum fluidisation (dimensionless)
$d_p$ =	D <sub>50</sub> – mean diameter of the particles (cm)
$d_p$ =	D <sub>10</sub> – coarsest diameter of the finest 10 wt% of the particles (cm)
$\rho_g$ =	Gas density (g.cm <sup>-3</sup> )
$\rho_s$ =	Solid density (g.cm <sup>-3</sup> )
$\mu$ =	Gas viscosity (g.cm <sup>-1</sup> .s <sup>-1</sup> )
$g$ =	Acceleration of gravity (980 cm.s <sup>-2</sup> )

For standard titania slag the following values were used:

		+150-300µm	+300-500µm	+500-700µm
$\phi_s$	The value for coal was used (Perry and Green, 1984)	0.63	0.63	0.63
$\varepsilon_{mf}$	Taken from Fig. 9-50 from the values of sharp sand (Perry and Green, 1984)	0.53	0.48	0.20
$d_p$	Experimentally determined	0.021	0.038	0.060
$d_p$	Experimentally determined	0.016	0.031	0.055

### Gas density

The following five mixtures of Air and CO<sub>2</sub> were considered:

Air	Air:CO <sub>2</sub>			CO <sub>2</sub>
	5.7:4.3	3.8:6.2	1.9:8.1	
21% O <sub>2</sub>	12% O <sub>2</sub>	8% O <sub>2</sub>	4% O <sub>2</sub>	

- at the following four temperatures: 20, 750, 800, 850 and 950°C.

The density,  $\rho$ , of CO<sub>2</sub> at these temperatures were calculated with the following equation:



$$\rho = \frac{n}{V} \cdot M = \frac{PM}{ZRT'} \quad [2]$$

with:

- P** = Atmospheric pressure (0.85 atm)  
**M** = Molecular mass of CO<sub>2</sub> (44.01 g.mol<sup>-1</sup>)  
**R** = Universal gas constant (8.31451 J.mol<sup>-1</sup>.K<sup>-1</sup>)  
**T'** = Gas temperature (K)  
**Z** = Compressibility factor (~1.00 at high temperatures and 0.996 at 20°C)

The density of dry air at 0.85 atm can be calculated from the published density at 1 atm (Holman, 1976, p503). Gas density at two pressures are related by the following equation:

$$\frac{\rho_2}{P_2} = \frac{\rho_1}{P_1} \quad [3]$$

The following table lists the calculated density of the different gas mixtures at 0.85 atm.

**Gas densities,  $\rho$ , at 0.85 atm ( $\times 10^{-3}$  g.cm<sup>-3</sup>)**

Temperature °C	Gas composition				
	Air	Air – CO <sub>2</sub> mixtures			CO <sub>2</sub>
		4% O <sub>2</sub>	8% O <sub>2</sub>	12% O <sub>2</sub>	
20	1.023	1.460	1.357	1.239	1.562
750	0.293	0.415	0.387	0.353	0.444
800	0.279	0.396	0.369	0.337	0.424
850	0.267	0.378	0.352	0.322	0.405
950	0.246	0.348	0.324	0.296	0.372

### Solid density

The density of standard titania slag was measured as 3.84 g.cm<sup>-3</sup>.

### Viscosity

The viscosity of air and CO<sub>2</sub> are listed in the following table (Perry & Green, 1984, p3-250). This shows that the variation in viscosity between the two gasses at the temperatures under consideration is very small.

**Gas viscosities,  $\mu$ , ( $\times 10^{-2}$  g.cm<sup>-1</sup>.s<sup>-1</sup>)**

Temperature °C	Gas	
	Air	CO <sub>2</sub>
750	0.044	0.048
800	0.045	0.049
850	0.047	0.051
950	0.048	0.055

Let  $\mu$  be 0.048 g.cm<sup>-1</sup>.s<sup>-1</sup> for all temperatures and compositions under consideration.

With all the physical properties known Equation 1 can be solved for  $u_{mf}$ .

### Gas flow rates at minimum fluidisation

The results for an 8% O<sub>2</sub> gas mixture are shown in the following table.

### Gas flow rates at minimum fluidisation

Temperature (°C)	$u_{mf}$ (cm.s <sup>-1</sup> )			Gas flow rates (+300-500µm)	
	+150-300µm	+300-500µm	+500-700µm	At T (cm <sup>3</sup> .min <sup>-1</sup> )	At 20°C (cm <sup>3</sup> .min <sup>-1</sup> )
750	2.89	6.35	0.746	1197	343
800	2.89	6.35	0.746	1197	343
850	2.89v	6.35	0.746	1197	343
950	2.89	6.35	0.746	1197	343

- The bed cross section was  $\Pi d^2/4 = 3.14(2)^2/4 = 3.14 \text{ cm}^2$
- The flow at two temperatures are related by the following equation:  $V_2 = V_1 \cdot T_2/T_1$

### Maximum fluidising velocity

The terminal fluidising velocity is given by the following equation (Kunii & Levenspiel, 1977):

$$u_t = d_p \cdot \sqrt[3]{\frac{4 (\rho_s - \rho_g)^2 g^2}{225 \rho_g \mu}} \quad [4]$$

for  $0.4 < Re_p < 500$ , where:

$$Re_p = \frac{d_p u_t \rho_g}{\mu} \quad [5]$$

The maximum fluidisation velocity was calculated for the +300-500µm fraction in an 8% O<sub>2</sub> gas mixture and is shown in the following table.

### Gas flow rates at maximum fluidisation

Temperature T (°C)	$u_t$ (cm.s <sup>-1</sup> )	Gas flow rate		$u/u_{mf}$
		At T (cm <sup>3</sup> .min <sup>-1</sup> )	at 20°C (cm <sup>3</sup> .min <sup>-1</sup> )	
750	343.04	64629	18510	54
800	348.53	65663	17930	55
850	354.06	66705	17403	56
950	363.98	68574	16429	57

- The bed cross section was  $\Pi d^2/4 = 3.14(2)^2/4 = 3.14 \text{ cm}^2$
- The flow at two temperatures are related by the following equation:  $V_2 = V_1 \cdot T_2/T_1$

Based on the above calculations the following conditions for fluidisation was chosen:

**Conditions of fluidisation for +300-500µm particles in an 8% O<sub>2</sub> atmosphere at 850°C compared with the incipient and terminal conditions**

Gas flow rate		$u_g$ (cm.s <sup>-1</sup> )	$u/u_{mf}$ (cm.s <sup>-1</sup> )	$u/u_t$ (cm.s <sup>-1</sup> )
At 20°C (cm <sup>3</sup> .min <sup>-1</sup> )	At 850°C (cm <sup>3</sup> .min <sup>-1</sup> )			
11000	42160	224	35.2	0.63

## APPENDIX XIV: Chemical composition profile data

### PFE1172: Particle 1

Distance from centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.49	0.25	0.04	0.02	1.29	1.39	8.96	0.03	92.82	0.16	0.02	106.47
5	1.45	0.22	0.04	0.00	1.30	1.37	8.84	0.05	92.84	0.14	0.01	106.26
10	1.35	0.23	0.04	0.02	1.21	1.27	8.78	0.07	92.97	0.16	0.01	106.11
15	1.38	0.25	0.03	0.02	1.29	1.36	8.26	0.15	93.18	0.14	0.02	106.07
20	1.46	0.24	0.04	0.08	1.30	1.35	8.69	0.06	93.21	0.14	0.01	106.57
25	1.35	0.26	0.04	0.01	1.26	1.34	8.58	0.05	93.00	0.15	0.02	106.05
30	1.30	0.26	0.04	0.01	1.33	1.39	8.46	0.02	93.40	0.13	0.01	106.34
35	1.22	0.23	0.03	0.06	1.28	1.31	8.32	0.04	93.32	0.14	0.01	105.96
40	1.09	0.24	0.03	0.02	1.45	1.31	8.25	0.03	93.76	0.13	0.01	106.33
45	1.13	0.22	0.03	0.02	1.30	1.38	8.14	0.02	94.17	0.14	0.01	106.56
50	1.05	0.20	0.04	0.04	1.33	1.42	8.04	0.04	94.11	0.15	0.01	106.41
55	1.06	0.22	0.04	0.04	1.37	1.42	7.53	0.04	94.22	0.13	0.01	106.07
60	1.09	0.22	0.04	0.00	1.35	1.40	7.91	0.03	94.20	0.15	0.01	106.39
65	1.11	0.23	0.04	0.00	1.24	1.39	7.95	0.04	94.17	0.16	0.00	106.32
70	1.12	0.22	0.04	0.01	1.38	1.38	7.98	0.02	94.33	0.14	0.01	106.63
75	1.16	0.23	0.03	0.08	1.35	1.38	8.01	0.04	94.36	0.15	0.01	106.79
80	1.16	0.26	0.04	0.05	1.40	1.27	8.06	0.02	94.48	0.14	0.01	106.87
85	1.14	0.25	0.04	0.03	1.37	1.44	8.03	0.03	94.20	0.13	0.01	106.66
90	1.12	0.22	0.04	0.06	1.38	1.40	8.13	0.04	94.02	0.13	0.00	106.53
95	1.10	0.25	0.04	0.01	1.42	1.32	8.05	0.04	94.33	0.14	0.02	106.70
100	1.08	0.25	0.03	0.01	1.36	1.35	8.08	0.04	94.03	0.15	0.01	106.39
105	1.07	0.28	0.04	0.01	1.40	1.37	8.21	0.04	93.78	0.14	0.01	106.35
110	1.05	0.22	0.04	0.06	1.36	1.31	8.18	0.03	93.68	0.15	0.02	106.09
115	1.00	0.24	0.04	0.02	1.40	1.36	8.14	0.03	93.93	0.14	0.01	106.29

Distance from centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
120	1.10	0.25	0.04	0.01	1.43	1.36	8.02	0.02	93.96	0.13	0.01	106.33
125	0.98	0.21	0.04	0.02	1.48	1.32	8.25	0.04	93.65	0.14	0.01	106.14
130	0.99	0.20	0.04	0.01	1.43	1.28	8.10	0.04	93.65	0.13	0.01	105.88
135	0.96	0.25	0.04	0.08	1.44	1.43	8.04	0.03	94.04	0.14	0.01	106.45
140	0.97	0.22	0.04	0.03	1.43	1.30	8.04	0.03	94.40	0.13	0.00	106.58
145	0.95	0.24	0.04	0.03	1.48	1.33	7.93	0.03	94.51	0.14	0.01	106.69
150	0.91	0.27	0.04	0.05	1.52	1.38	7.94	0.04	94.09	0.15	0.01	106.38
155	0.90	0.23	0.04	0.05	1.47	1.46	7.98	0.03	94.02	0.14	0.01	106.32
160	0.92	0.25	0.03	0.00	1.38	1.31	8.04	0.04	94.20	0.13	0.01	106.31
165	0.98	0.22	0.03	0.05	1.46	1.40	7.83	0.02	92.84	0.14	0.01	104.99
170	0.73	0.21	0.04	0.00	0.96	1.13	6.74	0.04	91.06	0.09	0.01	100.99
175	1.01	0.22	0.04	0.02	1.32	1.30	7.79	0.08	93.51	0.13	0.01	105.42
180	0.98	0.25	0.04	0.00	1.38	1.32	7.98	0.04	94.06	0.13	0.01	106.19
185	1.07	0.23	0.03	0.00	1.29	1.20	7.12	0.03	93.33	0.13	0.01	104.43
190	1.04	0.19	0.04	0.03	1.40	1.35	8.29	0.01	93.34	0.14	0.00	105.83
195	1.00	0.19	0.04	0.00	1.36	1.17	6.07	0.04	93.49	0.11	0.01	103.48
200	1.09	0.24	0.04	0.01	1.44	0.70	3.14	0.03	94.13	0.14	0.01	100.96
205	1.08	0.17	0.04	0.00	1.46	0.97	3.12	0.02	93.23	0.13	0.00	100.23
210	1.02	0.22	0.03	0.03	1.31	0.69	2.89	0.02	94.16	0.11	0.01	100.49
215	1.47	0.23	0.04	0.06	1.95	1.03	9.48	0.03	83.16	0.16	0.01	97.61
220	0.85	0.21	0.03	0.05	0.79	0.71	6.11	0.03	91.27	0.10	0.01	100.15
225	0.14	0.19	0.03	0.04	0.15	0.50	1.06	0.03	97.87	0.07	0.01	100.10
230	0.52	0.20	0.05	0.06	2.46	3.58	31.74	0.18	58.81	0.22	0.02	97.82

**PFE1172: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.18	0.22	0.03	0.06	1.46	1.41	8.91	0.05	92.72	0.15	0.01	106.20
5	1.12	0.22	0.03	0.07	1.51	1.37	8.87	0.02	92.35	0.14	0.02	105.72
10	1.14	0.22	0.04	0.00	1.46	1.32	8.84	0.03	92.61	0.15	0.00	105.82
15	1.09	0.26	0.04	0.00	1.37	1.39	8.82	0.04	92.77	0.14	0.02	105.94
20	1.15	0.20	0.04	0.06	1.51	1.42	8.76	0.03	92.84	0.14	0.01	106.16
25	1.13	0.23	0.04	0.00	1.58	1.46	8.54	0.02	93.09	0.12	0.01	106.21
30	1.09	0.26	0.04	0.03	1.52	1.46	8.52	0.02	92.95	0.14	0.02	106.05
35	1.05	0.25	0.04	0.02	1.49	1.33	8.47	0.03	92.93	0.15	0.01	105.76
40	1.04	0.26	0.04	0.01	1.35	1.34	6.91	0.03	93.58	0.13	0.01	104.69
45	1.07	0.24	0.04	0.06	1.54	1.36	8.45	0.03	93.24	0.13	0.01	106.16
50	1.01	0.22	0.04	0.07	1.58	1.40	8.53	0.02	93.45	0.13	0.00	106.44
55	1.02	0.21	0.05	0.01	1.53	1.42	8.43	0.03	93.31	0.14	0.01	106.14
60	1.03	0.23	0.04	0.02	1.57	1.49	8.34	0.03	93.08	0.12	0.01	105.96
65	1.02	0.15	0.04	0.06	1.53	1.30	6.95	0.03	93.32	0.13	0.01	104.55
70	1.14	0.25	0.04	0.06	1.66	1.32	8.00	0.01	92.95	0.13	0.01	105.58
75	1.02	0.21	0.04	0.05	1.69	1.39	8.40	0.02	93.22	0.13	0.02	106.19
80	0.94	0.19	0.04	0.00	1.58	1.47	8.45	0.03	92.98	0.13	0.01	105.81
85	0.99	0.21	0.04	0.01	1.51	1.44	8.45	0.02	93.19	0.14	0.01	105.99
90	1.01	0.20	0.04	0.07	1.61	1.46	8.50	0.02	93.15	0.13	0.00	106.20
95	0.97	0.21	0.04	0.05	1.54	1.41	8.41	0.02	93.48	0.13	0.02	106.27
100	0.99	0.22	0.04	0.03	1.63	1.39	8.41	0.01	93.20	0.13	0.01	106.04
105	1.01	0.21	0.04	0.01	1.64	1.37	8.41	0.03	92.83	0.14	0.00	105.67
110	0.91	0.23	0.04	0.07	1.71	1.44	10.35	0.01	91.09	0.12	0.01	105.98
115	1.00	0.20	0.04	0.00	1.72	1.53	8.51	0.03	92.99	0.13	0.01	106.15
120	0.96	0.20	0.03	0.00	1.63	1.41	8.59	0.03	92.93	0.12	0.01	105.89

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
125	0.98	0.21	0.03	0.08	1.72	1.32	8.59	0.03	93.10	0.12	0.01	106.19
130	0.96	0.26	0.04	0.10	1.68	1.34	8.60	0.02	93.07	0.14	0.00	106.20
135	0.95	0.21	0.04	0.12	1.69	1.37	8.67	0.03	92.68	0.12	0.01	105.89
140	0.99	0.22	0.04	0.08	1.63	1.34	8.32	0.02	92.14	0.13	0.00	104.92
145	0.98	0.23	0.03	0.05	1.68	1.42	8.75	0.02	92.72	0.13	0.01	106.02
150	0.99	0.26	0.04	0.04	1.59	1.40	8.92	0.01	92.32	0.12	0.01	105.69
155	1.03	0.21	0.03	0.07	1.53	1.43	9.10	0.02	92.21	0.14	0.01	105.78
160	1.06	0.23	0.04	0.04	1.64	1.46	9.02	0.04	92.12	0.14	0.01	105.80
165	1.00	0.20	0.03	0.08	1.61	1.40	9.06	0.03	92.49	0.13	0.00	106.05
170	1.04	0.27	0.04	0.04	1.68	1.41	9.06	0.03	92.19	0.14	0.01	105.90
175	1.13	0.20	0.03	0.02	1.61	1.31	7.91	0.02	91.32	0.14	0.01	103.70
180	0.94	0.20	0.03	0.02	1.22	0.84	3.54	0.02	93.03	0.10	0.01	99.95
185	1.18	0.23	0.04	0.03	1.77	1.02	7.33	0.03	89.92	0.13	0.00	101.67
190	0.76	0.16	0.04	0.05	0.76	0.69	1.34	0.03	96.83	0.11	0.00	100.76
195	1.22	0.19	0.03	0.02	1.26	0.95	2.35	0.02	94.51	0.15	0.01	100.70
200	1.21	0.22	0.04	0.01	1.42	0.99	3.49	0.01	93.45	0.12	0.01	100.96
205	1.13	0.21	0.03	0.01	1.33	0.97	4.66	0.01	91.48	0.15	0.01	100.00
210	1.11	0.23	0.03	0.02	0.94	1.21	4.27	0.02	92.48	0.21	0.00	100.52
215	0.61	0.18	0.03	0.05	0.90	0.68	4.18	0.03	93.90	0.12	0.01	100.68
220	0.93	0.22	0.03	0.00	1.30	1.54	7.20	0.04	88.76	0.17	0.02	100.20
225	0.32	0.22	0.03	0.00	0.45	0.66	1.99	0.02	96.45	0.13	0.02	100.30
230	0.87	0.16	0.03	0.04	1.65	1.12	23.55	0.01	58.52	0.13	0.00	86.07
235	1.07	0.18	0.03	0.07	0.93	0.96	9.21	0.04	84.42	0.15	0.01	97.07
240	2.44	0.18	0.04	0.11	1.77	1.53	63.81	0.02	17.13	0.00	0.02	87.05



**PFE1172: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.31	0.25	0.04	0.00	1.25	1.31	9.63	0.04	92.16	0.15	0.02	106.15
5	1.43	0.21	0.04	0.00	1.31	1.32	9.51	0.03	91.63	0.15	0.00	105.62
10	1.41	0.29	0.04	0.02	1.30	1.31	9.50	0.05	91.80	0.16	0.02	105.90
15	1.30	0.26	0.04	0.04	1.30	3.09	9.13	0.11	90.79	0.13	0.02	106.22
20	1.36	0.22	0.05	0.04	1.26	1.39	9.57	0.05	91.96	0.14	0.01	106.05
25	1.37	0.28	0.04	0.07	1.27	1.36	9.20	0.04	91.62	0.14	0.00	105.37
30	1.48	0.25	0.04	0.07	1.37	1.43	10.73	0.03	90.78	0.16	0.02	106.34
35	1.38	0.23	0.03	0.04	1.30	1.38	9.60	0.06	91.97	0.15	0.01	106.14
40	1.35	0.27	0.04	0.06	1.28	1.36	9.66	0.05	91.85	0.14	0.01	106.05
45	1.39	0.20	0.04	0.07	1.35	1.41	9.55	0.05	91.90	0.14	0.01	106.11
50	1.33	0.26	0.04	0.03	1.30	1.33	9.35	0.07	91.73	0.14	0.01	105.59
55	1.48	0.25	0.04	0.10	1.29	1.33	9.58	0.03	91.82	0.14	0.01	106.07
60	1.46	0.26	0.04	0.04	1.29	1.30	9.68	0.03	92.04	0.15	0.02	106.30
65	1.40	0.27	0.04	0.05	1.23	1.36	9.65	0.03	91.66	0.14	0.01	105.83
70	1.42	0.27	0.04	0.04	1.26	1.35	9.64	0.04	91.77	0.16	0.01	105.99
75	1.38	0.26	0.04	0.05	1.27	1.33	9.61	0.04	91.54	0.15	0.01	105.66
80	1.39	0.21	0.04	0.03	1.29	1.30	9.76	0.04	91.82	0.15	0.00	106.04
85	1.40	0.22	0.03	0.05	1.25	1.37	9.78	0.04	91.61	0.14	0.00	105.89
90	1.40	0.21	0.04	0.00	1.28	1.35	9.65	0.04	91.84	0.14	0.01	105.96
95	1.50	0.23	0.04	0.05	1.30	1.30	9.78	0.05	91.87	0.15	0.01	106.28
100	1.40	0.24	0.04	0.05	1.25	1.34	9.72	0.05	91.55	0.14	0.00	105.78
105	1.39	0.21	0.04	0.06	1.28	1.29	10.01	0.02	91.60	0.15	0.01	106.05
110	1.36	0.23	0.04	0.05	1.28	1.25	9.88	0.03	91.85	0.14	0.01	106.13
115	1.37	0.24	0.03	0.04	1.17	1.40	9.85	0.03	91.84	0.15	0.01	106.11
120	1.47	0.21	0.04	0.06	1.31	1.37	9.79	0.04	91.67	0.15	0.01	106.11
125	1.46	0.24	0.04	0.04	1.24	1.35	9.78	0.06	91.58	0.14	0.01	105.94
130	1.43	0.26	0.04	0.04	1.31	1.33	9.84	0.04	91.62	0.14	0.02	106.07
135	1.43	0.21	0.04	0.06	1.33	1.25	9.83	0.05	91.43	0.14	0.00	105.77
140	1.39	0.24	0.04	0.00	1.23	1.26	9.95	0.04	91.53	0.15	0.01	105.83

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
145	1.43	0.28	0.04	0.06	1.24	1.29	9.84	0.04	91.70	0.15	0.01	106.08
150	1.40	0.23	0.03	0.06	1.36	1.35	9.91	0.07	91.51	0.14	0.02	106.08
155	1.41	0.24	0.03	0.07	1.33	1.36	9.90	0.05	91.61	0.16	0.01	106.16
160	1.44	0.25	0.04	0.02	1.26	1.27	9.87	0.01	91.73	0.14	0.01	106.03
165	1.17	0.24	0.04	0.06	1.30	1.35	8.76	0.09	91.34	0.14	0.02	104.51
170	1.42	0.25	0.04	0.05	1.35	1.29	9.85	0.05	91.69	0.14	0.01	106.13
175	1.40	0.28	0.04	0.01	1.27	7.34	9.32	0.11	87.89	0.14	0.01	107.79
180	1.38	0.23	0.05	0.07	1.18	3.99	9.58	0.16	88.31	0.14	0.04	105.13
185	1.45	0.22	0.03	0.02	1.35	1.33	9.84	0.04	91.78	0.15	0.01	106.22
190	1.39	0.23	0.03	0.03	1.25	1.36	10.95	0.10	90.18	0.14	0.01	105.66
195	1.45	0.21	0.04	0.03	1.28	1.32	9.90	0.03	91.63	0.15	0.01	106.04
200	1.22	0.27	0.03	0.04	1.25	1.22	9.77	0.15	90.14	0.13	0.02	104.24
205	1.40	0.20	0.03	0.03	1.29	1.32	9.83	0.04	91.78	0.15	0.01	106.06
210	1.39	0.24	0.03	0.04	1.19	1.34	9.79	0.05	91.41	0.15	0.01	105.64
215	1.38	0.22	0.03	0.04	1.20	1.06	3.83	0.04	94.39	0.12	0.00	102.31
220	1.35	0.20	0.03	0.05	1.00	0.75	1.02	0.05	95.99	0.09	0.01	100.55
225	1.24	0.25	0.03	0.00	1.18	1.25	1.70	0.04	94.89	0.16	0.01	100.75
230	1.24	0.22	0.04	0.02	1.05	2.06	2.67	0.16	92.29	0.22	0.01	99.96
235	0.93	0.20	0.03	0.03	0.63	1.87	1.77	0.03	93.29	0.18	0.01	98.99
240	1.48	0.18	0.03	0.06	1.18	0.96	2.32	0.04	93.77	0.14	0.01	100.18
245	1.54	0.22	0.03	0.00	1.48	1.11	4.83	0.07	88.86	0.15	0.01	98.31
250	1.24	0.22	0.03	0.00	1.15	0.98	2.80	0.06	90.24	0.15	0.01	96.88
255	1.32	0.18	0.04	0.00	1.07	0.99	3.71	0.03	93.03	0.17	0.01	100.55
260	0.49	0.22	0.04	0.06	0.51	0.71	1.85	0.03	96.15	0.14	0.01	100.21
265	1.75	0.25	0.04	0.00	1.63	0.88	16.33	0.08	76.25	0.20	0.01	97.41
270	0.69	0.21	0.04	0.03	0.84	0.77	10.84	0.05	86.41	0.14	0.01	100.02
275	0.83	0.20	0.04	0.03	1.87	1.35	22.52	0.06	70.24	0.17	0.02	97.34
280	0.41	0.23	0.04	0.09	1.25	3.68	48.45	0.01	43.96	0.02	0.01	98.13

**PFE1173: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.94	0.23	0.04	0.02	1.53	1.39	7.19	0.02	94.85	0.12	0.01	106.33
5	0.90	0.22	0.03	0.01	1.64	1.45	7.23	0.04	94.82	0.13	0.01	106.48
10	0.94	0.22	0.04	0.06	1.66	1.36	7.25	0.02	95.05	0.13	0.02	106.75
15	0.90	0.22	0.04	0.06	1.58	1.43	7.21	0.02	94.74	0.13	0.00	106.34
20	0.92	0.24	0.04	0.00	1.52	1.42	7.20	0.03	94.65	0.14	0.01	106.16
25	1.00	0.28	0.04	0.01	1.60	1.42	7.18	0.03	94.64	0.13	0.01	106.35
30	0.94	0.21	0.03	0.05	1.52	1.43	7.34	0.02	94.76	0.14	0.01	106.46
35	0.96	0.25	0.03	0.03	1.49	1.35	7.41	0.02	94.05	0.12	0.01	105.71
40	1.04	0.26	0.04	0.04	1.51	1.38	7.38	0.02	94.46	0.14	0.01	106.28
45	1.01	0.23	0.04	0.10	1.47	1.32	7.52	0.02	94.30	0.12	0.00	106.12
50	0.96	0.28	0.03	0.04	1.48	1.34	7.53	0.02	94.39	0.13	0.01	106.19
55	1.07	0.21	0.04	0.01	1.53	1.41	7.47	0.03	94.28	0.14	0.01	106.21
60	1.06	0.24	0.04	0.08	1.50	1.43	7.49	0.05	94.64	0.13	0.00	106.66
65	1.03	0.23	0.04	0.06	1.52	1.37	7.53	0.03	94.35	0.13	0.01	106.29
70	0.99	0.26	0.04	0.04	1.38	1.37	7.67	0.02	94.29	0.14	0.00	106.19
75	1.16	0.24	0.04	0.04	1.47	1.43	7.11	0.03	93.76	0.13	0.02	105.42
80	0.96	0.26	0.04	0.07	1.50	1.40	7.65	0.02	94.05	0.14	0.01	106.10
85	1.07	0.26	0.04	0.04	1.44	1.43	7.76	0.04	93.86	0.14	0.01	106.09
90	0.70	0.24	0.03	0.11	1.37	1.36	11.85	0.04	89.98	0.12	0.01	105.80
95	1.03	0.26	0.04	0.07	1.39	1.42	7.92	0.02	93.78	0.15	0.01	106.08
100	0.87	0.24	0.04	0.08	1.36	1.33	6.66	0.03	93.58	0.12	0.01	104.33
105	1.06	0.22	0.04	0.01	1.47	1.41	8.95	0.03	92.71	0.13	0.00	106.02
110	1.09	0.23	0.04	0.04	1.48	1.43	8.04	0.04	93.59	0.13	0.01	106.11
115	1.07	0.29	0.04	0.02	1.42	1.34	8.05	0.04	93.92	0.13	0.01	106.34
120	1.19	0.26	0.04	0.01	1.43	1.40	8.16	0.03	94.05	0.14	0.02	106.71
125	1.01	0.22	0.04	0.05	1.34	1.26	7.11	0.04	93.15	0.13	0.01	104.36
130	1.11	0.22	0.04	0.03	1.38	1.13	6.31	0.03	93.04	0.13	0.01	103.42
135	1.23	0.24	0.03	0.07	1.68	1.16	5.60	0.03	92.70	0.15	0.01	102.89
140	1.16	0.23	0.04	0.01	1.47	0.97	4.67	0.04	93.18	0.12	0.01	101.88
145	1.05	0.20	0.04	0.04	1.19	0.96	3.95	0.05	93.86	0.11	0.01	101.47
150	0.99	0.19	0.04	0.03	1.24	0.88	3.51	0.04	94.28	0.12	0.01	101.31

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
155	1.15	0.25	0.04	0.09	1.39	0.90	3.74	0.04	93.42	0.13	0.00	101.14
160	1.46	0.22	0.04	0.04	1.80	1.51	6.57	0.04	90.08	0.13	0.01	101.89
165	1.33	0.22	0.04	0.03	1.44	1.01	4.68	0.04	92.42	0.13	0.01	101.34
170	1.13	0.22	0.04	0.00	1.32	1.35	5.41	0.03	91.20	0.12	0.01	100.80
175	0.34	0.25	0.03	0.01	0.50	0.72	1.73	0.03	96.56	0.16	0.01	100.35
180	0.84	0.25	0.03	0.05	0.80	0.74	1.76	0.03	96.50	0.13	0.01	101.16
185	1.33	0.24	0.04	0.02	1.48	0.96	4.68	0.04	92.06	0.15	0.02	101.02
190	1.28	0.23	0.03	0.03	1.44	1.52	5.74	0.04	90.84	0.17	0.01	101.31
195	1.31	0.19	0.04	0.07	1.22	0.76	5.17	0.03	91.88	0.11	0.00	100.78
200	1.36	0.21	0.03	0.06	1.39	0.92	5.20	0.04	91.88	0.14	0.01	101.22
205	1.57	0.21	0.04	0.01	1.94	0.96	7.36	0.03	88.79	0.14	0.01	101.05
210	1.49	0.23	0.04	0.01	1.51	0.79	5.59	0.04	90.99	0.13	0.00	100.82
215	1.31	0.20	0.04	0.03	1.06	0.86	6.48	0.03	90.62	0.13	0.01	100.75
220	3.75	0.22	0.04	0.04	3.07	18.51	27.13	0.13	48.00	0.49	0.11	101.48
225	0.51	0.16	0.04	0.06	1.01	0.86	7.02	0.02	90.17	0.06	0.01	99.89
230	1.58	0.18	0.04	0.01	1.51	1.10	8.07	0.06	87.83	0.14	0.01	100.51
235	0.74	0.24	0.04	0.05	0.53	1.72	1.38	0.10	96.36	0.16	0.02	101.32
240	1.92	0.23	0.04	0.00	1.80	1.07	4.79	0.02	90.86	0.15	0.01	100.89
245	0.71	0.19	0.03	0.04	0.85	1.05	3.73	0.05	93.79	0.17	0.01	100.61
250	1.24	0.21	0.03	0.05	1.16	0.99	3.90	0.03	92.80	0.14	0.02	100.56
255	1.31	0.22	0.04	0.04	1.21	0.99	4.28	0.04	92.71	0.16	0.01	101.01
260	1.25	0.22	0.04	0.03	1.23	0.92	4.43	0.05	92.21	0.13	0.01	100.51
265	1.32	0.22	0.04	0.04	1.24	2.09	13.59	0.05	77.47	0.15	0.01	96.21
270	1.22	0.19	0.03	0.06	0.77	0.92	4.31	0.06	92.83	0.14	0.02	100.54
275	0.16	0.24	0.04	0.08	0.16	0.55	0.50	0.01	99.06	0.12	0.01	100.92
280	1.22	0.19	0.03	0.03	1.28	1.77	8.27	0.09	85.84	0.18	0.01	98.91
285	0.54	0.22	0.04	0.01	0.24	0.63	1.84	0.03	96.02	0.12	0.00	99.69
290	3.39	0.24	0.04	0.00	2.76	1.76	37.29	0.02	52.92	0.10	0.00	98.52
295	1.14	0.21	0.03	0.03	1.36	2.00	8.31	0.05	87.61	0.16	0.02	100.91
300	0.18	0.19	0.04	0.05	0.05	13.06	1.14	0.20	84.13	0.11	0.08	99.24

**PFE1173: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.82	0.26	0.05	0.01	1.21	1.29	9.61	0.05	90.77	0.14	0.02	105.21
5	0.37	0.15	0.72	0.17	0.11	3.75	1.07	73.36	18.09	0.04	1.89	99.72
10	1.72	0.20	0.04	0.00	1.26	1.31	9.66	0.07	90.81	0.15	0.03	105.26
15	1.69	0.23	0.04	0.06	1.28	1.23	9.60	0.04	90.48	0.15	0.03	104.81
20	1.47	0.26	0.04	0.07	1.28	1.34	9.52	0.04	91.52	0.14	0.02	105.70
25	1.39	0.23	0.04	0.06	1.32	1.32	9.50	0.03	91.72	0.13	0.02	105.75
30	1.39	0.24	0.04	0.03	1.42	1.40	9.48	0.02	91.63	0.13	0.02	105.80
35	1.35	0.25	0.04	0.06	1.43	1.33	9.39	0.04	91.68	0.13	0.01	105.70
40	1.19	0.18	0.04	0.05	1.39	1.37	9.24	0.03	91.97	0.15	0.01	105.61
45	1.19	0.21	0.04	0.04	1.38	1.44	9.18	0.05	91.99	0.14	0.00	105.66
50	1.20	0.22	0.04	0.02	1.52	1.30	8.99	0.02	91.92	0.15	0.01	105.40
55	1.10	0.22	0.04	0.09	1.33	1.33	8.16	0.03	92.19	0.13	0.00	104.62
60	1.18	0.25	0.03	0.00	1.42	1.30	8.99	0.03	91.88	0.13	0.01	105.22
65	1.18	0.28	0.04	0.05	1.40	1.24	8.80	0.03	91.83	0.14	0.01	104.98
70	1.21	0.26	0.03	0.07	1.33	1.35	8.71	0.03	92.11	0.13	0.01	105.24
75	1.14	0.24	0.04	0.02	1.38	1.35	8.16	0.03	92.59	0.14	0.01	105.11
80	1.23	0.27	0.03	0.05	1.45	1.25	8.89	0.03	91.38	0.14	0.01	104.72
85	1.04	0.22	0.04	0.06	1.42	1.36	12.99	0.04	88.45	0.15	0.02	105.77

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
90	1.14	0.19	0.04	0.01	1.35	1.21	6.73	0.04	92.56	0.12	0.01	103.40
95	1.25	0.20	0.04	0.05	1.45	0.91	1.55	0.05	95.36	0.12	0.01	100.98
100	1.15	0.23	0.04	0.06	1.13	0.76	1.44	0.04	95.64	0.12	0.01	100.61
105	1.07	0.18	0.03	0.00	1.26	0.89	2.31	0.05	94.64	0.12	0.01	100.55
110	1.22	0.19	0.03	0.03	1.33	0.98	2.43	0.04	94.51	0.12	0.00	100.90
115	1.19	0.20	0.03	0.01	1.42	0.98	1.92	0.03	94.79	0.15	0.01	100.72
120	0.78	0.21	0.04	0.00	0.88	0.81	1.61	0.04	95.61	0.15	0.00	100.12
125	1.87	0.22	0.03	0.00	1.58	1.18	2.95	0.06	92.18	0.20	0.01	100.26
130	0.83	0.24	0.03	0.00	0.85	0.88	1.55	0.05	96.12	0.14	0.01	100.71
135	1.07	0.24	0.03	0.00	0.98	0.77	2.86	0.04	93.97	0.11	0.01	100.07
140	1.36	0.24	0.04	0.00	1.23	0.79	3.49	0.06	92.53	0.15	0.01	99.90
145	1.08	0.20	0.04	0.06	1.47	0.80	5.14	0.04	90.08	0.11	0.01	99.03
150	0.30	0.22	0.04	0.04	0.37	0.51	1.35	0.02	96.52	0.12	0.01	99.48
155	1.51	0.23	0.04	0.01	1.80	1.33	9.02	0.03	85.90	0.12	0.01	100.00
160	1.41	0.18	0.04	0.00	1.64	1.20	9.92	0.03	84.99	0.12	0.01	99.55
165	0.77	0.27	0.03	0.02	0.51	0.66	2.78	0.03	94.79	0.11	0.01	99.96
170	1.19	0.21	0.04	0.07	1.40	2.28	14.36	0.06	79.14	0.17	0.01	98.92

**PFE1173: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.84	0.23	0.04	0.03	1.62	1.30	6.93	0.03	95.06	0.13	0.01	106.21
5	0.88	0.26	0.04	0.03	1.68	1.40	7.13	0.01	94.99	0.12	0.00	106.54
10	0.83	0.23	0.04	0.06	1.62	1.43	7.03	0.02	95.00	0.12	0.00	106.38
15	0.86	0.21	0.04	0.03	1.61	1.46	7.08	0.02	94.64	0.13	0.01	106.08
20	0.92	0.25	0.03	0.08	1.56	1.40	7.14	0.02	94.37	0.13	0.01	105.90
25	0.89	0.21	0.04	0.05	1.61	1.38	7.23	0.02	94.90	0.13	0.01	106.46
30	0.90	0.26	0.04	0.06	1.55	1.39	7.31	0.03	94.37	0.13	0.01	106.04
35	0.95	0.24	0.04	0.04	1.66	1.39	7.40	0.02	94.55	0.13	0.01	106.44
40	0.91	0.25	0.03	0.08	1.56	1.41	7.58	0.02	94.30	0.12	0.00	106.26
45	0.90	0.25	0.04	0.07	1.57	1.33	7.42	0.01	93.70	0.13	0.00	105.43
50	0.93	0.23	0.04	0.01	1.55	1.36	7.84	0.03	93.94	0.12	0.00	106.03
55	0.88	0.22	0.04	0.05	1.50	1.44	7.89	0.02	93.76	0.13	0.01	105.93
60	0.95	0.22	0.06	0.94	1.78	1.36	7.61	0.02	92.81	0.12	0.09	105.96
65	1.01	0.20	0.03	0.03	1.62	1.40	8.11	0.03	93.09	0.13	0.01	105.66
70	0.90	0.27	0.03	0.02	1.59	1.26	7.91	0.02	92.95	0.14	0.01	105.11
75	0.89	0.22	0.03	0.01	1.39	1.27	4.67	0.01	93.65	0.12	0.01	102.27
80	0.94	0.22	0.03	0.04	1.47	1.02	3.53	0.01	94.67	0.11	0.00	102.05

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
85	1.06	0.21	0.03	0.02	1.48	1.05	3.88	0.03	93.55	0.12	0.01	101.44
90	0.98	0.20	0.03	0.04	1.34	0.80	2.72	0.04	94.77	0.13	0.00	101.05
95	1.10	0.21	0.03	0.04	1.35	0.88	3.20	0.03	93.89	0.12	0.01	100.85
100	0.96	0.18	0.04	0.05	1.25	0.87	2.94	0.01	94.51	0.13	0.01	100.96
105	1.10	0.18	0.03	0.06	1.46	0.79	4.03	0.04	92.77	0.10	0.01	100.56
110	1.15	0.20	0.03	0.02	1.24	0.93	2.76	0.04	94.25	0.11	0.01	100.74
115	1.62	0.21	0.03	0.02	1.75	1.23	4.54	0.03	91.05	0.15	0.01	100.65
120	1.29	0.21	0.04	0.00	1.42	0.90	3.97	0.03	92.67	0.11	0.02	100.65
125	1.08	0.16	0.03	0.03	0.97	1.31	5.13	0.05	88.91	0.15	0.00	97.81
130	1.61	0.21	0.03	0.02	1.59	1.23	6.51	0.03	88.48	0.16	0.02	99.89
135	1.41	0.20	0.03	0.03	1.73	1.20	7.94	0.04	85.57	0.15	0.02	98.32
140	3.51	0.21	0.03	0.09	2.93	3.10	20.16	0.05	68.68	0.18	0.01	98.94
145	2.36	0.21	0.03	0.10	1.91	2.00	14.38	0.06	78.34	0.13	0.01	99.52
150	1.66	0.17	0.03	0.01	1.66	2.09	18.10	0.06	74.92	0.14	0.03	98.87
155	2.29	0.16	0.04	0.08	1.37	3.30	34.43	0.04	55.86	0.04	0.01	97.62



**PFE986: Particle1**

Distance from Centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.80	0.25	0.04	0.03	1.71	1.42	4.63	0.01	95.41	0.13	0.01	104.44
5	0.64	0.23	0.04	0.05	1.30	1.37	3.47	0.01	96.24	0.11	0.02	103.47
10	0.78	0.23	0.03	0.05	1.36	1.21	12.88	0.02	89.94	0.10	0.01	106.61
15	0.82	0.21	0.04	0.02	1.67	1.27	4.54	0.03	95.75	0.12	0.00	104.47
20	0.85	0.26	0.03	0.05	1.72	1.47	6.73	0.02	94.83	0.13	0.01	106.10
25	0.45	0.25	0.03	0.00	0.72	0.73	6.36	0.01	93.90	0.07	0.01	102.54
30	0.79	0.24	0.04	0.03	1.54	1.37	6.46	0.02	95.14	0.11	0.01	105.74
35	0.81	0.24	0.03	0.07	1.72	1.45	6.29	0.02	95.01	0.12	0.01	105.77
40	0.78	0.22	0.03	0.01	1.92	1.56	4.55	0.04	95.16	0.12	0.01	104.39
45	0.70	0.23	0.03	0.01	1.58	1.36	4.16	0.01	95.50	0.10	0.01	103.70
50	0.67	0.23	0.03	0.02	1.35	1.20	3.37	0.00	96.03	0.10	0.01	103.01
55	0.83	0.26	0.04	0.05	1.62	1.22	4.63	0.03	94.68	0.11	0.01	103.48
60	0.77	0.22	0.03	0.04	1.74	1.22	3.46	0.03	95.17	0.10	0.01	102.78
65	0.65	0.22	0.04	0.02	1.22	0.78	1.59	0.02	96.77	0.08	0.00	101.36
70	0.53	0.22	0.04	0.06	1.10	0.70	0.81	0.02	97.30	0.07	0.01	100.85
75	0.82	0.23	0.04	0.01	1.34	0.76	0.78	0.03	96.89	0.07	0.00	100.97
80	0.82	0.19	0.04	0.04	1.37	0.85	0.56	0.01	97.20	0.10	0.01	101.17
85	0.87	0.21	0.04	0.00	1.44	0.88	0.57	0.05	96.67	0.10	0.01	100.85
90	0.55	0.22	0.04	0.02	0.92	0.73	0.30	0.02	97.81	0.07	0.01	100.68
95	0.62	0.22	0.03	0.05	0.92	0.78	0.41	0.02	97.58	0.10	0.01	100.74
100	0.66	0.18	0.03	0.01	0.94	0.74	0.34	0.02	97.46	0.09	0.02	100.49
105	0.66	0.19	0.03	0.01	1.12	0.93	0.43	0.02	96.92	0.11	0.01	100.43
110	0.79	0.21	0.04	0.03	1.28	0.86	0.50	0.03	96.98	0.11	0.01	100.83
115	0.64	0.26	0.03	0.00	0.95	0.70	0.41	0.03	97.01	0.08	0.00	100.09
120	1.10	0.23	0.04	0.02	1.99	0.97	0.94	0.03	95.24	0.13	0.01	100.70
125	0.74	0.25	0.04	0.00	1.27	0.90	0.65	0.03	96.35	0.10	0.01	100.33
130	0.70	0.19	0.03	0.00	1.18	0.70	0.57	0.01	96.83	0.09	0.00	100.31
135	0.94	0.22	0.03	0.01	1.31	0.78	0.79	0.02	96.14	0.10	0.01	100.32
140	1.10	0.23	0.03	0.00	1.83	1.05	1.38	0.00	91.99	0.13	0.00	97.75
145	0.29	0.22	0.03	0.01	1.07	0.83	1.19	0.01	94.12	0.15	0.00	97.93
150	0.70	0.24	0.03	0.01	0.66	0.86	0.83	0.03	94.51	0.07	0.01	97.94

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
155	1.40	0.21	0.04	0.03	1.88	0.71	1.87	0.04	93.58	0.12	0.01	99.87
160	0.30	0.21	0.04	0.00	0.43	0.51	0.49	0.04	97.14	0.04	0.00	99.21
165	0.70	0.19	0.03	0.00	0.61	0.80	1.88	0.03	94.61	0.12	0.01	99.00
170	1.06	0.19	0.04	0.01	1.56	0.68	2.97	0.03	92.34	0.10	0.00	98.98
175	0.99	0.17	0.04	0.00	2.25	3.03	11.23	0.06	77.58	0.18	0.02	95.54
180	0.88	0.23	0.03	0.00	1.54	1.93	9.27	0.01	85.83	0.15	0.00	99.87
185	0.67	0.24	0.03	0.00	1.06	0.60	2.20	0.01	95.55	0.06	0.01	100.43
190	0.78	0.22	0.04	0.01	1.19	0.62	2.56	0.03	94.35	0.06	0.01	99.87
195	0.57	0.19	0.03	0.04	1.04	0.67	3.02	0.03	94.25	0.10	0.00	99.93
200	0.96	0.22	0.04	0.00	1.61	1.84	7.35	0.02	87.54	0.17	0.01	99.75
205	1.13	0.20	0.04	0.00	1.83	0.88	5.09	0.02	91.15	0.09	0.01	100.43
210	0.34	0.23	0.04	0.01	0.58	0.68	1.79	0.01	96.41	0.10	0.01	100.19
215	1.45	0.19	0.04	0.00	1.88	1.01	5.99	0.04	88.52	0.13	0.01	99.25
220	1.19	0.23	0.03	0.00	1.55	0.80	4.16	0.02	91.86	0.09	0.01	99.93
225	0.40	0.24	0.04	0.00	0.47	0.62	1.91	0.01	96.12	0.11	0.01	99.93
230	0.94	0.20	0.04	0.00	1.35	0.74	4.14	0.02	92.61	0.11	0.01	100.15
235	1.01	0.23	0.04	0.00	1.57	1.29	6.98	0.03	88.98	0.14	0.00	100.26
240	1.45	0.22	0.04	0.05	2.31	3.00	14.40	0.05	78.68	0.14	0.01	100.36
245	1.29	0.25	0.04	0.06	1.80	1.71	15.22	0.02	79.68	0.11	0.01	100.18
250	0.50	0.20	0.03	0.00	0.61	0.90	4.31	0.03	73.58	0.08	0.00	80.25
255	0.23	0.22	0.04	0.00	0.17	0.43	0.99	0.01	96.16	0.08	0.00	98.33
260	0.51	0.20	0.04	0.06	0.98	1.07	5.73	0.04	91.29	0.12	0.01	100.05
265	1.14	0.22	0.04	0.00	1.81	1.63	13.63	0.03	81.14	0.15	0.02	99.80
270	1.44	0.25	0.04	0.07	1.71	1.60	21.98	0.03	72.41	0.13	0.01	99.67
275	0.43	0.22	0.03	0.05	0.65	0.65	4.64	0.01	92.56	0.09	0.01	99.34
280	0.20	0.24	0.04	0.02	0.31	1.13	2.28	0.03	95.02	0.09	0.02	99.37
285	0.81	0.27	0.04	0.01	0.87	3.13	27.41	0.03	68.32	0.05	0.01	100.95
290	0.76	0.23	0.03	0.00	0.14	3.32	25.12	0.01	67.81	0.03	0.01	97.48
295	0.38	0.24	0.04	0.00	0.04	1.37	8.86	0.02	85.54	0.03	0.00	96.50

**PFE986: Particle 2**

Distance from the centre (µm)	MnO %	NI O %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.16	0.21	0.03	0.00	0.39	0.54	0.99	0.02	98.07	0.03	0.01	100.45
5	0.42	0.20	0.04	0.04	0.71	0.64	1.79	0.02	96.79	0.05	0.00	100.70
10	0.64	0.22	0.04	0.03	1.17	0.84	2.01	0.01	95.15	0.08	0.00	100.18
15	0.71	0.22	0.04	0.00	1.13	0.62	1.66	0.02	95.71	0.08	0.01	100.19
20	1.05	0.23	0.04	0.02	1.87	0.83	2.69	0.04	93.49	0.08	0.03	100.36
25	1.31	0.25	0.04	0.04	2.55	1.42	5.52	0.03	89.61	0.13	0.00	100.90
30	0.81	0.22	0.03	0.04	1.77	0.83	4.65	0.01	91.61	0.08	0.00	100.04
35	1.14	0.19	0.04	0.00	2.45	1.45	7.55	0.01	87.32	0.12	0.00	100.28
40	0.21	0.20	0.04	0.01	0.12	0.57	0.49	0.03	97.63	0.10	0.02	99.39
45	0.62	0.22	0.03	0.00	1.58	1.21	2.87	0.03	93.67	0.12	0.00	100.34
50	0.60	0.19	0.04	0.01	1.20	0.68	2.13	0.02	95.28	0.08	0.01	100.24
55	0.84	0.24	0.04	0.02	1.81	1.28	2.45	0.03	93.53	0.11	0.00	100.35
60	0.71	0.21	0.04	0.05	1.59	1.38	1.75	0.01	94.20	0.10	0.01	100.05
65	0.92	0.20	0.03	0.00	1.62	0.78	0.87	0.01	95.38	0.11	0.00	99.92
70	1.20	0.19	0.04	0.00	1.75	0.77	0.86	0.02	95.18	0.12	0.01	100.13
75	1.14	0.24	0.03	0.03	1.34	0.63	0.62	0.02	96.43	0.10	0.01	100.59
80	0.48	0.22	0.04	0.00	0.72	0.63	0.35	0.01	97.91	0.08	0.01	100.45
85	0.51	0.23	0.03	0.01	0.72	0.59	0.23	0.03	97.64	0.05	0.00	100.05
90	1.03	0.24	0.03	0.00	1.30	0.77	0.50	0.01	96.43	0.10	0.01	100.42
95	0.33	0.25	0.04	0.01	0.45	0.66	0.22	0.02	98.59	0.07	0.01	100.63
100	0.78	0.23	0.04	0.05	0.84	0.97	0.39	0.01	96.66	0.09	0.01	100.07
105	0.35	0.19	0.03	0.06	0.43	0.65	0.23	0.02	98.46	0.07	0.00	100.50
110	0.16	0.20	0.03	0.02	0.22	0.64	0.11	0.02	98.84	0.06	0.01	100.31
115	0.58	0.21	0.04	0.03	0.78	0.64	0.32	0.03	97.73	0.08	0.00	100.43
120	0.28	0.19	0.03	0.02	0.36	0.57	0.13	0.01	98.40	0.06	0.01	100.05
125	0.92	0.20	0.04	0.05	1.29	0.84	0.43	0.02	96.75	0.11	0.03	100.67
130	0.58	0.23	0.04	0.03	0.79	0.69	0.19	0.01	98.16	0.10	0.00	100.79
135	0.93	0.25	0.04	0.00	1.12	0.70	0.26	0.02	97.18	0.09	0.01	100.60
140	0.43	0.25	0.03	0.06	0.55	0.55	0.09	0.01	98.56	0.04	0.01	100.58
145	0.45	0.24	0.04	0.03	0.47	0.51	0.06	0.01	98.68	0.05	0.01	100.55
150	0.22	0.23	0.03	0.06	0.26	0.50	0.00	0.02	99.27	0.05	0.01	100.65
155	0.62	0.26	0.04	0.04	0.73	0.68	0.13	0.02	97.66	0.10	0.01	100.27

Distance from the centre (µm)	MnO %	NI O %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
160	0.69	0.21	0.04	0.00	0.73	0.56	0.11	0.03	98.10	0.09	0.01	100.56
165	0.54	0.23	0.04	0.01	0.74	0.55	0.08	0.03	98.29	0.08	0.01	100.58
170	0.79	0.22	0.04	0.00	1.42	0.65	0.19	0.01	96.46	0.10	0.01	99.88
175	1.10	0.24	0.04	0.03	1.53	0.73	0.23	0.02	96.48	0.11	0.01	100.52
180	0.42	0.25	0.03	0.02	0.71	0.65	0.09	0.02	98.42	0.08	0.01	100.71
185	0.83	0.25	0.04	0.00	1.01	0.65	0.14	0.03	97.50	0.09	0.01	100.55
190	0.31	0.20	0.04	0.02	0.54	0.56	0.05	0.04	99.11	0.05	0.01	100.92
195	0.44	0.25	0.04	0.03	0.54	0.56	0.05	0.05	98.44	0.05	0.02	100.45
200	0.79	0.22	0.04	0.00	1.31	0.55	0.09	0.05	97.51	0.07	0.01	100.63
205	0.26	0.22	0.03	0.00	0.50	0.61	0.08	0.05	98.91	0.08	0.01	100.74
210	0.59	0.21	0.04	0.02	1.26	0.82	0.30	0.02	96.47	0.11	0.01	99.83
215	0.68	0.22	0.04	0.04	1.46	0.92	0.44	0.01	96.87	0.09	0.01	100.77
220	0.61	0.26	0.03	0.00	1.31	0.90	0.52	0.01	96.96	0.09	0.01	100.70
225	0.44	0.22	0.03	0.03	1.00	0.72	0.39	0.02	97.87	0.07	0.01	100.78
230	0.42	0.24	0.03	0.00	0.94	0.81	0.53	0.02	97.32	0.08	0.00	100.39
235	0.58	0.24	0.04	0.02	1.43	0.99	1.07	0.00	96.19	0.09	0.00	100.65
240	0.67	0.21	0.03	0.00	1.75	1.21	1.72	0.02	94.35	0.10	0.01	100.07
245	0.81	0.21	0.03	0.02	1.93	1.34	2.35	0.02	89.12	0.11	0.01	95.95
250	0.85	0.21	0.04	0.01	1.71	1.02	1.55	0.01	94.89	0.12	0.01	100.41
255	0.84	0.20	0.03	0.00	1.75	1.30	1.71	0.01	94.32	0.12	0.01	100.29
260	0.66	0.21	0.03	0.02	1.22	1.04	1.17	0.01	96.43	0.09	0.01	100.89
265	1.10	0.23	0.04	0.00	1.86	1.08	1.52	0.01	94.13	0.12	0.01	100.09
270	0.92	0.22	0.03	0.02	1.54	1.13	1.46	0.02	94.99	0.11	0.01	100.46
275	0.46	0.22	0.04	0.01	0.74	0.83	0.86	0.01	96.98	0.11	0.01	100.27
280	0.96	0.20	0.03	0.00	1.51	1.02	1.60	0.02	94.58	0.12	0.01	100.06
285	1.32	0.24	0.04	0.00	1.96	1.45	2.89	0.01	92.04	0.13	0.02	100.10
290	1.05	0.22	0.04	0.00	1.39	1.00	3.30	0.03	92.69	0.14	0.02	99.86
295	0.91	0.26	0.04	0.05	1.82	1.25	8.84	0.05	86.31	0.08	0.03	99.63
300	1.26	0.27	0.04	0.04	1.98	1.07	6.60	0.36	88.67	0.13	0.03	100.43
305	2.13	0.24	0.04	0.04	2.26	2.02	11.11	0.20	81.14	0.16	0.06	99.38

**PFE986: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.21	0.22	0.04	0.08	1.69	1.32	8.16	0.02	91.26	0.13	0.00	104.13
5	1.05	0.21	0.03	0.04	1.52	1.31	8.49	0.02	92.77	0.13	0.02	105.59
10	1.02	0.26	0.04	0.03	1.41	1.22	7.89	0.03	91.54	0.12	0.01	103.57
15	1.18	0.24	0.04	0.02	1.52	1.18	8.05	0.02	91.14	0.12	0.02	103.52
20	1.06	0.22	0.04	0.00	0.91	0.91	1.97	0.01	95.33	0.11	0.00	100.56
25	1.21	0.21	0.04	0.00	1.40	1.04	5.52	0.02	91.35	0.11	0.01	100.89
30	1.09	0.21	0.03	0.03	1.26	1.07	5.80	0.02	91.52	0.11	0.01	101.14
35	1.26	0.26	0.04	0.07	1.62	0.94	5.99	0.02	90.99	0.13	0.01	101.32
40	1.65	0.20	0.04	0.00	1.57	0.89	6.20	0.05	90.43	0.11	0.01	101.15
45	1.35	0.20	0.04	0.02	1.42	1.08	4.17	0.02	91.82	0.14	0.01	100.26
50	1.54	0.25	0.04	0.04	1.57	1.07	6.12	0.04	89.85	0.12	0.01	100.64
55	1.77	0.26	0.04	0.00	1.66	1.08	4.26	0.03	91.54	0.13	0.02	100.76
60	1.37	0.22	0.04	0.03	1.34	1.23	4.26	0.05	92.24	0.14	0.02	100.93
65	1.11	0.19	0.03	0.03	1.05	0.96	3.12	0.04	94.21	0.10	0.02	100.86
70	1.26	0.23	0.04	0.00	1.31	0.97	5.22	0.03	91.39	0.12	0.02	100.59
75	1.21	0.20	0.04	0.01	1.19	0.88	5.04	0.02	91.18	0.11	0.01	99.87
80	1.10	0.26	0.04	0.00	1.28	1.11	6.66	0.06	89.48	0.15	0.03	100.14
85	0.85	0.24	0.04	0.00	0.91	1.00	5.43	0.04	90.89	0.15	0.02	99.56
90	1.92	0.22	0.04	0.00	1.62	2.28	11.43	0.03	83.23	0.19	0.02	100.98
95	1.75	0.24	0.04	0.05	1.27	1.67	9.25	0.07	85.31	0.14	0.05	99.82
100	1.66	0.26	0.04	0.03	1.39	1.84	9.86	0.04	84.42	0.15	0.05	99.73

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
105	0.49	0.22	0.04	0.01	0.63	1.38	4.00	0.03	93.86	0.18	0.03	100.86
110	1.67	0.21	0.04	0.04	1.48	2.76	11.06	0.03	82.87	0.17	0.03	100.35
115	1.21	0.22	0.04	0.01	0.88	8.19	7.23	0.09	85.45	0.13	0.08	103.53
120	1.31	0.24	0.04	0.00	0.89	0.99	4.95	0.04	92.02	0.13	0.04	100.65
125	1.29	0.22	0.04	0.05	0.79	0.75	3.98	0.01	92.66	0.14	0.04	99.97
130	1.54	0.23	0.04	0.06	0.97	0.89	5.55	0.03	90.82	0.13	0.04	100.30
135	1.63	0.24	0.03	0.06	1.17	0.89	5.87	0.04	90.13	0.11	0.03	100.20
140	1.27	0.22	0.04	0.00	1.12	1.10	6.36	0.03	87.41	0.16	0.03	97.74
145	1.62	0.24	0.04	0.03	0.96	0.71	4.99	0.03	91.07	0.12	0.02	99.83
150	2.16	0.20	0.04	0.06	1.27	1.02	6.61	0.03	88.33	0.11	0.03	99.85
155	1.31	0.21	0.04	0.00	0.89	0.88	5.37	0.03	90.63	0.11	0.03	99.50
160	1.42	0.20	0.04	0.00	1.44	1.58	11.05	0.04	83.59	0.15	0.03	99.51
165	1.82	0.18	0.04	0.00	1.86	3.57	35.32	0.06	53.38	0.22	0.02	96.45
170	4.15	0.23	0.05	0.03	1.78	1.74	34.08	0.03	56.79	0.15	0.03	99.05
175	1.96	0.22	0.04	0.00	1.38	1.11	13.25	0.05	79.38	0.13	0.01	97.51
180	1.09	0.18	0.04	0.09	0.98	0.78	7.10	0.07	86.39	0.12	0.01	96.86
185	2.35	0.16	0.04	0.00	1.49	1.07	10.86	0.11	71.84	0.15	0.02	88.08
190	0.34	0.18	0.03	0.00	0.23	0.58	1.36	0.03	96.66	0.11	0.00	99.51
195	3.03	0.13	0.02	0.05	3.35	2.32	30.60	0.08	39.19	0.00	0.03	78.79
200	2.88	0.24	0.04	0.03	2.00	2.84	32.25	0.01	57.09	0.02	0.04	97.44

**PFE1384: Particle 1**

Distance from the centre (µm)	MnO %	NI0 %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.06	0.06	0.04	0.02	0.69	0.57	0.43	0.03	99.03	0.07	0.01	100.99
5	0.07	0.12	0.03	0.04	1.03	0.63	0.78	0.05	97.97	0.09	0.01	100.82
10	0.07	0.44	0.04	0.02	2.07	1.39	2.63	0.04	90.97	0.27	0.01	97.94
15	0.03	0.02	0.03	0.02	0.27	0.52	0.31	0.03	99.70	0.04	0.00	100.98
20	0.08	0.05	0.04	0.07	0.63	1.38	0.73	0.06	97.91	0.08	0.01	101.03
25	0.06	0.13	0.03	0.04	1.26	0.77	1.40	0.03	96.21	0.10	0.01	100.03
30	0.09	0.16	0.03	0.03	1.17	1.22	1.96	0.04	95.34	0.13	0.00	100.17
35	0.05	0.20	0.04	0.03	1.64	1.06	3.75	0.05	93.27	0.13	0.00	100.23
40	0.07	0.10	0.04	0.01	0.89	0.66	1.62	0.03	96.71	0.09	0.01	100.21
45	0.06	0.08	0.04	0.02	1.51	0.70	3.07	0.02	94.85	0.10	0.01	100.45
50	0.06	0.07	0.04	0.04	1.11	0.69	2.87	0.01	95.40	0.12	0.00	100.40
55	0.06	0.04	0.04	0.00	1.12	0.73	2.93	0.03	94.80	0.10	0.01	99.85
60	0.07	0.13	0.04	0.00	1.46	1.01	4.96	0.02	92.25	0.09	0.00	100.03
65	0.09	0.29	0.04	0.07	2.69	2.46	17.71	0.02	75.48	0.16	0.02	99.03
70	0.08	0.19	0.05	0.07	2.00	1.24	12.73	0.02	82.41	0.14	0.01	98.94
75	0.04	0.14	0.07	0.06	1.61	1.77	10.96	0.05	76.50	0.14	0.04	91.38
80	0.07	0.05	0.05	0.00	0.94	0.75	5.17	0.02	91.26	0.12	0.00	98.44

Distance from the centre (µm)	MnO %	NI0 %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
85	0.08	0.09	0.08	0.10	1.23	1.88	6.47	0.05	89.54	0.15	0.02	99.70
90	0.06	0.00	0.05	0.00	0.74	0.66	2.99	0.01	95.36	0.13	0.01	100.00
95	0.06	0.04	0.05	0.11	1.01	1.10	4.23	0.03	93.32	0.11	0.01	100.07
100	0.08	0.01	0.05	0.03	0.94	0.73	4.69	0.04	93.16	0.14	0.00	99.87
105	0.07	0.04	0.04	0.00	0.89	0.63	4.12	0.03	93.91	0.12	0.02	99.86
110	0.05	0.04	0.04	0.03	1.29	1.03	5.99	0.04	92.40	0.15	0.01	101.06
115	0.04	0.07	0.04	0.11	1.34	1.01	7.03	0.03	90.15	0.15	0.01	99.96
120	0.09	0.09	0.05	0.07	1.53	3.92	8.57	0.07	84.81	0.14	0.05	99.40
125	0.08	0.15	0.05	0.04	2.85	1.50	16.89	0.03	76.93	0.16	0.00	98.66
130	0.10	0.18	0.06	0.05	1.90	1.64	18.88	0.04	74.99	0.17	0.02	98.02
135	0.07	0.00	0.05	0.02	0.75	4.18	4.55	0.08	92.08	0.13	0.04	101.94
140	0.08	0.13	0.04	0.06	1.81	0.96	12.86	0.04	83.16	0.16	0.02	99.32
145	0.04	0.03	0.05	0.02	0.96	1.04	5.45	0.03	91.33	0.14	0.01	99.11
150	0.08	0.01	0.06	0.07	0.82	0.94	7.13	0.07	88.82	0.14	0.02	98.15
155	0.08	0.00	0.10	0.18	0.31	1.63	6.40	2.51	87.85	0.07	0.12	99.25
160	0.05	0.00	0.07	0.09	0.10	4.45	13.74	0.08	73.41	0.02	0.03	92.03



**PFE1384: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.09	0.15	0.04	0.02	1.44	0.75	0.28	0.01	97.22	0.07	0.01	100.07
5	0.08	0.30	0.04	0.01	2.50	1.09	0.38	0.03	95.30	0.13	0.00	99.86
10	0.06	0.09	0.03	0.00	0.93	0.70	0.14	0.02	98.19	0.06	0.01	100.25
15	0.09	0.20	0.04	0.01	1.53	0.91	0.14	0.02	97.26	0.08	0.01	100.29
20	0.08	0.04	0.03	0.02	0.59	0.79	0.08	0.02	98.51	0.05	0.01	100.22
25	0.06	0.06	0.04	0.00	0.84	0.70	0.06	0.02	98.72	0.05	0.01	100.56
30	0.05	0.05	0.03	0.03	0.76	0.73	0.04	0.01	98.88	0.06	0.01	100.65
35	0.07	0.17	0.04	0.00	1.68	0.96	0.09	0.01	96.85	0.10	0.00	99.98
40	0.08	0.03	0.04	0.03	0.59	0.71	0.04	0.03	98.70	0.06	0.00	100.32
45	0.08	0.10	0.04	0.00	1.00	0.78	0.08	0.02	97.75	0.07	0.01	99.93
50	0.06	0.01	0.04	0.00	0.55	0.58	0.03	0.02	99.28	0.05	0.01	100.61
55	0.05	0.15	0.04	0.00	1.42	0.90	0.08	0.02	97.77	0.09	0.00	100.53
60	0.10	0.22	0.03	0.00	1.71	0.85	0.10	0.02	97.11	0.11	0.01	100.25
65	0.08	0.22	0.03	0.02	1.80	0.88	0.12	0.03	97.07	0.10	0.01	100.37
70	0.03	0.06	0.04	0.00	0.47	0.65	0.02	0.02	99.31	0.04	0.00	100.65
75	0.06	0.16	0.04	0.04	0.92	0.70	0.06	0.04	98.17	0.08	0.00	100.26
80	0.07	0.12	0.04	0.04	0.87	0.54	0.06	0.03	98.90	0.07	0.00	100.72
85	0.09	0.15	0.04	0.01	0.98	0.62	0.08	0.00	97.93	0.11	0.01	100.01
90	0.10	0.20	0.04	0.00	1.15	0.61	0.08	0.02	97.98	0.09	0.00	100.28
95	0.05	0.25	0.03	0.02	1.03	0.63	0.12	0.02	97.89	0.10	0.01	100.14
100	0.07	0.45	0.04	0.00	2.16	0.98	0.26	0.04	95.56	0.16	0.00	99.71
105	0.08	0.42	0.04	0.00	2.02	1.07	0.41	0.04	95.73	0.13	0.00	99.95
110	0.08	0.17	0.04	0.00	1.37	0.96	0.40	0.15	93.81	0.10	0.01	97.07
115	0.04	0.03	0.04	0.00	0.48	0.62	0.29	0.02	98.78	0.05	0.00	100.36
120	0.07	0.29	0.03	0.01	1.57	0.95	1.36	0.04	95.41	0.16	0.00	99.88
125	0.07	0.10	0.04	0.04	0.37	0.58	0.54	0.03	97.47	0.07	0.01	99.30
130	0.05	0.07	0.07	0.11	0.23	2.80	0.66	0.10	91.39	0.10	0.05	95.63
135	0.04	0.23	0.10	0.09	1.71	25.45	14.22	0.25	49.60	0.11	0.21	92.01
140	0.05	0.29	0.04	0.04	1.59	0.79	14.89	0.01	82.02	0.10	0.00	99.82
145	0.07	0.21	0.04	0.02	1.48	0.88	8.41	0.02	89.87	0.10	0.01	101.10
150	0.05	0.42	0.04	0.04	2.28	1.07	10.34	0.02	86.23	0.15	0.00	100.65
155	0.07	0.05	0.04	0.02	0.19	0.47	0.91	0.01	99.06	0.08	0.01	100.92
160	0.06	0.30	0.04	0.11	1.38	0.74	5.27	0.03	92.47	0.09	0.00	100.48
165	0.09	0.07	0.04	0.00	0.47	0.59	1.82	0.03	97.44	0.09	0.00	100.63
170	0.09	0.05	0.04	0.00	0.18	0.38	0.74	0.03	99.02	0.09	0.01	100.62
175	0.09	0.48	0.04	0.03	1.97	0.97	9.02	0.05	87.66	0.14	0.01	100.46
180	0.09	0.39	0.04	0.05	1.54	0.74	5.05	0.04	92.66	0.11	0.00	100.72
185	0.10	0.25	0.03	0.09	1.13	0.68	3.38	0.04	95.14	0.12	0.00	100.95

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
190	0.10	0.02	0.04	0.00	0.26	0.47	0.88	0.03	98.99	0.10	0.00	100.89
195	0.05	0.68	0.04	0.06	2.10	0.78	5.86	0.02	91.50	0.12	0.00	101.21
200	0.07	0.50	0.04	0.01	1.74	0.90	6.10	0.05	90.77	0.11	0.00	100.28
205	0.05	0.05	0.04	0.03	0.10	0.51	0.68	0.03	99.44	0.11	0.00	101.04
210	0.05	0.14	0.04	0.03	0.31	0.53	1.18	0.03	98.19	0.12	0.00	100.62
215	0.08	0.59	0.04	0.00	1.46	0.81	4.24	0.03	93.40	0.12	0.01	100.78
220	0.06	0.27	0.04	0.00	0.73	0.70	2.41	0.03	95.96	0.08	0.00	100.28
225	0.06	0.43	0.03	0.01	0.90	0.74	2.85	0.05	95.32	0.11	0.02	100.53
230	0.09	0.58	0.04	0.08	1.25	0.74	4.27	0.05	93.51	0.13	0.02	100.75
235	0.07	0.51	0.03	0.00	1.07	0.77	4.02	0.08	93.54	0.14	0.01	100.23
240	0.08	0.17	0.04	0.04	0.27	0.60	2.60	0.08	96.30	0.14	0.02	100.34
245	0.11	1.03	0.04	0.03	1.59	1.03	8.72	0.04	86.75	0.12	0.03	99.50
250	0.08	0.70	0.12	0.10	1.24	1.99	6.33	13.88	75.31	0.14	0.95	100.81
255	0.10	0.76	0.04	0.00	1.42	1.51	7.87	0.03	88.60	0.13	0.02	100.48
260	0.09	0.94	0.04	0.02	2.23	1.17	9.85	0.03	85.90	0.13	0.01	100.40
265	0.06	0.37	0.04	0.04	0.86	0.87	5.51	0.04	92.72	0.11	0.01	100.62
270	0.09	0.44	0.04	0.05	1.05	0.89	5.67	0.01	92.50	0.11	0.01	100.86
275	0.09	0.48	0.04	0.00	1.38	1.19	6.16	0.03	91.75	0.12	0.02	101.26
280	0.07	0.81	0.04	0.04	1.78	1.16	11.13	0.03	85.53	0.15	0.01	100.73
285	0.07	0.52	0.04	0.00	1.11	0.80	5.35	0.03	93.42	0.12	0.01	101.47
290	0.07	0.40	0.04	0.06	0.97	0.84	4.28	0.05	93.65	0.12	0.01	100.50
295	0.06	0.55	0.04	0.04	1.32	0.89	4.77	0.05	93.18	0.12	0.01	101.02
300	0.05	0.74	0.04	0.00	1.37	1.03	7.02	0.05	90.20	0.13	0.01	100.64
305	0.07	0.63	0.04	0.00	1.06	1.24	5.66	0.07	92.12	0.14	0.01	101.04
310	0.09	0.21	0.04	0.02	0.25	1.03	1.99	0.06	96.57	0.14	0.02	100.43
315	0.08	0.21	0.04	0.00	0.16	0.55	1.05	0.09	98.34	0.13	0.01	100.65
320	0.06	0.34	0.04	0.02	0.41	0.70	2.25	0.05	96.67	0.11	0.01	100.66
325	0.05	0.85	0.04	0.00	1.00	0.84	4.52	0.07	92.74	0.12	0.02	100.25
330	0.06	0.06	0.20	0.13	0.00	15.05	0.66	8.54	70.62	0.03	0.22	95.56

**PFE1384: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.04	0.08	0.03	0.01	1.05	0.78	0.21	0.01	97.92	0.07	0.01	100.21
5	0.06	0.14	0.03	0.00	1.24	0.75	0.29	0.01	97.78	0.10	0.00	100.41
10	0.07	0.19	0.04	0.02	1.92	0.79	0.48	0.02	96.11	0.09	0.00	99.72
15	0.06	0.30	0.04	0.02	2.02	6.17	1.38	0.10	89.77	0.13	0.08	100.06
20	0.04	0.13	0.04	0.03	0.92	0.80	0.49	0.02	97.42	0.11	0.01	100.00
25	0.09	0.12	0.04	0.00	1.10	0.74	0.51	0.02	96.56	0.10	0.01	99.29
30	0.08	0.13	0.04	0.06	1.17	0.79	0.46	0.01	97.50	0.09	0.01	100.33
35	0.07	0.03	0.04	0.00	0.42	0.65	0.24	0.02	98.99	0.09	0.00	100.55
40	0.07	0.08	0.04	0.02	0.50	0.70	0.27	0.02	98.22	0.07	0.01	99.99
45	0.06	0.09	0.04	0.02	1.18	0.76	0.42	0.02	97.41	0.10	0.00	100.09
50	0.04	0.05	0.04	0.02	0.85	0.67	0.31	0.02	98.29	0.07	0.01	100.37
55	0.07	0.04	0.03	0.00	0.62	0.61	0.26	0.02	98.54	0.08	0.00	100.26
60	0.06	0.10	0.04	0.00	0.73	0.68	0.30	0.03	98.29	0.08	0.01	100.31
65	0.06	0.08	0.05	0.03	0.96	0.72	0.45	0.04	97.71	0.08	0.02	100.18
70	0.09	0.10	0.04	0.03	1.10	0.72	0.61	0.02	97.16	0.10	0.00	99.96
75	0.06	0.14	0.04	0.03	1.34	0.77	0.60	0.02	97.24	0.10	0.01	100.35
80	0.09	0.12	0.04	0.02	1.03	0.76	0.52	0.04	97.21	0.08	0.01	99.91
85	0.07	0.16	0.04	0.00	1.36	0.75	0.64	0.03	96.94	0.09	0.00	100.08
90	0.08	0.08	0.04	0.00	0.92	0.68	0.60	0.04	97.45	0.09	0.02	99.98
95	0.07	0.14	0.03	0.02	1.23	0.73	0.86	0.02	96.70	0.10	0.01	99.90
100	0.07	0.13	0.04	0.03	1.11	0.84	1.05	0.03	97.12	0.12	0.00	100.54
105	0.06	0.10	0.04	0.05	0.64	0.80	1.05	0.03	97.42	0.11	0.00	100.29
110	0.08	0.08	0.03	0.00	0.64	0.68	1.09	0.02	97.77	0.08	0.02	100.50
115	0.10	0.16	0.04	0.01	1.28	0.76	1.97	0.04	95.32	0.10	0.01	99.78
120	0.06	0.44	0.04	0.00	2.73	4.87	10.04	0.12	80.66	0.24	0.09	99.29
125	0.10	0.40	0.04	0.03	2.75	3.41	10.82	0.06	82.02	0.17	0.05	99.86
130	0.08	0.36	0.04	0.11	1.88	1.63	23.02	0.02	71.45	0.12	0.01	98.72
135	0.06	0.39	0.04	0.01	1.82	2.23	21.68	0.05	72.62	0.14	0.01	99.04
140	0.05	0.22	0.04	0.01	1.81	1.04	9.64	0.04	86.93	0.13	0.02	99.92

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
145	0.08	0.29	0.03	0.00	1.99	1.07	8.83	0.03	87.55	0.14	0.01	100.02
150	0.08	0.14	0.04	0.03	1.07	0.65	4.98	0.04	93.66	0.12	0.00	100.81
155	0.10	0.39	0.04	0.03	2.36	1.13	12.40	0.05	83.52	0.16	0.01	100.18
160	0.10	0.10	0.03	0.00	1.15	0.73	3.55	0.02	94.23	0.11	0.01	100.04
165	0.09	0.11	0.04	0.00	0.81	0.62	2.66	0.03	95.38	0.11	0.02	99.86
170	0.04	0.38	0.04	0.04	2.22	0.82	7.91	0.03	88.46	0.13	0.01	100.07
175	0.07	0.02	0.04	0.00	0.24	0.51	0.90	0.02	98.27	0.11	0.01	100.18
180	0.06	0.44	0.04	0.00	2.35	1.05	9.16	0.03	87.16	0.14	0.01	100.44
185	0.07	0.37	0.04	0.01	1.80	3.14	8.10	0.04	86.76	0.14	0.02	100.48
190	0.05	0.23	0.03	0.00	1.28	1.36	4.86	0.04	92.55	0.11	0.02	100.53
195	0.07	0.01	0.03	0.01	0.24	1.70	0.85	0.03	96.94	0.11	0.02	100.01
200	0.10	0.01	0.04	0.00	0.21	0.54	0.83	0.02	98.73	0.10	0.01	100.59
205	0.07	0.07	0.04	0.04	0.62	0.95	2.74	0.04	95.52	0.11	0.01	100.21
210	0.07	0.26	0.04	0.01	1.49	1.11	5.72	0.03	91.55	0.11	0.02	100.41
215	0.06	0.03	0.03	0.06	0.33	5.88	1.42	0.04	95.12	0.11	0.03	103.10
220	0.07	0.30	0.04	0.00	1.35	2.07	6.99	0.04	89.27	0.14	0.01	100.28
225	0.09	0.45	0.04	0.01	1.92	1.11	6.60	0.04	89.58	0.14	0.01	99.99
230	0.06	0.05	0.04	0.00	0.33	0.57	2.14	0.03	96.94	0.11	0.01	100.27
235	0.08	0.53	0.03	0.00	1.79	0.85	8.31	0.03	88.53	0.12	0.01	100.29
240	0.05	0.69	0.04	0.04	1.96	1.41	15.23	0.04	79.58	0.17	0.01	99.21
245	0.04	0.13	0.04	0.00	0.52	0.91	4.74	0.05	92.62	0.13	0.01	99.18
250	0.10	0.85	0.04	0.03	2.57	3.31	24.34	0.02	68.30	0.18	0.01	99.75
255	0.06	0.01	0.04	0.02	0.16	0.67	1.58	0.05	97.35	0.06	0.01	100.01
260	0.07	0.12	0.04	0.04	0.19	2.90	14.56	0.04	79.40	0.04	0.02	97.40
265	0.09	0.00	0.04	0.05	0.16	2.17	3.34	0.06	90.61	0.02	0.03	96.58
270	0.00	0.00	0.01	0.00	0.30	9.60	5.61	0.11	24.84	0.00	0.14	40.62
275	0.00	0.14	0.03	0.00	0.07	20.75	12.33	0.19	48.32	0.00	0.14	81.98

**PFE1377: Particle 1**

Distance from the centre (µm)	MnO %	NI0 %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.85	0.19	0.05	0.00	2.10	1.46	9.28	0.04	85.41	0.16	0.01	100.54
5	0.82	0.22	0.04	0.02	1.02	0.90	4.52	0.02	93.19	0.11	0.01	100.86
10	1.55	0.20	0.04	0.00	1.59	2.87	7.94	0.07	87.27	0.16	0.06	101.74
15	1.33	0.22	0.04	0.00	1.60	0.99	5.73	0.04	90.80	0.13	0.01	100.88
20	1.04	0.19	0.04	0.06	1.08	0.98	4.84	0.04	92.25	0.13	0.02	100.67
25	0.68	0.27	0.03	0.02	0.54	0.70	2.45	0.03	95.50	0.14	0.00	100.37
30	1.39	0.21	0.04	0.00	1.68	1.12	7.36	0.03	88.84	0.12	0.01	100.80
35	1.13	0.18	0.04	0.01	1.22	1.01	5.49	0.04	91.90	0.12	0.01	101.15
40	1.30	0.20	0.03	0.00	1.41	1.02	6.00	0.04	90.78	0.12	0.00	100.90
45	1.14	0.20	0.04	0.02	1.38	0.93	5.22	0.04	91.92	0.13	0.01	101.00
50	0.91	0.24	0.05	0.03	1.04	2.12	4.81	0.06	91.07	0.14	0.03	100.49
55	0.75	0.23	0.04	0.05	0.68	0.66	2.36	0.05	95.86	0.13	0.01	100.81
60	0.71	0.25	0.04	0.00	0.88	0.80	3.79	0.03	94.10	0.15	0.01	100.77
65	0.65	0.22	0.04	0.01	0.76	1.01	3.27	0.05	95.30	0.13	0.02	101.45
70	1.45	0.20	0.04	0.04	1.44	2.30	6.23	0.06	90.13	0.18	0.01	102.08
75	0.76	0.23	0.04	0.02	0.47	0.70	2.18	0.04	96.36	0.14	0.01	100.93
80	1.43	0.21	0.04	0.04	1.45	1.05	6.37	0.04	90.36	0.14	0.00	101.14
85	0.99	0.23	0.03	0.02	0.68	0.80	3.63	0.02	93.99	0.14	0.00	100.53
90	1.40	0.18	0.04	0.01	1.15	1.00	6.16	0.06	90.53	0.14	0.03	100.70
95	1.34	0.19	0.04	0.00	1.04	0.96	5.71	0.04	91.35	0.16	0.01	100.84
100	1.03	0.23	0.04	0.00	0.99	1.11	5.33	0.06	91.26	0.15	0.01	100.20
105	1.73	0.24	0.04	0.00	1.44	1.05	8.09	0.06	88.09	0.14	0.03	100.91

Distance from the centre (µm)	MnO %	NI0 %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
110	1.17	0.19	0.04	0.00	1.07	1.24	6.76	0.04	89.22	0.15	0.00	99.88
115	2.69	0.23	0.04	0.02	2.01	2.36	14.06	0.12	79.43	0.16	0.07	101.19
120	2.14	0.18	0.04	0.08	1.59	1.34	11.47	0.08	83.35	0.15	0.01	100.43
125	1.72	0.20	0.05	0.06	1.21	2.90	8.03	0.12	86.16	0.16	0.11	100.70
130	1.92	0.21	0.03	0.06	1.50	1.50	9.28	0.10	85.77	0.16	0.03	100.56
135	1.25	0.27	0.04	0.03	0.79	0.66	3.96	0.05	93.69	0.14	0.01	100.89
140	0.56	0.20	0.03	0.04	0.37	0.73	1.30	0.05	97.42	0.13	0.01	100.85
145	2.07	0.22	0.04	0.00	1.64	1.20	9.52	0.10	85.17	0.19	0.02	100.17
150	2.34	0.22	0.04	0.01	1.59	1.05	8.53	0.03	86.25	0.12	0.01	100.20
155	1.57	0.23	0.04	0.05	1.17	0.89	6.98	0.05	89.13	0.11	0.01	100.23
160	2.44	0.21	0.04	0.03	1.70	2.19	12.22	0.08	81.61	0.18	0.08	100.76
165	2.68	0.21	0.05	0.10	1.75	1.48	12.15	0.08	82.30	0.18	0.03	101.00
170	1.51	0.23	0.04	0.00	0.88	0.90	6.76	0.04	90.51	0.15	0.00	101.01
175	1.93	0.22	0.04	0.03	0.94	1.12	9.39	0.07	86.29	0.14	0.01	100.17
180	2.32	0.25	0.05	0.02	1.22	1.56	15.18	0.06	78.97	0.17	0.00	99.80
185	2.71	0.22	0.05	0.00	1.54	1.61	17.67	0.06	71.31	0.17	0.02	95.33
190	1.65	0.19	0.04	0.03	1.14	1.24	11.75	0.05	81.52	0.16	0.02	97.80
195	1.91	0.21	0.05	0.00	1.14	1.52	13.83	0.07	75.74	0.16	0.01	94.65
200	1.24	0.23	0.04	0.00	0.64	0.93	6.27	0.06	90.90	0.12	0.02	100.45
205	2.32	0.19	0.05	0.05	0.59	2.79	26.58	0.05	66.78	0.09	0.01	99.51
210	1.03	0.17	0.04	0.01	0.15	2.19	12.03	0.03	84.05	0.05	0.01	99.76

**PFE1377: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.13	0.21	0.04	0.00	1.22	0.88	3.80	0.05	93.86	0.14	0.04	101.35
5	1.17	0.21	0.03	0.02	1.23	0.88	4.11	0.04	93.03	0.15	0.01	100.87
10	0.80	0.20	0.04	0.01	0.65	2.74	2.32	0.07	94.30	0.13	0.03	101.27
15	1.30	0.21	0.04	0.04	1.36	0.86	4.48	0.05	92.52	0.14	0.02	101.02
20	1.03	0.21	0.04	0.00	0.71	0.64	2.36	0.01	95.25	0.13	0.01	100.39
25	0.98	0.20	0.04	0.02	0.90	0.72	3.03	0.05	95.54	0.11	0.01	101.60
30	1.40	0.21	0.06	0.04	1.86	1.29	5.23	0.75	89.45	0.11	0.02	100.43
35	1.02	0.27	0.04	0.01	1.49	0.82	3.29	0.02	93.93	0.10	0.02	101.00
40	0.96	0.21	0.04	0.00	1.37	0.79	2.71	0.04	94.70	0.13	0.03	100.97
45	1.04	0.24	0.04	0.02	1.53	0.77	2.32	0.05	94.74	0.10	0.03	100.86
50	0.84	0.24	0.04	0.00	1.19	0.76	1.96	0.04	95.26	0.11	0.02	100.45
55	0.81	0.21	0.04	0.00	1.05	0.65	1.68	0.07	95.87	0.10	0.03	100.52
60	0.67	0.21	0.04	0.00	1.04	0.73	1.71	0.01	96.29	0.07	0.03	100.79
65	1.06	0.23	0.04	0.02	1.04	0.80	2.13	0.03	95.62	0.13	0.03	101.13
70	1.60	0.22	0.04	0.00	2.04	1.05	3.83	0.05	92.10	0.15	0.04	101.11
75	1.40	0.22	0.04	0.02	2.23	0.91	3.24	0.01	93.46	0.10	0.02	101.63
80	0.75	0.19	0.04	0.00	0.89	0.80	1.48	0.03	96.19	0.12	0.03	100.51
85	0.91	0.18	0.04	0.01	1.17	1.18	2.13	0.03	94.71	0.14	0.03	100.53
90	1.20	0.22	0.04	0.03	1.36	0.76	2.34	0.02	95.28	0.12	0.02	101.39
95	0.91	0.21	0.04	0.01	1.27	0.80	2.01	0.03	95.39	0.10	0.04	100.80
100	1.49	0.21	0.04	0.03	2.55	2.07	4.21	0.04	90.26	0.15	0.18	101.23
105	1.29	0.21	0.04	0.03	1.80	0.99	3.74	0.03	92.82	0.14	0.01	101.11
110	0.35	0.21	0.04	0.00	0.22	3.58	0.53	0.04	94.86	0.11	0.03	99.95
115	1.30	0.22	0.04	0.03	1.65	0.89	3.16	0.03	93.56	0.13	0.02	101.02
120	0.56	0.20	0.04	0.01	0.77	1.68	1.60	0.04	95.79	0.12	0.17	100.97
125	1.34	0.24	0.03	0.04	1.58	0.98	3.54	0.04	93.26	0.13	0.02	101.21
130	1.21	0.27	0.04	0.00	1.96	1.24	4.56	0.05	91.52	0.15	0.02	101.01
135	0.97	0.22	0.04	0.01	1.26	1.16	3.16	0.02	94.25	0.13	0.02	101.23

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
140	2.22	0.23	0.04	0.12	3.15	2.45	9.60	0.05	83.09	0.17	0.04	101.15
145	1.15	0.21	0.04	0.05	2.35	1.96	5.03	0.03	90.38	0.16	0.01	101.38
150	0.55	0.21	0.03	0.03	0.80	1.05	1.71	0.01	96.22	0.13	0.01	100.75
155	0.84	0.25	0.03	0.04	1.76	3.65	4.96	0.03	90.45	0.16	0.02	102.18
160	1.14	0.22	0.04	0.01	1.80	1.50	5.59	0.03	91.07	0.13	0.01	101.53
165	1.33	0.20	0.03	0.00	2.46	2.28	13.90	0.05	80.25	0.21	0.02	100.73
170	0.29	0.15	0.08	0.29	0.29	26.56	1.67	0.47	62.18	0.06	0.35	92.39
175	1.00	0.23	0.04	0.02	1.22	1.68	10.02	0.03	86.72	0.14	0.01	101.11
180	1.12	0.26	0.03	0.00	1.39	1.05	11.16	0.02	85.27	0.12	0.01	100.42
185	1.76	0.24	0.04	0.00	2.76	1.73	14.74	0.03	79.00	0.15	0.01	100.45
190	0.58	0.22	0.03	0.01	0.88	0.85	2.96	0.01	94.95	0.10	0.02	100.60
195	0.70	0.22	0.04	0.05	1.26	0.91	3.82	0.04	94.54	0.11	0.01	101.69
200	0.66	0.18	0.04	0.00	1.03	1.50	3.56	0.06	93.30	0.12	0.03	100.47
205	1.39	0.16	0.05	0.17	3.12	17.69	12.72	0.28	69.20	0.24	0.37	105.39
210	1.13	0.21	0.06	0.23	1.55	4.53	12.22	0.14	79.36	0.14	0.19	99.75
215	1.65	0.21	0.04	0.01	2.28	2.04	15.31	0.05	78.88	0.13	0.03	100.60
220	0.28	0.20	0.03	0.00	0.29	0.89	1.33	0.02	98.04	0.09	0.01	101.17
225	0.77	0.24	0.04	0.00	1.64	0.89	3.93	0.04	93.92	0.11	0.01	101.58
230	0.56	0.22	0.04	0.02	1.03	1.27	3.39	0.04	94.63	0.11	0.01	101.32
235	2.18	0.22	0.04	0.09	4.72	2.89	16.47	0.03	74.82	0.14	0.02	101.61
240	1.76	0.24	0.05	0.06	1.83	1.96	19.07	0.02	73.88	0.14	0.01	99.02
245	1.16	0.21	0.03	0.00	1.46	1.09	12.97	0.03	78.54	0.12	0.00	95.62
250	1.60	0.21	0.04	0.02	2.25	2.87	28.74	0.02	64.78	0.16	0.01	100.69
255	0.56	0.19	0.05	0.00	1.22	2.49	11.42	0.03	84.43	0.13	0.01	100.53
260	1.12	0.21	0.04	0.04	1.19	4.30	30.90	0.00	63.92	0.03	0.02	101.77
265	0.51	0.17	0.04	0.05	0.23	4.42	13.19	0.03	78.77	0.02	0.03	97.47



**PFE1377: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.06	0.00	0.04	0.00	0.70	4.22	1.03	0.06	82.33	0.09	0.06	88.59
5	0.06	0.00	0.04	0.00	2.11	2.05	6.13	0.02	85.90	0.16	0.01	96.47
10	0.08	0.03	0.04	0.04	1.54	0.98	3.46	0.01	93.34	0.10	0.01	99.63
15	0.08	0.00	0.04	0.00	1.65	1.20	2.40	0.02	93.85	0.11	0.01	99.35
20	0.08	0.00	0.04	0.00	1.06	0.75	1.42	0.01	96.48	0.09	0.00	99.92
25	0.10	0.00	0.04	0.00	1.10	2.08	1.30	0.05	95.84	0.10	0.01	100.61
30	0.28	0.21	0.04	0.04	0.24	0.57	0.33	0.01	98.83	0.08	0.00	100.61
35	0.82	0.19	0.04	0.01	2.47	0.80	1.66	0.02	94.59	0.10	0.02	100.70
40	0.42	0.23	0.03	0.04	0.59	0.57	0.50	0.00	98.29	0.10	0.01	100.76
45	0.77	0.20	0.04	0.00	1.58	0.67	0.98	0.02	96.21	0.08	0.01	100.56
50	0.82	0.17	0.04	0.00	2.83	0.74	1.10	0.01	94.80	0.08	0.01	100.59
55	0.28	0.21	0.04	0.00	0.22	0.47	0.20	0.00	98.80	0.08	0.01	100.32
60	0.30	0.22	0.04	0.00	0.24	0.55	0.19	0.01	99.34	0.09	0.01	100.99
65	0.37	0.21	0.04	0.00	0.43	0.61	0.31	0.02	98.77	0.06	0.00	100.81
70	0.41	0.22	0.05	0.02	0.29	0.81	0.27	0.01	98.96	0.10	0.01	101.14
75	0.52	0.20	0.04	0.03	0.61	0.82	0.35	0.02	98.01	0.08	0.00	100.68
80	0.50	0.19	0.03	0.00	0.73	0.70	0.36	0.03	98.24	0.06	0.00	100.84
85	0.67	0.23	0.04	0.00	1.85	0.68	0.50	0.04	96.90	0.06	0.01	100.97
90	0.62	0.20	0.04	0.02	0.97	0.65	0.37	0.02	97.63	0.06	0.00	100.57
95	0.42	0.19	0.04	0.01	0.57	0.64	0.25	0.02	98.78	0.06	0.01	100.98
100	0.49	0.22	0.03	0.00	0.72	0.73	0.31	0.02	98.08	0.08	0.00	100.68
105	0.42	0.21	0.04	0.04	0.82	0.78	0.36	0.02	97.90	0.09	0.00	100.68
110	0.50	0.17	0.04	0.01	0.94	0.71	0.29	0.01	97.95	0.08	0.00	100.67
115	0.57	0.21	0.04	0.00	1.12	0.69	0.34	0.01	97.34	0.07	0.01	100.39
120	0.54	0.24	0.04	0.02	1.09	0.72	0.35	0.01	98.07	0.07	0.00	101.13
125	0.53	0.20	0.04	0.00	1.12	0.75	0.35	0.02	97.60	0.09	0.01	100.71
130	0.36	0.18	0.04	0.00	0.78	0.67	0.24	0.02	98.17	0.08	0.01	100.54
135	0.69	0.24	0.03	0.05	1.62	0.69	0.42	0.01	97.15	0.10	0.00	100.99
140	0.51	0.28	0.03	0.00	0.99	0.71	0.32	0.02	97.94	0.08	0.01	100.89
145	0.55	0.20	0.04	0.03	1.51	0.88	0.55	0.01	96.45	0.10	0.01	100.35
150	0.25	0.22	0.04	0.00	0.47	0.59	0.18	0.01	99.30	0.05	0.01	101.11

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
155	0.63	0.18	0.03	0.00	1.56	0.85	0.64	0.01	96.04	0.11	0.00	100.07
160	0.41	0.18	0.05	0.00	1.19	0.87	0.62	0.01	97.04	0.09	0.01	100.47
165	0.15	0.22	0.04	0.03	0.32	0.58	0.20	0.01	99.72	0.05	0.01	101.32
170	0.42	0.23	0.04	0.00	1.28	0.76	0.74	0.01	96.72	0.10	0.00	100.30
175	0.15	0.20	0.04	0.03	0.22	0.54	0.20	0.01	99.40	0.05	0.01	100.84
180	0.29	0.26	0.03	0.03	0.14	0.55	0.26	0.00	98.59	0.06	0.01	100.22
185	2.50	0.17	0.04	0.01	13.38	19.16	25.53	0.03	38.01	0.76	0.02	99.60
190	0.84	0.25	0.04	0.00	2.09	1.78	3.93	0.01	92.29	0.08	0.01	101.33
195	0.28	0.17	0.04	0.00	0.17	0.57	0.52	0.00	98.81	0.07	0.01	100.64
200	0.51	0.18	0.04	0.02	0.67	0.92	1.62	0.00	96.40	0.06	0.02	100.42
205	0.45	0.20	0.04	0.07	0.67	0.96	1.45	0.01	97.29	0.07	0.01	101.21
210	0.37	0.22	0.04	0.02	0.51	0.64	0.96	0.01	97.72	0.06	0.01	100.55
215	0.30	0.17	0.04	0.03	0.54	0.70	1.08	0.01	97.42	0.07	0.01	100.37
220	0.51	0.19	0.04	0.00	1.18	0.74	2.17	0.02	95.78	0.08	0.01	100.71
225	0.33	0.22	0.04	0.07	0.36	0.70	0.87	0.00	98.72	0.06	0.00	101.37
230	0.31	0.20	0.03	0.00	0.82	0.64	1.29	0.02	97.35	0.05	0.01	100.73
235	0.27	0.23	0.04	0.01	0.60	0.63	1.13	0.01	98.16	0.06	0.01	101.14
240	0.31	0.22	0.03	0.01	0.91	0.65	1.59	0.01	97.35	0.04	0.00	101.12
245	0.35	0.22	0.04	0.00	1.06	0.68	2.07	0.01	96.52	0.09	0.00	101.04
250	0.38	0.19	0.04	0.02	0.84	0.73	1.90	0.02	96.51	0.06	0.00	100.68
255	0.31	0.20	0.04	0.00	0.70	0.78	1.62	0.01	96.60	0.08	0.01	100.34
260	0.29	0.26	0.04	0.01	0.55	0.64	1.19	0.01	97.77	0.07	0.00	100.83
265	0.24	0.23	0.04	0.00	0.38	0.73	1.02	0.02	98.04	0.06	0.01	100.75
270	0.26	0.24	0.04	0.00	0.80	0.70	1.65	0.01	97.01	0.08	0.00	100.78
275	0.23	0.17	0.04	0.01	0.71	0.62	1.64	0.01	96.99	0.08	0.01	100.51
280	0.32	0.23	0.04	0.04	0.84	0.71	2.18	0.02	95.98	0.08	0.01	100.43
285	0.24	0.18	0.04	0.06	0.35	0.52	1.07	0.02	98.00	0.06	0.00	100.53
290	0.48	0.23	0.04	0.00	1.29	1.32	3.48	0.02	92.88	0.08	0.01	99.82
295	0.98	0.21	0.04	0.03	4.58	3.64	16.16	0.03	74.18	0.12	0.02	99.98

**PFE1356: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.61	0.20	0.03	0.00	1.51	1.18	7.12	0.03	92.32	0.16	0.01	104.17
5	5.30	0.20	0.04	0.08	4.06	2.92	22.91	0.04	70.04	0.24	0.01	105.83
10	2.76	0.19	0.04	0.05	1.95	1.13	11.13	0.04	86.83	0.13	0.02	104.28
15	1.95	0.19	0.04	0.04	1.82	1.23	10.87	0.05	87.91	0.15	0.01	104.26
20	1.86	0.20	0.04	0.06	1.70	1.46	10.37	0.11	87.93	0.16	0.02	103.91
25	1.32	0.25	0.03	0.02	1.04	1.09	8.81	0.18	89.45	0.13	0.02	102.33
30	1.88	0.21	0.04	0.02	1.69	2.66	16.19	0.39	77.60	0.16	0.07	100.90
35	0.30	0.19	0.03	0.03	0.24	2.01	2.26	0.41	97.58	0.09	0.15	103.29
40	0.38	0.22	0.04	0.07	0.38	2.72	1.90	0.11	94.98	0.09	0.06	100.93
45	1.43	0.22	0.03	0.02	1.44	0.83	8.14	0.02	91.19	0.09	0.02	103.44
50	1.09	0.22	0.03	0.01	1.54	1.03	6.89	0.04	93.03	0.13	0.01	104.02
55	1.09	0.21	0.03	0.00	1.78	0.96	6.88	0.02	92.37	0.13	0.02	103.50
60	1.59	0.20	0.04	0.11	2.30	3.42	10.38	0.14	82.29	0.14	0.13	100.72
65	0.72	0.20	0.03	0.00	1.37	0.94	4.70	0.04	95.33	0.11	0.01	103.45
70	0.44	0.20	0.03	0.04	0.83	0.89	2.91	0.03	98.24	0.10	0.01	103.72
75	0.64	0.22	0.03	0.00	0.74	0.76	2.85	0.03	98.47	0.11	0.01	103.85
80	0.50	0.25	0.04	0.04	0.79	0.89	2.14	0.04	98.54	0.12	0.01	103.36
85	0.83	0.24	0.03	0.00	1.06	0.96	4.15	0.08	95.81	0.12	0.02	103.29
90	0.76	0.21	0.03	0.04	0.62	0.73	2.21	0.05	98.71	0.09	0.01	103.48
95	0.37	0.19	0.04	0.04	0.47	2.13	1.38	0.65	96.09	0.11	0.06	101.51
100	1.91	0.18	0.04	0.07	2.11	2.99	7.64	0.18	87.55	0.17	0.07	102.90
105	1.07	0.21	0.04	0.01	1.01	0.84	3.67	0.03	96.69	0.12	0.03	103.69
110	0.24	0.19	0.04	0.01	0.22	0.96	0.63	0.10	98.50	0.09	0.03	101.01
115	0.42	0.18	0.03	0.03	0.71	1.40	1.39	0.15	96.02	0.12	0.08	100.54
120	0.69	0.19	0.03	0.08	1.59	1.56	3.70	0.05	93.70	0.12	0.02	101.73
125	0.69	0.17	0.04	0.04	1.03	2.90	2.66	0.07	95.27	0.08	0.09	103.03
130	0.33	0.21	0.03	0.02	0.94	0.93	1.44	0.01	99.31	0.08	0.01	103.32
135	3.63	0.21	0.04	0.07	3.26	0.91	10.24	0.01	86.61	0.13	0.02	105.12
140	0.49	0.25	0.03	0.06	1.20	0.88	1.77	0.02	98.41	0.11	0.01	103.23
145	0.42	0.19	0.04	0.03	0.89	0.76	1.11	0.01	100.18	0.08	0.02	103.71
150	1.70	0.20	0.04	0.06	2.00	1.02	4.41	0.02	93.81	0.13	0.01	103.40

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
155	0.90	0.21	0.04	0.08	0.96	0.75	2.10	0.02	98.22	0.08	0.01	103.36
160	0.27	0.20	0.03	0.05	0.47	0.70	0.86	0.01	100.46	0.07	0.00	103.12
165	0.32	0.19	0.03	0.02	0.88	0.79	1.12	0.03	99.99	0.08	0.01	103.47
170	0.52	0.21	0.03	0.00	1.12	0.85	1.41	0.03	98.62	0.11	0.01	102.91
175	0.55	0.21	0.04	0.02	1.02	0.93	1.34	0.03	99.09	0.10	0.00	103.32
180	0.62	0.22	0.03	0.04	1.09	0.98	1.48	0.02	98.67	0.13	0.01	103.29
185	1.19	0.22	0.03	0.03	1.08	0.94	1.96	0.04	97.58	0.11	0.02	103.19
190	0.52	0.17	0.03	0.02	0.88	0.85	1.17	0.03	99.30	0.11	0.01	103.09
195	0.77	0.23	0.03	0.02	2.11	5.93	2.19	0.03	92.97	0.27	0.00	104.55
200	0.26	0.16	0.04	0.01	0.56	0.68	0.68	0.03	100.88	0.06	0.01	103.37
205	0.29	0.19	0.03	0.02	0.72	0.80	0.93	0.03	100.22	0.09	0.01	103.33
210	1.46	0.19	0.03	0.00	1.07	0.72	3.20	0.02	96.89	0.09	0.01	103.69
215	0.64	0.16	0.04	0.01	1.11	0.94	1.99	0.04	97.32	0.12	0.02	102.39
220	0.32	0.20	0.04	0.05	1.26	1.19	6.07	0.03	94.28	0.12	0.01	103.57
225	1.19	0.22	0.04	0.01	2.00	1.21	4.84	0.03	93.49	0.16	0.01	103.20
230	1.98	0.16	0.03	0.04	3.95	4.49	13.67	0.05	79.19	0.28	0.02	103.86
235	2.32	0.22	0.04	0.04	4.15	2.08	18.14	0.03	77.28	0.17	0.00	104.47
240	2.69	0.21	0.04	0.05	4.42	4.84	18.94	0.04	70.45	0.24	0.02	101.94
245	0.51	0.06	0.05	0.00	1.04	3.22	4.52	0.22	50.23	0.14	0.05	60.03
250	0.44	0.20	0.04	0.00	0.68	0.92	2.94	0.03	97.48	0.09	0.02	102.83
255	0.54	0.20	0.04	0.06	0.82	0.82	3.40	0.03	96.50	0.11	0.02	102.53
260	0.87	0.22	0.03	0.03	1.04	1.00	6.13	0.05	93.82	0.14	0.01	103.35
265	1.51	0.21	0.03	0.00	1.75	0.90	11.90	0.02	87.47	0.16	0.01	103.97
270	1.35	0.21	0.04	0.00	1.55	1.21	12.81	0.03	85.30	0.15	0.01	102.64
275	0.82	0.19	0.04	0.03	0.99	1.43	10.63	0.03	88.13	0.13	0.01	102.43
280	1.00	0.18	0.04	0.03	0.98	0.89	10.86	0.06	88.64	0.10	0.01	102.79
285	0.46	0.21	0.03	0.04	0.40	0.74	4.63	0.04	95.95	0.06	0.01	102.55
290	1.48	0.21	0.03	0.05	1.75	5.69	18.75	0.06	72.92	0.09	0.03	101.05
295	0.00	0.00	0.00	0.00	0.00	0.00	0.49	0.00	8.40	0.00	0.00	8.89

**PFE1356: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.59	0.26	0.04	0.05	1.55	1.02	1.70	0.01	95.82	0.13	0.01	101.18
5	2.36	0.20	0.03	0.04	2.32	0.88	5.38	0.02	90.58	0.10	0.01	101.92
10	0.25	0.24	0.04	0.03	0.48	0.70	0.62	0.03	99.06	0.06	0.01	101.50
15	0.93	0.21	0.04	0.05	1.55	1.74	6.11	0.04	90.28	0.12	0.02	101.09
20	0.19	0.22	0.04	0.06	0.48	3.69	1.25	0.07	95.47	0.08	0.06	101.60
25	1.46	0.20	0.04	0.07	2.87	1.24	8.89	0.06	86.44	0.16	0.03	101.47
30	1.33	0.17	0.04	0.03	1.84	1.14	4.81	0.03	92.15	0.12	0.02	101.67
35	0.98	0.23	0.04	0.06	1.42	0.87	2.81	0.02	94.85	0.12	0.01	101.41
40	1.22	0.23	0.04	0.03	2.72	1.24	5.27	0.02	90.38	0.16	0.01	101.30
45	0.84	0.20	0.04	0.01	1.56	0.82	1.96	0.03	92.98	0.11	0.01	98.54
50	0.59	0.21	0.04	0.03	1.20	0.94	1.36	0.03	96.97	0.09	0.00	101.45
55	0.60	0.19	0.04	0.05	0.98	0.85	1.23	0.02	94.51	0.08	0.01	98.55
60	0.41	0.21	0.04	0.05	0.67	1.32	0.71	0.06	93.87	0.07	0.03	97.41
65	0.85	0.25	0.03	0.01	1.45	1.31	1.70	0.03	96.31	0.13	0.01	102.08
70	1.11	0.18	0.04	0.05	1.96	1.02	2.18	0.02	94.72	0.12	0.01	101.43
75	0.29	0.20	0.04	0.00	0.63	0.83	0.81	0.04	94.85	0.08	0.01	97.77
80	0.73	0.22	0.04	0.01	1.22	0.79	1.29	0.02	96.87	0.10	0.01	101.29
85	1.46	0.20	0.04	0.06	1.75	1.24	2.39	0.03	94.01	0.11	0.02	101.30
90	1.07	0.19	0.04	0.01	2.10	1.15	2.26	0.03	92.14	0.14	0.00	99.11
95	0.50	0.20	0.03	0.07	0.84	0.76	1.18	0.01	97.32	0.09	0.01	101.03
100	1.64	0.21	0.04	0.02	2.80	1.15	3.76	0.01	91.16	0.17	0.01	100.96

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
105	1.11	0.23	0.04	0.06	1.61	0.88	2.38	0.04	92.35	0.12	0.01	98.81
110	0.38	0.19	0.04	0.01	0.53	0.69	0.60	0.02	94.06	0.07	0.01	96.59
115	0.70	0.18	0.04	0.00	1.49	0.97	1.80	0.03	94.02	0.12	0.01	99.37
120	0.19	0.21	0.04	0.06	0.44	0.72	0.50	0.02	99.27	0.08	0.01	101.53
125	1.91	0.19	0.04	0.03	2.39	1.03	4.16	0.01	90.96	0.09	0.00	100.81
130	0.88	0.22	0.04	0.00	1.75	1.05	2.18	0.00	93.85	0.12	0.01	100.08
135	1.62	0.23	0.04	0.03	2.16	1.86	3.23	0.05	89.48	0.18	0.01	98.86
140	0.89	0.22	0.04	0.05	1.22	0.81	1.74	0.04	92.88	0.11	0.01	98.01
145	1.38	0.23	0.04	0.01	1.37	0.84	2.26	0.03	95.28	0.11	0.00	101.55
150	0.45	0.19	0.04	0.00	0.89	1.00	1.30	0.02	96.41	0.12	0.00	100.41
155	1.02	0.17	0.04	0.01	1.24	0.88	2.15	0.06	91.42	0.14	0.01	97.15
160	1.07	0.23	0.04	0.02	1.09	0.65	1.87	0.03	93.67	0.10	0.01	98.76
165	1.35	0.21	0.04	0.00	2.28	1.51	3.64	0.05	89.68	0.16	0.01	98.91
170	1.88	0.18	0.04	0.02	2.91	1.52	5.87	0.04	82.14	0.17	0.01	94.77
175	1.49	0.22	0.04	0.02	1.78	1.03	5.62	0.04	89.96	0.14	0.01	100.34
180	1.04	0.23	0.04	0.00	1.43	1.13	4.49	0.02	92.77	0.12	0.01	101.28
185	0.79	0.23	0.04	0.04	1.07	0.98	3.63	0.05	93.99	0.11	0.02	100.94
190	1.12	0.25	0.03	0.00	1.62	1.64	6.74	0.05	90.62	0.16	0.01	102.23
195	2.34	0.19	0.04	0.01	2.09	2.31	12.85	0.06	76.00	0.14	0.02	96.04
200	1.43	0.19	0.04	0.07	1.96	11.15	29.62	0.42	56.19	0.15	0.17	101.39

**PFE1356: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.53	0.19	0.04	0.01	1.90	1.06	6.20	0.01	91.45	0.15	0.01	102.56
5	0.46	0.27	0.04	0.00	0.61	0.67	1.50	0.05	97.74	0.09	0.02	101.45
10	1.74	0.25	0.04	0.01	2.10	9.19	7.82	0.14	84.54	0.15	0.06	106.03
15	1.76	0.22	0.04	0.03	2.22	1.26	7.27	0.01	89.51	0.16	0.01	102.49
20	1.63	0.24	0.04	0.04	2.48	1.15	7.49	0.01	89.33	0.21	0.01	102.61
25	0.96	0.24	0.04	0.02	1.03	0.69	3.75	0.02	95.39	0.10	0.01	102.25
30	0.86	0.18	0.05	0.01	2.21	1.62	4.92	0.04	90.63	0.18	0.03	100.73
35	1.30	0.19	0.04	0.01	1.05	1.03	3.90	0.05	93.68	0.11	0.01	101.37
40	0.45	0.22	0.04	0.04	0.44	0.67	1.41	0.01	98.58	0.08	0.02	101.96
45	0.96	0.23	0.03	0.01	1.58	1.20	4.92	0.02	92.50	0.16	0.02	101.63
50	0.65	0.20	0.04	0.09	1.57	1.34	3.45	0.03	94.46	0.14	0.02	101.96
55	0.86	0.22	0.04	0.03	1.70	4.96	4.28	0.13	86.09	0.17	0.06	98.53
60	1.06	0.26	0.04	0.00	0.96	0.76	3.74	0.03	94.36	0.11	0.02	101.52
65	0.92	0.24	0.04	0.00	0.96	0.99	3.49	0.01	94.54	0.14	0.01	101.15
70	1.89	0.21	0.04	0.06	1.44	0.91	5.00	0.03	93.03	0.12	0.02	102.73
75	0.51	0.21	0.03	0.04	0.30	0.60	0.80	0.03	99.07	0.09	0.03	101.72
80	2.82	0.24	0.04	0.05	2.37	1.09	8.18	0.04	88.11	0.21	0.03	103.17
85	1.53	0.22	0.04	0.09	0.63	3.90	3.38	0.12	93.53	0.10	0.12	103.65
90	2.33	0.24	0.04	0.03	1.04	0.79	4.95	0.04	92.61	0.13	0.04	102.23
95	0.95	0.23	0.04	0.00	0.76	0.79	2.23	0.04	96.42	0.14	0.03	101.62
100	0.54	0.23	0.03	0.01	0.31	0.71	1.00	0.05	98.38	0.08	0.05	101.39
105	4.95	0.22	0.04	0.06	2.44	1.43	14.70	0.07	78.57	0.15	0.09	102.72

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
110	1.47	0.23	0.06	0.00	0.28	2.44	2.29	7.70	75.11	0.07	6.30	95.94
115	0.75	0.26	0.04	0.07	0.42	0.69	1.49	0.06	96.61	0.09	0.11	100.59
120	0.73	0.23	0.05	0.02	0.22	2.77	1.36	0.13	93.90	0.10	0.11	99.62
125	0.59	0.22	0.04	0.01	0.28	0.62	0.81	0.06	98.77	0.10	0.02	101.52
130	1.44	0.22	0.04	0.02	0.61	0.61	2.55	0.10	94.93	0.09	0.06	100.66
135	3.18	0.22	0.04	0.00	1.80	1.21	6.73	0.23	85.49	0.15	0.02	99.06
140	1.53	0.20	0.04	0.02	1.90	1.64	4.66	0.05	89.80	0.19	0.03	100.05
145	3.03	0.21	0.04	0.03	2.02	1.05	9.96	0.06	84.49	0.16	0.01	101.07
150	0.26	0.17	0.04	0.01	0.27	2.14	0.80	0.09	95.25	0.09	0.03	99.13
155	0.62	0.19	0.04	0.00	0.34	3.99	2.32	0.13	89.47	0.10	0.04	97.24
160	1.56	0.22	0.03	0.01	1.18	1.04	6.87	0.04	91.20	0.14	0.02	102.31
165	2.18	0.23	0.04	0.05	1.61	1.17	10.98	0.04	86.00	0.15	0.01	102.45
170	3.80	0.19	0.04	0.06	3.14	2.72	23.27	0.06	68.29	0.20	0.01	101.77
175	0.22	0.20	0.03	0.05	0.31	0.69	1.28	0.04	98.45	0.10	0.01	101.36
180	2.67	0.22	0.05	0.07	2.13	1.45	18.40	0.06	79.14	0.17	0.04	104.38
185	2.01	0.23	0.04	0.02	1.74	1.75	17.63	0.05	78.62	0.15	0.01	102.25
190	2.31	0.24	0.03	0.02	1.68	1.13	16.44	0.03	81.17	0.15	0.02	103.23
195	0.22	0.19	0.05	0.03	0.11	3.73	1.08	0.10	92.38	0.06	0.04	98.00
200	3.14	0.17	0.05	0.00	1.54	12.04	23.37	0.19	50.78	0.12	0.09	91.49
205	0.01	0.00	0.01	0.00	0.00	6.41	0.86	0.09	8.21	0.00	0.04	15.62



**PFE1361: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.58	0.24	0.04	0.07	1.26	0.94	1.05	0.03	97.28	0.11	0.03	101.62
5	0.55	0.22	0.04	0.00	1.29	0.88	0.98	0.02	97.76	0.10	0.02	101.86
10	0.33	0.21	0.04	0.00	0.61	4.83	0.73	0.13	88.50	0.07	0.09	95.54
15	0.46	0.24	0.04	0.00	0.92	0.95	0.72	0.03	98.04	0.08	0.00	101.48
20	0.57	0.23	0.04	0.04	0.95	0.77	0.79	0.02	98.37	0.07	0.01	101.85
25	0.79	0.23	0.04	0.08	1.34	1.10	1.23	0.02	96.57	0.12	0.01	101.51
30	6.19	1.00	0.04	0.06	1.19	0.98	1.03	0.04	96.36	0.11	0.01	107.01
35	0.81	0.22	0.03	0.02	1.38	0.94	1.21	0.01	96.95	0.15	0.01	101.74
40	0.68	0.22	0.04	0.01	1.06	0.90	1.10	0.02	97.51	0.10	0.01	101.66
45	0.80	0.21	0.03	0.04	2.02	1.69	1.64	0.04	91.69	0.14	0.01	98.32
50	0.44	0.24	0.04	0.09	0.70	3.17	0.73	0.09	93.51	0.09	0.05	99.15
55	0.16	0.22	0.03	0.04	0.18	0.59	0.08	0.01	100.31	0.05	0.01	101.69
60	0.62	0.21	0.04	0.01	1.54	1.15	1.91	0.02	96.05	0.13	0.01	101.67
65	1.22	0.24	0.04	0.01	1.94	0.98	3.51	0.05	92.91	0.14	0.01	101.06
70	0.68	0.24	0.04	0.03	1.60	1.08	2.06	0.02	95.93	0.17	0.02	101.86
75	0.43	0.20	0.04	0.02	0.80	0.84	0.98	0.02	98.22	0.06	0.01	101.62
80	0.72	0.23	0.04	0.05	1.13	0.92	2.06	0.04	95.84	0.10	0.01	101.11
85	0.45	0.20	0.04	0.02	1.06	0.99	1.28	0.02	97.60	0.08	0.01	101.73
90	0.30	0.18	0.03	0.01	0.73	0.87	1.00	0.03	93.85	0.06	0.01	97.06
95	0.59	0.20	0.03	0.04	1.35	1.15	1.69	0.02	96.38	0.10	0.02	101.56
100	0.27	0.15	0.03	0.00	1.09	4.03	1.11	0.09	81.50	0.06	0.03	88.37
105	0.07	0.20	0.04	0.00	0.13	3.90	0.41	0.13	88.55	0.10	0.05	93.58

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
110	0.09	0.12	0.04	0.07	0.15	1.74	0.14	0.08	89.98	0.07	0.03	92.48
115	0.10	0.18	0.03	0.00	0.11	3.15	0.15	0.09	89.80	0.06	0.02	93.69
120	0.10	0.17	0.03	0.02	0.12	1.55	0.07	0.04	94.93	0.07	0.01	97.11
125	0.20	0.21	0.04	0.00	0.14	2.07	0.62	0.05	98.27	0.07	0.02	101.68
130	0.09	0.24	0.03	0.00	0.11	1.08	0.14	0.02	97.65	0.07	0.01	99.44
135	0.07	0.21	0.03	0.03	0.13	1.04	0.08	0.02	93.85	0.05	0.01	95.51
140	0.10	0.13	0.03	0.00	0.11	0.54	0.11	0.02	80.70	0.03	0.00	81.75
145	0.18	0.21	0.04	0.03	0.28	0.65	0.82	0.03	98.72	0.07	0.00	101.00
150	0.30	0.22	0.04	0.10	0.95	0.88	1.76	0.03	96.84	0.09	0.01	101.23
155	0.44	0.24	0.04	0.06	0.99	1.00	2.13	0.03	96.85	0.13	0.01	101.91
160	0.32	0.18	0.04	0.01	0.88	0.97	1.79	0.03	97.03	0.11	0.01	101.36
165	0.32	0.21	0.04	0.00	0.79	0.92	1.48	0.02	95.08	0.11	0.01	98.98
170	0.35	0.23	0.03	0.05	0.88	0.97	1.67	0.03	97.91	0.11	0.01	102.25
175	0.47	0.20	0.04	0.02	0.87	0.95	1.92	0.03	96.83	0.15	0.01	101.49
180	0.17	0.21	0.04	0.07	0.36	0.80	0.51	0.04	96.38	0.09	0.02	98.68
185	0.09	0.15	0.03	0.00	0.05	0.56	0.05	0.03	87.77	0.07	0.01	88.82
190	0.14	0.19	0.04	0.04	0.13	0.53	0.27	0.02	94.57	0.10	0.01	96.05
195	0.10	0.20	0.03	0.02	0.15	0.54	0.03	0.02	97.79	0.11	0.01	99.00
200	0.15	0.16	0.03	0.01	0.16	0.58	0.06	0.02	99.52	0.09	0.01	100.80
205	0.11	0.21	0.03	0.08	0.13	0.62	0.01	0.03	99.44	0.07	0.00	100.72
210	0.00	0.07	0.02	0.00	0.00	0.33	0.00	0.01	54.92	0.00	0.00	55.35

**PFE1361: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totale
0	0.11	0.24	0.04	0.01	0.05	1.08	0.00	0.07	94.42	0.08	0.02	96.10
5	0.15	0.19	0.04	0.02	0.13	2.29	0.32	0.06	98.26	0.08	0.03	101.55
10	0.10	0.18	0.04	0.05	0.09	0.83	0.35	0.05	94.92	0.07	0.01	96.68
15	0.12	0.20	0.03	0.03	0.15	0.63	0.11	0.02	95.95	0.06	0.01	97.31
20	0.06	0.19	0.03	0.00	0.05	0.66	0.02	0.04	93.71	0.06	0.01	94.82
25	0.06	0.19	0.04	0.01	0.08	0.66	0.01	0.02	93.00	0.05	0.01	94.12
30	0.12	0.18	0.04	0.01	0.06	1.73	0.09	0.07	90.53	0.05	0.05	92.92
35	0.09	0.20	0.03	0.03	0.07	0.62	0.00	0.02	95.27	0.07	0.01	96.42
40	0.05	0.19	0.04	0.03	0.13	1.01	0.04	0.11	92.29	0.06	0.03	93.98
45	0.07	0.15	0.03	0.00	0.14	0.79	0.04	0.13	95.08	0.07	0.05	96.54
50	0.10	0.21	0.04	0.03	0.10	0.83	0.07	0.08	96.47	0.08	0.02	98.04
55	0.07	0.21	0.03	0.08	0.14	0.63	0.04	0.06	97.86	0.09	0.02	99.24
60	0.07	0.21	0.04	0.00	0.15	0.51	0.05	0.04	97.57	0.07	0.01	98.71
65	0.10	0.20	0.04	0.00	0.18	0.77	0.22	0.04	96.36	0.06	0.01	97.98
70	0.09	0.24	0.04	0.06	0.12	0.60	0.00	0.03	98.66	0.08	0.02	99.93
75	0.12	0.20	0.04	0.00	0.19	2.23	0.24	0.13	93.18	0.06	0.08	96.46
80	0.10	0.20	0.04	0.04	0.20	0.69	0.17	0.06	97.15	0.08	0.02	98.74
85	0.12	0.25	0.04	0.00	0.13	0.55	0.04	0.04	96.50	0.09	0.01	97.74
90	0.14	0.19	0.03	0.00	0.37	1.83	0.30	0.06	85.95	0.15	0.02	89.02

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totale
95	0.09	0.21	0.04	0.04	0.13	0.83	0.04	0.08	95.27	0.08	0.01	96.81
100	0.11	0.14	0.03	0.00	0.18	4.03	0.17	0.10	75.30	0.11	0.05	80.22
105	0.10	0.14	0.03	0.00	0.00	0.47	0.00	0.09	71.73	0.04	0.02	72.61
110	0.09	0.23	0.04	0.04	0.23	0.71	0.30	0.03	98.15	0.07	0.00	99.89
115	0.12	0.19	0.03	0.00	0.20	0.61	0.22	0.03	94.48	0.06	0.00	95.94
120	0.19	0.21	0.04	0.00	0.42	0.69	0.62	0.02	99.39	0.04	0.02	101.65
125	0.19	0.18	0.03	0.02	0.47	0.81	0.96	0.04	94.65	0.07	0.01	97.42
130	0.07	0.17	0.04	0.00	0.03	1.98	0.06	0.13	87.91	0.06	0.06	90.49
135	0.09	0.18	0.04	0.06	0.10	0.67	0.09	0.02	97.74	0.07	0.01	99.07
140	0.07	0.20	0.03	0.00	0.14	1.46	0.08	0.05	89.10	0.09	0.02	91.23
145	0.08	0.15	0.03	0.00	0.28	1.50	0.44	0.06	77.44	0.09	0.01	80.08
150	0.09	0.23	0.04	0.03	0.14	0.61	1.83	0.00	97.58	0.09	0.00	100.64
155	0.10	0.21	0.03	0.01	0.08	0.91	0.00	0.04	93.30	0.08	0.01	94.75
160	5.83	0.46	0.03	0.00	0.05	0.91	0.00	0.04	83.40	0.07	0.02	90.79
165	0.10	0.18	0.03	0.00	0.08	0.62	0.01	0.03	94.25	0.09	0.01	95.40
170	0.09	0.16	0.04	0.00	0.10	0.58	0.01	0.03	95.52	0.09	0.01	96.62
175	0.15	0.23	0.04	0.04	0.18	0.53	0.29	0.02	99.18	0.08	0.01	100.75
180	0.36	0.21	0.04	0.00	1.10	5.63	1.50	0.06	85.47	0.10	0.02	94.48

### PFE361: Particle 3

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totale
0	0.11	0.21	0.04	0.00	0.16	0.52	0.15	0.13	98.71	0.07	0.05	100.15
5	0.10	0.25	0.03	0.03	0.13	0.70	0.05	0.06	99.20	0.08	0.04	100.66
10	0.12	0.18	0.03	0.07	0.11	0.94	0.05	0.05	99.78	0.08	0.04	101.44
15	0.09	0.22	0.04	0.05	0.06	1.71	0.05	0.13	94.48	0.06	0.16	97.04
20	0.10	0.21	0.04	0.04	0.06	0.68	0.16	0.07	97.61	0.06	0.04	99.07
25	0.11	0.24	0.03	0.00	0.14	0.95	0.02	0.07	99.81	0.08	0.02	101.45
30	0.08	0.24	0.04	0.01	0.12	0.48	0.08	0.10	98.97	0.07	0.04	100.21
35	0.11	0.22	0.03	0.00	0.13	0.60	0.04	0.09	96.90	0.06	0.05	98.22
40	0.11	0.24	0.04	0.03	0.10	3.20	0.03	0.14	96.22	0.07	0.16	100.34
45	0.12	0.20	0.04	0.01	0.11	0.59	0.10	0.03	95.74	0.05	0.01	96.99
50	0.11	0.22	0.04	0.08	0.13	0.64	0.11	0.05	98.49	0.07	0.02	99.96
55	0.10	0.25	0.03	0.00	0.11	0.58	0.02	0.08	98.83	0.08	0.01	100.08
60	0.08	0.23	0.04	0.00	0.08	0.48	0.05	0.03	99.32	0.06	0.02	100.39
65	0.08	0.24	0.03	0.00	0.13	0.69	0.02	0.09	98.87	0.09	0.02	100.24
70	0.07	0.21	0.04	0.04	0.15	0.56	0.06	0.08	99.03	0.07	0.01	100.31
75	0.10	0.24	0.03	0.05	0.11	0.87	0.02	0.11	98.58	0.07	0.05	100.22
80	0.15	0.21	0.04	0.01	0.14	0.72	0.26	0.06	97.76	0.04	0.03	99.41
85	0.12	0.20	0.04	0.00	0.14	0.62	0.10	0.04	97.84	0.08	0.03	99.21
90	0.09	0.18	0.04	0.03	0.09	4.51	0.10	0.16	94.12	0.07	0.08	99.47

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totale
95	0.11	0.20	0.03	0.00	0.20	2.02	0.28	0.08	97.57	0.05	0.06	100.59
100	0.08	0.19	0.04	0.02	0.11	0.74	0.15	0.07	99.54	0.09	0.01	101.03
105	0.23	0.19	0.05	0.10	0.23	0.66	0.50	0.05	98.04	0.09	0.01	100.14
110	0.09	0.25	0.04	0.00	0.16	0.69	0.08	0.06	99.11	0.09	0.02	100.58
115	0.05	0.24	0.03	0.03	0.07	10.50	0.10	0.23	87.69	0.06	0.08	99.08
120	0.11	0.17	0.04	0.02	0.10	0.62	0.09	0.05	96.90	0.07	0.01	98.17
125	0.10	0.22	0.03	0.00	0.07	1.22	0.13	0.05	92.77	0.08	0.03	94.69
130	0.14	0.20	0.04	0.03	0.19	1.27	0.31	0.06	97.63	0.09	0.01	99.96
135	0.07	0.21	0.03	0.00	0.13	1.49	0.13	0.08	90.23	0.07	0.02	92.45
140	0.09	0.17	0.03	0.02	0.10	2.21	0.11	0.18	79.73	0.06	0.07	82.77
145	0.10	0.20	0.04	0.01	0.13	0.72	0.14	0.06	98.25	0.11	0.01	99.77
150	0.07	0.20	0.03	0.01	0.13	0.73	0.04	0.05	96.85	0.09	0.01	98.22
155	0.03	0.19	0.04	0.01	0.06	0.73	0.06	0.07	85.54	0.06	0.01	86.79
160	0.10	0.18	0.03	0.00	0.01	0.64	0.07	0.06	92.48	0.09	0.02	93.69
165	0.07	0.25	0.03	0.07	0.13	0.63	0.05	0.04	98.35	0.10	0.01	99.73
170	0.05	0.21	0.03	0.00	0.11	0.92	0.36	0.04	88.65	0.05	0.01	90.42
175	0.10	0.21	0.04	0.00	0.00	0.83	0.02	0.04	93.04	0.09	0.01	94.37
180	0.09	0.22	0.04	0.00	0.00	2.50	0.10	0.11	92.66	0.05	0.06	95.82

**PFE1500: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.96	0.20	0.05	0.00	1.98	1.10	19.12	0.02	78.47	0.13	0.02	102.04
5	1.09	0.23	0.04	0.03	2.01	1.12	19.81	0.01	77.60	0.13	0.01	102.07
10	1.07	0.24	0.04	0.03	1.95	1.12	19.51	0.03	77.20	0.15	0.01	101.34
15	0.98	0.23	0.04	0.07	1.65	1.26	14.49	0.03	81.81	0.13	0.02	100.70
20	0.97	0.20	0.04	0.03	1.69	1.17	22.15	0.05	74.09	0.12	0.01	100.52
25	1.36	0.24	0.04	0.03	1.70	1.06	26.47	0.02	71.03	0.14	0.02	102.09
30	1.38	0.23	0.05	0.02	1.70	1.06	26.57	0.03	70.56	0.12	0.02	101.71
35	0.77	0.21	0.04	0.03	1.50	1.24	18.03	0.02	78.69	0.13	0.02	100.67
40	1.26	0.20	0.04	0.03	1.57	1.30	16.48	0.03	80.34	0.13	0.01	101.40
45	1.21	0.24	0.04	0.00	1.60	1.19	19.97	0.03	76.95	0.15	0.01	101.38
50	1.28	0.26	0.04	0.01	1.55	1.16	20.37	0.02	77.17	0.12	0.01	101.97
55	1.36	0.25	0.04	0.06	1.58	1.05	21.57	0.03	76.50	0.13	0.01	102.57
60	1.42	0.22	0.04	0.08	1.54	1.15	21.59	0.03	76.02	0.13	0.01	102.23
65	1.51	0.27	0.04	0.04	1.59	1.12	21.81	0.04	76.04	0.13	0.01	102.60
70	1.71	0.25	0.05	0.02	1.38	1.11	22.07	0.02	75.37	0.14	0.01	102.14
75	1.63	0.23	0.04	0.04	1.51	1.21	19.20	0.05	77.14	0.14	0.02	101.22
80	2.37	0.28	0.04	0.02	1.71	0.92	21.92	0.07	73.88	0.09	0.04	101.34
85	2.37	0.24	0.05	0.03	1.48	1.09	20.86	0.10	74.90	0.13	0.05	101.28
90	3.95	0.26	0.08	0.08	1.22	0.77	22.94	2.64	67.20	0.12	0.08	99.34
95	6.11	0.25	0.30	0.03	1.13	1.81	26.34	22.89	42.46	0.09	0.41	101.82
100	7.99	0.27	0.12	0.08	1.67	1.01	35.19	7.26	51.55	0.11	0.17	105.43
105	1.94	0.20	0.48	0.00	0.26	2.90	18.24	50.59	20.44	0.05	1.05	96.14
110	2.44	0.27	0.08	0.06	1.65	1.15	20.66	3.94	72.62	0.10	0.46	103.43

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
115	2.18	0.28	0.05	0.03	1.43	1.22	21.13	0.12	75.78	0.11	0.07	102.39
120	1.78	0.27	0.04	0.02	1.45	1.14	21.20	0.06	75.89	0.13	0.07	102.04
125	1.65	0.25	0.04	0.08	1.48	1.07	20.80	0.05	75.86	0.14	0.04	101.46
130	1.44	0.25	0.05	0.06	1.36	1.10	20.21	0.05	76.48	0.15	0.04	101.17
135	1.48	0.24	0.05	0.01	1.51	1.10	20.75	0.05	75.66	0.13	0.04	101.00
140	1.41	0.27	0.04	0.05	1.71	1.02	20.12	0.04	76.24	0.12	0.04	101.06
145	1.23	0.28	0.04	0.03	1.50	1.20	14.51	0.05	81.22	0.14	0.03	100.23
150	1.79	0.30	0.05	0.10	1.43	1.12	24.76	0.05	71.22	0.12	0.04	100.97
155	0.99	0.26	0.04	0.07	1.59	1.36	18.96	0.04	76.66	0.13	0.03	100.12
160	1.19	0.23	0.04	0.08	1.63	1.02	17.90	0.04	78.27	0.12	0.02	100.55
165	1.21	0.20	0.04	0.03	1.58	1.20	19.44	0.03	76.42	0.12	0.02	100.30
170	1.18	0.20	0.05	0.05	1.66	1.08	18.75	0.03	77.21	0.12	0.02	100.35
175	1.13	0.23	0.04	0.06	1.68	1.14	18.66	0.05	77.02	0.13	0.03	100.16
180	1.19	0.17	0.04	0.02	1.83	1.00	16.25	0.03	78.99	0.12	0.03	99.67
185	1.17	0.23	0.04	0.00	1.74	1.03	16.68	0.03	77.99	0.13	0.03	99.07
190	1.15	0.23	0.05	0.04	1.77	0.95	17.44	0.03	76.96	0.13	0.02	98.76
195	1.19	0.23	0.04	0.00	1.81	0.90	16.07	0.03	78.07	0.12	0.03	98.49
200	0.54	0.23	0.04	0.04	0.85	0.90	8.60	0.03	86.32	0.12	0.03	97.69
205	1.13	0.20	0.04	0.00	1.82	1.17	23.48	0.03	69.64	0.13	0.01	97.66
210	1.02	0.24	0.05	0.01	1.85	1.09	22.19	0.03	71.39	0.13	0.02	98.01
215	1.23	0.19	0.04	0.05	1.93	1.23	22.76	0.03	71.07	0.14	0.03	98.69
220	1.01	0.25	0.05	0.00	1.91	1.28	25.97	0.04	67.44	0.14	0.03	98.12



**PFE1500: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.69	0.19	0.04	0.05	2.36	1.23	15.73	0.02	82.84	0.12	0.00	103.26
5	0.69	0.19	0.05	0.05	2.32	1.11	15.89	0.02	82.92	0.11	0.01	103.35
10	0.67	0.21	0.04	0.06	2.32	1.17	16.36	0.00	82.22	0.13	0.01	103.19
15	0.77	0.20	0.04	0.06	2.22	1.15	16.12	0.01	82.45	0.13	0.02	103.15
20	0.74	0.23	0.04	0.01	2.37	1.14	15.92	0.02	82.24	0.11	0.00	102.82
25	0.65	0.22	0.04	0.07	2.24	1.06	16.80	0.03	81.57	0.11	0.02	102.80
30	0.74	0.22	0.04	0.05	2.11	5.77	18.98	0.18	73.39	0.14	0.17	101.78
35	0.59	0.24	0.04	0.04	2.06	1.20	15.37	0.01	82.29	0.12	0.02	101.99
40	0.67	0.22	0.04	0.05	2.26	1.12	16.59	0.02	81.46	0.12	0.01	102.56
45	0.67	0.24	0.03	0.02	2.39	1.14	16.81	0.01	81.47	0.12	0.00	102.90
50	0.71	0.23	0.04	0.07	2.21	1.16	16.02	0.01	81.83	0.12	0.01	102.41
55	0.77	0.22	0.03	0.05	2.29	1.16	17.70	0.01	81.21	0.12	0.00	103.56
60	0.70	0.18	0.03	0.00	2.42	1.14	17.69	0.02	81.38	0.12	0.01	103.67
65	0.70	0.24	0.04	0.05	2.35	1.15	17.54	0.01	81.51	0.12	0.02	103.72
70	0.71	0.20	0.04	0.04	2.30	1.13	17.50	0.01	81.59	0.12	0.01	103.64
75	0.69	0.22	0.04	0.01	2.33	1.16	17.81	0.01	81.35	0.11	0.02	103.73
80	0.67	0.23	0.03	0.05	2.22	1.13	18.02	0.01	80.98	0.11	0.00	103.46
85	0.68	0.22	0.04	0.03	2.21	1.27	18.12	0.02	80.90	0.13	0.02	103.63
90	0.85	0.22	0.04	0.05	2.19	1.16	18.70	0.01	80.51	0.12	0.01	103.84

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
95	0.88	0.24	0.04	0.05	2.19	1.13	19.02	0.02	79.52	0.12	0.01	103.21
100	1.03	0.23	0.04	0.04	1.89	1.16	19.57	0.03	79.18	0.13	0.03	103.33
105	1.92	0.21	0.05	0.01	1.68	1.05	19.33	0.46	76.73	0.13	0.05	101.60
110	1.76	0.23	0.04	0.00	1.67	1.18	14.18	0.08	81.99	0.12	0.02	101.26
115	1.32	0.23	0.04	0.06	1.65	1.12	18.33	0.04	79.61	0.12	0.01	102.53
120	1.19	0.27	0.03	0.01	1.63	1.15	17.67	0.06	80.17	0.13	0.01	102.31
125	1.13	0.27	0.04	0.10	1.79	1.07	16.52	0.03	80.80	0.12	0.02	101.89
130	1.12	0.21	0.03	0.04	1.78	1.13	15.89	0.05	80.99	0.13	0.01	101.37
135	1.09	0.21	0.04	0.05	1.84	1.21	15.02	0.04	81.88	0.13	0.01	101.52
140	0.98	0.20	0.03	0.04	1.89	1.14	14.38	0.03	82.88	0.14	0.01	101.69
145	0.92	0.22	0.04	0.04	1.99	1.13	13.68	0.03	83.32	0.12	0.02	101.51
150	0.92	0.21	0.04	0.00	1.78	1.13	12.49	0.02	84.30	0.14	0.01	101.03
155	0.90	0.22	0.03	0.06	2.04	1.13	12.65	0.03	84.07	0.13	0.01	101.26
160	0.89	0.26	0.03	0.03	1.84	1.07	12.52	0.03	83.22	0.12	0.03	100.04
165	0.87	0.23	0.03	0.07	1.89	1.12	12.98	0.02	82.88	0.13	0.02	100.22
170	0.87	0.24	0.04	0.03	1.82	1.05	12.42	0.01	83.25	0.13	0.01	99.87
175	0.81	0.26	0.03	0.03	1.88	1.33	14.41	0.03	81.07	0.13	0.02	100.00
180	0.92	0.24	0.04	0.07	2.36	1.46	21.35	0.03	73.16	0.13	0.02	99.77

**PFE1500: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.61	0.24	0.03	0.03	2.40	1.09	13.08	0.02	84.00	0.13	0.00	101.64
5	0.70	0.24	0.04	0.01	2.36	1.58	13.30	0.03	83.41	0.13	0.03	101.81
10	0.52	0.24	0.04	0.03	2.08	1.10	9.59	0.02	87.09	0.11	0.00	100.82
15	0.51	0.22	0.04	0.03	2.27	1.33	10.63	0.02	86.51	0.11	0.01	101.68
20	1.28	0.26	0.04	0.11	3.45	4.38	35.23	0.01	58.28	0.19	0.01	103.23
25	0.36	0.24	0.04	0.02	2.23	1.41	10.91	0.02	85.35	0.13	0.01	100.69
30	0.53	0.23	0.04	0.00	2.49	1.34	12.68	0.01	84.10	0.13	0.01	101.55
35	0.60	0.21	0.04	0.07	2.28	1.13	14.13	0.02	83.19	0.14	0.00	101.81
40	0.63	0.25	0.03	0.00	2.16	1.18	11.81	0.02	85.32	0.12	0.01	101.53
45	0.66	0.21	0.04	0.04	2.36	1.24	15.44	0.02	81.99	0.13	0.00	102.12
50	0.67	0.25	0.04	0.05	2.32	1.13	14.36	0.01	83.43	0.11	0.01	102.37
55	0.94	0.20	0.04	0.07	2.22	1.09	16.78	0.01	81.03	0.11	0.01	102.47
60	1.34	0.22	0.08	0.12	2.16	1.14	16.07	1.56	80.35	0.12	0.14	103.30
65	0.81	0.26	0.04	0.10	2.27	1.23	15.55	0.02	81.86	0.12	0.00	102.28
70	0.67	0.19	0.03	0.07	2.35	1.19	15.54	0.01	82.16	0.11	0.01	102.32
75	0.65	0.25	0.05	0.05	2.41	1.11	15.10	0.02	83.31	0.13	0.01	103.09
80	0.69	0.20	0.03	0.01	2.51	1.20	15.10	0.02	83.36	0.13	0.00	103.25
85	0.65	0.22	0.05	0.07	2.38	1.27	15.04	0.02	83.22	0.12	0.01	103.03
90	0.66	0.22	0.04	0.03	2.51	1.14	14.66	0.00	83.60	0.13	0.00	102.98
95	0.73	0.23	0.04	0.01	2.50	1.18	14.38	0.02	83.73	0.12	0.00	102.95
100	0.68	0.17	0.04	0.03	2.55	1.24	14.10	0.01	84.25	0.12	0.00	103.18
105	0.57	0.19	0.05	0.08	2.48	1.21	14.80	0.01	83.82	0.12	0.00	103.31
110	0.66	0.22	0.04	0.00	2.56	1.22	14.07	0.02	84.31	0.12	0.01	103.24

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
115	0.64	0.24	0.03	0.05	2.55	1.20	14.80	0.03	83.69	0.12	0.01	103.36
120	0.63	0.22	0.04	0.00	2.59	1.25	14.45	0.02	83.14	0.13	0.02	102.48
125	0.62	0.22	0.03	0.07	2.52	1.22	14.40	0.02	84.59	0.12	0.01	103.81
130	0.61	0.22	0.04	0.03	2.41	1.21	14.33	0.01	84.79	0.12	0.00	103.78
135	0.62	0.24	0.03	0.03	2.39	1.25	14.70	0.01	84.33	0.12	0.01	103.73
140	0.59	0.23	0.04	0.01	2.51	1.18	12.80	0.02	84.63	0.11	0.01	102.13
145	0.60	0.24	0.04	0.06	2.43	1.14	13.57	0.01	83.95	0.12	0.01	102.17
150	0.59	0.25	0.04	0.06	2.36	1.12	13.13	0.02	84.30	0.13	0.01	101.99
155	0.62	0.25	0.04	0.01	2.41	1.14	13.46	0.01	84.10	0.12	0.01	102.15
160	0.62	0.21	0.04	0.03	2.37	1.10	12.95	0.02	83.80	0.12	0.01	101.26
165	0.62	0.22	0.04	0.05	2.27	1.07	12.16	0.02	84.98	0.11	0.01	101.54
170	0.65	0.22	0.04	0.00	2.53	1.22	12.99	0.02	83.57	0.12	0.01	101.36
175	0.70	0.21	0.04	0.01	2.27	1.19	12.49	0.01	83.99	0.11	0.01	101.02
180	0.78	0.27	0.04	0.02	2.12	1.16	15.24	0.01	81.67	0.10	0.01	101.42
185	1.16	0.21	0.04	0.04	1.91	0.98	16.12	0.01	80.50	0.12	0.01	101.08
190	1.05	0.22	0.04	0.03	1.68	1.67	18.56	0.03	72.04	0.13	0.01	95.44
195	1.03	0.22	0.04	0.08	1.37	1.70	23.27	0.06	72.24	0.12	0.02	100.15
200	1.30	0.21	0.05	0.07	1.83	1.43	25.58	0.05	69.07	0.12	0.02	99.70
205	1.35	0.25	0.05	0.02	1.45	1.13	20.08	0.04	74.83	0.12	0.01	99.31
210	1.27	0.19	0.04	0.00	1.50	1.09	23.06	0.04	66.92	0.13	0.01	94.26
215	1.13	0.24	0.04	0.06	1.61	1.03	18.64	0.06	74.65	0.13	0.06	97.63

**PFE1501: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.70	0.21	0.04	0.04	2.37	1.20	16.57	0.01	82.44	0.13	0.02	103.74
5	0.67	0.23	0.04	0.00	2.54	1.16	16.44	0.01	82.57	0.13	0.00	103.78
10	0.71	0.23	0.04	0.02	2.45	1.17	16.30	0.01	83.07	0.13	0.01	104.13
15	0.71	0.24	0.04	0.05	2.38	1.27	16.23	0.02	82.51	0.12	0.00	103.55
20	0.68	0.25	0.04	0.00	2.44	1.26	16.01	0.01	82.88	0.12	0.00	103.68
25	0.65	0.20	0.04	0.03	2.40	1.24	15.99	0.02	82.73	0.12	0.01	103.43
30	0.65	0.21	0.05	0.08	2.35	1.18	15.23	0.02	83.01	0.12	0.01	102.90
35	0.66	0.24	0.04	0.07	2.38	1.13	15.66	0.01	82.76	0.12	0.01	103.08
40	0.72	0.25	0.04	0.04	2.44	1.19	15.47	0.03	82.79	0.12	0.01	103.09
45	0.68	0.25	0.03	0.03	2.34	1.35	15.88	0.01	83.06	0.13	0.00	103.75
50	0.73	0.26	0.04	0.03	2.24	1.20	15.89	0.00	82.92	0.13	0.00	103.44
55	0.77	0.27	0.04	0.00	2.18	1.17	16.17	0.02	82.65	0.13	0.01	103.40
60	0.79	0.21	0.04	0.00	2.37	1.17	16.03	0.02	82.82	0.13	0.01	103.57
65	0.76	0.28	0.05	0.07	2.18	1.18	16.32	0.03	83.17	0.13	0.01	104.17
70	0.75	0.22	0.04	0.01	2.14	1.20	16.41	0.03	82.77	0.13	0.01	103.71
75	0.80	0.21	0.04	0.03	2.13	1.15	16.56	0.01	82.70	0.13	0.01	103.77
80	0.85	0.21	0.04	0.03	2.02	1.19	16.56	0.02	83.10	0.14	0.01	104.17
85	0.87	0.24	0.04	0.04	2.10	1.18	16.82	0.02	82.39	0.13	0.00	103.83
90	0.77	0.27	0.04	0.04	2.06	1.18	16.94	0.01	82.38	0.13	0.00	103.84
95	0.81	0.25	0.04	0.06	1.97	1.10	17.30	0.02	81.80	0.13	0.02	103.49
100	0.78	0.25	0.04	0.05	2.04	1.18	17.32	0.03	81.75	0.13	0.00	103.57

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
105	0.75	0.28	0.03	0.04	2.06	1.19	17.17	0.03	82.00	0.12	0.01	103.67
110	0.82	0.22	0.04	0.00	2.02	1.15	17.58	0.02	82.04	0.13	0.01	104.03
115	0.80	0.26	0.04	0.10	1.94	1.20	17.86	0.02	81.51	0.13	0.01	103.86
120	0.90	0.24	0.04	0.06	2.00	1.19	16.72	0.02	81.88	0.13	0.01	103.17
125	0.82	0.25	0.04	0.08	2.03	1.21	17.56	0.01	81.37	0.13	0.01	103.49
130	0.82	0.28	0.04	0.01	1.92	1.05	15.77	0.03	82.42	0.12	0.02	102.47
135	0.93	0.27	0.04	0.03	1.94	1.11	13.91	0.04	83.71	0.11	0.05	102.14
140	0.95	0.25	0.03	0.00	2.12	1.12	13.80	0.04	83.27	0.14	0.02	101.74
145	0.92	0.23	0.04	0.02	1.99	1.14	13.22	0.04	83.65	0.14	0.01	101.39
150	0.95	0.20	0.04	0.01	1.93	1.11	14.18	0.03	83.02	0.12	0.01	101.60
155	0.96	0.21	0.04	0.00	1.97	1.10	15.79	0.03	81.10	0.12	0.01	101.33
160	1.10	0.21	0.04	0.01	1.70	1.59	12.25	0.03	84.45	0.12	0.02	101.53
165	1.01	0.21	0.04	0.05	2.00	1.10	16.08	0.05	80.50	0.13	0.01	101.16
170	0.95	0.26	0.04	0.02	1.65	1.10	16.74	0.04	80.10	0.14	0.00	101.03
175	1.11	0.23	0.03	0.04	1.63	1.13	17.61	0.03	79.64	0.13	0.02	101.58
180	1.09	0.22	0.05	0.02	1.64	0.99	25.45	0.04	70.65	0.14	0.01	100.30
185	0.96	0.21	0.04	0.00	1.99	1.62	28.98	0.05	65.53	0.11	0.01	99.51
190	1.00	0.19	0.05	0.00	1.61	1.20	27.90	0.15	65.45	0.12	0.03	97.68
195	1.23	0.21	0.06	0.07	1.96	1.31	26.88	0.55	66.22	0.13	0.05	98.66

**PFE1501: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.80	0.21	0.04	0.02	1.90	1.17	18.34	0.03	80.54	0.13	0.01	103.18
5	0.86	0.23	0.05	0.02	1.88	1.12	18.28	0.01	80.47	0.13	0.01	103.07
10	0.83	0.22	0.04	0.05	1.98	1.15	18.48	0.01	80.59	0.14	0.01	103.51
15	0.85	0.23	0.04	0.09	1.94	1.12	18.58	0.02	80.43	0.12	0.01	103.45
20	0.86	0.23	0.04	0.04	2.00	1.13	18.80	0.01	80.15	0.12	0.02	103.38
25	0.85	0.24	0.04	0.01	1.86	1.23	18.39	0.03	80.16	0.14	0.01	102.94
30	0.87	0.25	0.05	0.04	1.86	1.20	18.53	0.02	80.11	0.12	0.01	103.05
35	0.81	0.23	0.04	0.05	1.98	1.16	18.48	0.03	80.38	0.13	0.01	103.30
40	0.89	0.26	0.05	0.00	1.91	1.19	18.56	0.03	80.44	0.14	0.01	103.46
45	0.86	0.21	0.04	0.09	2.00	1.17	18.59	0.02	80.32	0.13	0.00	103.42
50	0.82	0.24	0.04	0.02	1.89	1.14	18.73	0.02	80.06	0.14	0.01	103.09
55	0.83	0.23	0.04	0.02	1.92	1.18	18.81	0.03	79.97	0.13	0.00	103.15
60	0.92	0.25	0.04	0.08	1.91	1.14	18.93	0.02	79.68	0.14	0.01	103.10
65	0.94	0.22	0.05	0.06	1.89	1.15	19.01	0.02	79.69	0.12	0.02	103.17
70	0.92	0.25	0.04	0.00	1.89	1.20	19.13	0.03	79.82	0.13	0.00	103.39
75	0.90	0.24	0.04	0.07	1.82	1.18	19.03	0.03	79.07	0.13	0.01	102.52
80	0.96	0.22	0.04	0.03	1.96	1.18	19.09	0.02	79.42	0.12	0.01	103.06
85	0.94	0.21	0.04	0.00	1.75	1.08	17.36	0.03	80.58	0.13	0.01	102.14

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
90	0.96	0.24	0.04	0.04	1.89	1.05	14.51	0.02	82.49	0.13	0.01	101.38
95	0.98	0.25	0.04	0.05	1.88	1.08	12.66	0.04	83.75	0.13	0.01	100.86
100	0.93	0.21	0.04	0.01	1.84	1.11	12.70	0.03	83.94	0.12	0.00	100.92
105	0.97	0.23	0.04	0.04	1.80	1.16	15.02	0.03	81.82	0.13	0.01	101.24
110	1.00	0.26	0.04	0.05	1.84	1.05	15.05	0.03	81.89	0.12	0.01	101.31
115	0.85	0.27	0.04	0.04	1.90	1.07	15.03	0.02	81.53	0.14	0.01	100.88
120	1.04	0.20	0.03	0.03	1.91	1.17	13.48	0.03	80.50	0.12	0.00	98.52
125	0.96	0.22	0.04	0.02	1.84	1.08	18.80	0.03	77.82	0.11	0.00	100.94
130	0.97	0.18	0.04	0.04	1.91	1.07	15.23	0.03	81.18	0.12	0.00	100.77
135	1.00	0.23	0.03	0.07	1.96	1.17	14.11	0.03	82.08	0.14	0.00	100.81
140	1.04	0.22	0.04	0.02	2.01	1.21	15.55	0.02	80.76	0.13	0.01	101.01
145	0.60	0.25	0.04	0.03	1.55	1.21	18.92	0.03	77.65	0.12	0.01	100.40
150	0.41	0.27	0.05	0.10	1.47	1.15	55.05	0.01	39.72	0.08	0.00	98.29
155	1.37	0.17	0.04	0.02	1.98	0.94	16.73	0.05	77.95	0.11	0.02	99.38
160	1.04	0.21	0.04	0.00	1.76	0.79	15.81	0.04	79.05	0.11	0.02	98.89
165	0.93	0.21	0.04	0.05	0.29	5.13	33.02	0.12	51.68	0.03	0.03	91.52



**PFE1502: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.69	0.27	0.04	0.03	1.31	1.21	15.73	0.03	81.11	0.13	0.01	100.55
5	1.21	0.24	0.04	0.10	1.27	0.87	29.06	0.03	68.35	0.10	0.02	101.29
10	1.45	0.27	0.04	0.06	1.41	0.89	29.95	0.03	67.84	0.13	0.01	102.07
15	0.98	0.27	0.04	0.07	1.54	1.19	19.59	0.02	77.62	0.14	0.01	101.46
20	1.02	0.22	0.04	0.00	1.56	1.81	18.75	0.03	77.51	0.17	0.02	101.13
25	1.06	0.28	0.04	0.03	1.71	1.17	21.25	0.04	76.12	0.16	0.01	101.87
30	1.05	0.25	0.04	0.00	1.63	1.16	21.34	0.03	76.95	0.15	0.01	102.59
35	1.09	0.23	0.04	0.00	1.58	1.23	21.04	0.04	77.50	0.14	0.01	102.90
40	1.03	0.22	0.04	0.01	1.68	1.16	19.38	0.03	78.27	0.14	0.00	101.95
45	0.98	0.24	0.04	0.08	1.68	1.19	20.45	0.03	78.05	0.14	0.02	102.90
50	0.98	0.26	0.03	0.02	1.79	1.24	19.99	0.02	78.67	0.15	0.01	103.16
55	0.94	0.20	0.05	0.03	1.81	1.13	19.09	0.02	79.52	0.13	0.00	102.92
60	0.86	0.24	0.04	0.08	1.86	1.07	19.22	0.04	78.62	0.14	0.01	102.18
65	0.94	0.24	0.04	0.00	1.98	1.21	19.20	0.02	79.32	0.13	0.01	103.09
70	0.84	0.27	0.05	0.04	1.95	1.22	19.16	0.03	79.52	0.14	0.01	103.21
75	0.85	0.26	0.04	0.08	1.91	1.12	18.94	0.01	79.55	0.16	0.01	102.93
80	0.83	0.23	0.04	0.04	1.93	1.16	19.12	0.02	79.48	0.14	0.02	102.98
85	0.87	0.25	0.04	0.03	1.81	1.12	17.46	0.02	80.44	0.15	0.01	102.19
90	0.86	0.25	0.04	0.04	1.89	1.18	18.33	0.02	79.73	0.14	0.01	102.47
95	0.83	0.24	0.04	0.09	1.96	1.19	18.45	0.02	80.68	0.13	0.01	103.64
100	0.80	0.21	0.04	0.01	1.96	1.20	18.52	0.03	80.22	0.15	0.01	103.14
105	0.85	0.24	0.04	0.03	1.99	1.16	18.52	0.02	80.50	0.13	0.01	103.50
110	0.80	0.24	0.04	0.08	1.93	1.26	18.16	0.03	80.74	0.15	0.01	103.44
115	0.78	0.25	0.04	0.05	1.96	1.23	18.07	0.02	80.69	0.14	0.02	103.23
120	0.80	0.24	0.04	0.01	2.00	1.20	18.00	0.01	81.09	0.15	0.00	103.54
125	0.79	0.28	0.04	0.00	2.06	1.20	17.96	0.03	80.95	0.14	0.01	103.44
130	0.79	0.22	0.04	0.08	2.01	1.22	17.77	0.03	81.28	0.14	0.00	103.57
135	0.76	0.23	0.04	0.02	2.17	1.21	17.77	0.01	81.18	0.15	0.02	103.56

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
140	0.79	0.24	0.04	0.02	2.11	1.21	16.57	0.02	81.37	0.14	0.00	102.51
145	0.77	0.23	0.04	0.00	2.00	1.22	14.88	0.02	82.71	0.14	0.01	102.02
150	0.72	0.20	0.04	0.06	2.13	1.24	12.60	0.01	84.02	0.15	0.00	101.16
155	0.74	0.24	0.04	0.00	2.12	1.17	14.06	0.03	82.78	0.15	0.01	101.33
160	0.83	0.26	0.04	0.02	2.10	1.12	13.24	0.02	83.11	0.13	0.01	100.88
165	0.76	0.24	0.04	0.01	1.96	1.09	11.45	0.01	84.56	0.14	0.02	100.29
170	0.83	0.22	0.04	0.05	2.14	1.16	12.14	0.03	84.05	0.14	0.00	100.78
175	0.83	0.23	0.05	0.02	2.14	1.23	13.63	0.02	82.57	0.13	0.00	100.85
180	0.74	0.23	0.04	0.08	2.15	1.15	12.10	0.01	83.56	0.13	0.01	100.19
185	0.77	0.23	0.04	0.05	2.14	1.11	10.90	0.01	85.27	0.14	0.01	100.66
190	0.77	0.26	0.04	0.00	2.24	1.18	11.97	0.02	83.86	0.14	0.00	100.48
195	0.72	0.22	0.04	0.04	2.13	1.18	11.27	0.03	84.89	0.13	0.00	100.66
200	0.73	0.27	0.04	0.03	1.96	1.11	10.06	0.03	85.95	0.15	0.00	100.34
205	0.78	0.22	0.04	0.02	2.16	1.00	9.96	0.01	85.28	0.13	0.01	99.60
210	0.68	0.26	0.04	0.02	2.06	0.99	10.95	0.01	85.13	0.15	0.01	100.30
215	0.75	0.22	0.04	0.04	1.98	1.12	12.52	0.01	82.64	0.13	0.01	99.46
220	0.73	0.20	0.03	0.05	2.10	1.01	13.04	0.01	82.34	0.14	0.01	99.66
225	0.65	0.24	0.04	0.00	2.05	1.15	15.13	0.01	80.23	0.12	0.00	99.63
230	0.49	0.24	0.05	0.08	2.23	1.36	26.93	0.00	67.49	0.11	0.01	98.98
235	0.80	0.24	0.05	0.00	1.95	0.98	39.97	0.01	45.94	0.13	0.00	90.07
240	0.81	0.21	0.05	0.00	2.19	1.75	15.79	0.02	76.77	0.15	0.04	97.78
245	0.66	0.22	0.04	0.02	2.10	1.16	15.76	0.03	78.29	0.13	0.02	98.41
250	0.63	0.24	0.04	0.02	2.15	0.96	13.23	0.02	81.74	0.13	0.01	99.18
255	0.62	0.22	0.04	0.02	2.09	1.33	17.62	0.02	76.20	0.12	0.02	98.31
260	0.44	0.22	0.05	0.08	1.48	1.08	24.81	0.05	69.85	0.10	0.03	98.17

**PFE1502: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.75	0.27	0.03	0.03	2.19	1.08	13.53	0.02	84.04	0.13	0.01	102.07
5	0.76	0.23	0.04	0.00	2.08	1.08	14.22	0.01	83.70	0.14	0.00	102.26
10	0.68	0.21	0.04	0.01	2.18	1.24	14.72	0.02	82.70	0.13	0.01	101.94
15	0.70	0.24	0.04	0.00	2.16	1.23	13.70	0.03	83.70	0.14	0.01	101.93
20	0.75	0.28	0.04	0.00	2.05	1.17	14.51	0.01	83.20	0.14	0.01	102.14
25	0.73	0.25	0.04	0.00	2.01	1.17	13.65	0.00	83.62	0.14	0.00	101.62
30	0.82	0.25	0.04	0.05	2.02	1.23	13.03	0.02	83.51	0.14	0.01	101.12
35	0.73	0.23	0.04	0.00	1.91	1.09	12.91	0.03	83.87	0.15	0.00	100.96
40	0.80	0.28	0.04	0.04	2.03	1.14	13.52	0.01	82.31	0.15	0.01	100.30
45	0.76	0.23	0.04	0.00	2.01	1.18	14.03	0.02	81.80	0.13	0.00	100.20
50	0.83	0.24	0.03	0.07	2.29	1.13	13.27	0.02	81.89	0.13	0.01	99.88
55	0.80	0.25	0.03	0.00	1.82	1.27	12.40	0.02	82.27	0.14	0.01	99.01
60	0.82	0.26	0.04	0.07	1.89	1.05	13.83	0.03	81.48	0.13	0.00	99.60
65	1.02	0.25	0.04	0.00	1.83	1.77	17.05	0.06	77.26	0.13	0.04	99.46
70	0.83	0.26	0.04	0.01	1.94	1.18	13.93	0.03	82.43	0.13	0.00	100.78
75	0.80	0.24	0.04	0.01	1.90	1.13	15.16	0.02	81.45	0.14	0.00	100.87
80	0.94	0.19	0.04	0.04	1.68	1.15	16.59	0.01	79.80	0.16	0.01	100.59

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
85	0.94	0.22	0.04	0.03	1.76	1.11	16.57	0.03	80.15	0.14	0.01	101.01
90	0.98	0.19	0.04	0.00	1.72	1.24	17.03	0.03	79.22	0.15	0.01	100.61
95	0.98	0.25	0.04	0.01	1.64	1.13	17.61	0.03	78.63	0.15	0.00	100.46
100	1.02	0.28	0.04	0.02	1.56	1.10	17.27	0.03	78.99	0.17	0.01	100.49
105	1.02	0.27	0.05	0.04	1.62	1.13	18.84	0.03	77.81	0.15	0.00	100.97
110	1.02	0.24	0.04	0.04	1.51	1.23	19.16	0.02	77.40	0.15	0.00	100.81
115	1.08	0.25	0.05	0.04	1.53	1.18	19.66	0.03	76.18	0.16	0.01	100.15
120	1.27	0.25	0.04	0.06	1.51	1.31	19.06	0.03	76.63	0.16	0.02	100.32
125	1.16	0.24	0.04	0.03	1.53	1.16	19.91	0.04	76.01	0.15	0.01	100.28
130	1.34	0.25	0.04	0.01	1.37	1.22	16.13	0.03	79.04	0.15	0.00	99.59
135	1.34	0.22	0.05	0.06	1.36	1.48	18.68	0.05	76.31	0.17	0.03	99.74
140	1.20	0.25	0.04	0.00	1.26	1.20	21.74	0.03	73.85	0.14	0.01	99.71
145	0.73	0.24	0.32	0.36	0.80	2.42	16.79	21.83	53.42	0.10	0.66	97.67
150	2.26	0.27	0.05	0.00	0.71	0.50	22.16	0.01	69.33	0.11	0.07	95.48
155	4.09	0.23	0.04	0.06	1.21	0.59	35.60	0.00	54.15	0.14	0.05	96.15
160	4.23	0.22	0.05	0.02	1.21	0.97	40.71	0.01	47.60	0.14	0.05	95.21

### PFE1502: Particle 2

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.59	0.22	0.04	0.03	1.38	0.97	15.44	0.04	82.91	0.14	0.01	101.77
5	1.03	0.29	0.04	0.03	2.02	1.16	18.82	0.04	78.88	0.15	0.00	102.45
10	1.06	0.22	0.04	0.03	1.86	1.26	18.18	0.02	79.54	0.14	0.00	102.35
15	0.95	0.23	0.04	0.03	1.74	1.22	20.22	0.03	78.48	0.13	0.00	103.07
20	1.01	0.25	0.04	0.06	1.66	1.07	20.05	0.03	78.51	0.14	0.01	102.84
25	0.97	0.21	0.04	0.07	1.71	1.12	19.45	0.02	79.39	0.14	0.01	103.14
30	1.04	0.27	0.04	0.03	1.73	1.12	19.46	0.02	78.85	0.14	0.01	102.71
35	1.02	0.26	0.04	0.05	1.65	1.13	19.71	0.03	78.80	0.14	0.01	102.83
40	1.00	0.22	0.04	0.04	1.55	1.22	18.89	0.04	80.00	0.14	0.01	103.14
45	1.08	0.22	0.04	0.03	1.68	1.16	20.87	0.03	79.17	0.15	0.01	104.45
50	0.98	0.22	0.04	0.07	1.57	1.20	21.24	0.02	78.44	0.14	0.01	103.93
55	1.07	0.23	0.03	0.05	1.59	1.17	21.13	0.01	78.57	0.13	0.00	103.98
60	1.04	0.32	0.05	0.03	1.63	1.19	21.15	0.03	78.77	0.13	0.00	104.33
65	1.04	0.24	0.05	0.09	1.43	1.14	21.12	0.03	78.33	0.12	0.01	103.59
70	1.04	0.23	0.04	0.05	1.64	1.19	21.03	0.03	78.56	0.15	0.00	103.95
75	1.01	0.18	0.04	0.01	1.62	1.16	21.27	0.03	78.38	0.13	0.01	103.84
80	0.97	0.20	0.04	0.07	1.55	1.17	20.46	0.04	79.11	0.14	0.01	103.74
85	1.01	0.26	0.04	0.08	1.65	1.19	20.95	0.03	78.62	0.14	0.01	103.98
90	1.01	0.27	0.05	0.07	1.64	1.26	20.83	0.03	78.46	0.14	0.01	103.76
95	1.03	0.21	0.04	0.00	1.68	1.21	20.97	0.03	78.67	0.14	0.01	103.98
100	1.00	0.25	0.04	0.05	1.67	1.27	20.97	0.03	78.67	0.14	0.01	104.10
105	1.01	0.25	0.04	0.13	1.69	1.12	21.10	0.03	78.42	0.12	0.00	103.91
110	1.06	0.27	0.04	0.05	1.55	1.19	21.04	0.03	78.81	0.14	0.01	104.17
115	0.96	0.22	0.04	0.02	1.58	1.12	20.57	0.04	79.09	0.14	0.01	103.78
120	1.01	0.25	0.04	0.05	1.61	1.16	21.21	0.03	78.38	0.13	0.01	103.88
125	1.02	0.23	0.05	0.06	1.62	1.18	21.00	0.01	78.48	0.14	0.01	103.80
130	1.07	0.18	0.05	0.07	1.53	1.13	20.77	0.03	78.67	0.13	0.01	103.64
135	0.99	0.30	0.04	0.04	1.37	1.09	21.19	0.04	78.09	0.14	0.01	103.28
140	1.01	0.27	0.04	0.02	1.47	1.05	20.48	0.04	78.49	0.14	0.00	103.00
145	1.03	0.22	0.05	0.00	1.52	1.15	20.86	0.04	78.12	0.14	0.01	103.14
150	1.16	0.24	0.04	0.04	1.36	1.13	20.33	0.04	78.67	0.12	0.01	103.14
155	1.04	0.23	0.04	0.00	1.50	1.11	20.23	0.04	78.53	0.13	0.01	102.85

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
160	1.06	0.20	0.03	0.08	1.53	1.10	18.92	0.03	79.29	0.14	0.01	102.38
165	1.14	0.20	0.04	0.01	1.65	1.15	19.23	0.02	79.16	0.13	0.01	102.73
170	1.04	0.20	0.04	0.05	1.63	1.14	19.37	0.02	79.22	0.13	0.01	102.85
175	1.01	0.23	0.05	0.07	1.69	1.10	18.79	0.01	79.32	0.13	0.01	102.40
180	1.05	0.26	0.05	0.04	1.82	1.26	18.29	0.04	79.62	0.14	0.01	102.56
185	0.95	0.23	0.04	0.09	1.73	1.16	17.69	0.02	80.10	0.14	0.01	102.16
190	0.99	0.22	0.04	0.03	1.84	1.09	17.62	0.03	80.12	0.13	0.00	102.11
195	0.98	0.25	0.04	0.02	1.90	1.16	17.03	0.03	80.53	0.14	0.01	102.08
200	1.04	0.23	0.05	0.09	1.97	1.15	18.00	0.02	79.70	0.14	0.01	102.40
205	0.99	0.24	0.04	0.03	1.91	1.14	18.04	0.03	80.04	0.12	0.00	102.58
210	0.96	0.28	0.04	0.08	1.90	1.22	16.66	0.02	81.33	0.14	0.01	102.63
215	1.08	0.22	0.04	0.01	1.92	1.10	15.45	0.03	82.12	0.14	0.02	102.11
220	1.04	0.26	0.04	0.01	2.02	1.10	16.58	0.02	81.52	0.15	0.01	102.75
225	1.01	0.25	0.04	0.07	1.93	1.13	17.02	0.02	80.35	0.12	0.00	101.94
230	1.16	0.28	0.06	0.05	2.01	4.89	15.59	0.19	78.24	0.17	0.03	102.66
235	1.27	0.21	0.04	0.00	2.41	1.17	15.65	0.03	81.17	0.14	0.01	102.10
240	0.95	0.26	0.04	0.04	1.83	1.09	16.44	0.03	80.82	0.13	0.00	101.63
245	1.05	0.21	0.04	0.06	1.95	1.09	15.95	0.03	80.91	0.13	0.02	101.43
250	1.13	0.22	0.04	0.11	2.15	1.11	15.74	0.04	80.67	0.14	0.00	101.34
255	1.14	0.21	0.04	0.00	2.20	1.25	17.00	0.03	79.76	0.13	0.00	101.76
260	0.54	0.23	0.04	0.00	0.98	0.96	9.27	0.03	89.18	0.12	0.01	101.36
265	1.03	0.20	0.04	0.01	1.84	1.43	18.35	0.06	77.07	0.16	0.01	100.18
270	0.86	0.21	0.04	0.05	1.89	1.16	27.24	0.02	68.61	0.14	0.01	100.23
275	0.52	0.21	0.04	0.00	1.64	1.81	41.91	0.03	52.82	0.13	0.00	99.13
280	1.15	0.21	0.05	0.04	1.66	1.78	42.10	0.06	50.65	0.12	0.02	97.83
285	1.15	0.23	0.04	0.04	1.78	0.90	19.53	0.30	75.20	0.12	0.00	99.28
290	0.95	0.26	0.04	0.00	1.71	0.92	19.08	0.03	76.94	0.14	0.00	100.06
295	0.98	0.25	0.04	0.04	1.87	2.76	20.21	0.06	73.23	0.13	0.04	99.61
300	0.88	0.22	0.04	0.01	1.62	2.26	23.36	0.09	70.56	0.14	0.02	99.19
305	0.60	0.24	0.04	0.04	0.19	2.59	25.22	0.03	70.60	0.03	0.01	99.59

**PFE1502: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.41	0.24	0.04	0.06	1.66	1.39	17.52	0.11	80.46	0.12	0.09	103.09
5	1.27	0.26	0.04	0.03	1.64	1.27	17.96	0.09	80.47	0.12	0.07	103.22
10	1.19	0.24	0.04	0.13	1.65	1.26	17.50	0.08	80.63	0.13	0.06	102.90
15	1.21	0.23	0.04	0.02	1.78	1.40	16.94	0.11	80.90	0.12	0.07	102.82
20	1.25	0.28	0.05	0.03	1.80	1.23	15.67	0.06	82.06	0.13	0.04	102.59
25	1.08	0.25	0.04	0.02	1.64	1.19	14.61	0.06	83.35	0.13	0.03	102.39
30	1.15	0.21	0.04	0.06	1.84	1.13	15.51	0.04	82.38	0.14	0.01	102.51
35	1.12	0.26	0.04	0.00	1.71	1.22	15.98	0.05	81.77	0.12	0.01	102.28
40	0.87	0.22	0.04	0.02	1.35	1.12	11.79	0.05	85.92	0.14	0.02	101.53
45	1.09	0.23	0.04	0.06	1.91	1.07	16.17	0.05	82.01	0.14	0.01	102.77
50	1.12	0.26	0.04	0.04	1.86	1.10	16.97	0.04	81.16	0.11	0.01	102.72
55	1.05	0.25	0.04	0.04	1.86	1.13	15.70	0.03	82.36	0.13	0.01	102.60
60	0.94	0.25	0.04	0.04	1.86	1.13	15.96	0.03	81.91	0.13	0.01	102.29
65	1.06	0.24	0.04	0.03	1.90	1.19	16.02	0.02	81.41	0.11	0.01	102.04
70	1.00	0.22	0.04	0.04	2.02	1.21	15.91	0.03	81.81	0.12	0.00	102.41
75	1.35	0.22	0.04	0.03	2.35	1.26	12.22	0.04	84.11	0.14	0.02	101.75
80	0.51	0.23	0.04	0.01	1.00	0.90	5.67	0.03	92.35	0.12	0.02	100.86
85	0.97	0.24	0.04	0.02	2.31	1.31	13.62	0.02	83.80	0.13	0.02	102.46
90	0.92	0.27	0.04	0.07	2.37	1.10	13.80	0.03	83.72	0.12	0.02	102.46
95	0.41	0.26	0.04	0.00	0.55	0.82	2.40	0.02	96.71	0.13	0.01	101.35

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
100	0.87	0.25	0.04	0.08	2.28	1.13	14.65	0.02	82.46	0.12	0.00	101.90
105	0.89	0.20	0.04	0.00	2.18	1.16	15.91	0.01	81.07	0.12	0.01	101.59
110	0.97	0.20	0.05	0.05	2.31	1.78	18.64	0.05	77.23	0.10	0.03	101.40
115	1.11	0.21	0.03	0.00	2.11	0.89	13.39	0.02	83.63	0.11	0.02	101.51
120	0.44	0.27	0.04	0.00	1.14	1.45	6.19	0.08	90.90	0.12	0.06	100.69
125	1.04	0.21	0.04	0.00	1.85	1.14	23.68	0.05	71.56	0.11	0.02	99.69
130	1.02	0.25	0.04	0.04	1.53	1.03	21.42	0.03	74.83	0.11	0.02	100.31
135	1.02	0.18	0.04	0.00	1.25	1.04	16.49	0.06	79.25	0.11	0.03	99.46
140	1.24	0.21	0.04	0.03	1.83	1.16	21.08	0.03	74.39	0.11	0.01	100.14
145	0.98	0.22	0.04	0.03	1.85	1.07	14.23	0.05	81.53	0.13	0.02	100.13
150	1.12	0.25	0.04	0.08	2.28	2.14	15.62	0.06	78.57	0.12	0.02	100.30
155	0.73	0.21	0.04	0.00	1.25	1.30	11.82	0.03	84.63	0.11	0.02	100.13
160	0.74	0.18	0.04	0.00	1.42	1.91	13.98	0.05	81.06	0.11	0.02	99.51
165	0.98	0.22	0.04	0.04	2.25	1.27	23.58	0.04	71.72	0.13	0.01	100.28
170	1.03	0.22	0.04	0.01	1.93	1.30	20.26	0.02	74.96	0.13	0.03	99.92
175	0.70	0.20	0.04	0.08	1.90	1.48	12.82	0.04	83.41	0.13	0.02	100.80
180	0.96	0.26	0.04	0.06	2.39	1.58	22.82	0.02	71.47	0.13	0.02	99.76
185	0.74	0.26	0.04	0.03	2.04	3.88	21.64	0.06	70.70	0.12	0.05	99.58
190	0.72	0.25	0.04	0.07	2.02	1.53	19.52	0.02	75.15	0.11	0.02	99.43



**PFE1504: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.35	0.25	0.04	0.01	1.71	1.08	18.85	0.02	78.67	0.12	0.03	102.13
5	1.50	0.21	0.04	0.01	1.71	1.04	19.95	0.04	77.38	0.12	0.03	102.02
10	1.97	0.21	0.09	0.00	1.42	1.31	15.81	3.39	77.95	0.13	0.09	102.37
15	1.41	0.20	0.38	0.00	0.79	3.63	10.09	39.15	50.95	0.08	0.54	107.23
20	1.47	0.23	0.04	0.04	1.67	1.12	18.27	0.06	78.82	0.11	0.03	101.85
25	1.12	0.19	0.04	0.08	1.84	1.21	17.54	0.02	79.79	0.13	0.00	101.95
30	0.93	0.21	0.04	0.00	1.93	1.05	17.29	0.11	75.27	0.12	0.01	96.96
35	0.95	0.21	0.04	0.09	2.07	1.14	19.25	0.02	78.53	0.12	0.01	102.42
40	0.95	0.25	0.04	0.06	1.96	1.07	19.56	0.03	77.76	0.12	0.01	101.80
45	0.94	0.21	0.04	0.04	1.87	1.11	18.67	0.01	79.28	0.12	0.01	102.31
50	1.08	0.22	0.04	0.08	1.78	1.17	18.66	0.03	79.26	0.12	0.00	102.43
55	1.21	0.26	0.04	0.06	1.59	1.17	18.70	0.02	78.67	0.12	0.01	101.86
60	1.29	0.23	0.04	0.06	1.44	1.10	19.94	0.06	77.06	0.13	0.03	101.37
65	1.43	0.23	0.04	0.03	1.28	8.39	19.15	0.11	71.66	0.11	0.08	102.50
70	1.71	0.21	0.04	0.02	1.31	2.98	21.59	0.08	74.67	0.12	0.05	102.77
75	1.56	0.26	0.05	0.10	1.33	1.06	21.86	0.05	76.25	0.12	0.03	102.67
80	1.08	0.25	0.04	0.02	0.53	1.11	7.62	0.11	89.33	0.12	0.06	100.25
85	1.75	0.27	0.04	0.03	1.40	1.12	21.24	0.08	76.43	0.12	0.04	102.51
90	1.64	0.19	0.04	0.03	1.45	2.80	18.32	0.10	77.81	0.13	0.08	102.60
95	1.95	0.25	0.04	0.04	1.46	1.08	19.20	0.09	77.77	0.14	0.07	102.10
100	2.15	0.20	0.04	0.04	1.45	1.06	20.99	0.08	76.04	0.13	0.07	102.25
105	2.30	0.22	0.05	0.03	1.45	1.07	21.76	0.28	74.94	0.12	0.09	102.31
110	2.43	0.22	0.08	0.11	1.53	1.79	18.10	12.11	67.53	0.10	0.12	104.11
115	1.93	0.28	0.05	0.10	1.47	1.98	17.06	1.04	76.47	0.11	0.07	100.55
120	1.44	0.19	0.04	0.04	1.18	7.51	15.36	0.20	73.01	0.10	0.12	99.18
125	1.45	0.22	0.04	0.06	1.49	1.27	16.38	0.04	80.64	0.13	0.03	101.75
130	1.21	0.23	0.04	0.00	1.72	1.27	16.95	0.04	79.43	0.12	0.03	101.03
135	1.11	0.24	0.04	0.01	1.86	1.21	17.72	0.04	79.58	0.12	0.00	101.93
140	0.91	0.19	0.04	0.07	1.81	1.19	15.95	0.03	80.92	0.13	0.01	101.26
145	0.84	0.21	0.04	0.03	1.98	1.19	15.58	0.03	81.15	0.14	0.01	101.20
150	0.79	0.21	0.04	0.03	2.00	1.16	15.93	0.02	80.11	0.13	0.01	100.41
155	0.79	0.21	0.04	0.04	2.06	1.26	16.82	0.02	79.48	0.14	0.00	100.86
160	0.83	0.23	0.04	0.02	2.33	1.08	16.77	0.01	79.89	0.13	0.00	101.32
165	0.80	0.20	0.04	0.08	2.29	1.15	15.06	0.01	81.62	0.13	0.01	101.38
170	0.82	0.24	0.04	0.01	1.96	1.19	14.53	0.02	81.76	0.13	0.01	100.72
175	0.84	0.23	0.05	0.00	1.99	1.12	14.41	0.03	82.20	0.12	0.01	100.99
180	0.91	0.19	0.04	0.03	1.99	1.13	12.27	0.01	84.20	0.13	0.01	100.89

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
185	0.94	0.24	0.04	0.00	1.92	1.13	12.08	0.03	84.21	0.13	0.02	100.74
190	0.80	0.21	0.05	0.06	1.77	1.03	11.45	0.03	85.02	0.14	0.01	100.57
195	0.59	0.23	0.04	0.06	1.33	0.98	9.63	0.04	87.23	0.11	0.01	100.24
200	0.95	0.25	0.04	0.03	1.97	1.10	11.92	0.03	84.45	0.14	0.00	100.88
205	0.71	0.21	0.04	0.04	1.80	1.07	11.36	0.03	84.99	0.13	0.01	100.39
210	0.76	0.21	0.04	0.00	2.03	1.15	12.19	0.01	83.76	0.14	0.00	100.30
215	0.84	0.15	0.04	0.00	2.16	1.08	11.88	0.01	84.45	0.12	0.01	100.74
220	0.63	0.18	0.04	0.07	1.76	1.22	12.05	0.01	84.80	0.14	0.01	100.89
225	0.69	0.25	0.04	0.06	2.03	0.99	11.55	0.02	84.75	0.14	0.01	100.53
230	0.91	0.20	0.04	0.00	2.40	1.13	12.53	0.02	83.44	0.13	0.01	100.79
235	0.80	0.18	0.04	0.06	1.99	1.24	11.67	0.02	84.84	0.14	0.00	100.97
240	0.80	0.22	0.04	0.00	2.21	1.25	12.42	0.02	83.85	0.12	0.01	100.92
245	0.74	0.21	0.04	0.03	2.04	1.08	11.71	0.02	84.72	0.12	0.00	100.71
250	0.71	0.19	0.04	0.00	2.05	1.17	12.85	0.01	83.33	0.13	0.01	100.49
255	0.77	0.21	0.04	0.00	1.91	1.13	13.54	0.02	82.80	0.12	0.01	100.56
260	0.77	0.23	0.04	0.06	2.16	1.06	14.83	0.02	81.43	0.13	0.00	100.72
265	0.70	0.25	0.05	0.06	2.05	1.05	15.79	0.02	80.48	0.11	0.01	100.57
270	0.84	0.22	0.03	0.13	2.53	1.15	24.30	0.02	70.65	0.12	0.01	100.01
275	0.68	0.23	0.04	0.01	2.07	1.54	27.05	0.01	68.19	0.13	0.01	99.95
280	0.96	0.21	0.04	0.03	2.00	1.45	41.00	0.02	52.71	0.10	0.01	98.53
285	0.54	0.25	0.04	0.03	1.94	1.17	36.94	0.01	57.27	0.10	0.00	98.29
290	0.45	0.23	0.06	0.08	1.71	2.36	39.94	0.03	53.33	0.12	0.00	98.31
295	0.58	0.23	0.04	0.13	2.03	1.06	39.01	0.00	56.26	0.08	0.00	99.40
300	0.95	0.21	0.04	0.03	1.89	1.34	19.92	0.02	75.78	0.13	0.01	100.31
305	0.71	0.20	0.04	0.06	1.58	1.02	17.11	0.01	79.21	0.12	0.01	100.07
310	0.67	0.23	0.05	0.05	1.65	1.34	17.90	0.02	78.23	0.13	0.02	100.27
315	0.86	0.22	0.04	0.04	0.20	1.53	23.60	0.04	72.21	0.03	0.01	98.77

**PFE1504: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.84	0.23	0.05	0.05	2.12	1.10	15.51	0.02	80.07	0.12	0.00	100.10
5	0.76	0.23	0.04	0.00	1.95	1.86	16.96	0.02	77.77	0.16	0.02	99.77
10	0.94	0.26	0.04	0.02	2.53	1.20	17.00	0.01	78.28	0.13	0.02	100.43
15	0.52	0.23	0.04	0.04	1.24	1.55	6.29	0.01	89.91	0.13	0.01	99.95
20	0.61	0.23	0.04	0.02	1.89	1.00	8.84	0.01	87.24	0.14	0.01	100.01
25	0.61	0.24	0.04	0.00	1.97	2.53	13.44	0.04	81.52	0.16	0.02	100.58
30	0.79	0.22	0.04	0.04	2.59	1.11	13.95	0.03	81.75	0.11	0.00	100.63
35	0.78	0.26	0.04	0.02	2.45	1.15	14.03	0.02	81.34	0.12	0.00	100.20
40	0.51	0.28	0.05	0.00	1.26	1.06	8.51	0.03	88.05	0.14	0.01	99.90
45	0.72	0.23	0.04	0.05	2.27	1.29	17.00	0.01	77.98	0.14	0.00	99.72
50	0.70	0.23	0.04	0.04	1.92	1.18	14.86	0.02	80.35	0.14	0.02	99.48
55	0.43	0.22	0.04	0.03	1.40	0.82	8.52	0.02	87.53	0.11	0.01	99.12
60	0.82	0.22	0.04	0.01	2.46	1.13	14.80	0.01	80.18	0.12	0.01	99.79
65	0.41	0.21	0.04	0.01	1.57	1.14	9.83	0.01	86.38	0.13	0.01	99.73
70	0.64	0.21	0.04	0.05	2.45	1.12	11.04	0.02	84.70	0.12	0.00	100.37
75	0.77	0.23	0.05	0.02	2.28	1.23	11.90	0.02	83.88	0.13	0.00	100.49
80	0.79	0.24	0.04	0.05	1.86	0.84	9.05	0.02	87.07	0.12	0.01	100.09
85	0.52	0.20	0.05	0.03	1.30	0.66	4.30	0.01	93.17	0.13	0.01	100.38
90	0.71	0.21	0.04	0.01	1.85	1.35	11.88	0.02	83.56	0.14	0.01	99.78
95	0.81	0.24	0.04	0.03	1.81	10.00	18.95	0.13	64.60	0.11	0.09	96.79
100	0.72	0.27	0.04	0.04	2.02	1.20	18.10	0.02	76.76	0.13	0.01	99.30
105	0.99	0.27	0.04	0.07	3.11	1.40	26.51	0.02	67.48	0.12	0.01	100.01
110	0.72	0.26	0.04	0.07	1.32	10.79	26.31	0.11	58.98	0.14	0.08	98.82
115	0.38	0.20	0.04	0.00	1.23	11.21	20.14	0.12	64.72	0.11	0.11	98.24
120	1.02	0.25	0.04	0.05	1.91	3.84	29.45	0.07	62.04	0.14	0.04	98.86

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
125	0.62	0.22	0.04	0.02	2.26	1.15	21.95	0.02	74.03	0.12	0.01	100.44
130	0.70	0.21	0.04	0.04	2.30	0.95	20.12	0.02	75.90	0.13	0.02	100.42
135	0.72	0.20	0.04	0.06	1.63	0.73	9.52	0.02	87.79	0.12	0.01	100.84
140	1.07	0.25	0.05	0.00	2.99	1.39	24.04	0.03	69.44	0.13	0.02	99.40
145	0.63	0.21	0.04	0.00	1.40	1.02	9.30	0.01	87.21	0.11	0.00	99.93
150	0.76	0.24	0.04	0.00	2.20	1.17	14.93	0.03	80.20	0.14	0.01	99.73
155	0.93	0.23	0.03	0.06	2.34	1.03	15.49	0.02	79.97	0.12	0.01	100.22
160	0.65	0.23	0.04	0.00	2.07	1.08	17.55	0.02	76.97	0.12	0.01	98.73
165	0.71	0.25	0.04	0.03	2.38	1.66	26.56	0.02	67.18	0.13	0.00	98.96
170	0.72	0.24	0.05	0.02	1.41	1.40	26.13	0.01	68.62	0.11	0.01	98.71
175	1.08	0.27	0.05	0.03	2.10	1.44	31.55	0.02	62.63	0.14	0.01	99.32
180	0.55	0.24	0.04	0.00	1.82	1.02	12.64	0.02	83.50	0.10	0.00	99.92
185	0.85	0.21	0.04	0.06	1.62	1.37	11.28	0.04	84.65	0.12	0.02	100.25
190	0.88	0.23	0.05	0.00	1.78	0.93	12.45	0.02	83.73	0.11	0.00	100.18
195	0.45	0.19	0.04	0.00	1.27	2.74	7.64	0.03	88.86	0.10	0.01	101.32
200	0.30	0.21	0.03	0.00	0.61	0.70	4.02	0.00	94.54	0.09	0.00	100.50
205	0.60	0.24	0.05	0.07	1.46	0.88	11.03	0.03	85.77	0.11	0.01	100.25
210	0.58	0.22	0.05	0.00	1.56	0.97	12.62	0.03	84.52	0.11	0.01	100.66
215	0.48	0.21	0.04	0.00	1.11	0.81	12.18	0.02	84.03	0.10	0.01	98.97
220	0.99	0.21	0.05	0.05	2.24	3.21	41.46	0.02	49.41	0.14	0.02	97.79
225	0.75	0.22	0.04	0.03	2.31	1.06	17.97	0.02	76.97	0.14	0.01	99.53
230	0.74	0.22	0.04	0.00	2.01	1.11	17.04	0.04	77.91	0.11	0.01	99.24
235	0.52	0.20	0.05	0.04	0.32	1.47	15.47	0.01	80.49	0.05	0.03	98.63
240	0.52	0.26	0.04	0.00	0.00	7.30	18.91	0.11	70.37	0.05	0.08	97.64

**PFE1504: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.15	0.21	0.04	0.02	1.66	1.14	14.74	0.03	83.69	0.12	0.01	102.81
5	1.05	0.23	0.04	0.01	1.61	1.10	14.10	0.02	83.70	0.13	0.02	102.00
10	0.98	0.24	0.03	0.02	1.69	1.05	14.33	0.03	83.80	0.11	0.02	102.30
15	1.09	0.21	0.04	0.03	1.73	1.02	15.24	0.04	82.91	0.15	0.01	102.47
20	1.06	0.23	0.04	0.00	1.56	1.12	13.66	0.02	84.17	0.16	0.01	102.01
25	1.08	0.24	0.04	0.02	1.86	1.19	15.14	0.03	83.00	0.14	0.01	102.73
30	1.18	0.22	0.04	0.03	1.65	1.06	14.45	0.03	83.49	0.15	0.00	102.30
35	1.16	0.21	0.04	0.05	1.74	1.17	14.68	0.03	83.06	0.14	0.01	102.29
40	1.01	0.22	0.04	0.00	1.60	1.14	13.52	0.03	84.37	0.13	0.01	102.09
45	1.12	0.22	0.04	0.01	1.64	1.19	14.42	0.03	82.50	0.13	0.01	101.30
50	1.19	0.26	0.04	0.00	1.76	1.07	14.39	0.03	83.25	0.13	0.01	102.12
55	1.01	0.20	0.04	0.01	1.68	1.03	14.24	0.05	83.17	0.13	0.00	101.57
60	1.04	0.22	0.04	0.01	1.48	1.00	13.65	0.03	83.64	0.13	0.01	101.26
65	0.99	0.25	0.04	0.07	1.48	1.02	13.94	0.03	83.63	0.13	0.01	101.58
70	1.05	0.23	0.04	0.03	1.73	1.10	15.27	0.03	81.92	0.13	0.02	101.54

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
75	1.27	0.27	0.04	0.01	1.57	1.16	17.45	0.03	79.29	0.18	0.01	101.29
80	0.88	0.25	0.04	0.00	1.57	1.00	20.66	0.02	75.73	0.13	0.01	100.29
85	0.86	0.24	0.05	0.04	1.75	1.08	41.81	0.04	52.39	0.11	0.01	98.36
90	0.76	0.26	0.04	0.06	1.83	1.16	43.37	0.03	51.14	0.12	0.01	98.78
95	0.99	0.26	0.05	0.03	1.55	6.89	34.15	0.08	53.79	0.12	0.05	97.97
100	1.09	0.27	0.04	0.00	1.63	2.36	25.25	0.05	68.53	0.11	0.02	99.36
105	0.56	0.21	0.03	0.02	0.89	0.60	13.02	0.03	85.32	0.10	0.01	100.78
110	0.84	0.27	0.04	0.02	1.20	0.73	16.20	0.03	80.84	0.12	0.00	100.28
115	1.09	0.28	0.04	0.03	1.79	1.01	21.69	0.04	73.76	0.13	0.01	99.85
120	0.81	0.23	0.04	0.05	1.24	0.84	15.03	0.03	81.12	0.11	0.02	99.51
125	1.09	0.21	0.05	0.00	1.80	1.42	24.29	0.03	71.20	0.14	0.01	100.23
130	0.86	0.26	0.05	0.02	1.67	1.28	21.42	0.03	74.60	0.12	0.01	100.31
135	0.77	0.23	0.04	0.01	1.03	1.23	28.47	0.04	67.96	0.08	0.02	99.86
140	0.45	0.23	0.04	0.00	0.11	1.10	13.27	0.01	84.36	0.05	0.01	99.61

**PFE1505: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.66	0.24	0.04	0.03	2.82	1.07	18.24	0.02	76.57	0.10	0.01	99.80
5	0.61	0.23	0.04	0.05	2.37	4.69	15.90	0.09	74.98	0.13	0.08	99.17
10	0.66	0.22	0.04	0.02	2.34	1.06	15.50	0.00	79.72	0.11	0.01	99.69
15	0.36	0.22	0.04	0.01	1.40	0.87	9.37	0.01	87.35	0.11	0.01	99.73
20	0.77	0.21	0.04	0.00	2.18	1.10	17.75	0.03	77.18	0.13	0.01	99.39
25	0.84	0.18	0.04	0.03	2.26	1.03	17.66	0.03	77.13	0.12	0.02	99.34
30	0.83	0.20	0.04	0.02	2.04	1.10	16.43	0.03	79.21	0.14	0.01	100.04
35	0.78	0.25	0.04	0.05	1.63	1.11	15.07	0.05	80.09	0.14	0.01	99.21
40	0.97	0.24	0.04	0.03	1.99	1.51	17.19	0.01	77.02	0.14	0.02	99.15
45	1.00	0.20	0.04	0.05	1.87	3.73	15.71	0.05	78.03	0.15	0.02	100.84
50	1.21	0.25	0.04	0.03	1.77	1.15	17.77	0.04	76.41	0.13	0.02	98.81
55	0.68	0.21	0.75	0.56	0.71	3.89	6.52	45.03	39.47	0.05	0.47	98.32
60	1.30	0.22	0.04	0.01	1.87	1.22	16.73	0.04	77.72	0.13	0.01	99.28
65	0.97	0.21	0.04	0.05	1.37	0.95	12.36	0.02	83.08	0.15	0.01	99.21
70	0.86	0.20	0.03	0.00	1.92	1.05	15.14	0.03	80.08	0.14	0.01	99.45
75	0.84	0.21	0.04	0.05	1.84	1.15	15.05	0.04	80.26	0.14	0.01	99.63
80	0.76	0.21	0.03	0.02	2.36	1.03	15.38	0.02	79.88	0.14	0.01	99.84
85	0.78	0.21	0.04	0.02	2.62	0.98	15.96	0.02	78.62	0.12	0.01	99.38
90	0.65	0.22	0.04	0.01	2.33	1.10	15.91	0.02	78.76	0.10	0.01	99.15
95	0.67	0.22	0.04	0.00	2.75	1.07	16.84	0.03	77.97	0.11	0.00	99.68
100	0.50	0.22	0.04	0.03	1.38	1.01	12.28	0.02	83.04	0.11	0.01	98.64
105	0.61	0.21	0.04	0.01	1.82	6.67	17.45	0.13	73.37	0.14	0.10	100.55

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
110	0.56	0.22	0.04	0.01	2.19	2.10	14.97	0.03	79.38	0.13	0.02	99.62
115	0.73	0.19	0.04	0.00	2.58	5.79	18.32	0.14	69.60	0.15	0.13	97.66
120	0.52	0.21	0.03	0.02	1.47	1.13	9.80	0.02	85.54	0.12	0.01	98.85
125	0.94	0.21	0.04	0.05	2.95	2.10	18.24	0.02	74.50	0.17	0.02	99.25
130	0.56	0.19	0.03	0.00	1.39	0.79	8.69	0.02	87.60	0.14	0.01	99.42
135	0.33	0.22	0.04	0.00	0.70	0.68	6.27	0.03	90.90	0.13	0.00	99.29
140	1.30	0.23	0.04	0.02	2.72	1.36	29.50	0.02	64.32	0.20	0.00	99.71
145	0.78	0.18	0.04	0.01	1.59	1.36	16.43	0.04	77.83	0.16	0.01	98.43
150	1.06	0.23	0.04	0.02	2.14	1.22	23.41	0.03	71.42	0.17	0.01	99.73
155	1.30	0.24	0.04	0.00	2.14	1.45	25.48	0.03	68.41	0.18	0.01	99.29
160	0.92	0.23	0.04	0.01	1.84	3.64	20.94	0.05	72.05	0.17	0.04	99.94
165	0.66	0.22	0.04	0.00	1.78	3.03	15.17	0.12	77.83	0.12	0.06	99.02
170	0.84	0.17	0.04	0.00	1.65	6.87	24.76	0.13	62.39	0.15	0.07	97.06
175	0.97	0.21	0.04	0.01	1.91	1.95	27.86	0.03	67.17	0.14	0.02	100.30
180	0.98	0.21	0.04	0.04	1.54	1.69	28.41	0.04	65.88	0.13	0.02	98.97
185	1.04	0.22	0.04	0.07	1.70	3.09	34.10	0.05	58.53	0.13	0.02	99.00
190	0.53	0.20	0.04	0.01	0.87	0.96	14.05	0.03	81.89	0.11	0.01	98.69
195	0.87	0.25	0.04	0.00	1.36	1.12	19.09	0.03	76.03	0.12	0.02	98.93
200	1.03	0.26	0.04	0.02	1.89	1.21	21.80	0.03	73.63	0.14	0.01	100.06
205	0.92	0.22	0.05	0.04	1.14	1.39	25.92	0.06	69.37	0.12	0.04	99.27



**PFE1505: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.48	0.26	0.04	0.02	1.97	1.28	23.21	0.03	70.52	0.15	0.01	98.96
5	1.13	0.25	0.04	0.01	2.00	1.16	18.50	0.01	75.72	0.11	0.01	98.94
10	1.11	0.21	0.04	0.08	2.00	1.34	18.13	0.03	76.66	0.13	0.01	99.75
15	1.14	0.19	0.03	0.00	2.11	1.04	14.92	0.04	80.58	0.13	0.00	100.17
20	1.44	0.22	0.04	0.04	2.45	1.11	17.23	0.02	77.26	0.11	0.01	99.92
25	1.09	0.19	0.04	0.05	1.59	1.06	15.76	0.03	80.17	0.11	0.01	100.09
30	1.05	0.21	0.04	0.05	1.75	1.27	16.63	0.03	78.87	0.13	0.01	100.03
35	1.05	0.18	0.03	0.07	1.83	1.11	16.62	0.02	79.60	0.12	0.01	100.64
40	0.96	0.21	0.04	0.07	1.54	0.99	15.22	0.04	80.71	0.12	0.01	99.91
45	1.08	0.19	0.04	0.05	1.84	0.92	15.99	0.03	79.51	0.12	0.01	99.77
50	0.52	0.20	0.04	0.01	0.82	0.85	8.97	0.04	87.48	0.11	0.01	99.05
55	1.01	0.21	0.04	0.06	1.85	1.08	18.11	0.04	77.50	0.12	0.01	100.02
60	0.32	0.25	0.03	0.02	0.50	0.62	4.73	0.02	92.82	0.10	0.00	99.42
65	0.28	0.30	0.04	0.05	0.40	0.72	3.23	0.02	94.94	0.09	0.01	100.06
70	0.84	0.22	0.03	0.04	1.57	0.99	12.82	0.02	82.74	0.12	0.01	99.39
75	0.77	0.23	0.04	0.03	1.59	2.21	12.21	0.05	82.88	0.13	0.03	100.16
80	0.77	0.20	0.04	0.03	1.77	1.02	10.67	0.04	85.52	0.13	0.01	100.20
85	0.62	0.21	0.03	0.02	1.49	0.96	8.01	0.03	88.35	0.12	0.00	99.84
90	0.18	0.25	0.04	0.03	0.14	0.47	1.00	0.00	98.30	0.10	0.00	100.50
95	0.87	0.23	0.04	0.00	2.55	1.10	12.28	0.03	82.84	0.11	0.02	100.06

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
100	1.12	0.21	0.04	0.01	3.17	4.81	19.21	0.04	72.15	0.13	0.04	100.91
105	0.82	0.21	0.04	0.03	2.37	1.10	12.17	0.02	82.43	0.12	0.01	99.33
110	0.81	0.27	0.03	0.03	2.99	1.12	12.70	0.02	81.92	0.12	0.01	100.02
115	0.70	0.20	0.04	0.04	2.28	1.20	12.33	0.01	82.97	0.12	0.00	99.88
120	0.61	0.19	0.04	0.02	1.83	1.01	10.18	0.03	85.51	0.11	0.01	99.54
125	0.91	0.23	0.04	0.05	2.93	1.49	17.71	0.02	76.07	0.13	0.01	99.59
130	0.53	0.22	0.04	0.02	1.44	0.93	10.06	0.02	86.05	0.11	0.01	99.41
135	0.51	0.23	0.04	0.01	1.62	0.92	9.71	0.01	87.02	0.11	0.01	100.18
140	0.71	0.22	0.04	0.03	2.34	1.20	13.24	0.03	82.23	0.12	0.01	100.15
145	0.84	0.20	0.04	0.00	2.76	1.69	21.76	0.01	72.57	0.14	0.02	100.01
150	0.62	0.22	0.04	0.02	2.10	1.09	13.78	0.02	81.55	0.12	0.01	99.55
155	0.72	0.21	0.04	0.06	2.68	1.14	18.08	0.03	76.60	0.12	0.02	99.68
160	0.56	0.22	0.04	0.00	2.01	1.11	16.37	0.02	79.11	0.12	0.00	99.54
165	0.73	0.23	0.04	0.06	2.94	1.99	40.28	0.01	51.72	0.11	0.01	98.12
170	0.46	0.22	0.04	0.03	1.64	1.51	41.31	0.05	52.92	0.09	0.01	98.28
175	0.64	0.21	0.04	0.00	1.73	0.85	15.54	0.02	79.83	0.09	0.01	98.95
180	0.35	0.19	0.04	0.05	0.98	0.94	7.91	0.03	88.88	0.11	0.02	99.48
185	0.60	0.24	0.04	0.07	1.57	1.89	18.38	0.04	77.61	0.09	0.03	100.55

**PFE1505: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.84	0.24	0.04	0.05	1.99	1.04	12.79	0.02	82.47	0.14	0.02	99.63
5	0.75	0.18	0.04	0.03	2.10	1.09	11.58	0.03	83.94	0.14	0.01	99.89
10	0.70	0.21	0.03	0.02	2.11	1.09	10.16	0.02	85.48	0.12	0.01	99.93
15	0.88	0.23	0.04	0.06	2.37	1.13	12.11	0.04	83.00	0.13	0.00	99.98
20	0.70	0.23	0.04	0.05	1.89	1.12	10.56	0.04	85.14	0.12	0.01	99.89
25	0.77	0.23	0.04	0.00	1.97	1.10	10.76	0.03	84.58	0.13	0.01	99.61
30	0.73	0.24	0.03	0.08	1.95	1.17	11.13	0.03	84.72	0.12	0.01	100.21
35	0.67	0.21	0.03	0.03	2.01	1.05	10.60	0.03	85.54	0.12	0.01	100.30
40	0.78	0.22	0.03	0.04	1.91	1.12	11.94	0.03	83.31	0.13	0.00	99.51
45	0.73	0.22	0.04	0.04	2.05	1.15	11.42	0.02	83.86	0.12	0.02	99.66
50	0.73	0.22	0.03	0.05	1.87	1.03	11.34	0.02	84.32	0.12	0.01	99.75
55	0.70	0.21	0.03	0.01	2.20	1.04	12.46	0.02	83.04	0.12	0.01	99.84
60	0.68	0.19	0.04	0.03	2.12	1.17	12.06	0.02	83.06	0.14	0.01	99.51
65	0.67	0.22	0.04	0.00	2.13	1.03	11.11	0.01	84.65	0.14	0.01	100.01
70	0.73	0.21	0.04	0.02	1.95	1.11	11.27	0.03	84.17	0.12	0.02	99.66
75	0.89	0.20	0.03	0.08	2.94	0.99	13.25	0.01	81.39	0.11	0.00	99.89
80	0.72	0.23	0.04	0.00	2.32	0.96	12.83	0.02	82.67	0.13	0.01	99.94
85	0.78	0.19	0.04	0.00	2.33	1.14	15.05	0.01	80.07	0.12	0.01	99.73
90	0.87	0.24	0.04	0.03	2.98	2.07	16.50	0.05	75.84	0.16	0.02	98.78
95	0.78	0.25	0.04	0.04	2.68	0.89	13.96	0.02	81.22	0.10	0.01	99.96
100	0.86	0.20	0.05	0.04	2.62	3.61	18.68	0.04	73.59	0.15	0.04	99.89
105	0.66	0.24	0.04	0.01	1.96	0.83	12.81	0.02	83.16	0.13	0.01	99.84
110	0.60	0.20	0.03	0.00	1.59	0.77	10.95	0.03	85.72	0.11	0.01	100.01
115	0.59	0.22	0.05	0.01	1.37	1.66	9.29	0.03	84.80	0.09	0.01	98.12
120	0.64	0.21	0.04	0.00	1.51	0.81	9.86	0.04	85.96	0.11	0.01	99.18

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
125	0.59	0.22	0.04	0.00	1.68	6.24	11.74	0.07	80.15	0.12	0.06	100.91
130	0.70	0.25	0.04	0.02	2.41	1.38	16.48	0.01	78.58	0.13	0.01	100.00
135	0.74	0.21	0.04	0.04	2.27	2.81	16.23	0.05	76.60	0.12	0.03	99.13
140	0.87	0.21	0.04	0.03	3.03	1.53	21.96	0.03	72.64	0.12	0.01	100.45
145	0.96	0.22	0.04	0.00	2.58	1.22	19.63	0.04	74.43	0.12	0.02	99.26
150	0.52	0.20	0.04	0.00	1.56	7.51	12.48	0.06	79.66	0.11	0.03	102.16
155	0.72	0.24	0.04	0.05	2.35	7.88	18.31	0.12	67.36	0.13	0.05	97.27
160	0.85	0.22	0.04	0.01	2.88	1.19	19.96	0.03	75.24	0.11	0.01	100.55
165	0.75	0.20	0.04	0.03	2.77	1.29	16.08	0.03	79.23	0.14	0.02	100.57
170	1.01	0.20	0.05	0.00	2.84	2.37	22.65	0.04	69.96	0.15	0.03	99.29
175	0.75	0.23	0.04	0.03	3.24	1.38	13.74	0.03	80.86	0.14	0.01	100.43
180	0.67	0.25	0.04	0.01	2.60	1.15	12.06	0.03	83.79	0.13	0.01	100.74
185	0.84	0.25	0.03	0.03	2.87	1.45	22.14	0.01	71.29	0.13	0.02	99.04
190	0.80	0.23	0.05	0.05	2.18	2.73	25.82	0.04	65.98	0.12	0.04	98.05
195	0.61	0.25	0.04	0.04	2.40	2.17	21.03	0.04	72.36	0.13	0.01	99.07
200	0.77	0.23	0.03	0.08	2.78	1.43	26.72	0.02	67.78	0.16	0.00	99.99
205	0.61	0.19	0.04	0.02	1.83	1.43	14.17	0.03	81.04	0.12	0.01	99.48
210	0.46	0.19	0.03	0.00	1.98	0.96	14.08	0.02	83.35	0.12	0.00	101.20
215	0.53	0.22	0.04	0.01	1.57	0.83	14.51	0.03	81.32	0.09	0.00	99.13
220	0.62	0.21	0.04	0.03	2.16	4.29	18.69	0.05	73.58	0.10	0.02	99.79
225	0.77	0.20	0.04	0.02	2.33	1.23	21.40	0.03	73.71	0.11	0.02	99.86
230	0.53	0.20	0.04	0.05	1.07	1.26	20.82	0.03	74.62	0.08	0.03	98.71
235	0.56	0.23	0.04	0.00	0.16	1.86	23.24	0.02	72.60	0.04	0.02	98.76
240	0.43	0.20	0.03	0.00	0.04	1.73	10.22	0.05	80.64	0.02	0.02	93.38

**PFE1612: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.74	0.24	0.03	0.03	2.33	1.20	14.44	0.01	83.94	0.14	0.00	103.09
5	0.66	0.23	0.04	0.00	2.09	1.17	13.48	0.02	84.71	0.14	0.01	102.54
10	0.85	0.16	0.04	0.00	1.79	1.08	9.16	0.02	77.27	0.06	0.00	90.44
15	0.78	0.20	0.04	0.03	2.31	1.09	12.50	0.02	85.40	0.12	0.01	102.48
20	0.78	0.25	0.03	0.00	2.37	1.08	14.56	0.01	83.31	0.14	0.01	102.53
25	0.81	0.20	0.04	0.00	2.29	1.14	15.25	0.02	82.35	0.12	0.02	102.23
30	1.04	0.24	0.04	0.08	2.06	0.78	26.85	0.00	72.34	0.10	0.01	103.53
35	0.83	0.24	0.04	0.00	2.35	1.09	15.22	0.02	82.51	0.13	0.00	102.43
40	0.69	0.23	0.03	0.03	1.99	1.15	13.22	0.02	84.69	0.13	0.01	102.21
45	0.83	0.22	0.03	0.05	2.18	1.12	13.58	0.01	83.84	0.13	0.00	102.00
50	0.73	0.23	0.03	0.03	2.15	1.27	13.84	0.03	83.65	0.14	0.01	102.10
55	0.84	0.27	0.04	0.00	2.26	1.19	14.03	0.01	83.86	0.14	0.01	102.64
60	0.79	0.23	0.04	0.00	2.37	1.30	13.96	0.01	83.44	0.13	0.01	102.27
65	0.79	0.22	0.04	0.06	2.16	1.12	14.80	0.01	83.18	0.12	0.00	102.50
70	0.87	0.22	0.04	0.03	2.01	1.25	12.90	0.00	84.54	0.12	0.01	101.98
75	0.84	0.19	0.04	0.00	2.05	1.09	12.83	0.01	84.98	0.13	0.01	102.16
80	0.83	0.20	0.04	0.06	2.01	1.15	12.78	0.01	84.76	0.14	0.01	101.98
85	0.83	0.22	0.04	0.00	1.94	1.14	12.94	0.02	84.56	0.14	0.00	101.83

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
90	0.81	0.23	0.04	0.00	1.98	1.26	12.36	0.01	85.39	0.12	0.01	102.21
95	0.63	0.29	0.03	0.02	1.91	1.34	12.57	0.03	84.47	0.17	0.00	101.45
100	1.31	0.24	0.04	0.04	1.69	0.61	21.83	0.00	76.79	0.07	0.00	102.62
105	0.83	0.22	0.04	0.01	2.19	1.05	16.94	0.03	80.30	0.14	0.01	101.75
110	0.80	0.22	0.04	0.05	1.90	1.21	12.84	0.01	84.20	0.14	0.01	101.42
115	0.77	0.24	0.04	0.08	1.68	1.03	19.10	0.02	78.43	0.14	0.03	101.55
120	0.60	0.26	0.04	0.04	1.85	1.24	17.33	0.02	80.01	0.15	0.02	101.56
125	1.63	0.24	0.04	0.10	3.35	0.71	36.06	0.01	61.51	0.09	0.01	103.73
130	1.43	0.21	0.04	0.04	3.73	2.54	28.95	0.01	63.47	0.23	0.00	100.64
135	0.28	0.13	0.03	0.00	1.03	0.98	14.64	0.04	60.72	0.07	0.00	77.93
140	0.95	0.22	0.05	0.00	1.95	1.09	24.48	0.03	69.73	0.14	0.02	98.65
145	0.47	0.19	0.04	0.00	1.13	0.72	8.84	0.03	73.24	0.09	0.00	84.74
150	0.96	0.23	0.05	0.03	1.60	1.36	15.58	0.03	81.15	0.12	0.01	101.11
155	0.71	0.27	0.04	0.01	1.28	1.46	14.55	0.02	84.05	0.09	0.02	102.49
160	0.46	0.26	0.04	0.00	0.62	1.14	8.74	0.02	87.21	0.06	0.01	98.56
165	0.45	0.17	0.04	0.00	0.24	0.81	11.39	0.05	71.17	0.02	0.01	84.36

**PFE1612: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.86	0.24	0.04	0.02	1.93	1.19	18.69	0.03	80.07	0.14	0.01	103.21
5	0.77	0.18	0.04	0.05	1.89	1.18	18.90	0.02	79.41	0.13	0.02	102.60
10	0.85	0.25	0.04	0.05	1.81	1.11	18.58	0.02	80.36	0.15	0.02	103.23
15	0.93	0.25	0.03	0.03	1.82	1.14	19.65	0.03	79.13	0.12	0.01	103.14
20	0.99	0.19	0.04	0.01	1.77	1.15	19.90	0.04	78.54	0.12	0.01	102.76
25	0.95	0.22	0.03	0.05	1.93	1.17	18.49	0.03	80.04	0.13	0.01	103.04
30	0.53	0.21	0.04	0.03	0.59	0.98	8.93	0.09	88.92	0.09	0.04	100.44
35	0.92	0.24	0.04	0.05	1.16	0.80	18.47	0.05	80.52	0.10	0.01	102.36
40	0.87	0.24	0.04	0.01	2.06	1.31	17.95	0.03	79.98	0.15	0.01	102.64
45	0.93	0.23	0.04	0.00	1.91	1.21	19.58	0.03	78.55	0.14	0.01	102.61
50	0.92	0.27	0.04	0.12	1.70	1.20	18.82	0.03	79.70	0.13	0.01	102.94
55	0.98	0.23	0.03	0.00	1.72	1.22	17.96	0.02	80.27	0.15	0.01	102.58
60	0.95	0.25	0.04	0.03	1.64	1.12	19.28	0.03	78.93	0.11	0.00	102.38
65	1.18	0.23	0.04	0.03	1.66	1.13	20.70	0.03	78.17	0.14	0.01	103.31
70	1.17	0.21	0.04	0.02	1.40	1.17	20.24	0.03	78.94	0.13	0.00	103.35
75	1.13	0.24	0.04	0.04	1.50	1.12	18.83	0.04	79.56	0.15	0.01	102.66
80	1.04	0.20	0.04	0.03	1.47	1.17	18.38	0.03	79.96	0.16	0.01	102.47
85	0.64	0.20	0.04	0.00	0.66	0.81	8.84	0.05	89.93	0.08	0.02	101.27
90	0.99	0.23	0.04	0.04	1.89	1.09	17.41	0.02	80.27	0.15	0.00	102.13
95	0.88	0.22	0.04	0.00	1.95	1.38	16.68	0.02	81.32	0.12	0.01	102.62
100	0.84	0.25	0.04	0.04	1.74	1.12	15.04	0.02	83.04	0.15	0.01	102.28

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
105	0.82	0.25	0.03	0.04	1.80	1.27	14.53	0.02	83.56	0.11	0.01	102.44
110	0.99	0.23	0.04	0.06	1.80	4.33	19.12	0.04	77.94	0.12	0.02	104.66
115	0.74	0.19	0.04	0.03	2.20	1.26	15.64	0.02	82.28	0.14	0.01	102.54
120	0.82	0.27	0.04	0.06	2.19	1.21	14.80	0.04	83.11	0.13	0.01	102.68
125	0.73	0.20	0.04	0.03	2.05	1.13	14.94	0.03	82.80	0.13	0.00	102.09
130	0.66	0.20	0.04	0.02	2.16	1.26	14.04	0.03	84.26	0.14	0.01	102.82
135	0.66	0.24	0.04	0.02	2.09	1.26	14.13	0.02	83.30	0.13	0.01	101.92
140	0.98	0.18	0.04	0.01	2.04	0.80	23.50	0.01	75.47	0.08	0.01	103.11
145	0.51	0.20	0.04	0.04	2.16	1.45	11.36	0.03	76.66	0.18	0.01	92.62
150	0.72	0.24	0.04	0.03	2.44	1.20	15.88	0.01	79.38	0.16	0.00	100.10
155	0.83	0.25	0.04	0.00	2.29	0.85	19.74	0.03	78.52	0.13	0.00	102.66
160	0.68	0.22	0.04	0.00	1.65	0.73	9.78	0.01	87.09	0.10	0.00	100.31
165	1.07	0.14	0.03	0.00	2.86	0.79	14.83	0.02	79.06	0.13	0.01	98.95
170	0.72	0.22	0.03	0.00	2.39	0.85	13.69	0.03	76.30	0.11	0.00	94.33
175	0.88	0.18	0.04	0.04	2.05	0.85	13.44	0.02	80.66	0.12	0.02	98.29
180	0.22	0.32	0.07	0.23	0.75	1.89	94.95	0.01	33.17	0.09	0.02	131.71
185	0.27	0.35	0.05	0.12	0.89	2.44	67.74	0.01	49.98	0.09	0.03	121.97
190	0.68	0.21	0.04	0.02	1.83	1.12	16.26	0.01	81.37	0.10	0.01	101.64
195	0.83	0.21	0.04	0.05	1.64	1.18	18.50	0.02	79.50	0.10	0.02	102.09
200	1.20	0.23	0.04	0.00	2.38	5.20	23.48	0.01	70.69	0.08	0.05	103.35



**PFE1612: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.02	0.18	0.04	0.00	1.44	1.18	19.98	0.05	80.02	0.13	0.02	104.06
5	1.13	0.18	0.04	0.04	1.53	1.11	21.24	0.04	78.65	0.12	0.01	104.09
10	0.99	0.22	0.04	0.03	1.45	1.15	21.12	0.02	79.07	0.12	0.01	104.23
15	0.99	0.22	0.04	0.03	1.57	1.19	20.96	0.04	79.26	0.12	0.01	104.41
20	0.89	0.21	0.04	0.04	1.61	1.27	19.05	0.02	79.63	0.13	0.00	102.90
25	0.92	0.21	0.03	0.04	1.74	1.18	18.43	0.03	80.12	0.11	0.01	102.81
30	0.90	0.21	0.03	0.06	1.85	1.20	18.30	0.01	80.36	0.13	0.01	103.07
35	1.23	0.24	0.04	0.01	1.83	0.77	26.48	0.01	72.42	0.12	0.01	103.17
40	1.23	0.25	0.04	0.05	2.23	1.09	27.22	0.02	72.42	0.13	0.01	104.68
45	0.87	0.19	0.04	0.02	2.05	1.19	19.17	0.03	79.82	0.13	0.02	103.52
50	0.91	0.22	0.04	0.03	2.01	1.16	18.97	0.02	80.67	0.13	0.01	104.18
55	0.81	0.23	0.03	0.02	2.02	1.14	19.00	0.01	81.20	0.13	0.01	104.60
60	0.75	0.23	0.04	0.05	2.12	1.11	19.31	0.03	80.75	0.13	0.01	104.51
65	0.78	0.27	0.04	0.05	2.04	1.11	18.67	0.03	80.64	0.12	0.01	103.76
70	0.70	0.23	0.04	0.06	2.24	1.10	17.86	0.02	82.01	0.13	0.01	104.38
75	0.72	0.20	0.04	0.03	2.32	1.17	18.30	0.02	81.18	0.12	0.01	104.09
80	0.69	0.23	0.04	0.04	2.32	1.20	17.54	0.01	81.58	0.13	0.01	103.78
85	0.66	0.21	0.04	0.03	2.41	1.19	16.99	0.01	81.39	0.13	0.01	103.05
90	0.67	0.24	0.04	0.02	2.25	1.07	16.60	0.00	82.07	0.13	0.00	103.07
95	0.72	0.21	0.04	0.06	2.40	1.08	15.85	0.02	82.61	0.12	0.01	103.10
100	0.74	0.25	0.04	0.06	2.20	1.22	15.70	0.04	82.73	0.11	0.01	103.09
105	0.70	0.21	0.04	0.00	2.33	1.08	15.80	0.03	82.33	0.15	0.01	102.68
110	0.61	0.25	0.04	0.05	2.32	1.16	14.94	0.02	82.93	0.13	0.00	102.46
115	0.65	0.24	0.04	0.04	2.27	1.08	13.97	0.03	83.85	0.13	0.01	102.28

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
120	0.84	0.25	0.04	0.05	1.70	1.20	17.62	0.06	81.97	0.08	0.01	103.80
125	0.72	0.24	0.04	0.04	2.38	1.23	13.92	0.03	83.22	0.14	0.01	101.95
130	0.64	0.25	0.04	0.00	2.26	1.21	12.78	0.01	84.24	0.11	0.00	101.52
135	0.65	0.23	0.03	0.00	2.32	1.32	12.62	0.03	84.57	0.12	0.00	101.89
140	0.66	0.19	0.04	0.04	2.64	1.23	13.32	0.03	83.88	0.14	0.01	102.17
145	0.83	0.26	0.04	0.03	1.66	0.60	15.66	0.02	82.70	0.06	0.01	101.89
150	0.77	0.25	0.04	0.03	2.45	1.47	14.30	0.02	82.52	0.15	0.01	102.01
155	0.52	0.23	0.04	0.05	2.35	1.19	12.43	0.02	85.10	0.18	0.00	102.12
160	0.96	0.24	0.04	0.02	2.51	1.07	18.24	0.02	79.04	0.15	0.01	102.30
165	0.96	0.19	0.04	0.07	1.34	0.64	12.39	0.03	85.94	0.06	0.01	101.67
170	0.80	0.24	0.04	0.03	2.35	1.42	12.71	0.03	84.07	0.15	0.00	101.82
175	0.95	0.17	0.04	0.00	1.88	0.94	16.24	0.02	81.32	0.14	0.01	101.70
180	1.02	0.25	0.03	0.02	1.90	1.03	18.17	0.03	79.68	0.11	0.01	102.25
185	0.78	0.24	0.03	0.00	1.45	1.12	15.88	0.02	83.19	0.08	0.02	102.81
190	1.11	0.21	0.04	0.02	2.21	1.08	19.46	0.02	78.76	0.13	0.00	103.03
195	0.99	0.29	0.04	0.00	2.06	0.97	16.59	0.03	81.30	0.14	0.01	102.40
200	0.84	0.18	0.04	0.03	1.58	0.83	15.61	0.00	83.34	0.09	0.00	102.54
205	0.94	0.24	0.03	0.00	2.26	1.02	17.57	0.02	80.62	0.14	0.01	102.85
210	0.62	0.18	0.04	0.02	1.82	1.25	12.28	0.03	85.26	0.17	0.01	101.66
215	0.31	0.21	0.04	0.02	0.62	1.09	4.75	0.02	92.96	0.09	0.02	100.13
220	0.99	0.26	0.04	0.02	1.62	1.33	11.48	0.04	85.22	0.13	0.01	101.12
225	1.19	0.22	0.04	0.00	1.85	0.68	12.10	0.05	83.99	0.11	0.02	100.24
230	0.39	0.15	0.02	0.00	0.77	1.65	18.53	0.06	57.17	0.03	0.01	78.79

**PFE1617: Particle 1**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.84	0.21	0.04	0.05	2.08	1.18	18.70	0.02	81.14	0.13	0.01	104.41
5	0.86	0.23	0.04	0.13	1.94	1.20	19.34	0.02	81.06	0.13	0.01	104.96
10	0.84	0.24	0.04	0.05	2.07	1.21	19.38	0.01	80.59	0.13	0.01	104.55
15	0.75	0.22	0.04	0.01	1.97	1.12	19.38	0.02	80.85	0.12	0.01	104.49
20	0.83	0.25	0.04	0.01	1.90	1.12	18.29	0.02	80.92	0.12	0.01	103.52
25	0.82	0.21	0.04	0.04	1.95	1.12	18.23	0.02	81.09	0.12	0.00	103.65
30	0.81	0.23	0.04	0.02	1.97	1.24	18.84	0.02	80.50	0.12	0.00	103.78
35	0.83	0.26	0.03	0.06	1.89	1.21	19.20	0.01	81.24	0.13	0.00	104.85
40	0.87	0.21	0.04	0.04	1.86	1.15	18.97	0.02	81.43	0.11	0.01	104.71
45	0.88	0.26	0.05	0.05	1.96	1.20	19.01	0.03	80.50	0.12	0.00	104.05
50	0.86	0.23	0.04	0.09	1.87	1.11	19.13	0.02	80.31	0.12	0.01	103.78
55	0.93	0.26	0.04	0.09	2.03	1.19	19.56	0.02	79.76	0.12	0.01	104.00
60	0.89	0.22	0.04	0.05	1.94	1.21	18.70	0.02	80.42	0.13	0.00	103.62
65	0.96	0.21	0.04	0.01	1.88	1.19	18.41	0.01	80.26	0.12	0.01	103.10
70	0.85	0.21	0.04	0.00	1.75	1.15	18.65	0.01	80.67	0.12	0.01	103.44
75	0.93	0.18	0.04	0.04	1.85	1.08	18.21	0.02	80.85	0.13	0.00	103.32
80	0.90	0.20	0.04	0.02	1.83	1.11	17.23	0.02	81.39	0.14	0.01	102.90
85	0.88	0.17	0.03	0.02	1.78	1.12	18.24	0.03	80.87	0.12	0.00	103.25
90	0.93	0.22	0.04	0.02	1.78	1.22	17.05	0.03	81.88	0.14	0.01	103.31
95	0.91	0.20	0.04	0.03	1.78	1.09	17.98	0.03	80.47	0.13	0.02	102.66
100	0.94	0.22	0.04	0.00	1.72	1.16	16.57	0.03	81.57	0.14	0.00	102.38
105	0.93	0.21	0.04	0.04	1.76	1.11	17.48	0.02	81.16	0.12	0.01	102.87
110	0.95	0.25	0.04	0.04	1.48	1.38	15.72	0.04	82.75	0.11	0.04	102.79

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
115	1.07	0.21	0.04	0.08	1.74	1.13	17.33	0.04	81.07	0.14	0.01	102.85
120	0.89	0.20	0.04	0.00	1.57	1.10	15.30	0.04	83.49	0.11	0.02	102.74
125	1.01	0.22	0.04	0.01	1.70	1.18	17.96	0.03	80.08	0.12	0.01	102.35
130	0.42	0.23	0.04	0.00	0.82	6.15	8.86	0.17	84.07	0.10	0.11	100.97
135	0.53	0.22	0.03	0.00	1.42	1.09	10.46	0.04	87.80	0.13	0.01	101.73
140	0.34	0.23	0.04	0.00	0.93	2.99	8.40	0.14	84.09	0.08	0.05	97.28
145	0.16	0.23	0.04	0.00	0.40	1.15	2.47	0.09	94.84	0.08	0.05	99.49
150	0.22	0.16	0.04	0.02	0.58	8.94	3.29	0.16	83.94	0.07	0.10	97.52
155	0.07	0.20	0.05	0.00	0.15	6.19	0.83	0.21	91.91	0.04	0.08	99.72
160	0.10	0.23	0.04	0.00	0.24	2.52	0.80	0.07	95.08	0.05	0.02	99.15
165	0.13	0.20	0.03	0.01	0.46	2.71	2.30	0.09	88.62	0.04	0.02	94.60
170	0.12	0.18	0.04	0.00	0.52	3.11	2.79	0.16	84.80	0.07	0.08	91.87
175	0.05	0.23	0.03	0.03	0.17	2.58	0.40	0.09	92.04	0.05	0.03	95.68
180	0.10	0.17	0.03	0.00	0.33	6.07	1.65	0.19	82.64	0.09	0.07	91.36
185	0.11	0.17	0.03	0.00	0.46	4.91	2.57	0.12	85.46	0.09	0.04	93.96
190	0.06	0.22	0.03	0.00	0.10	2.53	0.66	0.17	87.03	0.07	0.05	90.91
195	0.04	0.18	0.03	0.00	0.06	1.34	0.48	0.06	92.14	0.08	0.01	94.41
200	0.03	0.14	0.03	0.00	0.09	1.02	0.36	0.10	90.17	0.06	0.02	92.02
205	0.05	0.16	0.04	0.00	0.03	6.01	0.32	0.22	77.03	0.06	0.06	83.96
210	0.05	0.16	0.04	0.00	0.11	15.11	0.84	0.56	75.57	0.09	0.25	92.78
215	0.06	0.22	0.04	0.04	0.13	2.07	0.11	0.37	94.02	0.03	0.18	97.27
220	0.07	0.21	0.04	0.01	0.21	2.56	0.64	0.19	96.64	0.04	0.10	100.70

**PFE1617: Particle 2**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	0.77	0.24	0.04	0.03	2.03	1.13	13.85	0.03	84.01	0.14	0.01	102.26
5	0.79	0.21	0.04	0.08	2.07	1.13	14.26	0.02	84.21	0.12	0.00	102.93
10	0.76	0.24	0.04	0.05	2.04	1.17	14.32	0.02	83.88	0.13	0.00	102.65
15	0.77	0.18	0.04	0.03	2.12	1.20	14.73	0.02	83.40	0.13	0.01	102.64
20	0.77	0.18	0.03	0.09	2.12	1.14	15.48	0.02	82.86	0.13	0.01	102.83
25	0.82	0.19	0.04	0.05	2.03	1.24	14.47	0.01	83.72	0.14	0.01	102.72
30	0.79	0.24	0.04	0.02	2.07	0.94	16.07	0.02	79.78	0.13	0.01	100.11
35	0.78	0.22	0.04	0.06	2.05	1.12	16.26	0.02	82.53	0.12	0.01	103.20
40	0.78	0.25	0.04	0.01	2.08	1.15	16.62	0.03	82.24	0.13	0.02	103.35
45	0.85	0.20	0.04	0.02	1.95	1.10	15.75	0.01	82.62	0.15	0.01	102.68
50	0.78	0.25	0.04	0.02	1.83	1.10	15.98	0.02	82.82	0.13	0.01	102.97
55	0.84	0.22	0.04	0.00	1.95	1.12	15.26	0.02	82.90	0.12	0.01	102.48
60	0.81	0.17	0.04	0.07	1.94	1.07	15.41	0.01	82.94	0.13	0.00	102.57
65	0.84	0.22	0.05	0.03	2.00	1.17	15.22	0.02	83.09	0.14	0.00	102.78
70	0.95	0.25	0.04	0.06	1.87	1.08	14.80	0.02	83.25	0.12	0.00	102.44

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
75	0.85	0.18	0.03	0.00	1.90	1.16	14.01	0.02	83.66	0.13	0.01	101.95
80	0.95	0.25	0.04	0.02	2.03	1.25	14.12	0.02	83.39	0.14	0.01	102.21
85	0.92	0.22	0.04	0.04	1.86	1.09	12.62	0.01	84.73	0.14	0.01	101.67
90	0.89	0.28	0.04	0.00	2.00	1.43	12.95	0.03	84.51	0.14	0.00	102.26
95	1.06	0.20	0.04	0.02	2.29	1.70	13.64	0.03	82.39	0.13	0.02	101.51
100	0.89	0.22	0.03	0.03	1.94	1.48	11.76	0.03	84.90	0.15	0.02	101.46
105	0.84	0.18	0.03	0.05	1.88	1.44	11.92	0.03	84.99	0.13	0.01	101.49
110	0.20	0.21	0.03	0.04	1.15	1.59	5.91	0.04	89.65	0.13	0.01	98.97
115	0.03	0.18	0.04	0.00	0.13	0.76	0.46	0.06	89.03	0.05	0.02	90.74
120	0.04	0.20	0.03	0.00	0.12	0.91	0.17	0.06	92.04	0.05	0.00	93.62
125	0.06	0.12	0.02	0.00	0.15	1.55	0.55	0.09	66.68	0.10	0.00	69.31
130	0.05	0.21	0.03	0.00	0.13	0.87	0.43	0.10	86.71	0.04	0.04	88.60
135	0.06	0.20	0.03	0.00	0.12	1.59	0.38	0.13	95.85	0.06	0.05	98.48
140	0.10	0.23	0.04	0.00	0.45	4.99	1.74	0.14	91.38	0.09	0.07	99.22

**PFE1617: Particle 3**

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
0	1.46	0.25	0.04	0.05	1.49	1.13	21.95	0.05	77.13	0.14	0.03	103.73
5	1.81	0.28	0.05	0.06	1.55	1.12	21.94	0.08	76.55	0.13	0.03	103.59
10	3.24	0.28	0.05	0.08	1.86	0.98	20.67	0.45	75.29	0.13	0.10	103.13
15	3.17	0.25	0.05	0.07	1.89	1.15	19.36	0.62	73.23	0.12	0.07	99.96
20	1.61	0.21	0.05	0.11	1.29	6.54	19.48	0.17	70.99	0.11	0.13	100.68
25	1.51	0.24	0.04	0.00	1.57	1.16	22.14	0.05	76.96	0.13	0.02	103.81
30	1.49	0.23	0.04	0.04	1.58	1.19	21.89	0.04	77.14	0.13	0.01	103.78
35	1.29	0.23	0.04	0.06	1.63	1.24	21.63	0.05	77.68	0.12	0.01	103.99
40	1.24	0.29	0.04	0.08	1.58	1.17	21.37	0.03	78.20	0.13	0.01	104.14
45	1.05	0.21	0.04	0.01	1.80	1.09	20.97	0.02	78.64	0.12	0.02	103.96
50	1.05	0.24	0.04	0.00	1.86	1.09	20.35	0.03	78.80	0.14	0.01	103.61
55	0.99	0.26	0.04	0.07	1.81	1.23	17.98	0.03	80.40	0.12	0.01	102.94
60	1.00	0.22	0.04	0.06	1.98	1.21	18.24	0.02	79.69	0.13	0.01	102.59
65	0.92	0.24	0.04	0.06	1.87	1.07	17.38	0.02	80.31	0.12	0.01	102.03
70	0.84	0.23	0.04	0.05	1.93	1.18	16.90	0.02	80.98	0.13	0.01	102.32
75	0.83	0.26	0.03	0.05	1.99	1.20	18.19	0.03	79.98	0.12	0.01	102.69
80	0.92	0.24	0.04	0.02	1.96	1.19	17.86	0.02	80.50	0.14	0.01	102.88
85	0.85	0.22	0.04	0.04	1.89	1.12	17.67	0.05	79.72	0.14	0.00	101.72
90	0.87	0.21	0.04	0.03	1.84	1.09	17.41	0.03	80.91	0.12	0.00	102.55
95	0.86	0.22	0.04	0.01	1.86	1.18	18.46	0.03	80.38	0.12	0.01	103.16
100	0.91	0.25	0.05	0.02	1.82	1.16	18.65	0.02	80.28	0.13	0.01	103.27
105	0.88	0.20	0.05	0.05	1.75	1.14	19.36	0.02	79.74	0.14	0.01	103.34

Distance from the centre (µm)	MnO %	NiO %	K <sub>2</sub> O %	Na <sub>2</sub> O %	MgO %	Al <sub>2</sub> O <sub>3</sub> %	FeO %	SiO <sub>2</sub> %	TiO <sub>2</sub> %	Cr <sub>2</sub> O <sub>3</sub> %	CaO %	Totals
110	0.91	0.26	0.04	0.00	1.78	1.10	19.85	0.03	79.48	0.11	0.01	103.57
115	0.94	0.26	0.04	0.01	1.82	1.11	19.97	0.04	79.80	0.13	0.00	104.11
120	0.97	0.26	0.04	0.09	1.78	1.17	20.11	0.04	79.23	0.12	0.02	103.84
125	0.97	0.24	0.04	0.03	1.73	1.18	20.22	0.03	79.16	0.11	0.01	103.72
130	0.77	0.19	0.03	0.01	1.38	0.83	17.13	0.02	72.86	0.11	0.00	93.34
135	0.93	0.25	0.05	0.02	1.64	1.09	19.33	0.03	79.16	0.14	0.00	102.64
140	1.00	0.24	0.04	0.05	1.69	1.19	19.20	0.03	78.97	0.13	0.01	102.55
145	1.05	0.24	0.04	0.05	1.70	1.18	19.30	0.04	78.71	0.13	0.02	102.45
150	0.90	0.22	0.04	0.02	1.29	0.95	15.33	0.03	78.09	0.11	0.01	96.97
155	1.08	0.24	0.04	0.04	1.64	1.06	13.90	0.03	82.96	0.14	0.02	101.12
160	0.84	0.21	0.04	0.02	0.94	0.84	9.09	0.03	84.07	0.08	0.01	96.17
165	0.86	0.22	0.04	0.04	1.01	1.07	9.08	0.04	88.68	0.14	0.01	101.18
170	0.83	0.21	0.04	0.00	1.12	0.91	9.26	0.03	88.65	0.13	0.01	101.19
175	0.74	0.20	0.04	0.02	1.33	1.10	10.17	0.04	85.97	0.13	0.01	99.73
180	0.12	0.22	0.03	0.02	0.15	0.62	0.40	0.03	95.65	0.03	0.02	97.28
185	0.17	0.14	0.03	0.00	0.42	0.87	4.05	0.06	73.49	0.05	0.01	79.28
190	0.05	0.14	0.03	0.00	0.01	0.89	0.26	0.10	79.66	0.03	0.02	81.18
195	0.05	0.16	0.04	0.00	0.08	0.73	0.17	0.09	87.72	0.05	0.03	89.11
200	0.07	0.23	0.03	0.00	0.03	1.38	0.30	0.12	93.38	0.09	0.02	95.66
205	0.13	0.26	0.04	0.00	0.07	3.51	0.22	0.19	94.04	0.07	0.06	98.58
210	0.09	0.23	0.04	0.02	0.18	8.14	0.41	0.19	90.55	0.07	0.09	100.00



## APPENDIX XV. Reduction leach logsheets

### BTS Experimental log sheet

		PFE2420								
Date:	99/01/12	%								
Test no.	J1	TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
Temperature (°C)	105	88.2	8.18	1.21	0.90	0.26	0.41	0.11	1.30	1.48
R.p.m.		%								
Feed sample no.	PFE2420	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
Solution volume (L)	0.50	52.81	6.36	0.73	0.48	0.19	0.23	0.08	0.82	0.69
Solution description	HCl 20%									
Solid mass (g)	50.12									
LYNN OOND OKS.	45 min	%								
Add 31.5gSnCl <sub>2</sub> .2H <sub>2</sub> O At 0H		TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
		92.0	6.01	1.10	0.83	0.16	0.41	0.11	1.21	1.54
Final volume (liter)	0.475	%								
Number of samples	1	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
Volume of samples (l)	0.025	55.09	4.67	0.66	0.44	0.11	0.23	0.08	0.77	0.72
Dry residue mass (g)	47.43									
Residue sample no.	PFE2422									

Experimental results		g/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
0.5	PFE2421	1.120	2.260	0.105	0.057	0.071	0.025	0.005	0.139	0.021
	<b>Accountability</b>	1.007	1.032	0.997	0.986	0.944	1.049	1.012	1.041	1.013

Extraction		%								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
0.5	PFE2421	2.116	35.450	14.370	11.938	38.112	10.828	6.895	16.853	3.028

**BTS Experimental log sheet**

		PFE2420								
Date:	99/01/12	%								
Test no.	J2	TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
Temperature (°C)	105	88.2	8.18	1.21	0.90	0.26	0.41	0.11	1.30	1.48
R.p.m.		%								
Feed sample no.	PFE2420	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
Solution volume (L)	0.50	52.81	6.36	0.73	0.48	0.19	0.23	0.08	0.82	0.69
Solution description	HCl 20%									
Solid mass (g)	49.25									
LYNN OOND OKS.	45 min	%								
Add 31.5gSnCl <sub>2</sub> .2H AT 0Hr		TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
		92.1	5.78	1.08	0.82	0.13	0.41	0.11	1.18	1.53
Final Volume(L)	0.475	%								
Number of samples	1	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
Volume of samples (l)	0.025	55.15	4.49	0.65	0.43	0.09	0.23	0.08	0.75	0.72
Dry residue mass (g)	46.16									
Residue sample no.	PFE2424									

**Experimental results**

		g/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.0	PFE2423	1.360	2.360	0.110	0.062	0.082	0.028	0.005	0.161	0.027
	Accountability	1.004	1.020	0.982	0.979	0.894	1.055	0.999	1.039	1.007

**Extraction**

		%								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.0	PFE2423	2.614	37.672	15.321	13.215	44.794	12.341	6.477	19.866	3.962

**BTS Experimental log sheet**

PFE2420

Date:	99/01/12	%							
Test no.	J3	TiO2	FeO	MgO	Al2O3	CaO	V2O3	Cr2O3	MnO
Temperature (°C)	105	88.2	8.18	1.21	0.90	0.26	0.41	0.11	1.30
R.p.m.		%							
Feed sample no.	PFE2420	Ti	Fe	Mg	Al	Ca	V	Cr	Mn
Solution volume (L)	0.50	52.81	6.36	0.73	0.48	0.19	0.23	0.08	0.82
Solution description	HCl 20%								
Solid mass (g)	50.59								
LYNN OOND OKS.	45 min	%							
Add 31.5gSnCl2.2H AT 0Hr		TiO2	FeO	MgO	Al2O3	CaO	V2O3	Cr2O3	MnO
		92.7	5.63	1.07	0.81	0.14	0.40	0.10	1.16
FinalVolume(L)	0.475	%							
Number of samples	1	Ti	Fe	Mg	Al	Ca	V	Cr	Mn
Volume of samples (l)	0.025	55.51	4.38	0.64	0.43	0.10	0.22	0.07	0.73
Dry residue mass (g)	47.15								
Residue sample no.	PFE2426								

**Experimental results**

		g/l							
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE2425	1.620	2.610	0.123	0.070	0.105	0.032	0.006	0.188
	<b>Accountability</b>	1.008	1.027	0.983	0.977	1.032	1.040	0.925	1.046

**Extraction**

		%							
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn
0.0		0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1.5	PFE2425	3.032	40.559	16.678	14.525	55.839	13.731	8.144	22.583

**BTS Experimental log sheet**

<b>Date:</b>	98/12/02	<b>Feed</b>								
<b>Test no.</b>	J9	<b>%</b>								
<b>Temperature (°C)</b>	105	<b>TiO<sub>2</sub></b>	<b>FeO</b>	<b>MgO</b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>CaO</b>	<b>V<sub>2</sub>O<sub>5</sub></b>	<b>Cr<sub>2</sub>O<sub>3</sub></b>	<b>MnO</b>	<b>SiO<sub>2</sub></b>
<b>Stirring speed (rpm)</b>	Boiling	88.20	8.18	1.21	0.90	0.26	0.41	0.11	1.30	1.48
<b>Feed sample no.</b>	PFE2420	<b>%</b>								
<b>Solution volume (L)</b>	0.5	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
<b>Solution description</b>	HCl 20%	52.81	6.36	0.73	0.48	0.19	0.23	0.08	0.82	0.69
<b>Solid mass (g)</b>	39.93	<b>Residue</b>								
<b>LYNN OOND OX</b>	45 min	<b>%</b>								
<b>Ad SnCl<sub>2</sub>.2H<sub>2</sub>O at 0H</b>		<b>TiO<sub>2</sub></b>	<b>FeO</b>	<b>MgO</b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>CaO</b>	<b>V<sub>2</sub>O<sub>5</sub></b>	<b>Cr<sub>2</sub>O<sub>3</sub></b>	<b>MnO</b>	<b>SiO<sub>2</sub></b>
<b>Final volume (L)</b>	0.425	87.90	5.79	1.06	0.79	0.15	0.44	0.12	1.15	1.49
<b>Number of samples</b>	3	<b>%</b>								
<b>Volume of samples (L)</b>	0.025	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
<b>Dry residue mass (g)</b>	37.08	52.63	4.5	0.64	0.42	0.11	0.25	0.08	0.73	0.70
<b>Residue sample no.</b>	2616									
<b>Additives</b>	25.2gSnCl <sub>2</sub> .2H <sub>2</sub> O									

<b>Solution samples</b>		<b>mg/l</b>								
<b>Time(h)</b>	<b>Sample no.</b>	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
0.0		0	0	0	0	0	0	0	0	0
0.5	PFE2613	853	1760	82	45	59	18	3.8	107	13
1.5	PFE2614	1270	2090	103	56	78	23	5.2	149	28
2.0	PFE2615	1400	2170	110	60	90	24	5.1	163	34
<b>Accountability (%)</b>		0.958	1.080	0.999	0.970	1.128	1.125	1.097	1.064	0.994
<b>Time (h)</b>	<b>Solution based extraction</b>	<b>%</b>								
0.0		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.5	PFE2613	2.0	34.7	14.1	11.8	39.8	9.8	6.3	16.3	2.4
1.5	PFE2614	3.0	41.2	17.7	14.7	52.6	12.5	8.7	22.7	5.1
2.0	PFE2615	3.3	42.7	18.9	15.8	60.6	13.0	8.5	24.8	6.2



**BTS Experimental log sheet**

<b>Date:</b>	98/12/02	<b>Feed</b>								
<b>Test no.</b>		<b>%</b>								
<b>Temperature (°C)</b>	105	<b>TiO2</b>	<b>FeO</b>	<b>MgO</b>	<b>Al2O3</b>	<b>CaO</b>	<b>V2O5</b>	<b>Cr2O3</b>	<b>MnO</b>	<b>SiO2</b>
<b>Stirring speed (rpm)</b>	Boiling	88.20	8.18	1.21	0.90	0.26	0.41	0.11	1.30	1.48
<b>Feed sample no.</b>	PFE2420	<b>%</b>								
<b>Solution volume (L)</b>	0.5	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
<b>Solution description</b>	HCl 20%	52.81	6.36	0.73	0.48	0.19	0.23	0.08	0.82	0.69
<b>Solid mass (g)</b>	40.62	<b>Residue</b>								
<b>LYNN OOND OX</b>	45 min	<b>%</b>								
<b>Add SnCl2.2H2O at 0H</b>		<b>TiO2</b>	<b>FeO</b>	<b>MgO</b>	<b>Al2O3</b>	<b>CaO</b>	<b>V2O5</b>	<b>Cr2O3</b>	<b>MnO</b>	<b>SiO2</b>
<b>Final volume (L)</b>	0.4	89.50	5.70	1.06	0.79	0.13	0.43	0.12	1.13	1.46
<b>Number of samples</b>	4	<b>%</b>								
<b>Volume of samples (L)</b>	0.025	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
<b>Dry residue mass (g)</b>	37.46	53.59	4.43	0.64	0.42	0.09	0.24	0.08	0.72	0.68
<b>Residue sample no.</b>	2621									
<b>Additives</b>	25.2gSnCL2.2H2O									

<b>Solution samples</b>		<b>mg/l</b>								
<b>Time(h)</b>	<b>Sample no.</b>	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
0.0		0	0	0	0	0	0	0	0	0
0.5	PFE2617	896	1790	84	46	59	18	3.2	110	14
1.5	PFE2618	1250	2050	101	55	74	23	5.1	146	26
2.0	PFE2619	1400	2140	108	58	87	25	5.4	161	34
3.0	PFE2620	1580	2240	116	64	98	27	6.4	179	38
<b>Accountability (%)</b>		0.971	1.069	0.999	0.971	1.086	1.107	1.106	1.060	0.974
<b>Time (h)</b>	<b>Solution based extraction</b>	<b>%</b>								
0.0		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.5	PFE2617	2.1	34.7	14.2	11.9	39.1	9.6	5.2	16.5	2.5
1.5	PFE2618	2.9	39.7	17.1	14.2	49.0	12.3	8.3	21.8	4.6
2.0	PFE2619	3.3	41.4	18.2	15.0	57.6	13.4	8.8	24.1	6.0
3.0	PFE2620	3.7	43.4	19.6	16.5	64.9	14.4	10.5	26.8	6.8

## BTS Experimental log sheet

Date:	98/12/02	<b>Feed</b>								
Test no.		<b>%</b>								
Temperature (°C)	105	TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
Stirring speed (rpm)	Boiling	88.20	8.18	1.21	0.90	0.26	0.41	0.11	1.30	1.48
Feed sample no.	PFE2420	<b>%</b>								
Solution volume (L)	0.5	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
Solution description	HCl 20%	52.81	6.36	0.73	0.48	0.19	0.23	0.08	0.82	0.69
Solid mass (g)	40.13	<b>Residue</b>								
LYNN OOND OX	45 min	<b>%</b>								
Add SnCl <sub>2</sub> .2H <sub>2</sub> O at 0H		TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>
		88.40	5.56	1.02	0.77	0.11	0.42	0.11	1.10	1.45
		<b>%</b>								
		Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
Final volume (L)	0.375	52.93	4.32	0.61	0.41	0.08	0.24	0.08	0.70	0.68
Number of samples	5									
Volume of samples (L)	0.025									
Dry residue mass (g)	36.62									
Residue sample no.	2627									
Additives	25.2gSnCl <sub>2</sub> .2H <sub>2</sub> O									

Solution samples		mg/l								
Time(h)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si
0.0		0	0	0	0	0	0	0	0	0
0.5	PFE2622	887	1790	85	46	59	18	3.2	110	15
1.5	PFE2623	1210	2020	102	54	82	22	4.6	144	27
2.0	PFE2624	1370	2130	110	59	87	24	5	161	32
3.0	PFE2625	1550	2220	117	64	97	27	6.1	178	37
4.0	PFE2626	1740	2310	132	70	110	29	6	194	42
<b>Accountability (%)</b>		0.953	1.062	0.985	0.956	1.085	1.085	1.008	1.052	0.965
Time (h)	Solution based extraction	<b>%</b>								
0.0		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.5	PFE2622	2.1	35.1	14.5	12.0	39.6	9.7	5.3	16.7	2.7
1.5	PFE2624	2.9	39.6	17.4	14.1	55.0	11.9	7.6	21.8	4.9
2.0	PFE2625	3.2	41.7	18.8	15.4	58.3	13.0	8.3	24.4	5.8
3.0	PFE2626	3.7	43.5	20.0	16.7	65.0	14.6	10.1	27.0	6.7
4.0	PFE2627	4.1	45.3	22.6	18.3	73.7	15.7	9.9	29.4	7.6

**BTS Experimental log sheet**

<b>Date:</b>	99/01/06	<b>Feed</b>								
<b>Test no.</b>		<b>%</b>								
<b>Temperature (°C)</b>	105	<b>TiO<sub>2</sub></b>	<b>FeO</b>	<b>MgO</b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>CaO</b>	<b>V<sub>2</sub>O<sub>5</sub></b>	<b>Cr<sub>2</sub>O<sub>3</sub></b>	<b>MnO</b>	<b>SiO<sub>2</sub></b>
<b>Stirring speed (rpm)</b>	Boiling									
<b>Feed sample no.</b>	PFE2420	<b>%</b>								
<b>Solution volume (L)</b>	0.5	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
<b>Solution description</b>	HCl 20%	<b>Residue</b>								
<b>Solid mass (g)</b>	50.19	<b>%</b>								
<b>LYNN OOND OX</b>	45 min	<b>TiO<sub>2</sub></b>	<b>FeO</b>	<b>MgO</b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>CaO</b>	<b>V<sub>2</sub>O<sub>5</sub></b>	<b>Cr<sub>2</sub>O<sub>3</sub></b>	<b>MnO</b>	<b>SiO<sub>2</sub></b>
<b>Add SnCl<sub>2</sub>.2H<sub>2</sub>O at 0H</b>										
		<b>%</b>								
<b>Final volume (L)</b>	0.35	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
<b>Number of samples</b>	6	53.29	4.36	0.65	0.40	0.19	0.25	0.08	0.68	0.74
<b>Volume of samples (L)</b>	0.025									
<b>Dry residue mass (g)</b>	45.54									
<b>Residue sample no.</b>	2612									
<b>Additives</b>	31.5gSnCl <sub>2</sub> .2H <sub>2</sub> O									

<b>Solution samples</b>		<b>mg/l</b>								
<b>Time(h)</b>	<b>Sample no.</b>	<b>Ti</b>	<b>Fe</b>	<b>Mg</b>	<b>Al</b>	<b>Ca</b>	<b>V</b>	<b>Cr</b>	<b>Mn</b>	<b>Si</b>
0.0	PFE2606	486	1570	68	34	51	14	1.7	73	4.7
0.5	PFE2607	1170	2150	99	53	67	23	3.3	130	14
1.0	PFE2608	1540	2430	116	61	83	28	4.9	164	27
2.0	PFE2609	1930	2680	133	72	104	33	6.3	199	37
4.0	PFE2610	2420	2860	150	84	126	39	7.4	239	47
8.0	PFE2611	3240	3160	178	106	152	49	9.9	290	46
<b>Accountability (%)</b>		0.966	1.063	1.021	0.945	1.635	1.154	1.016	1.044	1.030
<b>Time (h)</b>	<b>Solution based extraction</b>	<b>%</b>								
0.0	PFE2606	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.5	PFE2607	1.3	9.1	4.2	4.0	8.6	3.9	2.1	6.9	1.3
1.0	PFE2608	2.0	9.1	6.6	5.6	17.2	6.1	4.2	11.0	3.2
2.0	PFE2609	2.7	13.5	8.9	7.9	28.4	8.2	6.1	15.3	4.7
4.0	PFE2610	3.6	17.4	11.2	10.5	40.2	10.8	7.5	20.1	6.1
8.0	PFE2611	5.2	24.9	15.0	15.1	54.1	15.1	10.9	26.3	5.9

BTS Experimental log sheet

<b>Date:</b>	99/06/22	<b>Solid feed description:</b> K6-S-35, 710-850µm, ox 1.5 h at 850°C											
<b>Test no.</b>													
<b>Temperature (°C)</b>	105												
<b>Stirring speed (rpm)</b>	Boiling												
<b>Feed sample no.</b>	BTSJ165	%											
<b>Solid mass (g)</b>	31.61	TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	Total		
		82.00	10.57	1.38	0.89	0.48	0.42	0.12	1.35	1.93	99.14		
<b>Residue sample no.</b>	BTSJ173	TiO2	FeO	MgO	Al2O3	CaO	V2O5	Cr2O3	MnO	SiO2	Total		
<b>Dry residue mass (g)</b>	27.99	88.20	6.09	0.85	0.70	0.20	0.41	0.11	0.97	2.18	99.71		
<b>Solution volume (L)</b>	0.5												
<b>Solution description</b>	HCl 20%												
<b>Additives</b>													
<b>Final volume (L)</b>	0.325												
<b>Number of samples</b>	7												
<b>Volume of samples (L)</b>	0.025												
<b>Solution samples</b>		mg/l											
<b>Time(min)</b>	<b>Sample no.</b>	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	Fe(II)	Fe(III)	
0.0		0	0	0	0	0	0	0	0	0	0	0	0
10.0	BTSJ166	143	1080	142	25	112	5.3	1.6	58	22	0	1080	
20.0	BTSJ167	219	1250	146	28	114	6	1.8	71	22	0	1250	
30.0	BTSJ168	310	1380	153	35	117	7	2.1	85	26	0	1380	
60.0	BTSJ169	535	1850	187	51	142	10	3.2	130	29	0	1850	
120.0	BTSJ170	804	2090	197	64	147	13	4.6	172	52	195	1895	
240.0	BTSJ171	1290	2770	254	100	187	20	7.6	261	65	279	2491	
480.0	BTSJ172	1625	3240	301	127	215	25	11	340	75	363	2877	
<b>Accountability (%)</b>		99.4	104.7	104.9	104.6	125.2	100.2	98.0	114.9	111.1			
<b>Time (min)</b>	<b>Solution based extraction</b>	%											
0.0		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
10.0	BTSJ166	0.5	20.8	27.0	8.4	51.6	3.6	3.1	10.7	3.9			
20.0	BTSJ167	0.7	24.1	27.8	9.4	52.6	4.0	3.5	13.1	3.9			
30.0	BTSJ168	1.0	26.6	29.1	11.8	53.9	4.7	4.0	15.7	4.6			
60.0	BTSJ169	1.7	35.6	35.6	17.1	65.5	6.7	6.2	24.1	5.1			
480.0	BTSJ172	5.2	62.4	57.3	42.6	99.1	16.8	21.2	62.9	13.1			
<b>Solids based extraction (%)</b>		4.8	49.0	45.5	30.4	63.1	13.6	18.8	36.4	0.0			



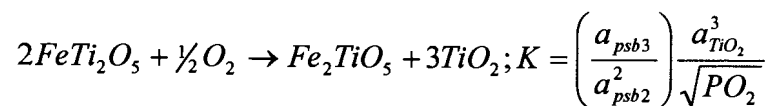
BTS Experimental log sheet

Date:	98/01/25	<b>Solid feed description:</b> LYNN OOND OK 45 min										
Test no.	J6	Leach1: 60 min in SnCl <sub>2</sub>										
Temperature (°C)	105	Leach 2: 8 h in 20% HCl										
Stirring speed (rpm)	Boiling											
Feed sample no.	PFE2420	%										
Solid mass (g)	49.93	TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	Total	
	46.74	88.20	8.18	1.21	0.90	0.26	0.41	0.11	1.30	1.48	102.05	
Residue sample no.	PFE2605	TiO <sub>2</sub>	FeO	MgO	Al <sub>2</sub> O <sub>3</sub>	CaO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	SiO <sub>2</sub>	Total	
Dry residue mass (g)	45.46	89.40	5.65	1.07	0.70	0.27	0.43	0.11	1.07	1.52	100.22	
Solution volume (L)	0.5											
Solution description	HCl 20%											
Additives	31.5gSnCl <sub>2</sub> .2H <sub>2</sub> O											
Final volume (L)	0.4											
	0.35											
Number of samples	4, 6											
Volume of samples (L)	0.025											
<b>Solution samples</b>		mg/l										
Time(m)	Sample no.	Ti	Fe	Mg	Al	Ca	V	Cr	Mn	Si	Fe(II)	Fe(III)
0	-	0	0	0	0	0	0	0	0	0		
10.0	PFE2594	906	1940	84	46	56	19	3.2	107	7.9		
20.0	PFE2595	1110	2150	95	53	63	22	3.5	126	12		
30.0	PFE2596	1240	2180	99	54	66	24	4.1	136	18		
60.0	PFE2597	1660	2560	120	66	86	29	5.4	176	30		
	PFE2599										0	35
90.0	PFE2600	203	137	15	10	26	2.8	0.1	20	4.6	0	137
120.0	PFE2601	328	222	21	18	31	4.6	0.1	34	12	167	55
180.0	PFE2602	487	330	29	28	46	7	1.4	51	19	195	51
300.0	PFE2603	702	424	40	41	62	10	3.2	71	31	279	145
540.0	PFE2604	989	558	55	58	80	14	4	98	38	558	0
<b>Accountability (%)</b>		<b>96.8</b>	<b>109.6</b>	<b>102.8</b>	<b>10.4</b>	<b>175.7</b>	<b>112.7</b>	<b>102.2</b>	<b>105.4</b>	<b>102.1</b>		
Time(m)	Solution based extraction	%										
0	-	0	0	0	0	0	0	0	0	0		
10.0	PFE2594	1.7	30.6	11.5	9.7	30.2	8.3	4.3	13.0	1.1		
20.0	PFE2595	2.1	33.9	13.1	11.1	33.9	9.6	4.7	15.3	1.7		
30.0	PFE2596	2.4	34.3	13.6	11.4	35.6	10.4	5.5	16.6	2.6		
60.0	PFE2597	3.1	40.3	16.5	13.9	46.3	12.6	7.2	21.4	4.3		
90.0	PFE2600	3.5	42.5	18.5	16.0	60.3	13.8	7.3	23.9	5.0		
120.0	PFE2601	3.8	43.8	19.4	17.7	63.0	14.6	7.3	25.6	6.1		
180.0	PFE2602	4.1	45.5	20.5	19.8	71.1	15.7	9.1	27.6	7.1		
300.0	PFE2603	4.5	47.0	22.0	22.5	79.7	17.0	11.4	30.1	8.8		
540.0	PFE2604	5.0	49.1	24.0	26.1	89.4	18.7	12.5	33.3	9.8		

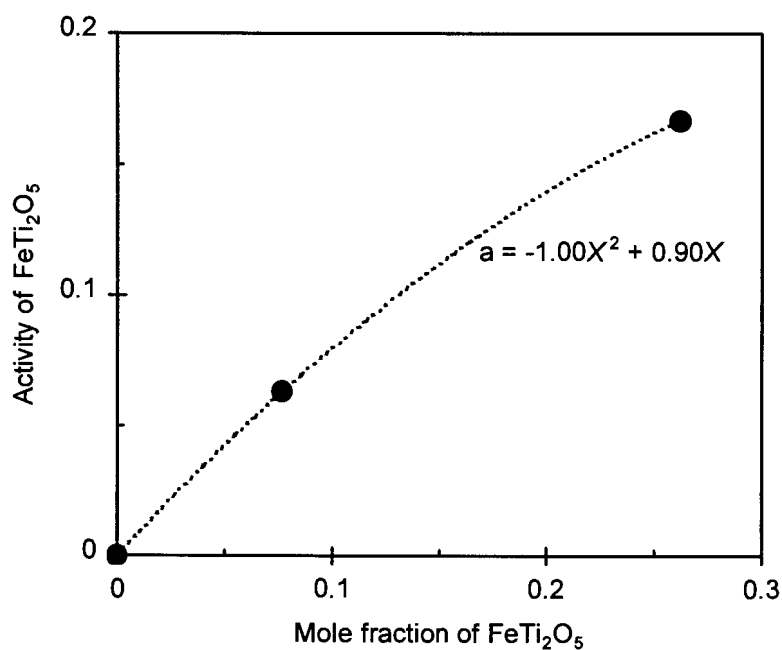
## APPENDIX XVI. Estimation of the oxygen isobars for oxidation and reduction at 850°C

### Oxidation

The phase diagrams indicate that the equilibrium slag composition during oxidation will fall in the TiO<sub>2</sub>-pseudobrookite solid solution phase field, towards the ferric pseudobrookite side. To determine the partial oxygen pressures in this region the activity of ferrous pseudobrookite in the M<sub>3</sub>O<sub>5</sub> phase was estimated by using the data of Webster and Bright (1961) on the positions of tie lines in the TiO<sub>2</sub>-(FeTi<sub>2</sub>O<sub>5</sub>-Fe<sub>2</sub>TiO<sub>5</sub> solid solution) two-phase field at 1200 °C, assuming the activities not to be strongly dependent on temperature. In this phase field, the following reaction is taken to fix the oxygen partial pressure:



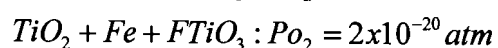
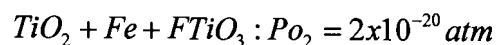
where “psb2” indicates the ferrous pseudobrookite species FeTi<sub>2</sub>O<sub>5</sub> and “psb3” the ferric pseudobrookite species Fe<sub>2</sub>TiO<sub>5</sub>. To estimate the activities close to the Fe<sub>2</sub>TiO<sub>5</sub> composition it was assumed that the activity of Fe<sub>2</sub>TiO<sub>5</sub> in the FeTi<sub>2</sub>O<sub>5</sub>-Fe<sub>2</sub>TiO<sub>5</sub> solid solution follows ideal behaviour. The activity of FeTi<sub>2</sub>O<sub>5</sub> was then calculated from the oxygen isobars given by Webster and Bright (1961) (assuming  $a_{TiO_2} \approx 1$ ), using literature data on the free energy of TiO<sub>2</sub> and FeTi<sub>2</sub>O<sub>5</sub> (Eriksson and Pelton, 1996) and Fe<sub>2</sub>TiO<sub>5</sub> (FACT) (to calculate the equilibrium constant). The results are shown below:



The figure shows that the available data can be presented by a simple parabolic equation. Using this relationship (and still assuming that  $a_{\text{Fe}_2\text{TiO}_5} = X_{\text{Fe}_2\text{TiO}_5}$ ), the  $\text{M}_3\text{O}_5$  composition which would be stable in a gas mixture containing 5%  $\text{O}_2$  was calculated for a temperature of 850 °C. This yielded a result of  $X_{\text{Fe}_2\text{TiO}_5} = 0.997$ , i.e. the equilibrium oxidation product is expected to be essentially pure  $\text{Fe}_2\text{TiO}_5$  and  $\text{TiO}_2$ .

### Reduction

For reduction in pure CO, it was assumed that the partial oxygen pressure was limited by equilibrium with pure carbon, i.e. by the reaction  $\text{CO} = \text{C} + \frac{1}{2}\text{O}_2$ . This reaction yields a partial oxygen pressure (based on the available free energy data, Kubaschewski et al., 1993) of  $1.7 \times 10^{-20}$  atm (for  $P_{\text{CO}} = 0.86$  atm). The phase diagram indicates that possible equilibrium phases under reducing conditions include the  $\text{TiO}_2 + \text{Fe} + \text{FeTiO}_3$  combination, the  $\text{TiO}_2 + \text{Ti}_{20}\text{O}_{39} + \text{Fe}$  combination or combinations of lower-oxygen Magneli phases with metallic iron. Using the available free energy data for the relevant pure phases (Eriksson and Pelton, 1996 and FACT) the equilibrium partial oxygen pressures were calculated as:

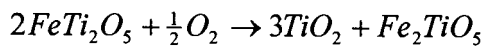


This calculation shows that pure CO can reduce the slag phase in to the rutile-metallic iron-ilmenite phase field, but that further reduction into the  $\text{TiO}_2 + \text{Ti}_{20}\text{O}_{39} + \text{Fe}$  phase field is not possible.

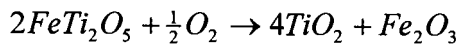


## APPENDIX XVII. Formation of hematite or ferric pseudobrookite during oxidation

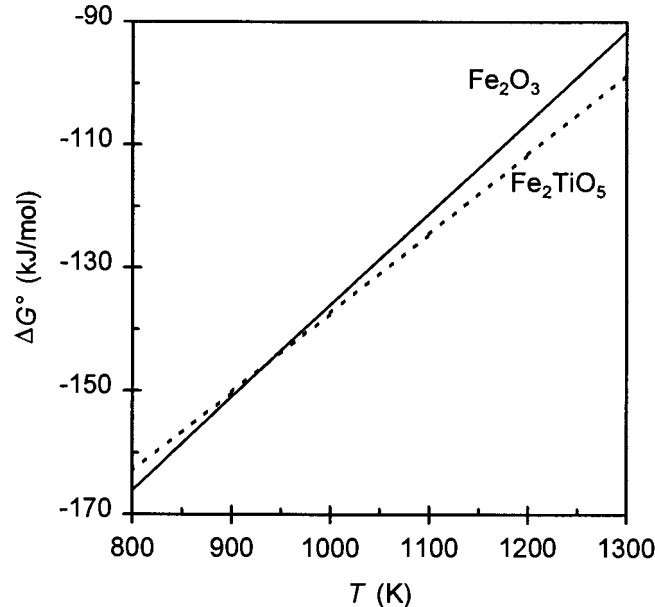
The experimental results show formation of hematite during oxidation at lower temperatures, despite the phase diagrams showing ferric pseudobrookite to be the stable product at the reigning partial oxygen pressure. This effect can be rationalised, by considering the standard free energy changes for two possible oxidation reactions of ferrous pseudobrookite:



and



The standard free energy changes for these two reactions based on the available free energy data (Webster and Bright, 1961, Erick and Pelton, 1996 and FACT) are compared in the figure below, in which the legends “Fe<sub>2</sub>O<sub>3</sub>” and Fe<sub>2</sub>TiO<sub>5</sub>” represent the respective forms in which iron is present after different oxidation reactions.



As the figure shows, the additional decrease in free energy if ferric pseudobrookite rather than hematite is formed, is small at the recommended roasting temperature of 1123 K (850 °C). This difference does increase at the higher temperatures. This thermodynamic effect, possibly together with kinetic

effects related to nucleation of the product phases, is presumed to be responsible for the very different morphology obtained by oxidation at the higher temperature of 1050°C (1323K).

## APPENDIX XVIII: Detailed results from the Mössbauer analysis

Mössbauer hyperfine interaction parameters of selected iron-titanium compounds and iron oxides are listed in the following table:

Phase	Composition	Fe <sup>3+</sup>	Fe <sup>2+</sup>
Ilmenite	FeTiO <sub>3</sub>	$\delta_{\text{Fe}} = 0.27 \text{ mm/s}; \Delta = 0.29 \text{ mm/s}$	$\delta_{\text{Fe}} = 1.04 \text{ mm/s}; \Delta = 0.70 \text{ mm/s}$
Pseudobrookite	Fe <sub>2</sub> TiO <sub>5</sub>	$\delta_{\text{Fe}} = 0.39 \text{ mm/s}; \Delta = 0.70 \text{ mm/s}$	
Ferro-pseudobrookite	FeTi <sub>2</sub> O <sub>5</sub>	$\delta_{\text{Fe}} = 1.05 \text{ mm/s}; \Delta = 3.15 \text{ mm/s}$ $\delta_{\text{Fe}} = 1.02 \text{ mm/s}; \Delta = 1.96 \text{ mm/s}$ $\delta_{\text{Fe}} = 1.17 \text{ mm/s}; \Delta = 2.83 \text{ mm/s}$ $\delta_{\text{Fe}} = 1.05 \text{ mm/s}; \Delta = 3.20 \text{ mm/s}$ $\delta_{\text{Fe}} = 1.02 \text{ mm/s}; \Delta = 2.18 \text{ mm/s}$	
Ulvöspinel	Fe <sub>2</sub> TiO <sub>4</sub>		$\delta_{\text{Fe}} = 1.16 \text{ mm/s}; \Delta = 1.52 \text{ mm/s}$
Armalcolite	Fe <sub>0.5</sub> Mg <sub>0.5</sub> Ti <sub>2</sub> O <sub>5</sub>		$\delta_{\text{Fe}} = 1.05 \text{ mm/s}; \Delta = 3.15 \text{ mm/s}$ $\delta_{\text{Fe}} = 1.03 \text{ mm/s}; \Delta = 2.01 \text{ mm/s}$
Hematite	Fe <sub>2</sub> O <sub>3</sub>	$\delta_{\text{Fe}} = 0.37 \text{ mm/s}; \Delta = -0.20 \text{ mm/s}$	

The samples that were submitted for analysis and the experimental conditions they were exposed to are listed in the following table:

Sample No.	Roast temperature	Oxidation time	Reduction time
PFE3060	850°C	15 min	
PFE3061	850°C	30 min	
PFE3062	850°C	60 min	
PFE3063	850°C	120 min	
PFE3064	850°C	240 min	
PFE3068	850°C	120 min	5 min
PFE3069	850°C	120 min	10 min
PFE3070	850°C	120 min	20 min
PFE3065	1050°C	30 min	
PFE3066	1050°C	60 min	
PFE3067	1050°C	120 min	
PFE3071	1050°C	30 min	20 min
PFE3072	1050°C	60 min	20 min
PFE3073	1050°C	120 min	20 min

\* Leaching was conducted in 20% boiling HCl.

The hyperfine interaction parameters are presented in the following table:

Sample	Hyperfine interaction parameters			% Abundance	Attribution
	$\delta_{Fe}$ (mm/s)	$\Delta$ (mm/s)	$B_{hf}$ (T)		
PFE3060	0.42(3)	-0.24(2)	50.79(2)	26(2)	Fe <sup>3+</sup> , Hematite-like compound
	0.37(3)	0.64(4)		35(3)	Fe <sup>3+</sup> compound (Pseudobrookite)
	1.08(2)	3.01(3)		32(2)	Fe <sup>2+</sup> compound (Ferropseudobrookite)
	1.28(3)	0.75(3)		7(4)	Fe <sup>2+</sup> compound (Ilmenite-like)
PFE3061	0.41(2)	-0.25(2)	50.9(3)	25(2)	Fe <sup>3+</sup> , Hematite-like compound
	0.37(3)	0.69(3)		55(4)	Fe <sup>3+</sup> compound (Pseudobrookite)
	1.08(3)	3.05(5)		15(5)	Fe <sup>2+</sup> compound (Ferropseudobrookite)
	1.16(2)	0.75(3)		5(3)	Fe <sup>2+</sup> compound (Ilmenite-like)
PFE3062	0.38(2)	-0.23(2)	50.0(2)	25(3)	Fe <sup>3+</sup> , Hematite-like compound
	0.38(3)	0.71(3)		68(3)	Fe <sup>3+</sup> compound (Pseudobrookite)
	1.09(2)	3.07(3)		7(3)	Fe <sup>2+</sup> compound (Ferropseudobrookite)
PFE3063	0.36(5)	-0.34(3)	50.8(2)	20(2)	Fe <sup>3+</sup> , Hematite-like compound
	0.38(3)	0.72(2)		77(3)	Fe <sup>3+</sup> compound (Pseudobrookite)
	1.19(3)	3.07(2)		3(2)	Fe <sup>2+</sup> compound (Ferropseudobrookite)
PFE3064	0.38(2)	-0.29(2)	50.8	21(3)	Fe <sup>3+</sup> , Hematite-like compound
	0.38(3)	0.72(3)		79(4)	Fe <sup>3+</sup> compound (Pseudobrookite)
PFE3068	0.30(2)	0.54(3)		26(3)	Fe <sup>3+</sup> Compound (Pseudobrookite)
	1.02(2)	2.75(2)		15(5)	Fe <sup>2+</sup> Compound (Ferropseudobrookite)
	1.10(3)	0.73		59(3)	Fe <sup>2+</sup> compound (Ilmenite-like)
PFE3069	0.30(3)	0.55(2)		12(3)	Fe <sup>3+</sup> compound (Pseudobrookite)
	1.05(2)	2.8(3)		15(5)	Fe <sup>2+</sup> compound (Ferropseudobrookite)
	1.09(2)	0.70(3)		73(5)	Fe <sup>2+</sup> compound (Ilmenite-like)



PFE3070	1.01(2)	2.2(3)		17(5)	Fe <sup>2+</sup> compound (Ferropseudobrookite-like)
	1.08(3)	0.75(3)		62(3)	Fe <sup>2+</sup> , ilmenite-like compound
	0.00(2)	-0.03(3)	33.06(3)	21(5)	Fe metallic
PFE3065	0.38(3)	0.73(4)		84(5)	Fe <sup>3+</sup> compound (Pseudobrookite)
	0.36(2)	-0.31(3)	49.8(4)	16(4)	Fe <sup>3+</sup> compound (Hematite-like)
PFE3066	0.38(2)	0.58(5)		61(5)	Fe <sup>3+</sup> compound (Pseudobrookite-like)
	0.38(3)	0.97(5)		39(3)	
PFE3067	0.39(2)	0.56(3)		59(5)	Fe <sup>3+</sup> compound (Pseudobrookite-like)
	0.38(2)	0.97(3)		41(5)	
PFE3071	0.32(2)	0.59(3)		21(4)	Fe <sup>3+</sup> compound (Pseudobrookite-like)
	1.10(2)	0.75(3)		64(5)	Fe <sup>2+</sup> compound (Ilmenite-like)
	1.06(2)	2.62(3)		15(5)	Fe <sup>2+</sup> compound (Ferropseudobrookite)
PFE3072	0.33(3)	0.61(2)		20(5)	Fe <sup>3+</sup> compound (Pseudobrookite-like)
	1.02(2)	0.74(3)		65(5)	Fe <sup>2+</sup> compound (ilmenite-like)
	0.91(5)	2.35(5)		15(4)	Fe <sup>2+</sup> compound (ferropseudobrookite)
PFE3073	0.31(3)	0.55(4)		21(2)	Fe <sup>3+</sup> compound (Pseudobrookite-like)
	1.10(3)	0.73(3)		69(2)	Fe <sup>2+</sup> compound (ilmenite-like)
	0.97(2)	2.4(5)		10(4)	Fe <sup>2+</sup> :compound (Ferropseudobrookite)

Error is quoted in brackets

$\delta$  is the isomeric (chemical) shift

$\Delta$  is the quadrupole splitting (site asymmetry)

$B_{hf}$  is the internal magnetic field

The Figures on the following pages give the room temperature spectra of the samples:

