

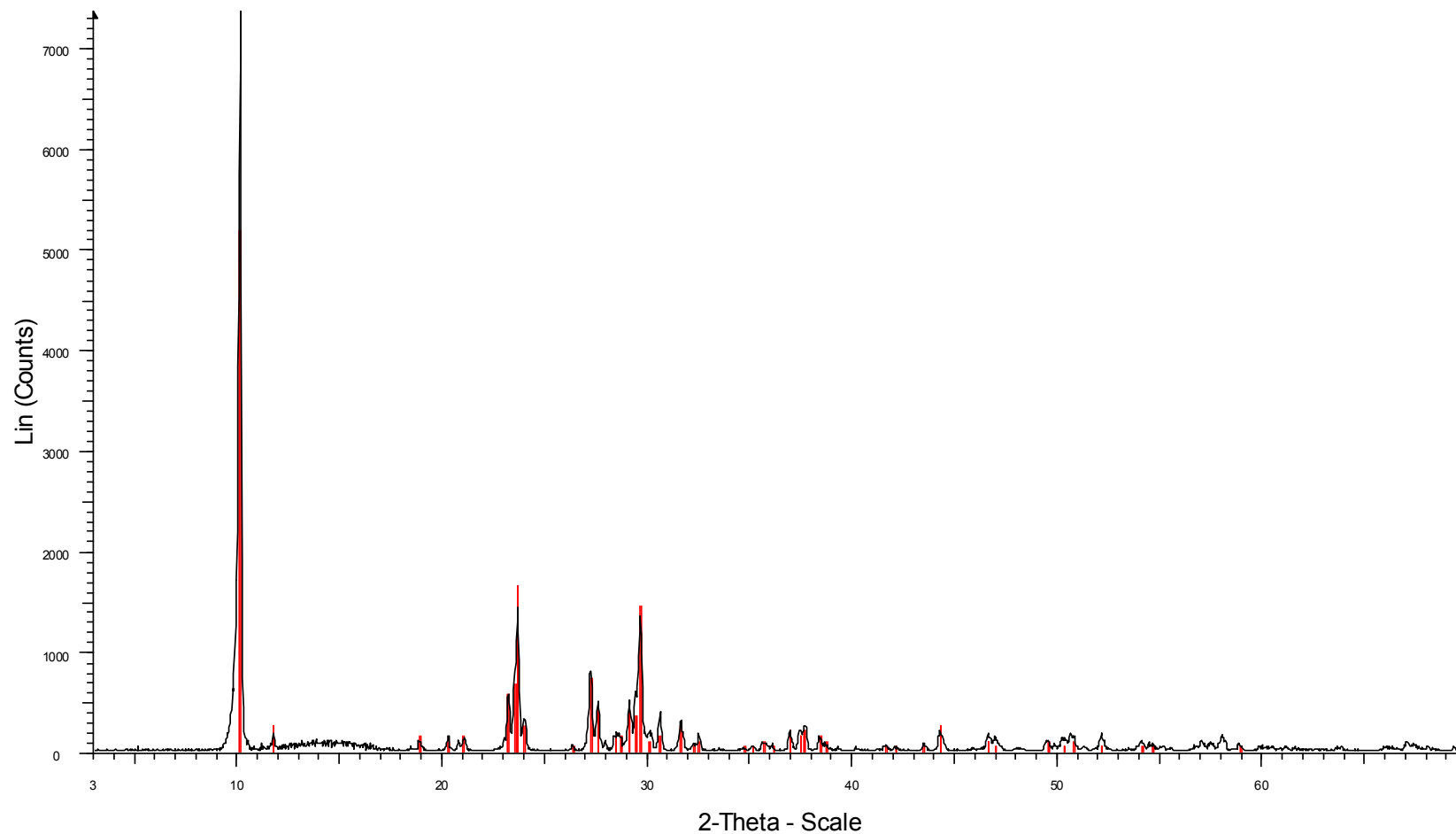
## **APPENDIX A**

### **XRD results for x-compound experiments**

**(See Chapter 3.3.2 page 3-5)**

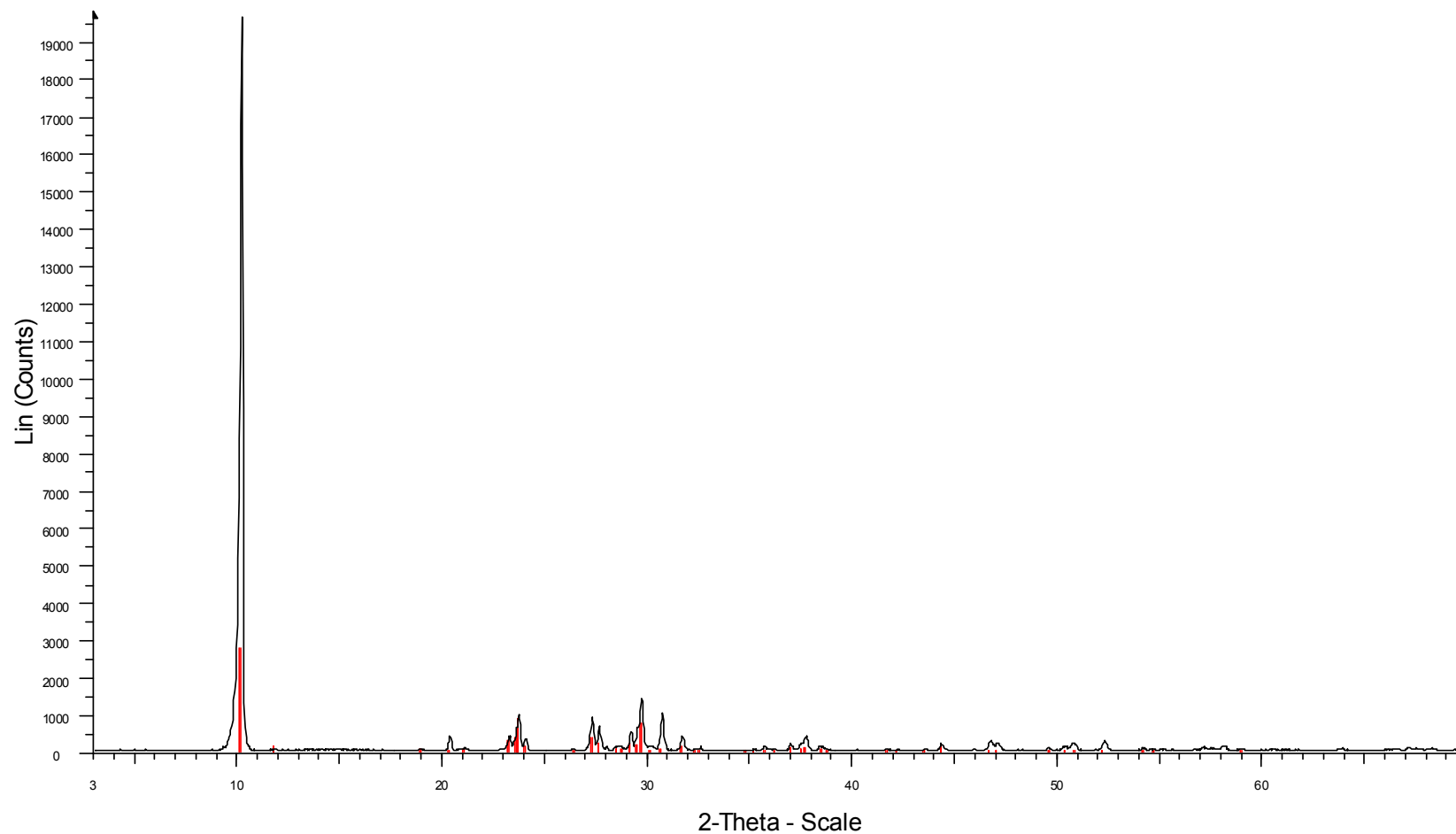
**List of experiments (see Table 2: X-compound experimental conditions and results, page 3-6):**

Experiment 1:	Reference experiment with 30 minute stirring
Experiment 2:	Reference experiment with 24 hour stirring
Experiment 3:	30 minute stirring with additional $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$
Experiment 4:	30 minute stirring with additional x-compound
Experiment 5:	30 minute stirring with additional gypsum
Experiment 6:	30 minute stirring with additional NaOH
Experiment 7:	30 minute stirring with $\text{K}_2\text{SiF}_6$ added as potassium source
Experiment 8:	30 minute stirring with additional $\text{H}_2\text{SiF}_6$



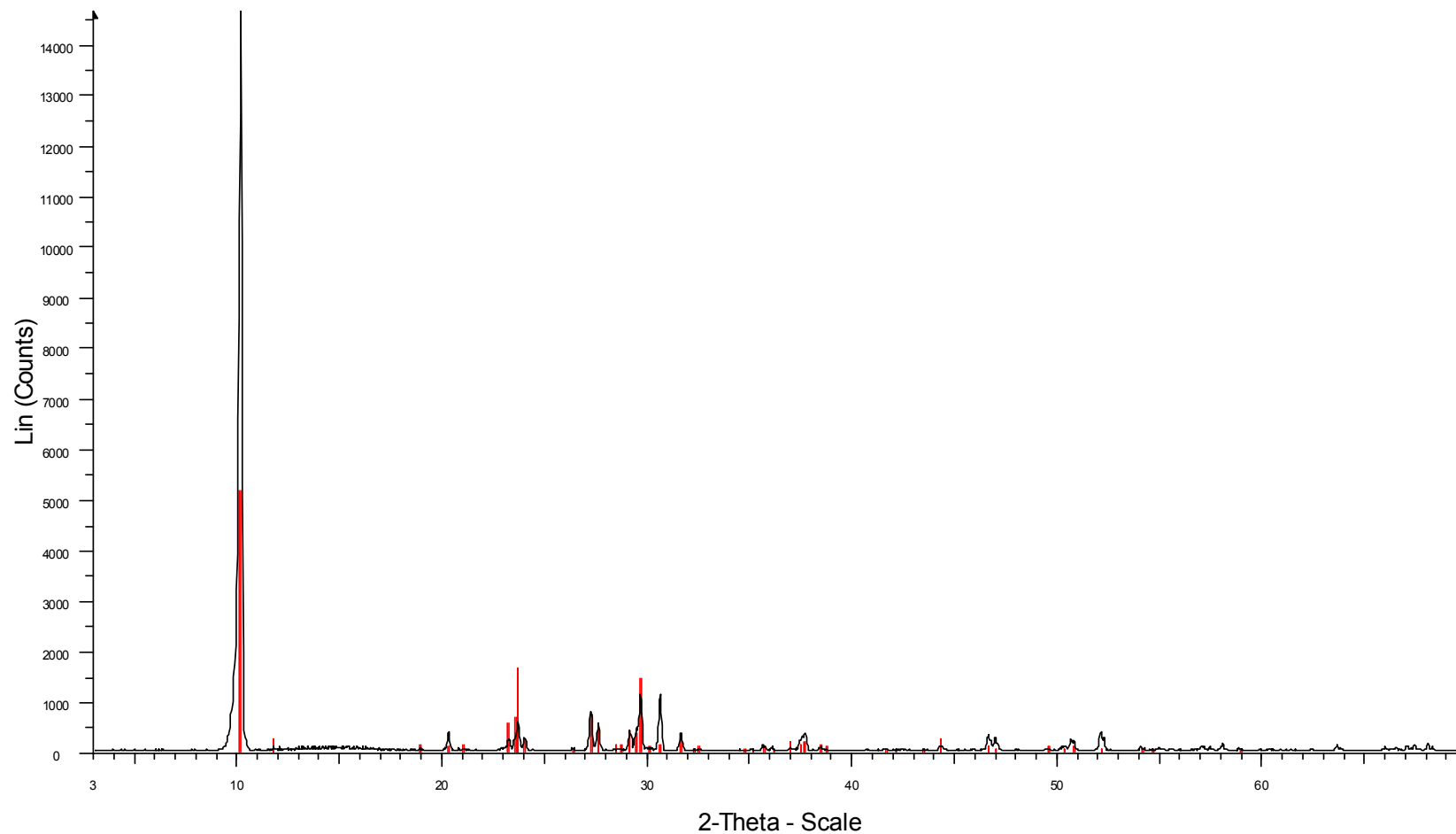
Op1. 5 filtr. na 1 week - File: kruger20026.raw - Type: 2Th/Thlocked - Start: 3.000 ° - End: 69.990 ° - Step: 0.030 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Creation: 6/22/00 2:03:33 PM  
38-1444 (I) - Potassium Iron Hydrogen Phosphate Hydrate - Fe<sub>3</sub>KH<sub>14</sub>(PO<sub>4</sub>)<sub>8</sub>·4H<sub>2</sub>O - Y: 70.48 % - d x by: 0.9979 - WL: 1.5406 - 0 -

### XRD results of x-compound experiment 1



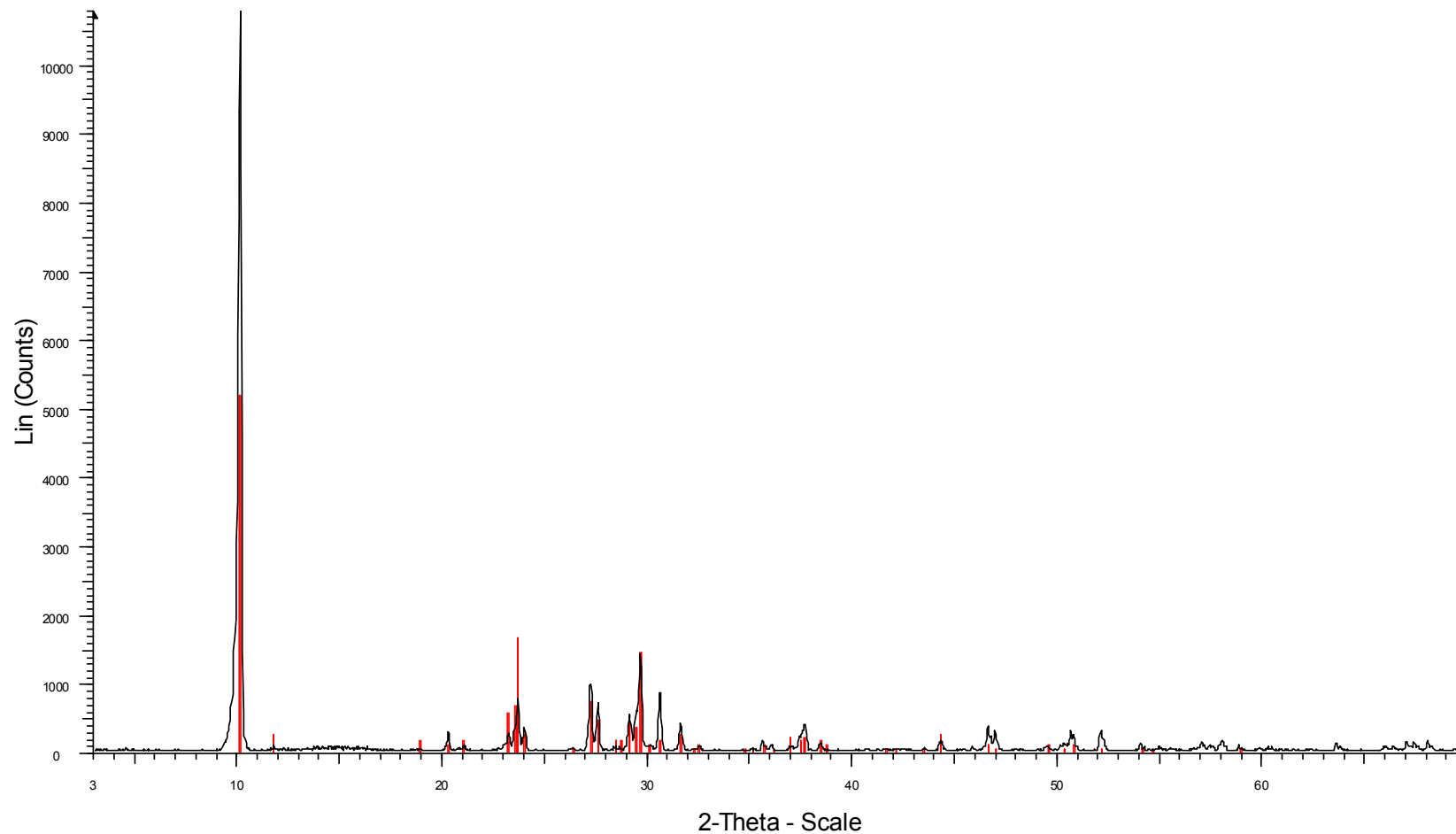
Op1. 6 filtr. na 1 week - File: kruger20035.raw - Type: 2Th/Thlocked - Start: 3.000 ° - End: 69.990 ° - Step: 0.030 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Creation: 6/22/00 1:01:51 PM  
38-1444 (I) - Potassium Iron Hydrogen Phosphate Hydrate - Fe<sub>3</sub>KH<sub>14</sub>(PO<sub>4</sub>)<sub>8</sub>·4H<sub>2</sub>O - Y: 14.30 % - d x by: 0.9979 - WL: 1.5406 - 0 -

### XRD results of x-compound experiment 2



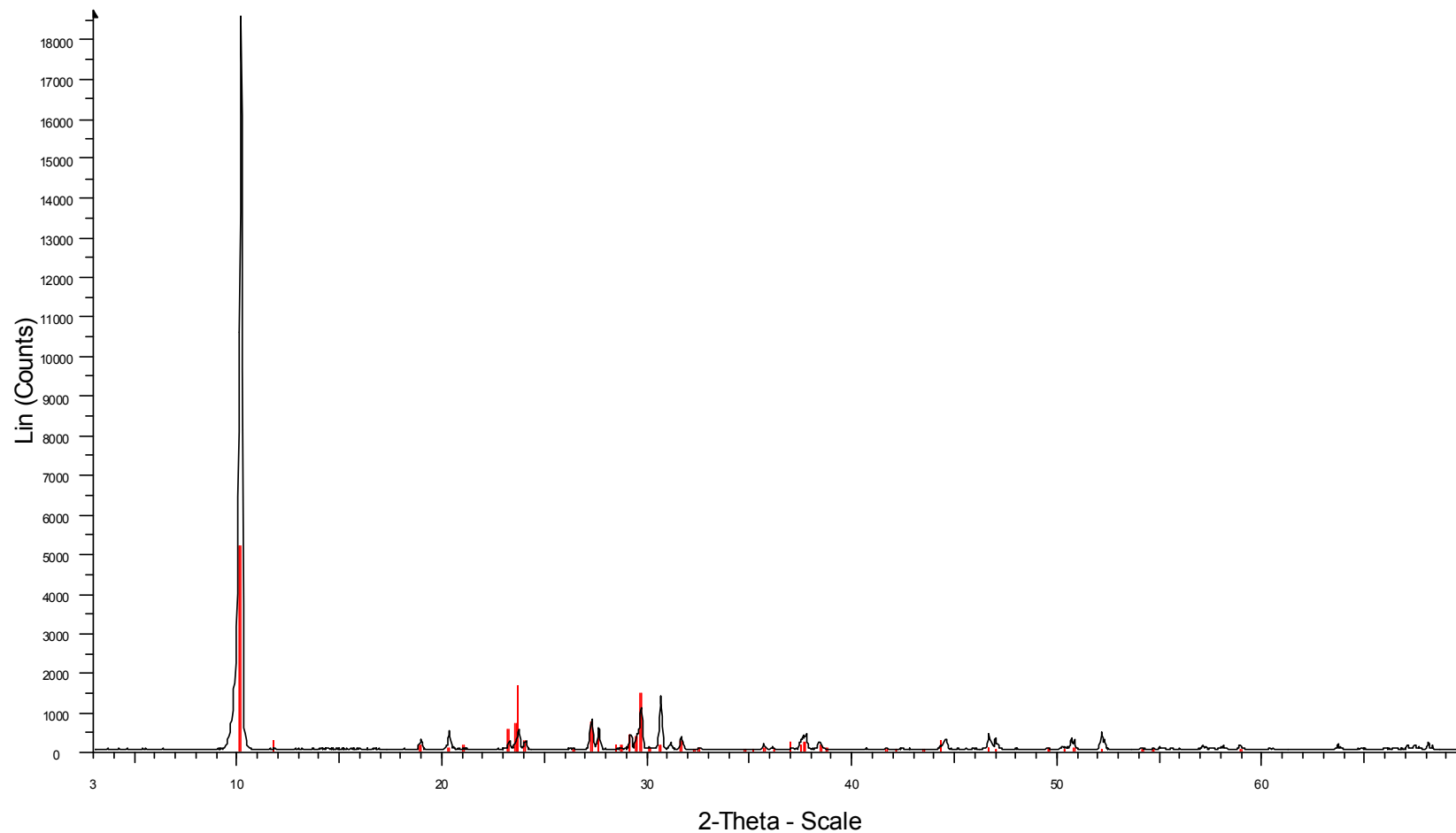
Op1. 7 filtr. na 1 week - File: kruger20029.raw - Type: 2Th/Thlocked - Start: 3.000 ° - End: 69.990 ° - Step: 0.030 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Creation: 6/22/00 5:08:06 PM  
38-1444 (I) - Potassium Iron Hydrogen Phosphate Hydrate - Fe<sub>3</sub>KH<sub>14</sub>(PO<sub>4</sub>)<sub>8</sub>·4H<sub>2</sub>O - Y: 35.44 % - d x by: 0.9979 - WL: 1.5406 - 0 -

### XRD results of x-compound experiment 3



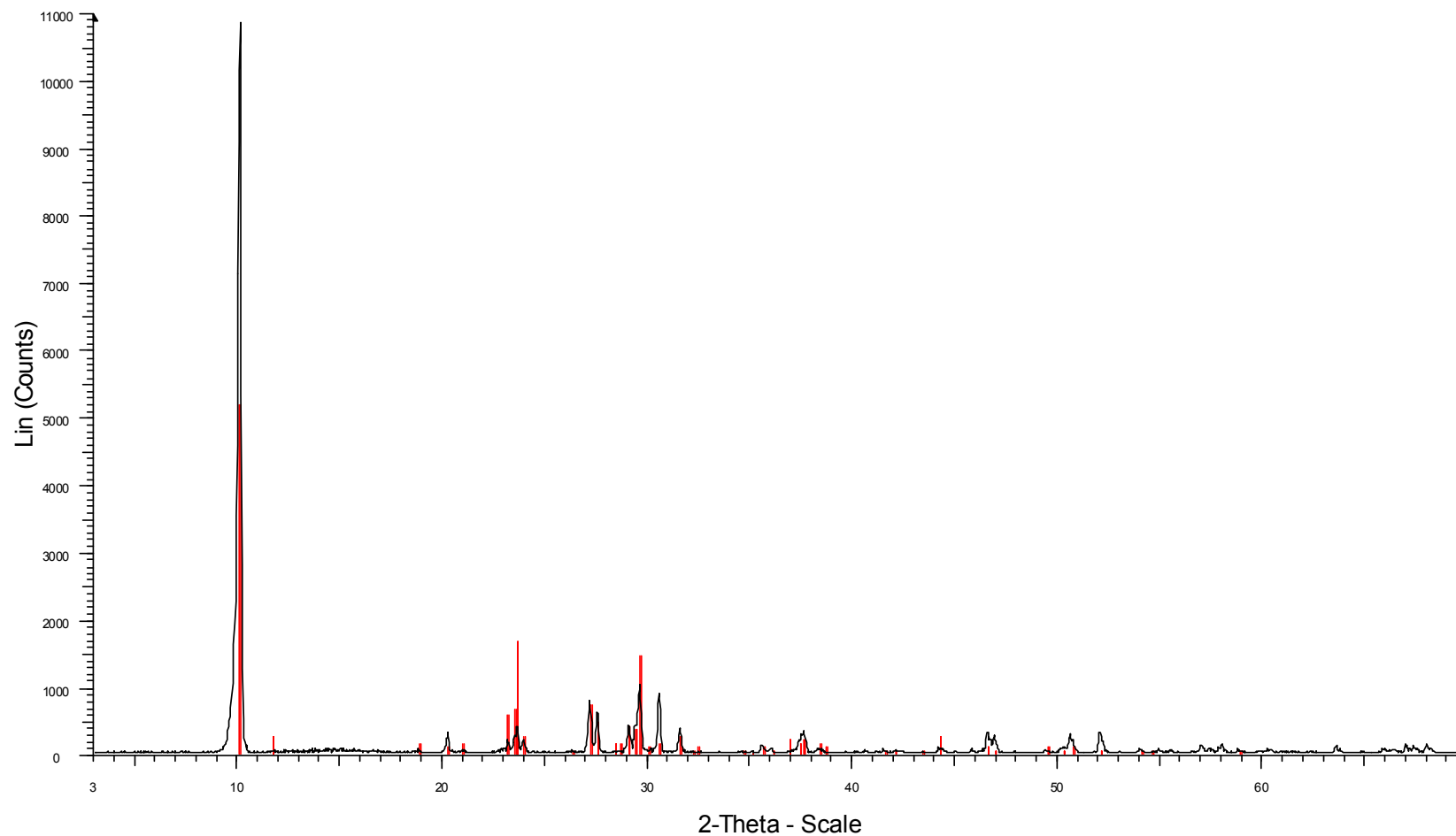
Op1. 8 filtr. na 1 week - File: kruger20028.raw - Type: 2Th/Thlocked - Start: 3.000 ° - End: 69.990 ° - Step: 0.030 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Creation: 6/22/00 4:06:34 PM  
38-1444 (I) - Potassium Iron Hydrogen Phosphate Hydrate - Fe<sub>3</sub>KH<sub>14</sub>(PO<sub>4</sub>)<sub>8</sub>·4H<sub>2</sub>O - Y: 48.17 % - d x by: 0.9979 - WL: 1.5406 - 0 -

### XRD results of x-compound experiment 4



Op1. 9 filtr. na 1 week - File: kruger20031.raw - Type: 2Th/Thlocked - Start: 3.000 ° - End: 69.990 ° - Step: 0.030 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Creation: 6/22/00 7:11:12 PM  
38-1444 (I) - Potassium Iron Hydrogen Phosphate Hydrate - Fe<sub>3</sub>KH<sub>14</sub>(PO<sub>4</sub>)<sub>8</sub>·4H<sub>2</sub>O - Y: 27.93 % - d x by: 0.9979 - WL: 1.5406 - 0 -

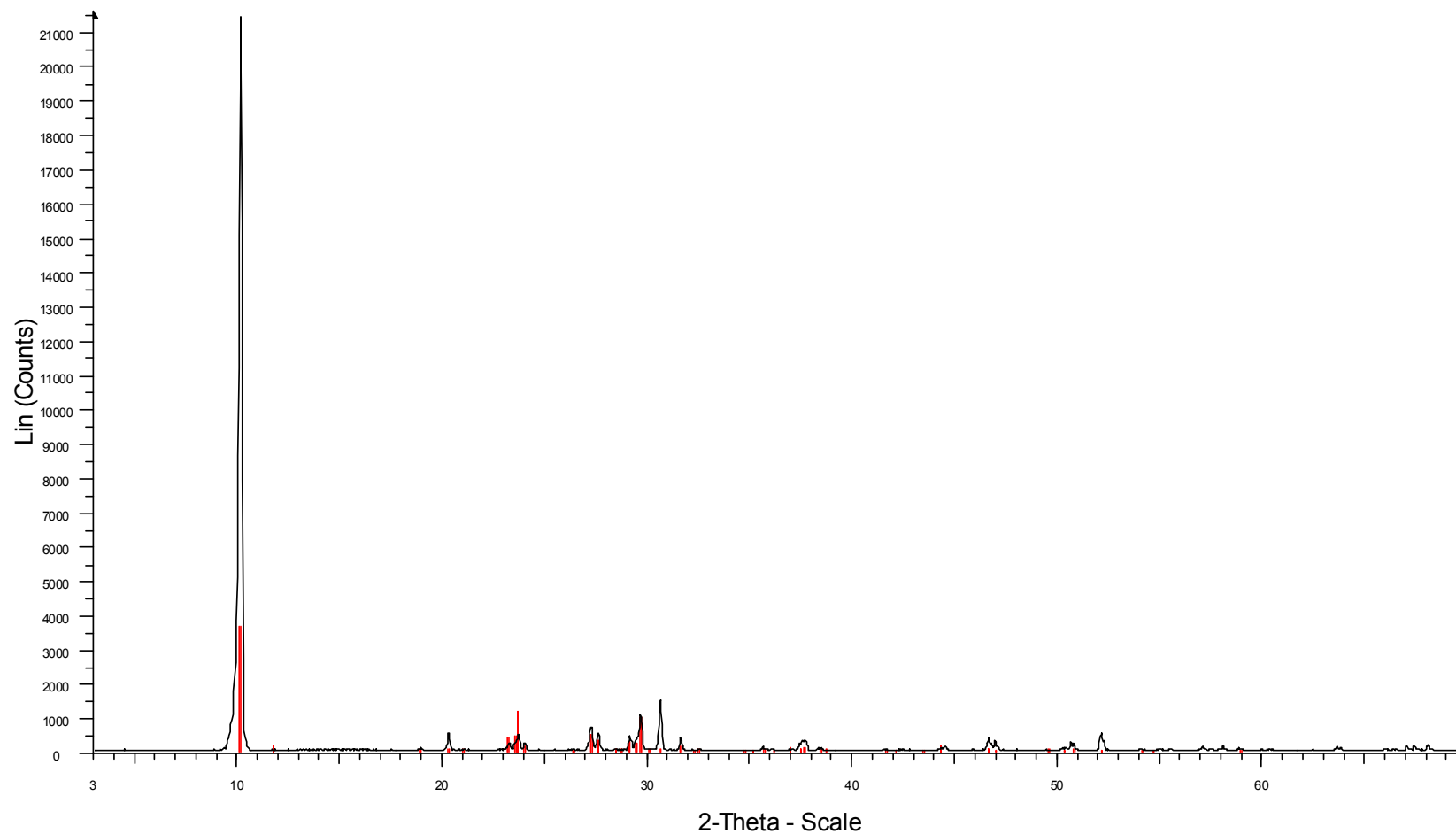
### XRD results of x-compound experiment 5



Op1. 10 filtr. na 1 week - File: kruger20033.raw - Type: 2Th/Th locked - Start: 3.000 ° - End: 69.990 ° - Step: 0.030 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Creation: 6/22/00 9:14:14 PM  
38-1444 (I) - Potassium Iron Hydrogen Phosphate Hydrate -  $\text{Fe}_3\text{KH}_{14}(\text{PO}_4)_8 \cdot 4\text{H}_2\text{O}$  - Y: 47.87 % - d x by: 0.9979 - WL: 1.5406 - 0 -

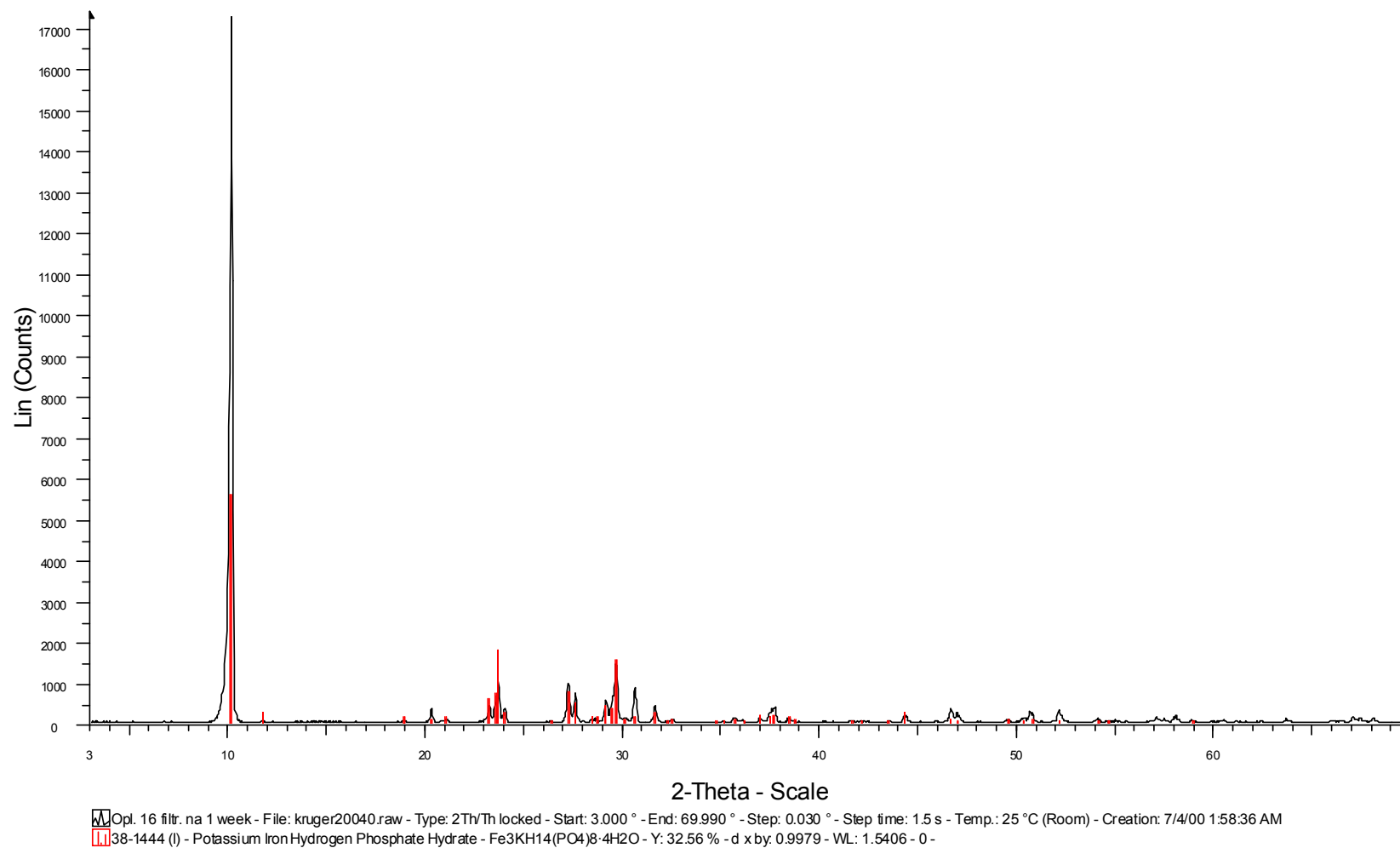
### XRD results of x-compound experiment 6





Op1. 11 filtr. na 1 week - File: kruger20032.raw - Type: 2Th/Th locked - Start: 3.000 ° - End: 69.990 ° - Step: 0.030 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Creation: 6/22/00 8:12:44 PM  
38-1444 (I) - Potassium Iron Hydrogen Phosphate Hydrate - Fe<sub>3</sub>KH<sub>14</sub>(PO<sub>4</sub>)<sub>8</sub>·4H<sub>2</sub>O - Y: 17.13 % - d x by: 0.9979 - WL: 1.5406 - 0 -

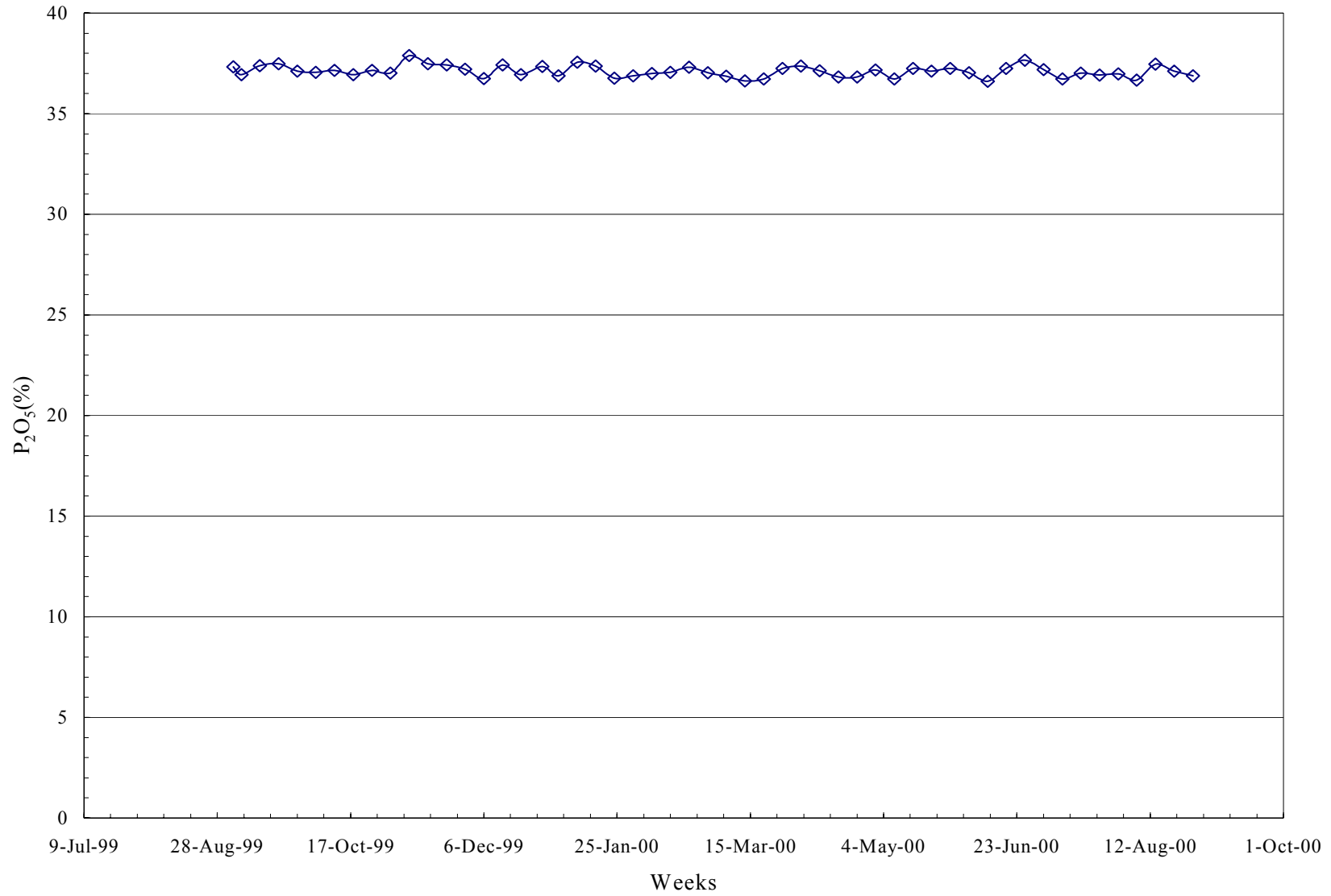
**XRD results of x-compound experiment 7**

**XRD results of x-compound experiment 8**

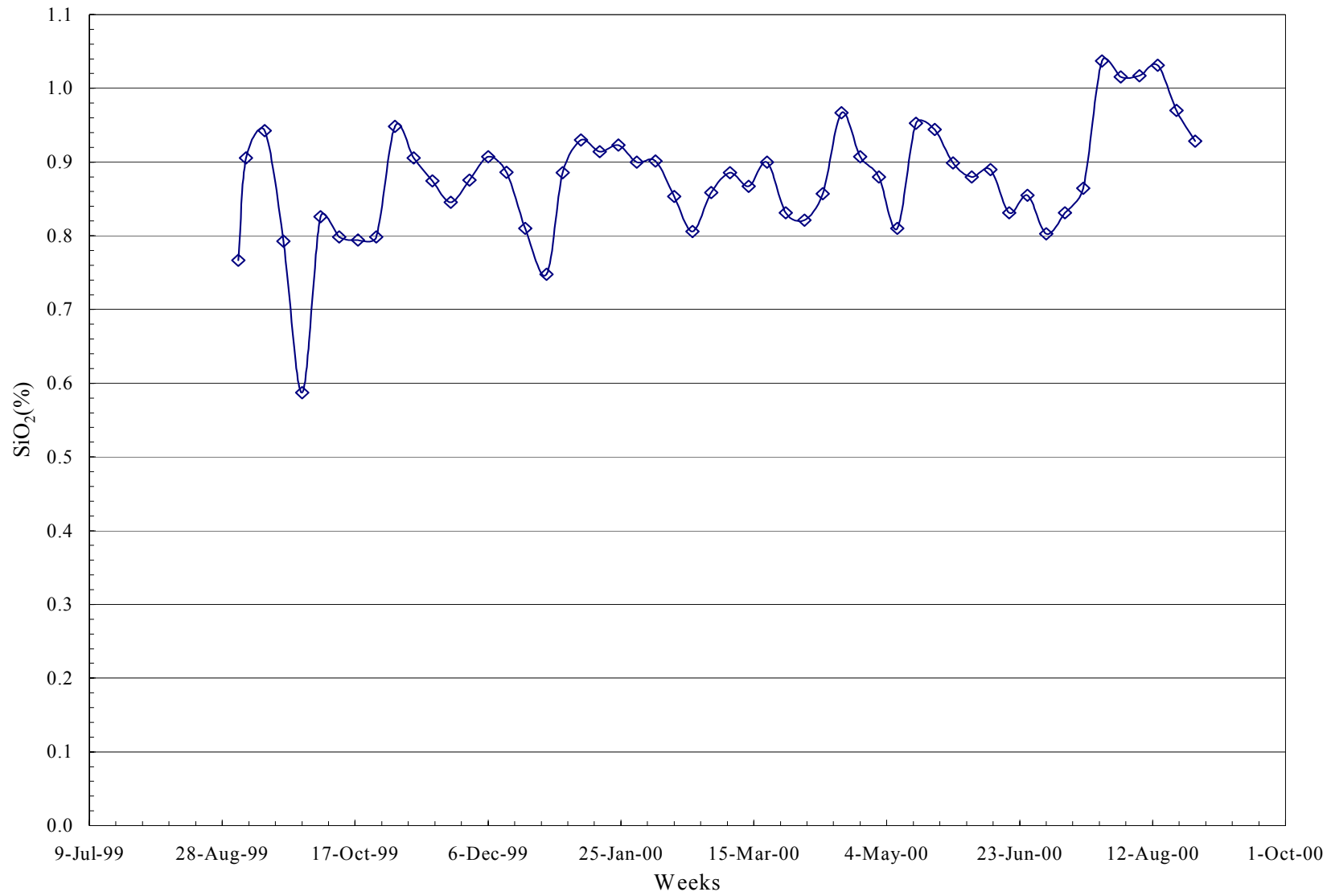
## **APPENDIX B**

### **Composition of phosphate rock monitored over time**

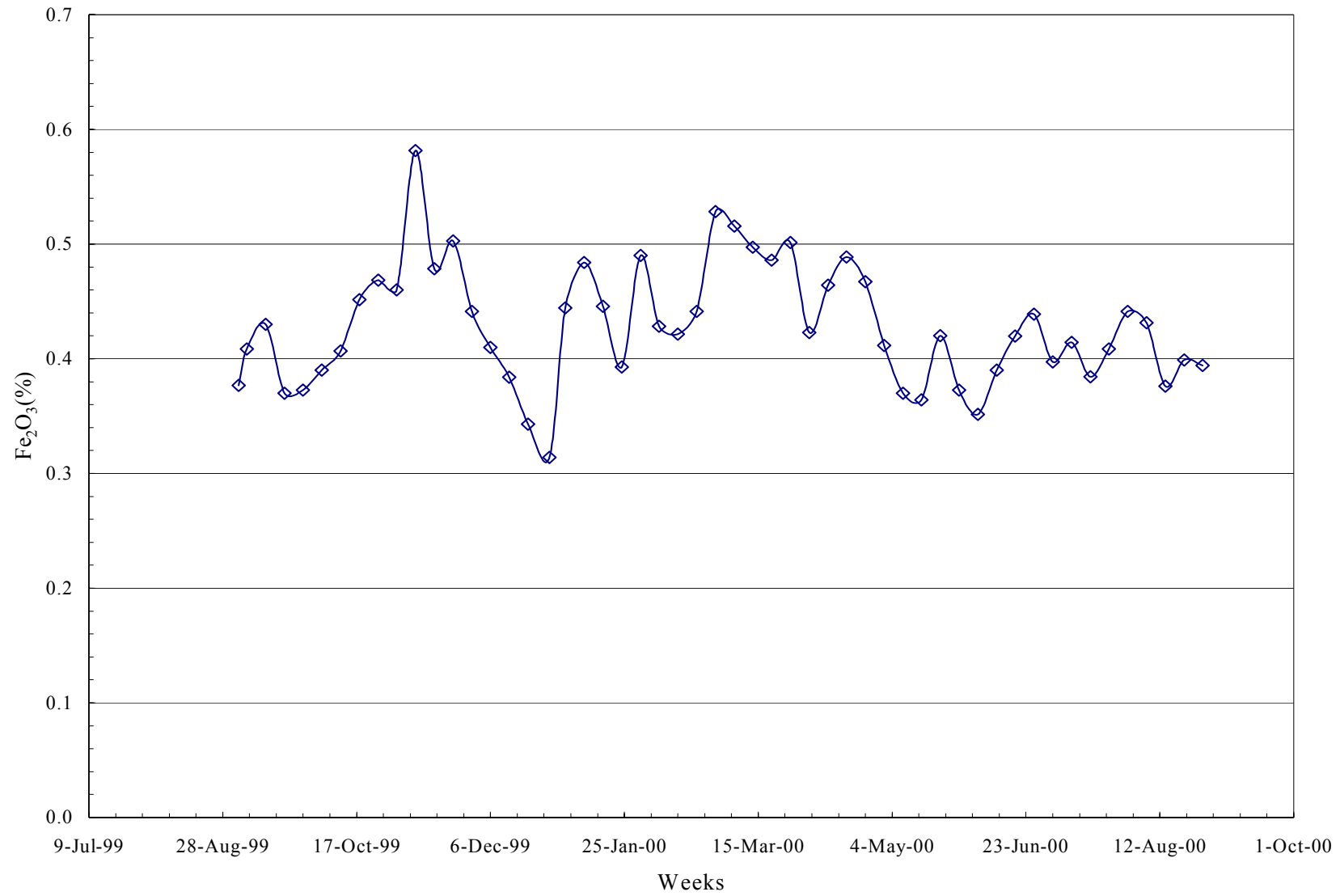
**(See Chapter 3.4.2, p 3-13)**

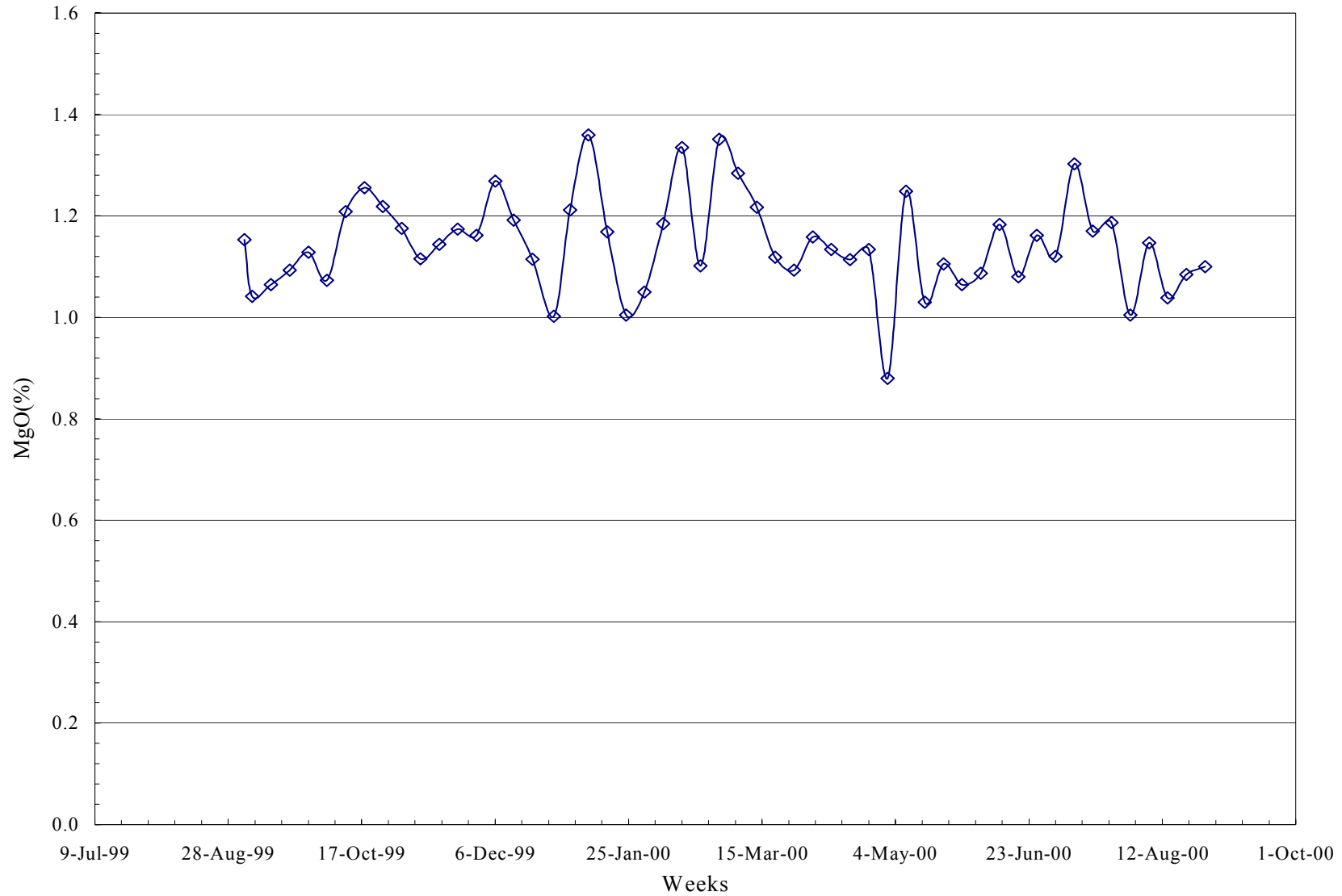


**P<sub>2</sub>O<sub>5</sub> concentration variations in the rock**

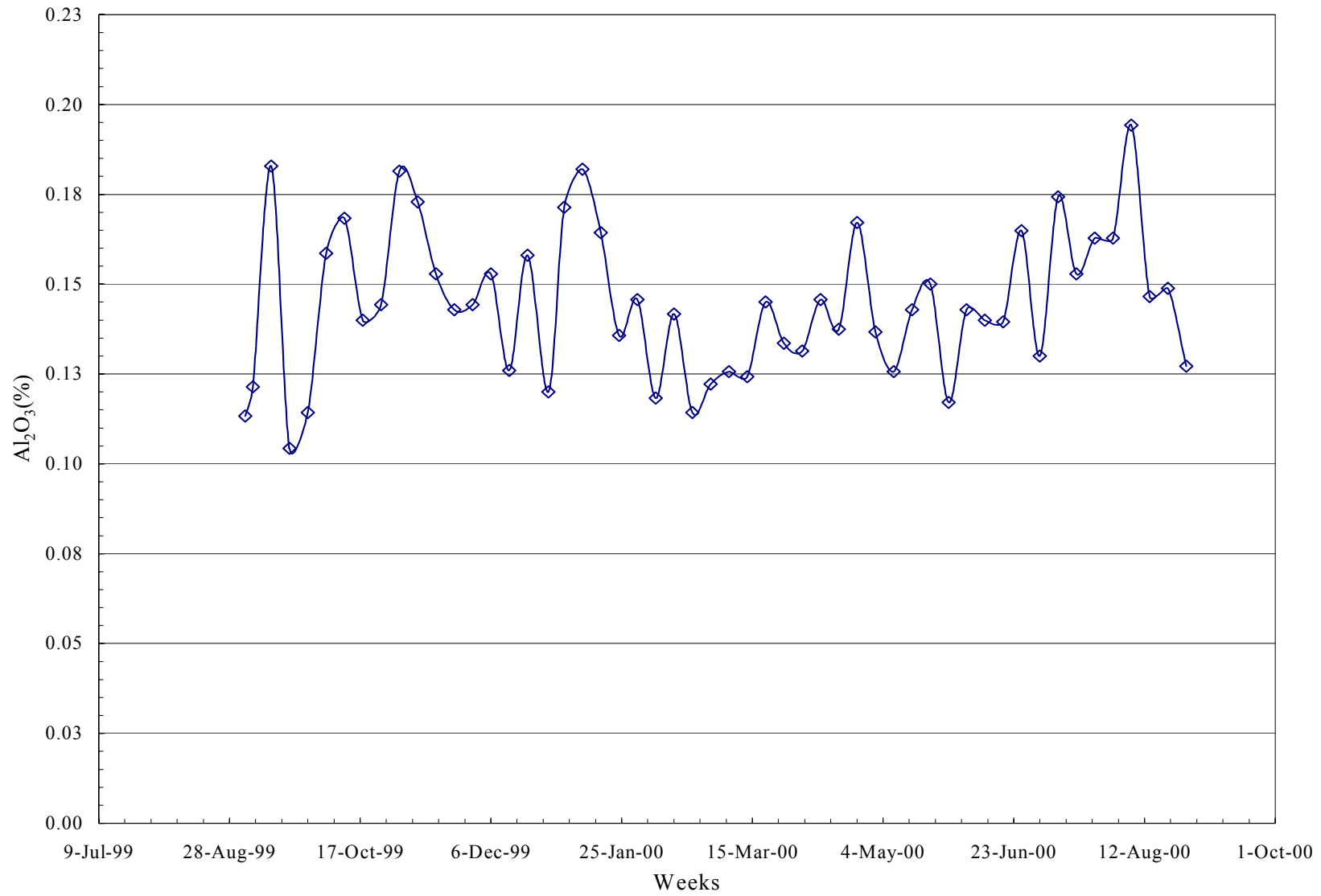


**Silica concentration variations in the rock**

**Iron concentration variations in the rock**

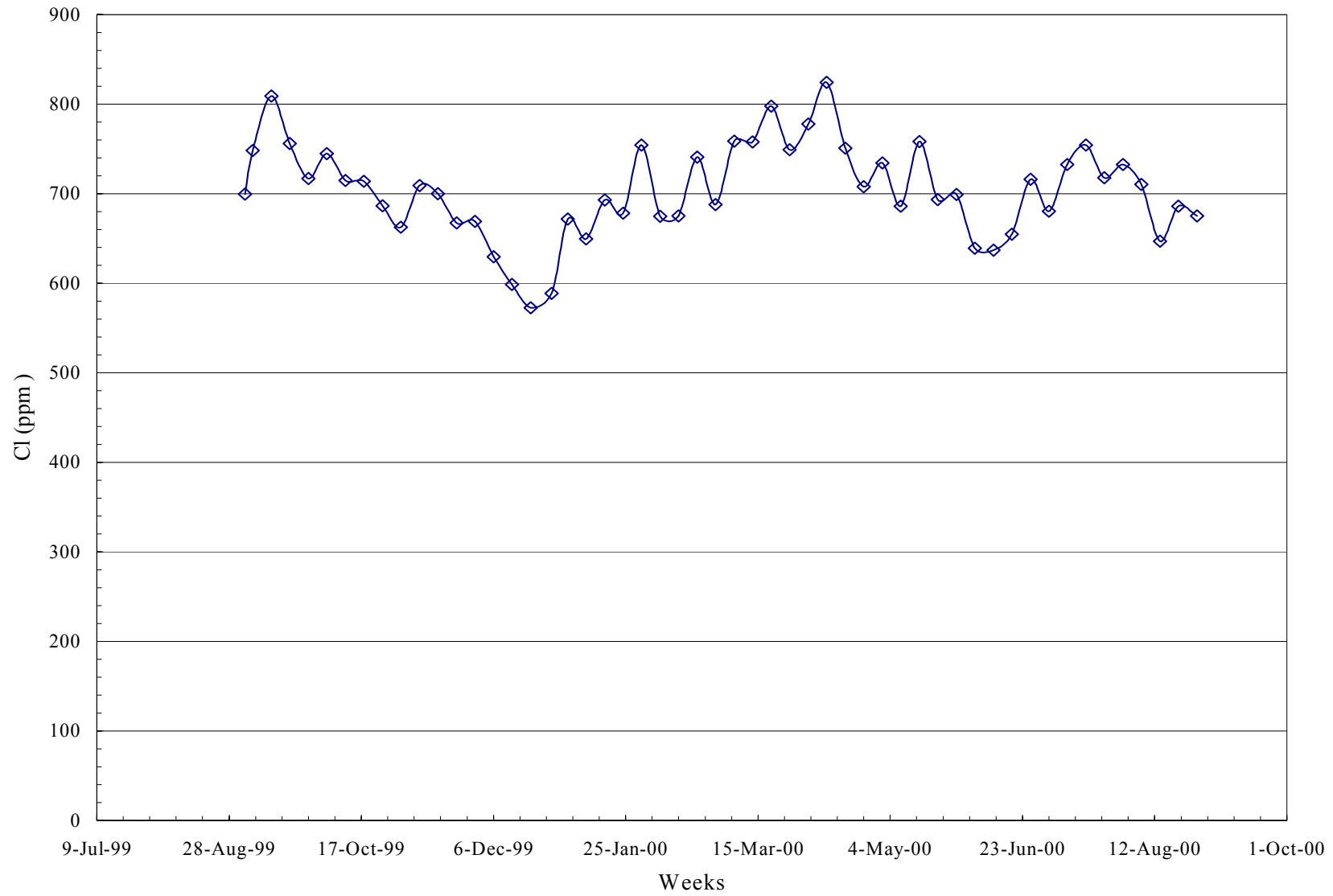


**Magnesium concentration variation in the rock**

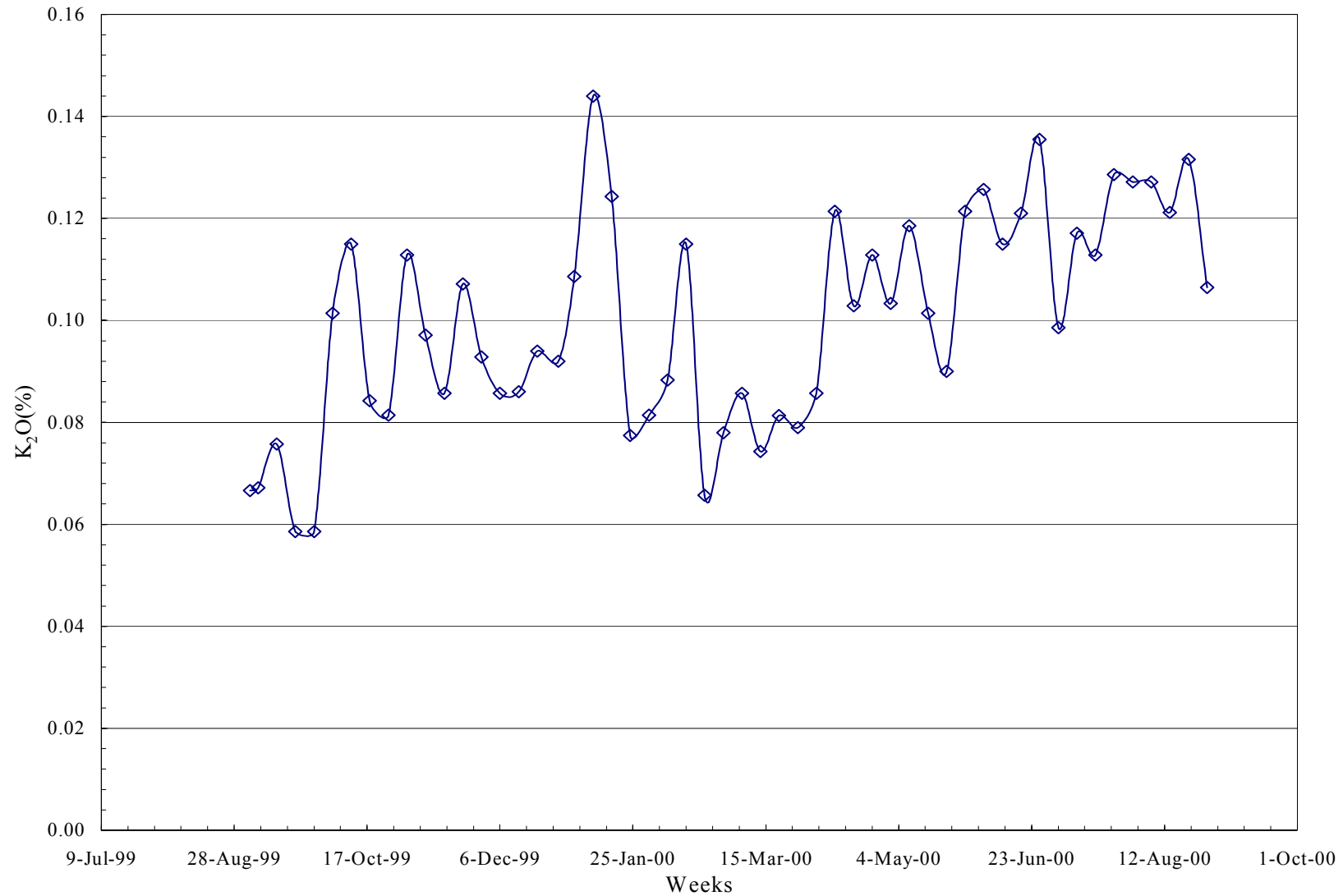


**Aluminium concentration variation in the rock**





**Chlorine concentration variation in the rock**

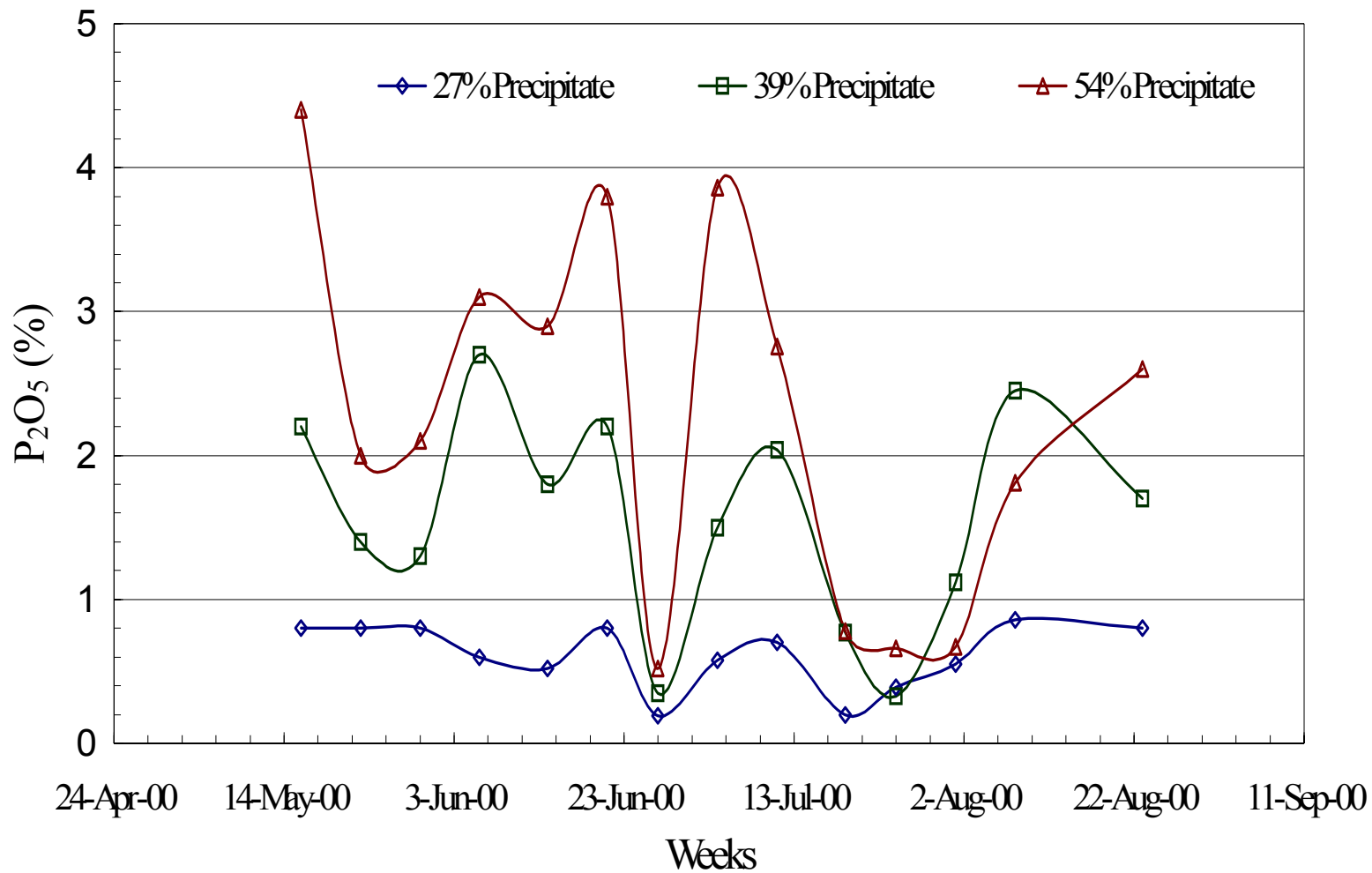


**Potassium concentration variation in the rock**

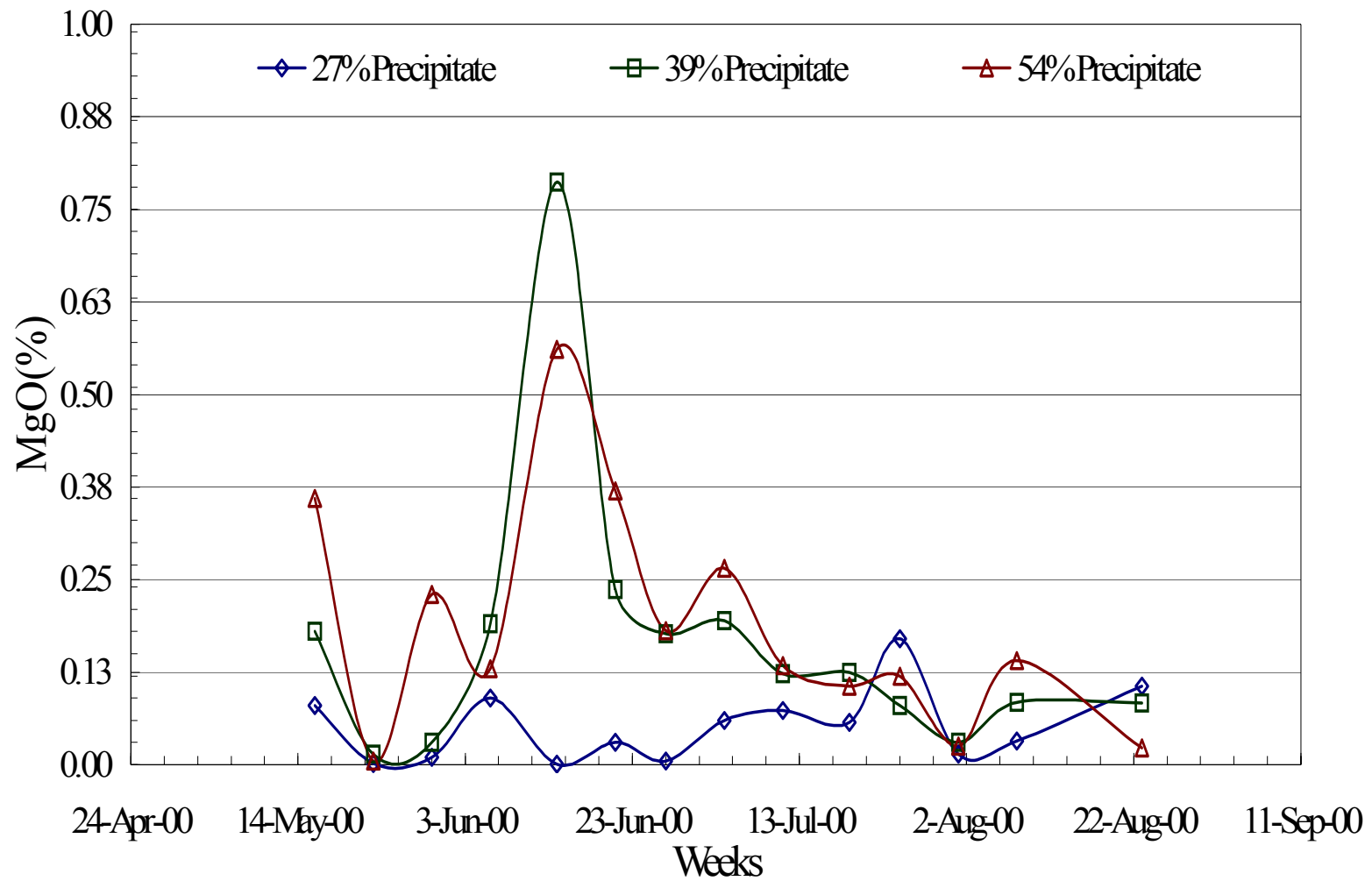
## **APPENDIX C**

**Compositional analyses of precipitate taken from  
27%, 39% and 54% P<sub>2</sub>O<sub>5</sub> acid over time**

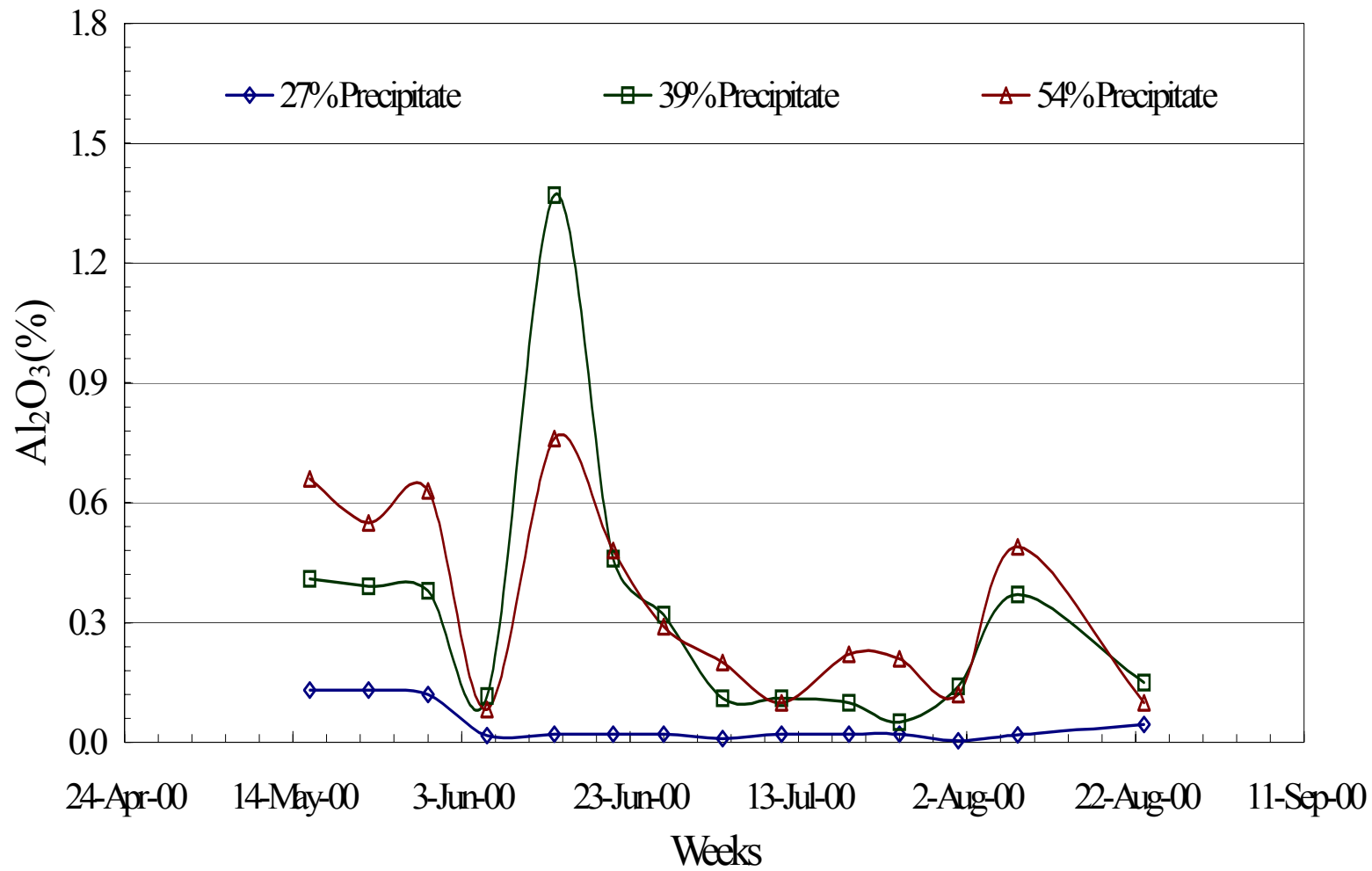
**(See Chapter 3.4.2, p 3-13)**



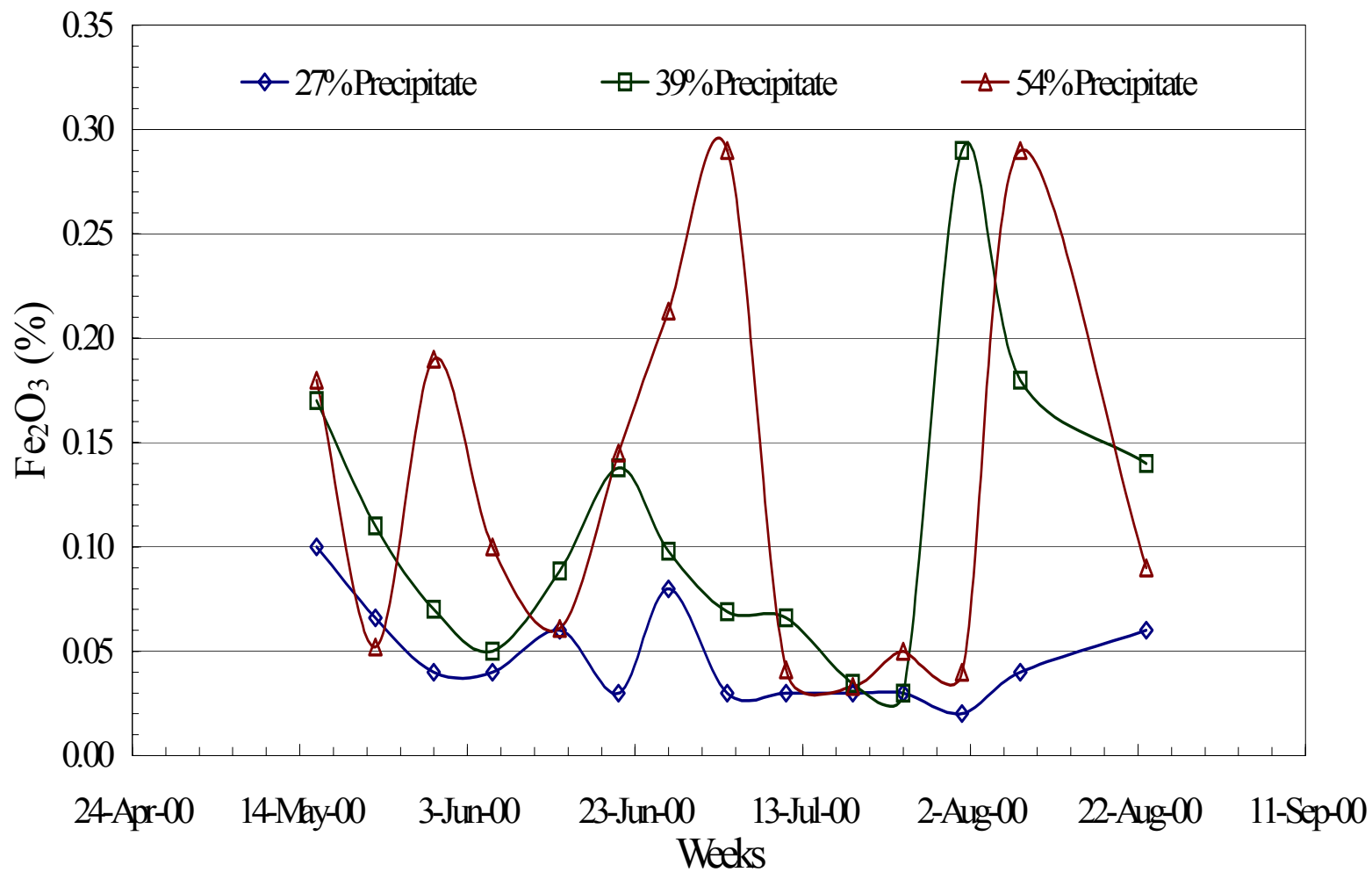
P<sub>2</sub>O<sub>5</sub> content of precipitate taken from different concentration acids



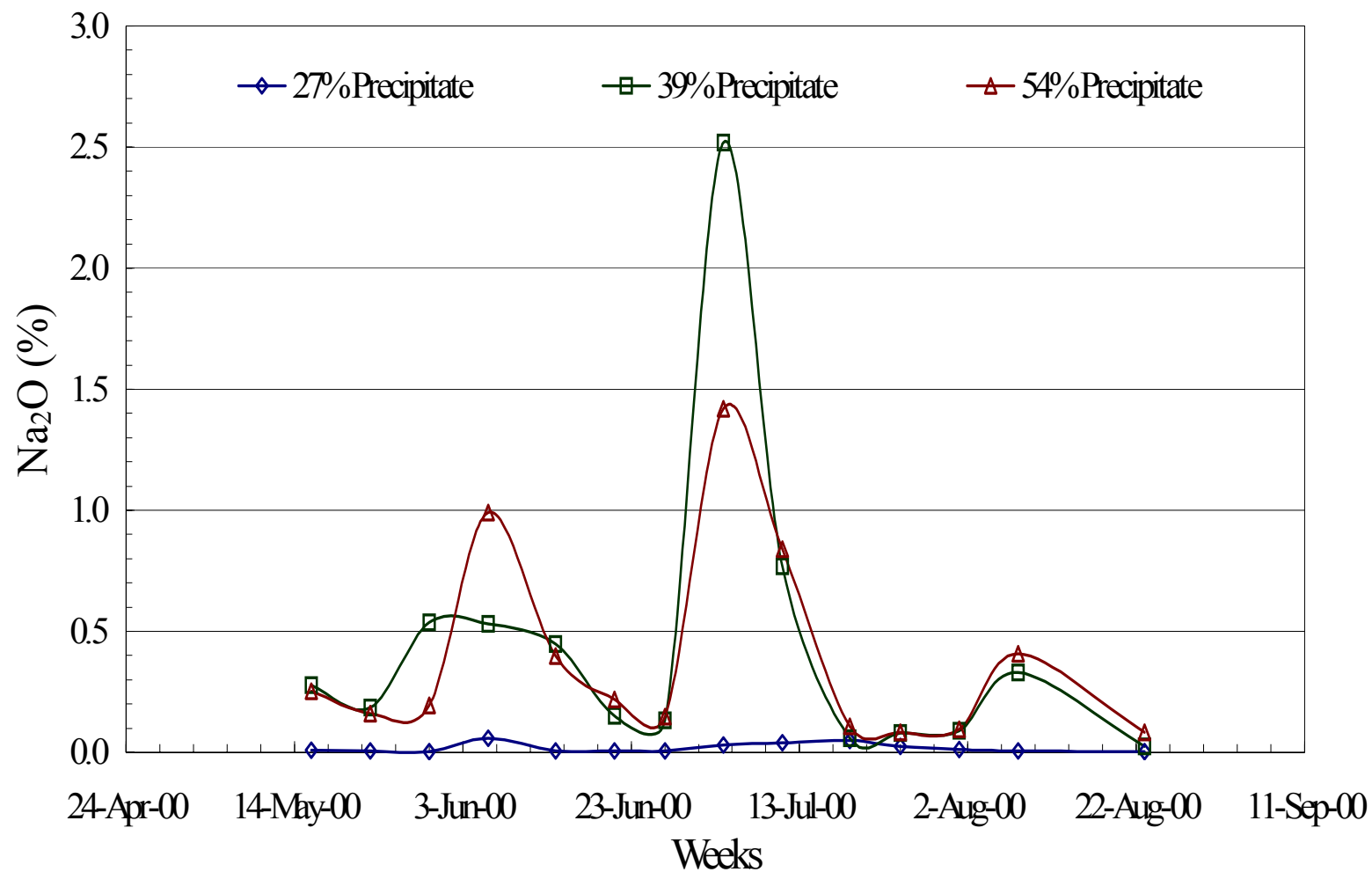
MgO content of precipitate taken from different concentration acids



Al<sub>2</sub>O<sub>3</sub> content of precipitate taken from different concentration acids

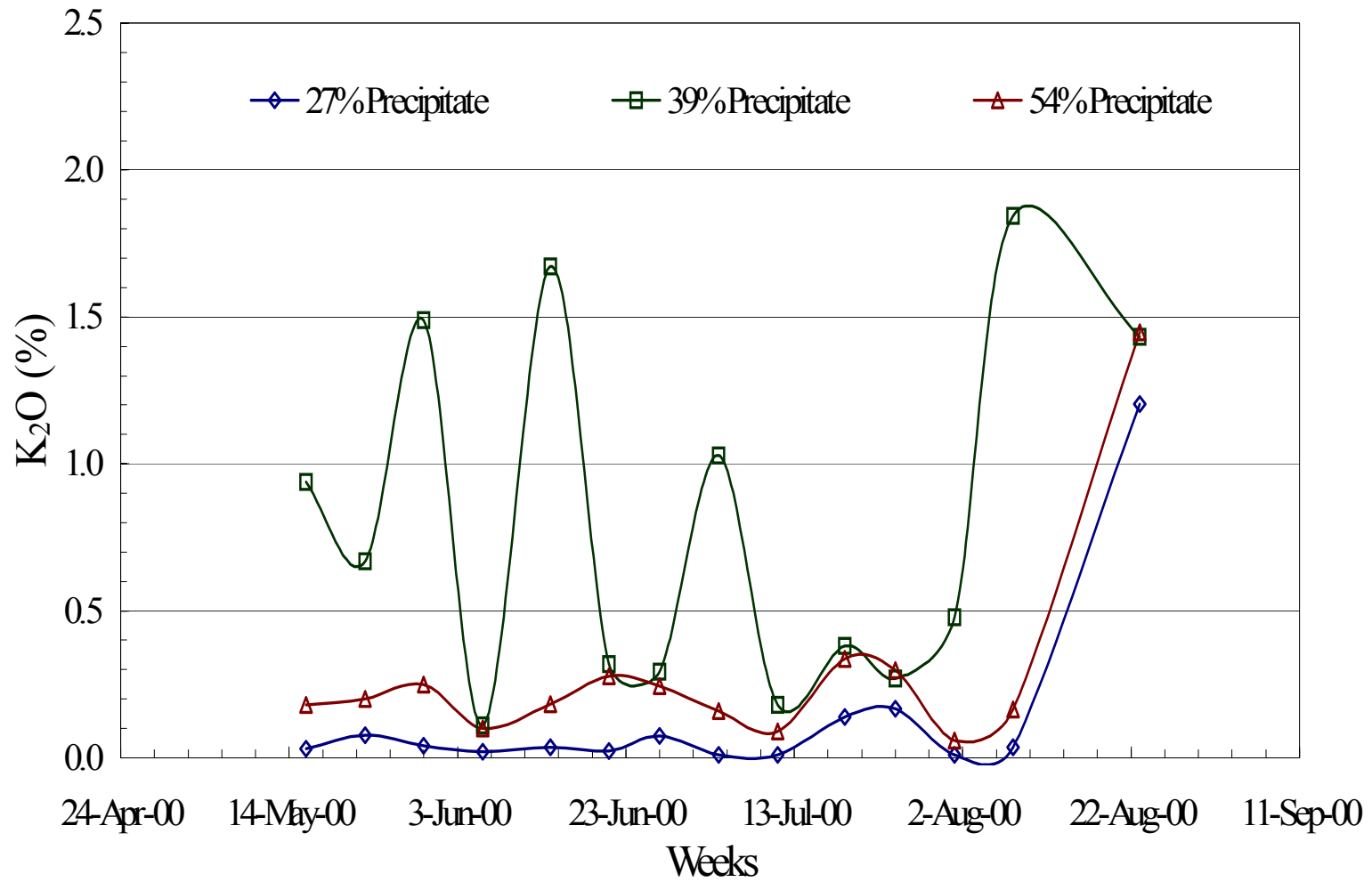


**Fe<sub>2</sub>O<sub>3</sub> content of precipitate taken from different concentration acids**

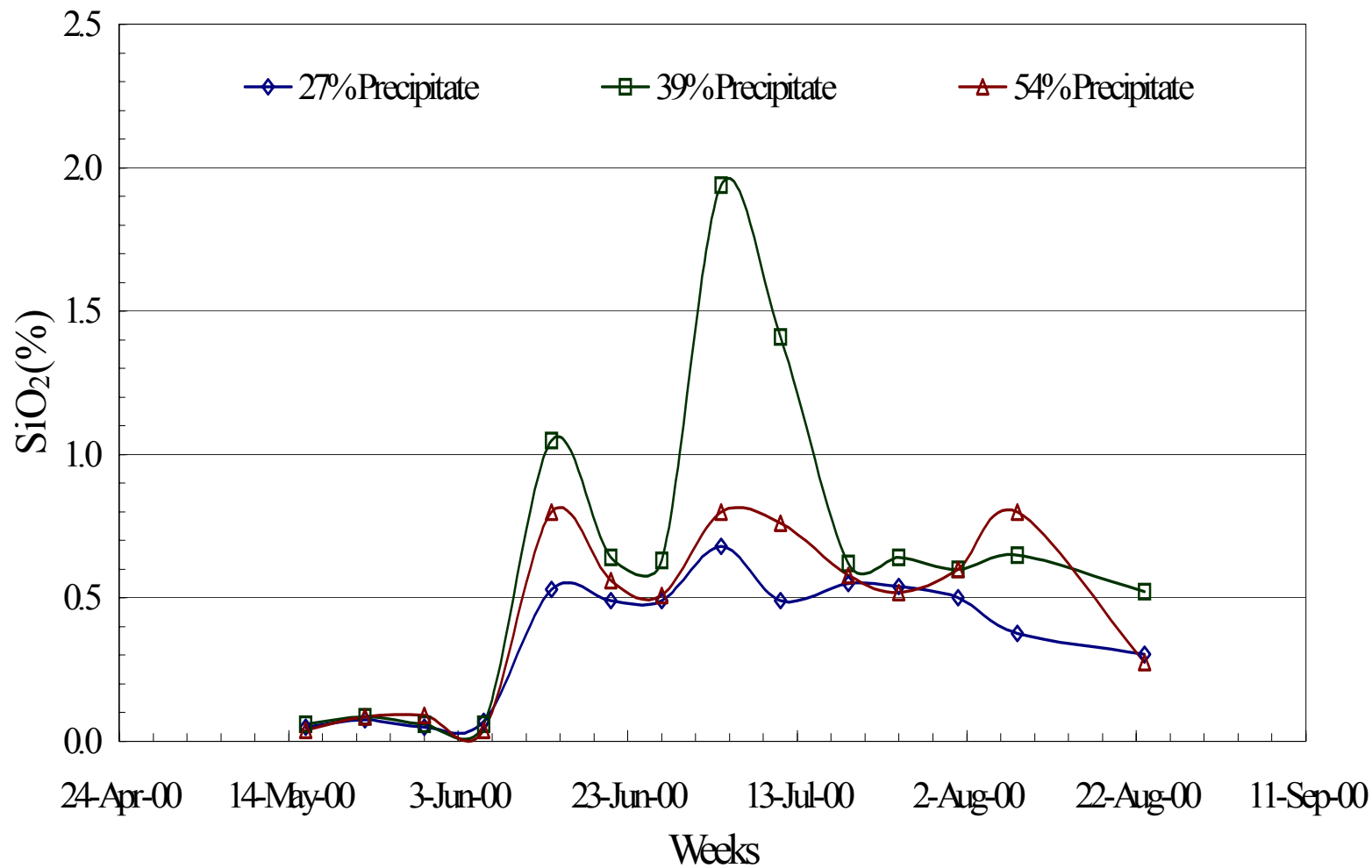


Na<sub>2</sub>O content of precipitate taken from different concentration acids





**K<sub>2</sub>O content of precipitate taken from different concentration acids**

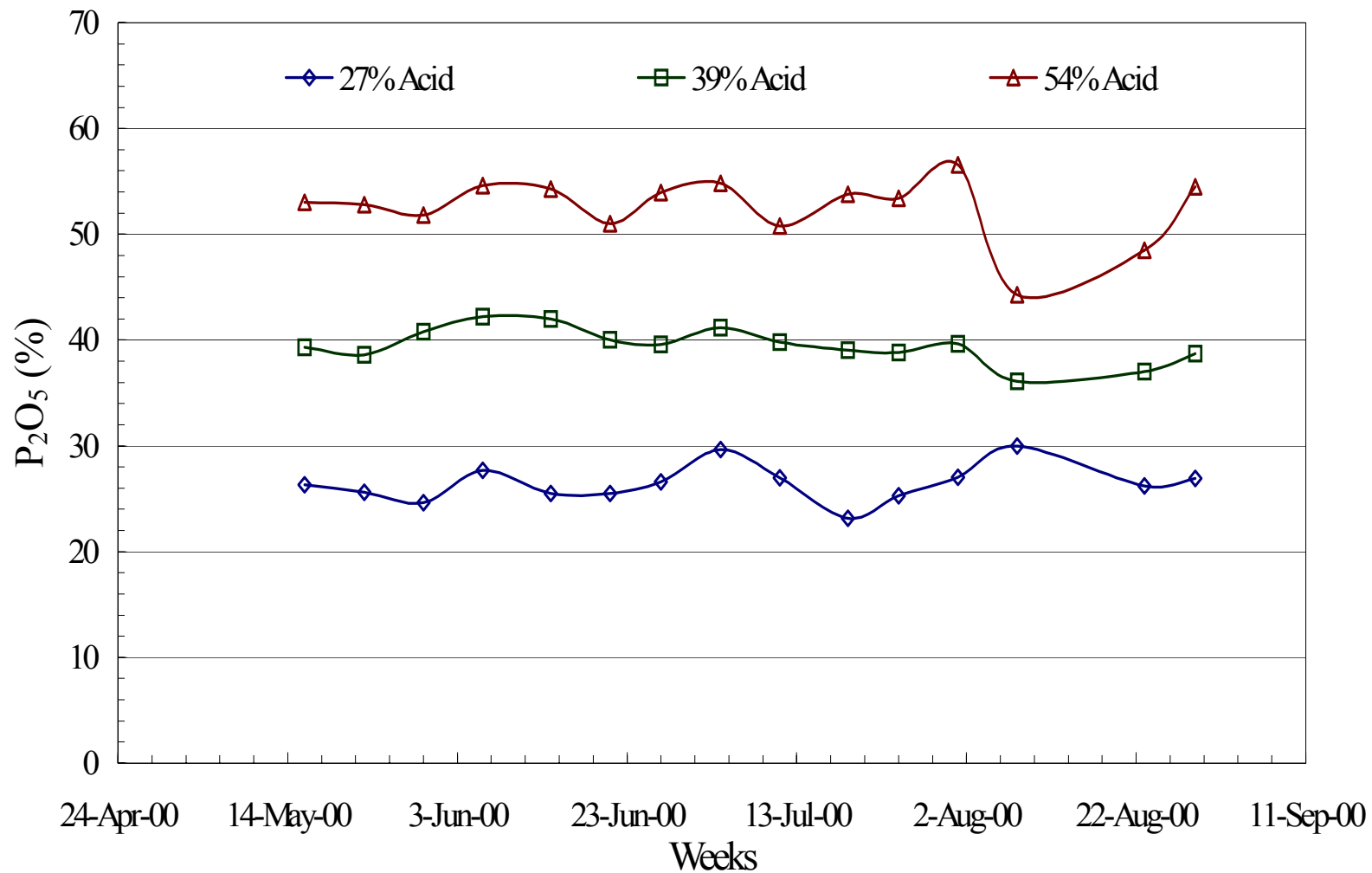


SiO<sub>2</sub> content of precipitate taken from different concentration acids

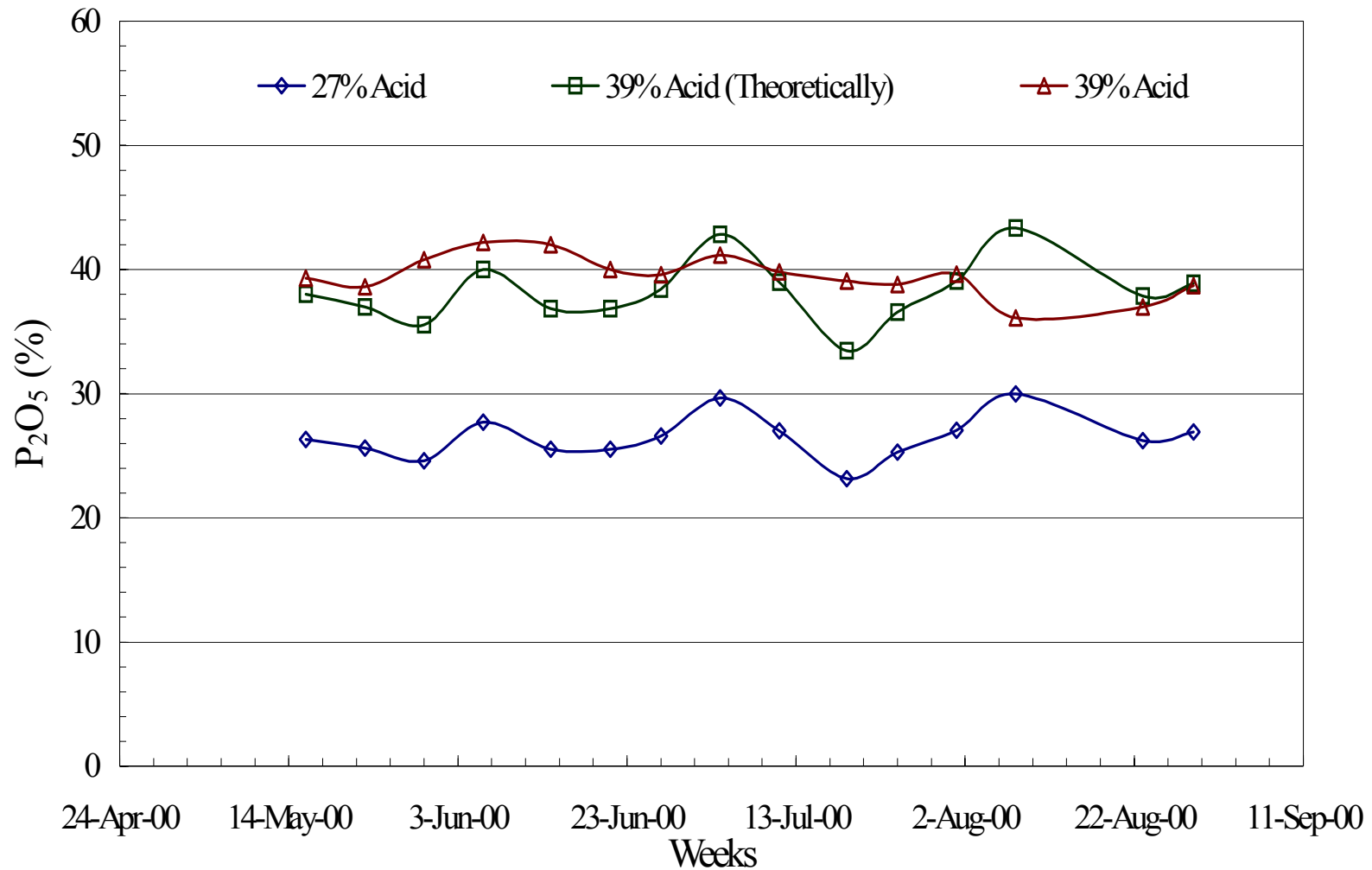
## **APPENDIX D**

### **Compositional analyses of 27%, 39% and 54% P<sub>2</sub>O<sub>5</sub> acid over time**

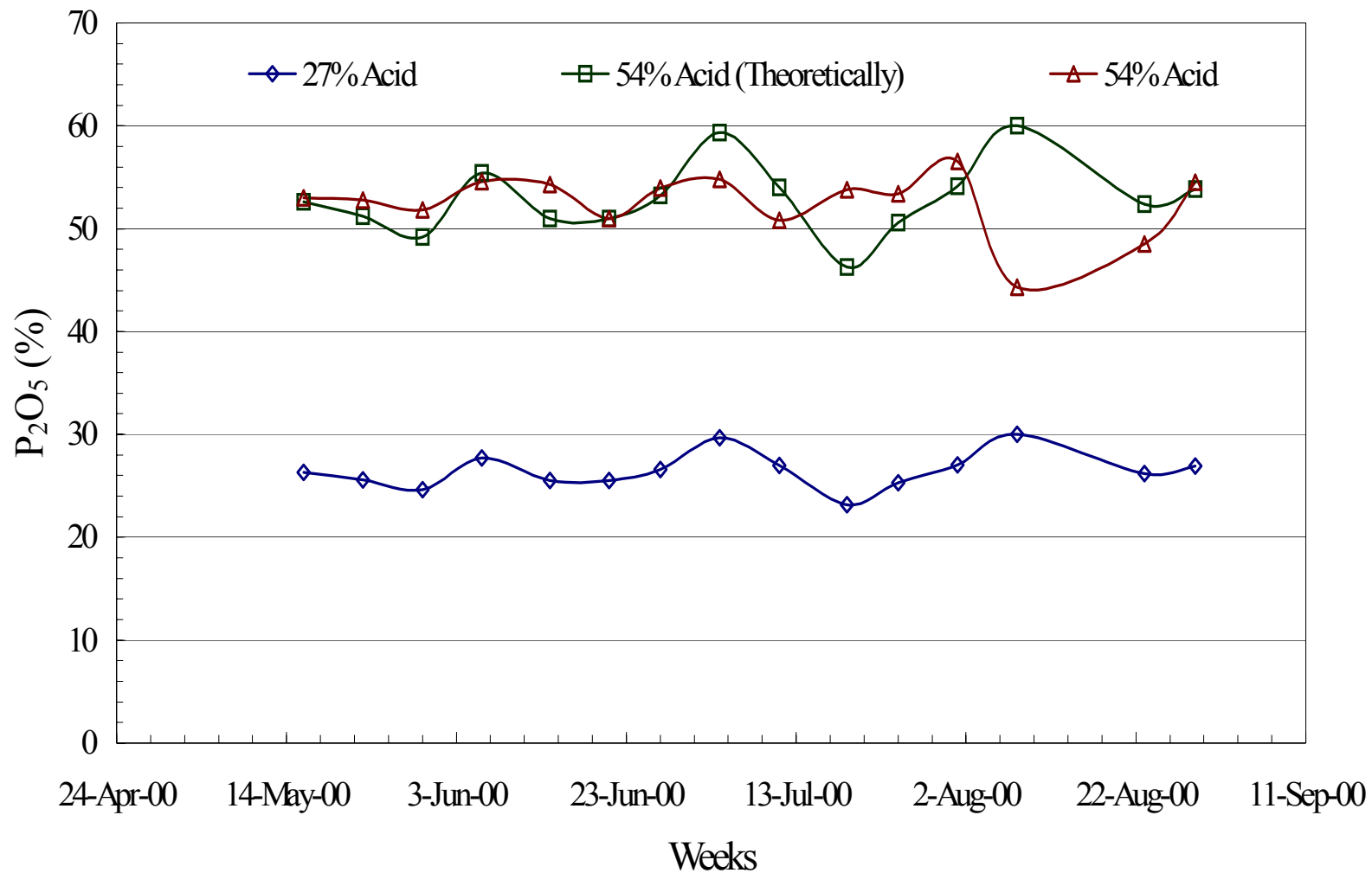
**(See Chapter 3.4.2, p 3-13)**



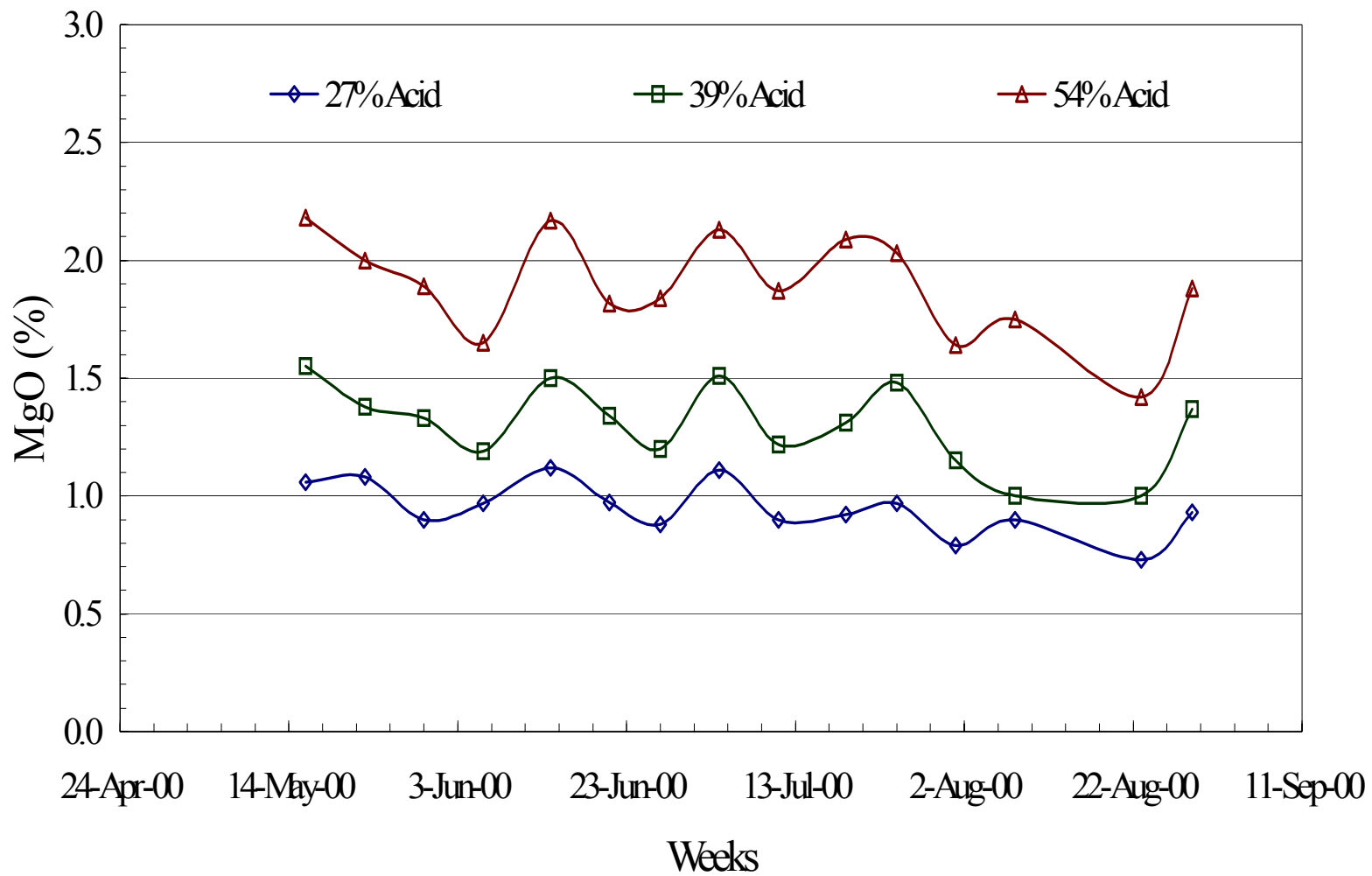
% P<sub>2</sub>O<sub>5</sub> variations over the time period investigated



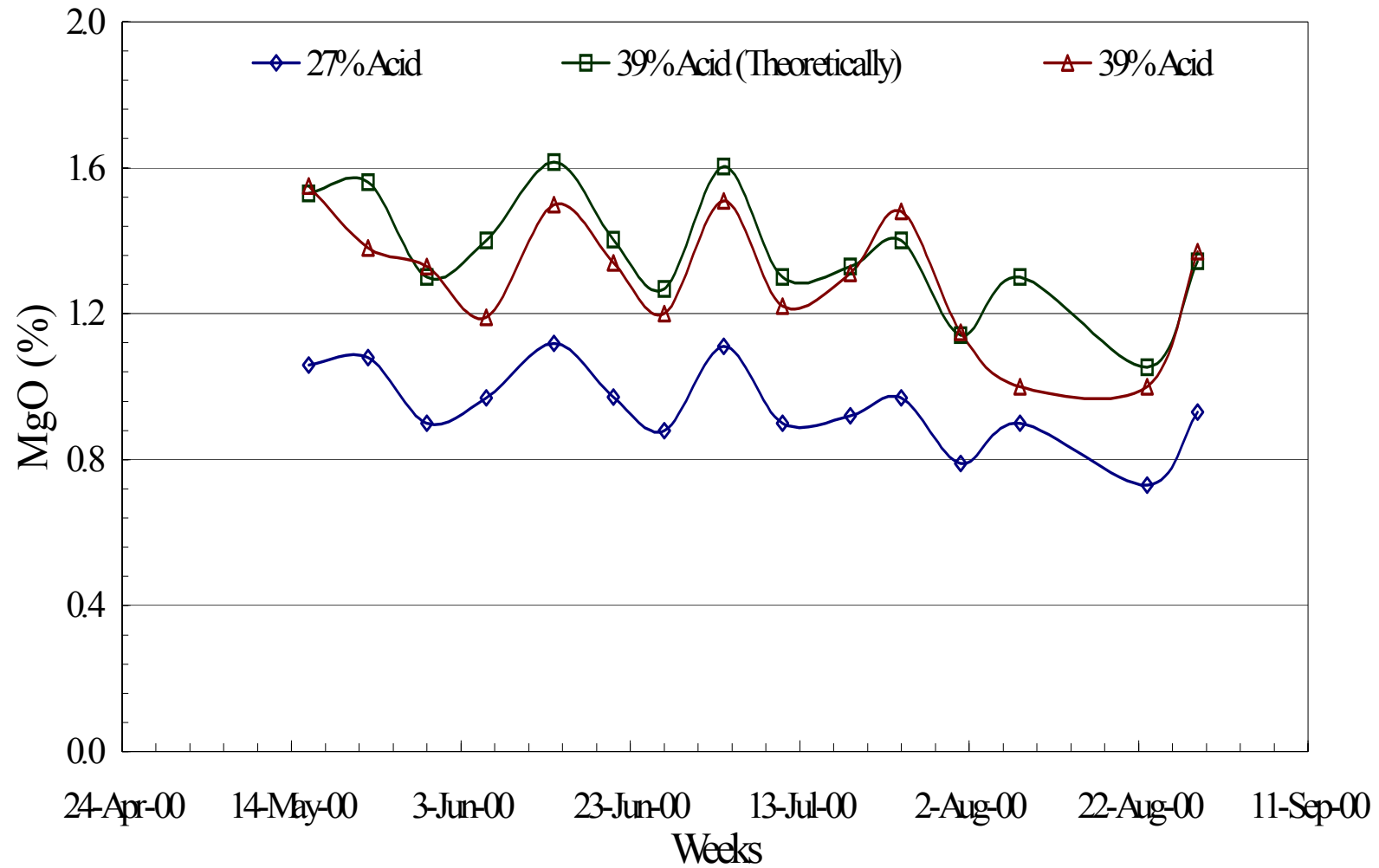
Comparing 39 %  $P_2O_5$  theoretical and 39 %  $P_2O_5$  when 27 %  $P_2O_5$  is concentrated



Comparing 54 %  $P_2O_5$  theoretical and 54 %  $P_2O_5$  when 27 %  $P_2O_5$  is concentrated

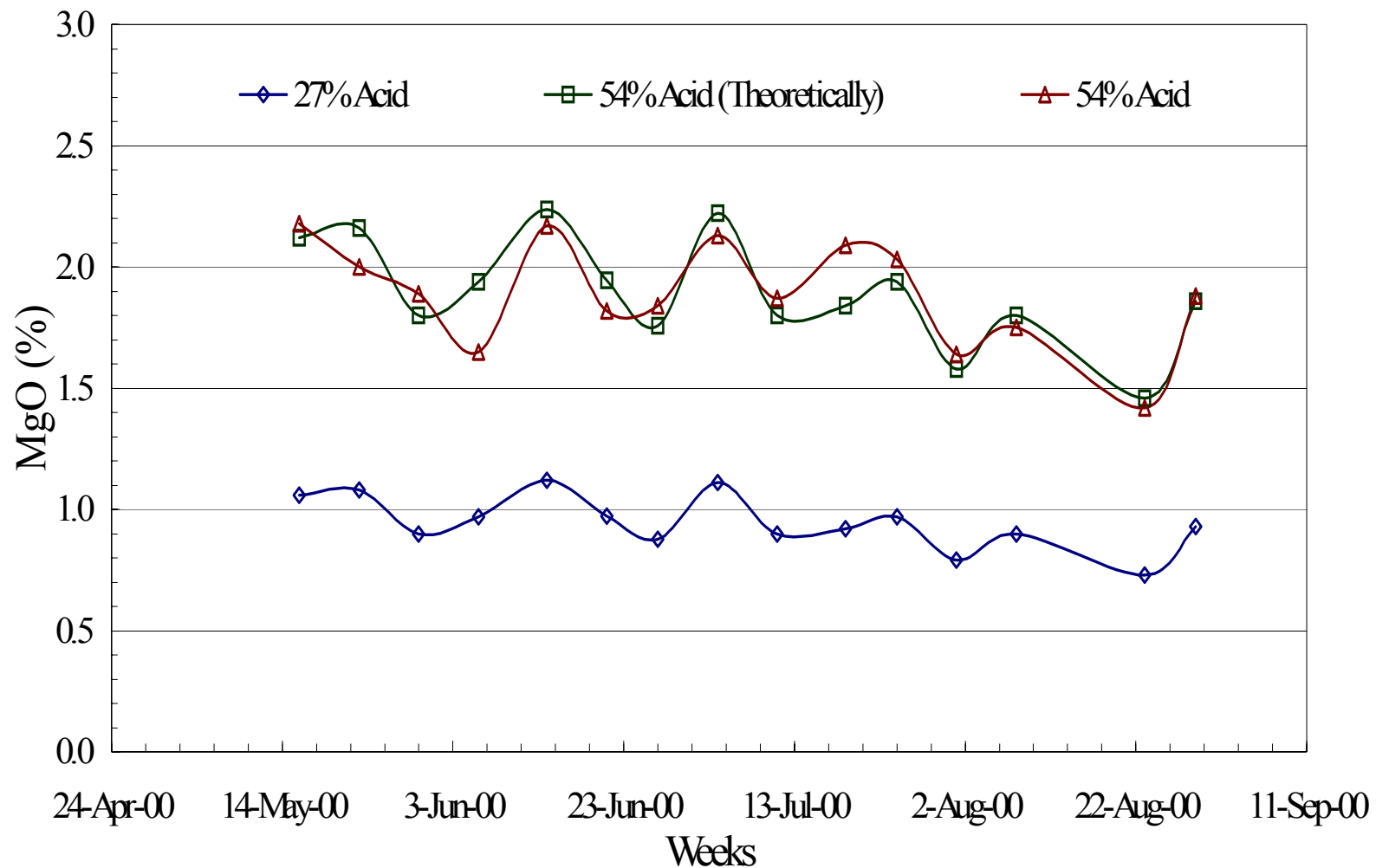


**% MgO variations in the different acid concentrations over the time period investigated**

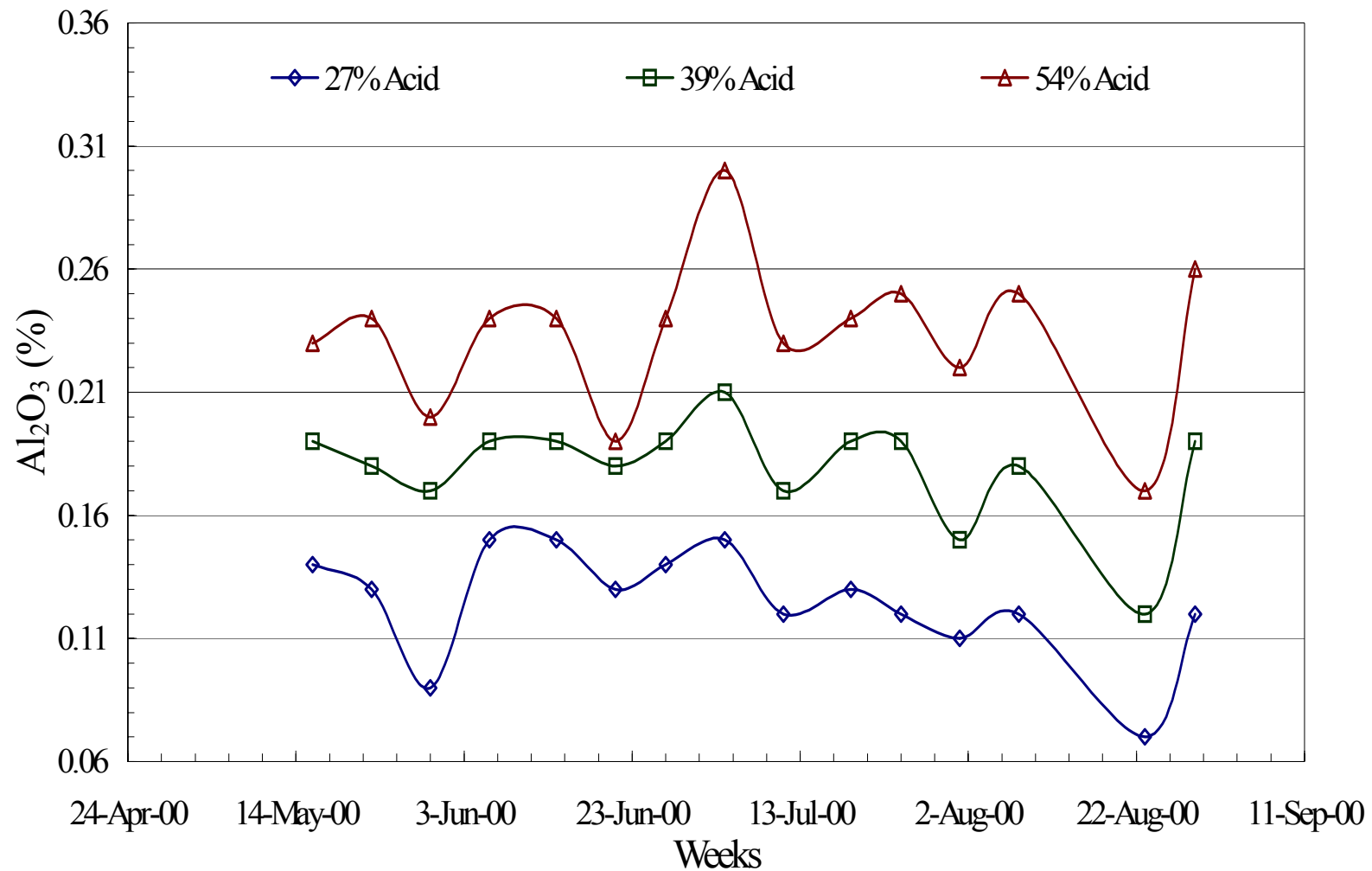


Comparing MgO concentration in 39 %  $P_2O_5$  theoretical and 39 %  $P_2O_5$  when 27 %  $P_2O_5$  is concentrated

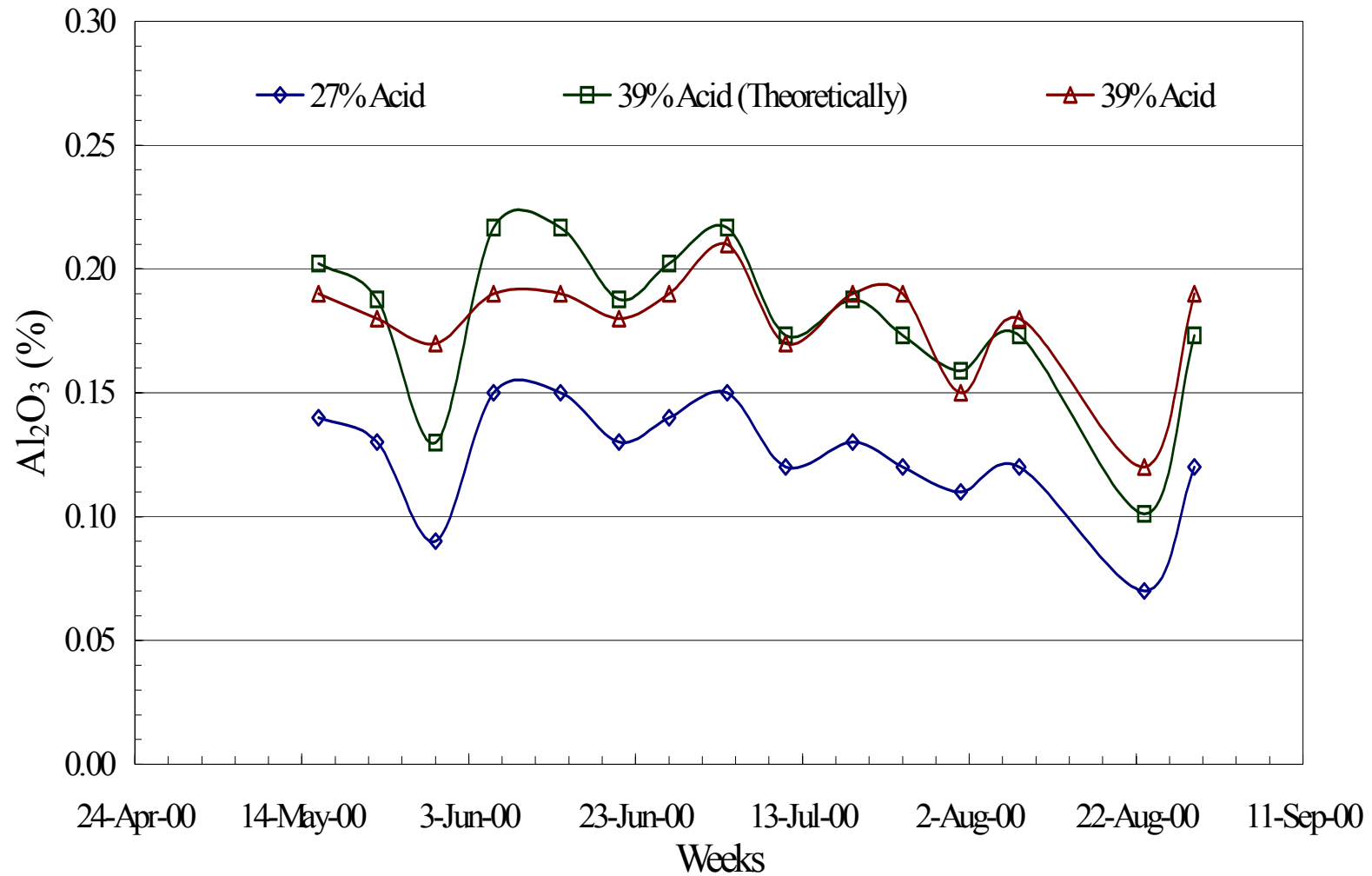




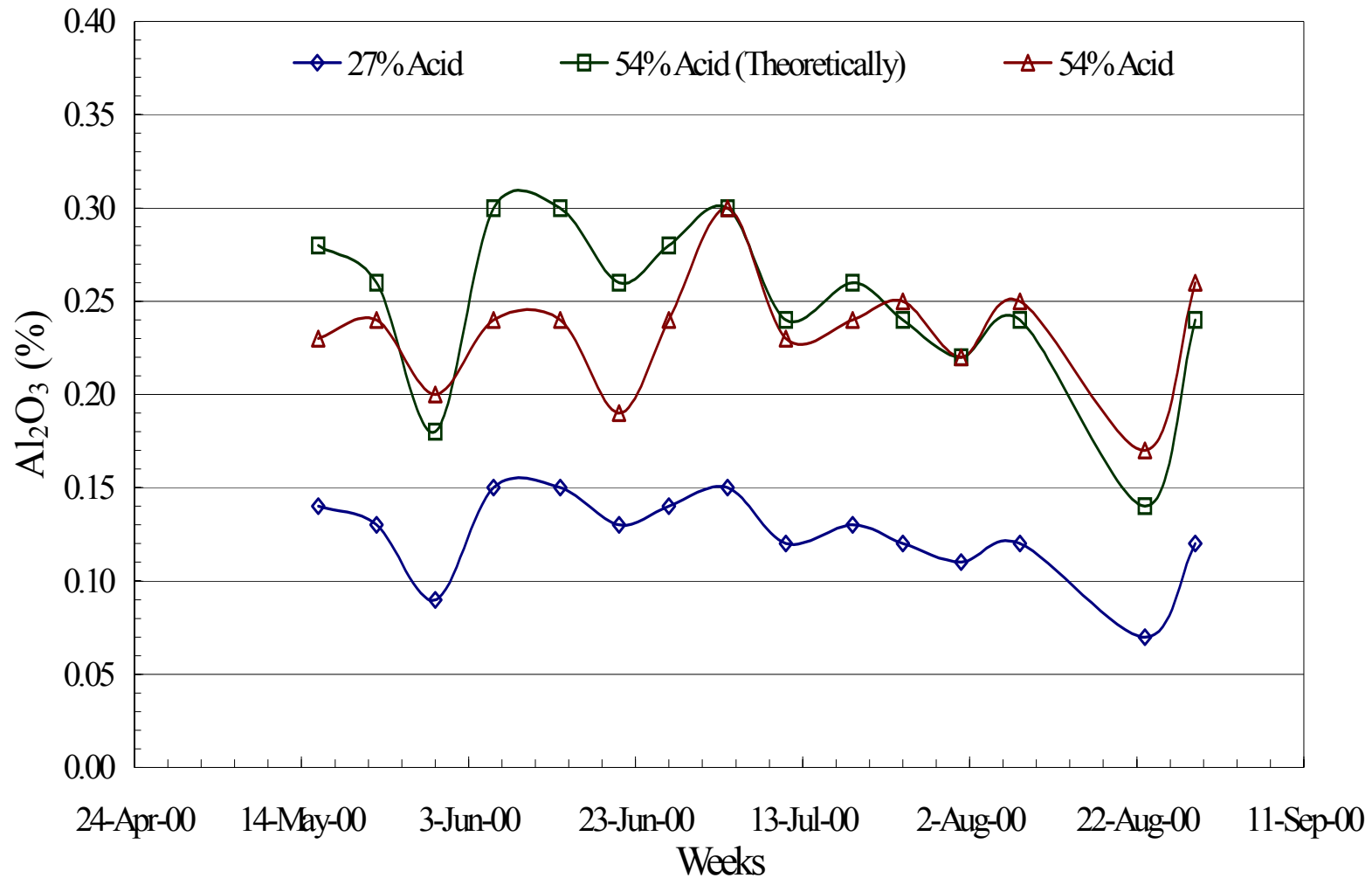
Comparing MgO concentration in 54 % P<sub>2</sub>O<sub>5</sub> theoretical and 54 % P<sub>2</sub>O<sub>5</sub> when 27 % P<sub>2</sub>O<sub>5</sub> is concentrated



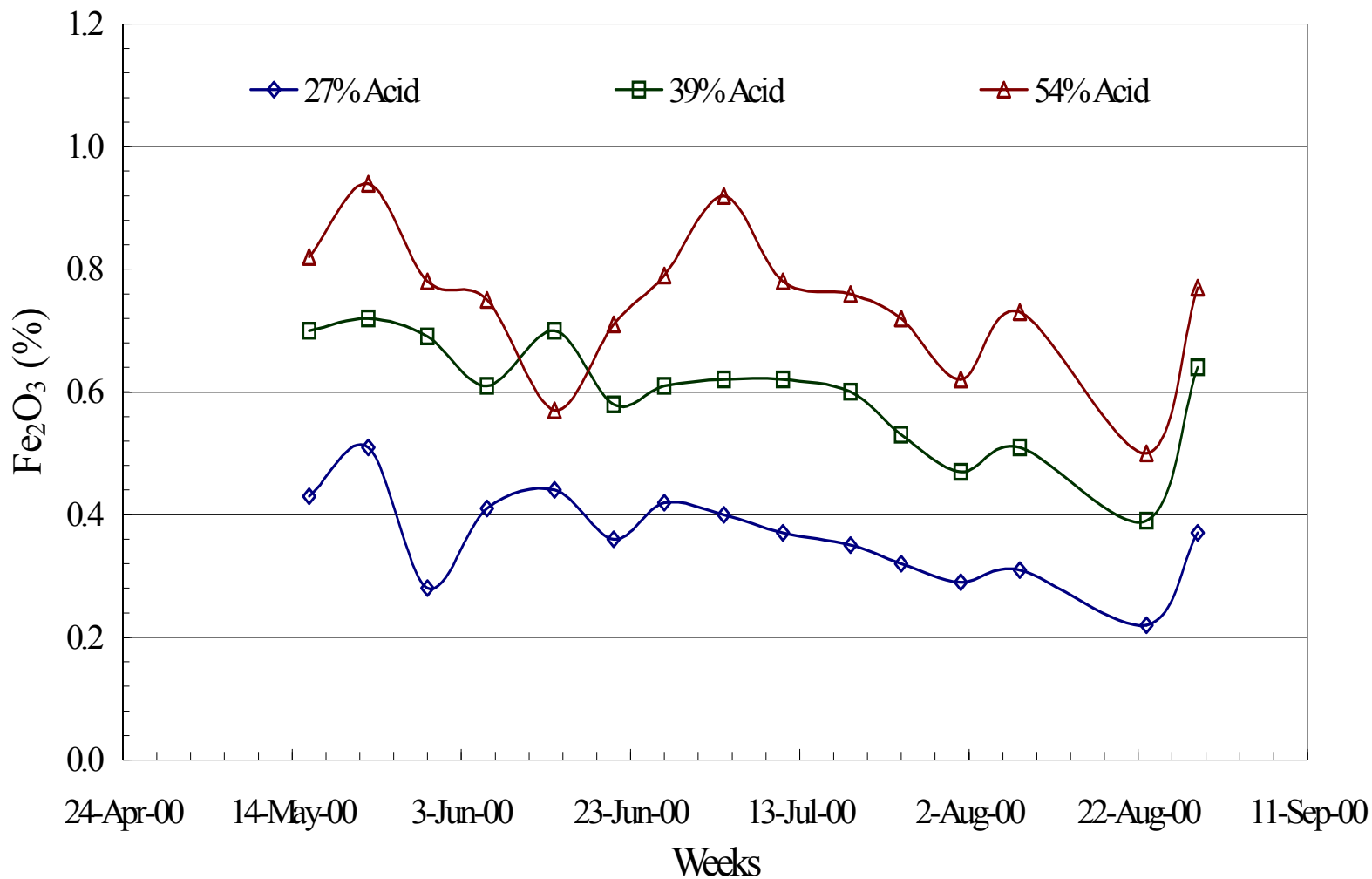
**%  $\text{Al}_2\text{O}_3$  variations in the different acid concentrations over the time period investigated**



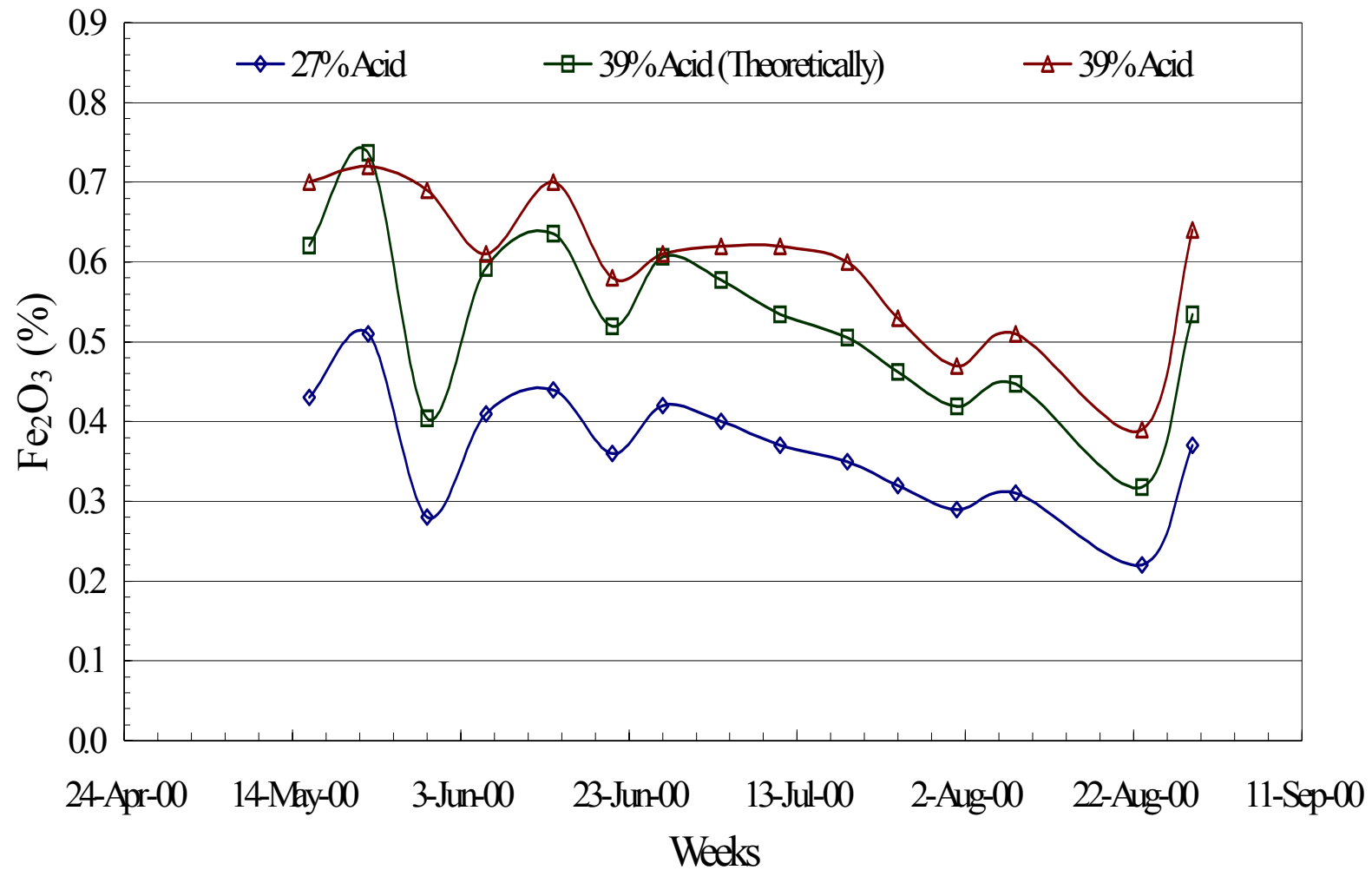
Comparing  $\text{Al}_2\text{O}_3$  concentration in 39 %  $\text{P}_2\text{O}_5$  theoretical and 39 %  $\text{P}_2\text{O}_5$  when 27 %  $\text{P}_2\text{O}_5$  is concentrated



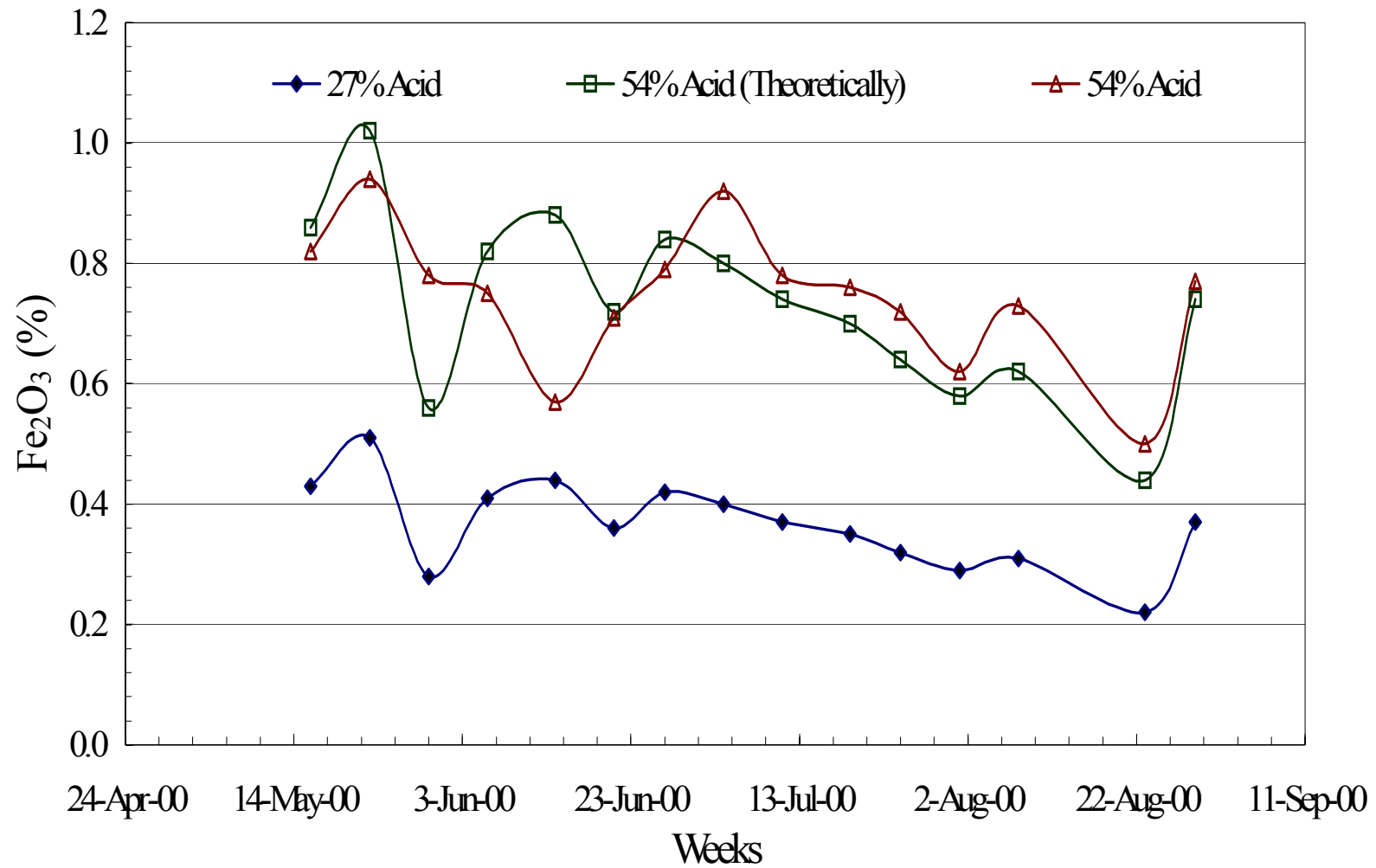
Comparing  $\text{Al}_2\text{O}_3$  concentration in 54 %  $\text{P}_2\text{O}_5$  theoretical and 54 %  $\text{P}_2\text{O}_5$  when 27 %  $\text{P}_2\text{O}_5$  is concentrated



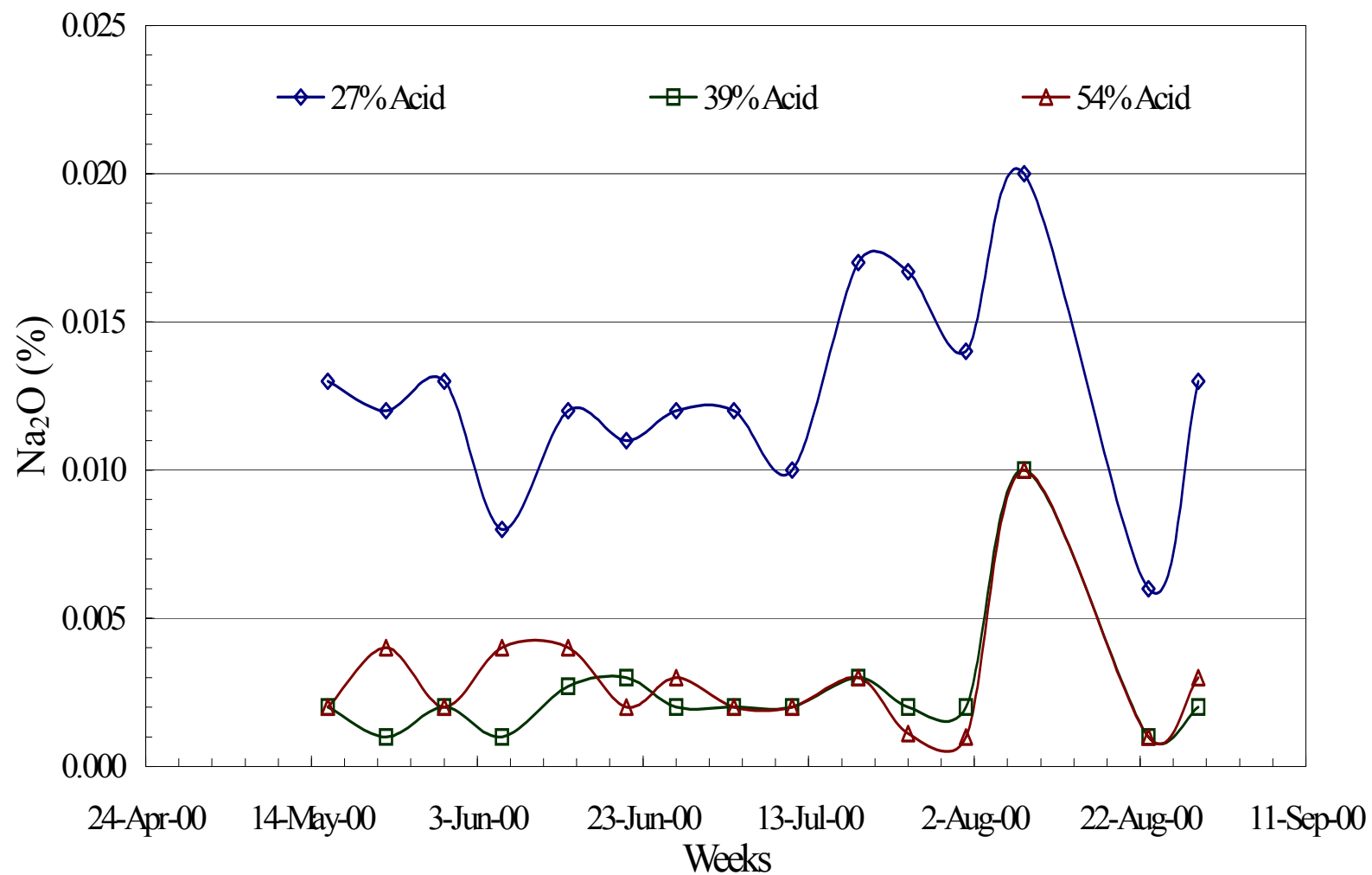
**% Fe<sub>2</sub>O<sub>3</sub> variations in the different acid concentrations over the time period investigated**



Comparing  $\text{Fe}_2\text{O}_3$  concentration in 39 %  $\text{P}_2\text{O}_5$  theoretical and 39 %  $\text{P}_2\text{O}_5$  when 27 %  $\text{P}_2\text{O}_5$  is concentrated

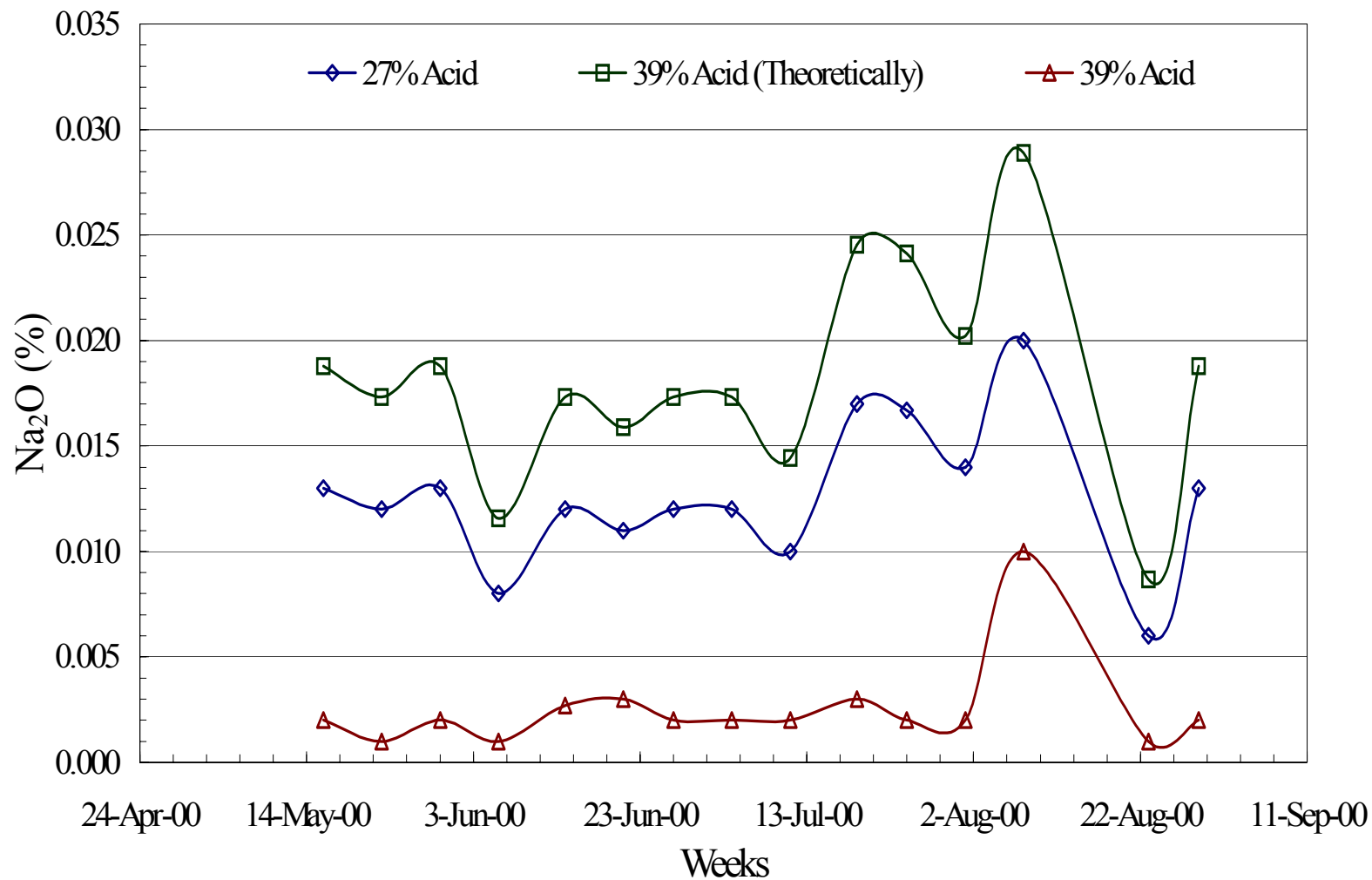


Comparing Fe<sub>2</sub>O<sub>3</sub> concentration in 54 % P<sub>2</sub>O<sub>5</sub> theoretical and 54 % P<sub>2</sub>O<sub>5</sub> when 27 % P<sub>2</sub>O<sub>5</sub> is concentrated

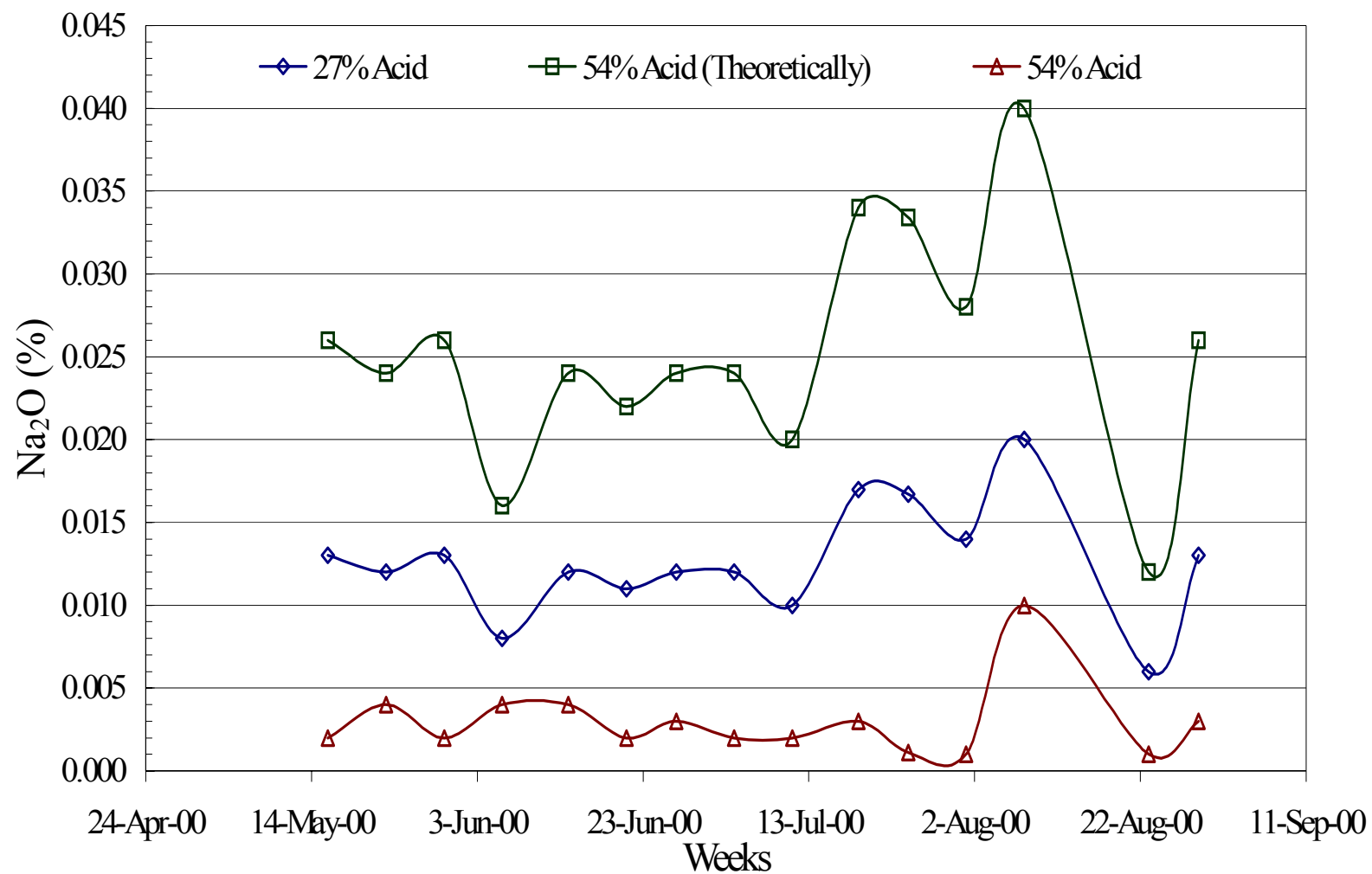


**%  $\text{Na}_2\text{O}$  variations in the different acid concentrations over the time period investigated**

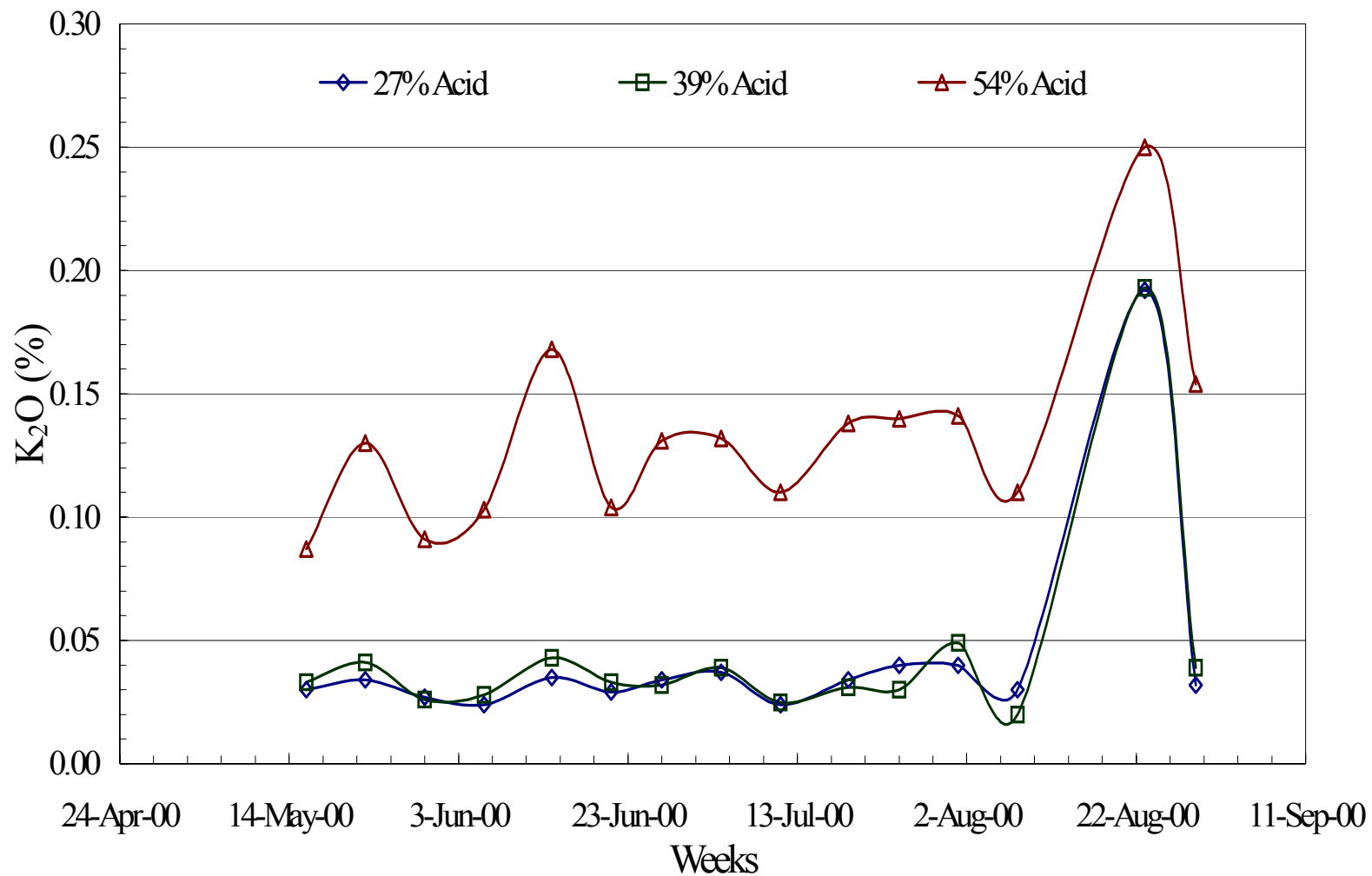




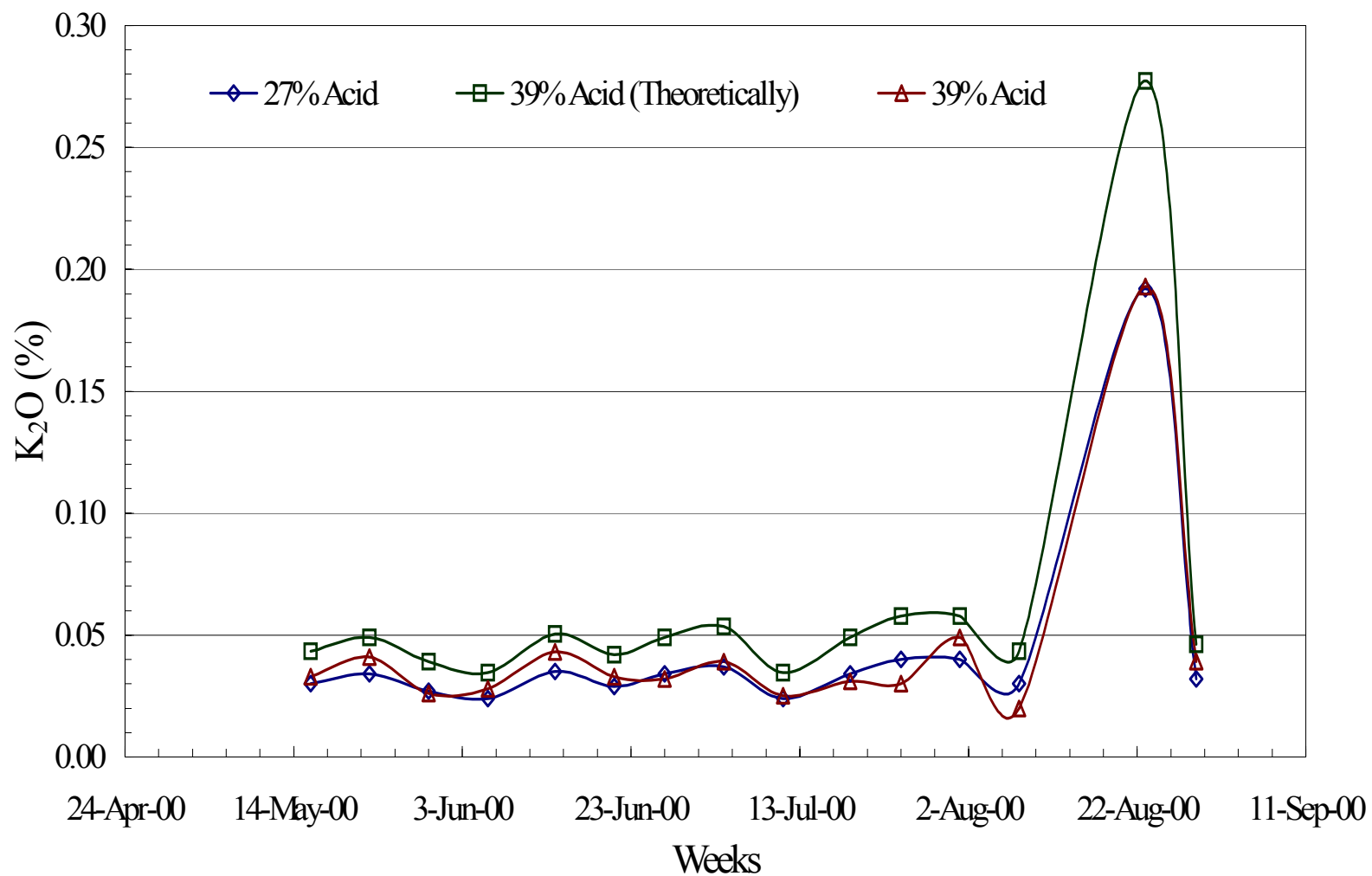
Comparing Na<sub>2</sub>O concentration in 39 % P<sub>2</sub>O<sub>5</sub> theoretical and 39 % P<sub>2</sub>O<sub>5</sub> when 27 % P<sub>2</sub>O<sub>5</sub> is concentrated



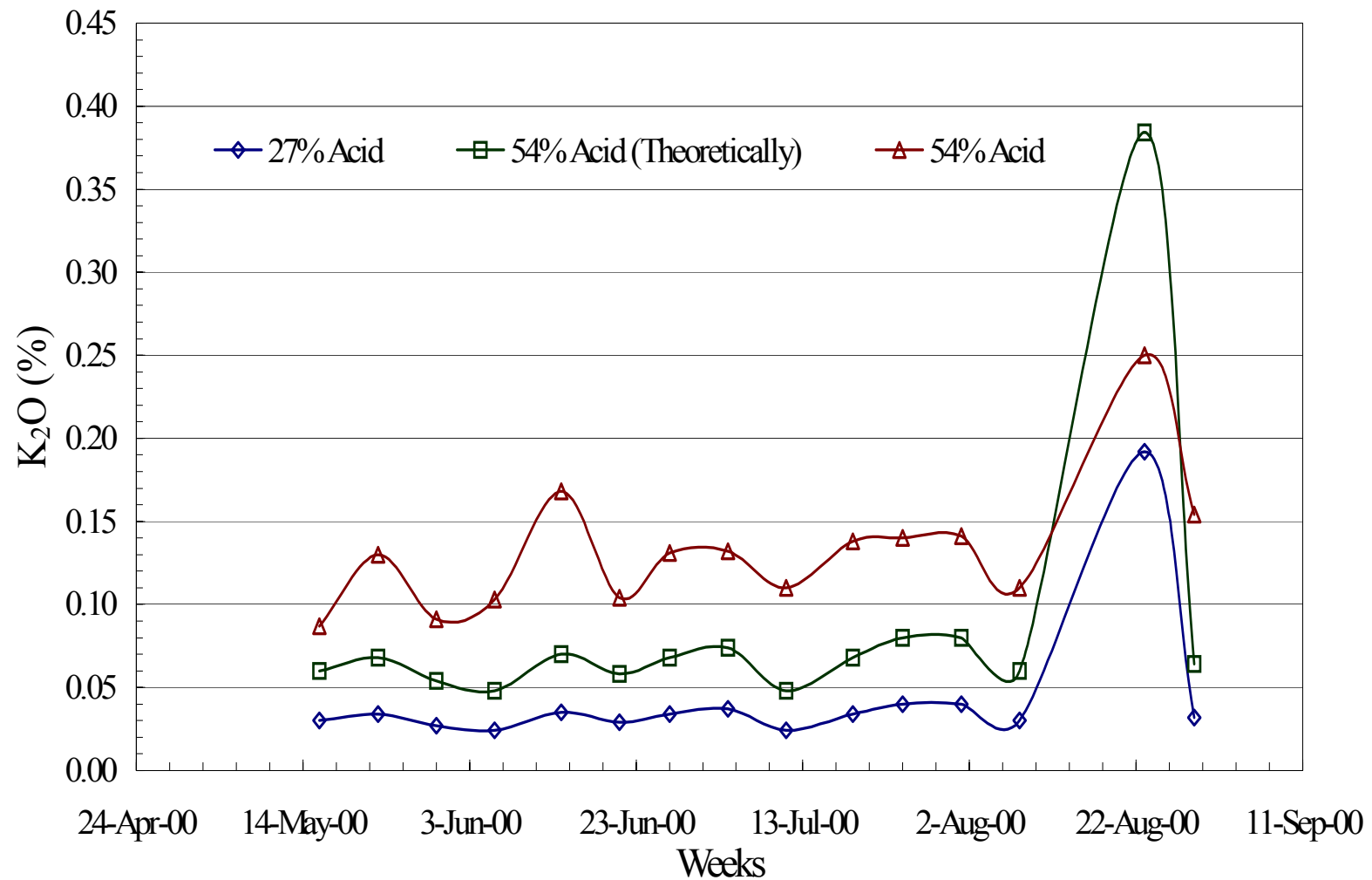
Comparing  $\text{Na}_2\text{O}$  concentration in 54 %  $\text{P}_2\text{O}_5$  theoretical and 54 %  $\text{P}_2\text{O}_5$  when 27 %  $\text{P}_2\text{O}_5$  is concentrated



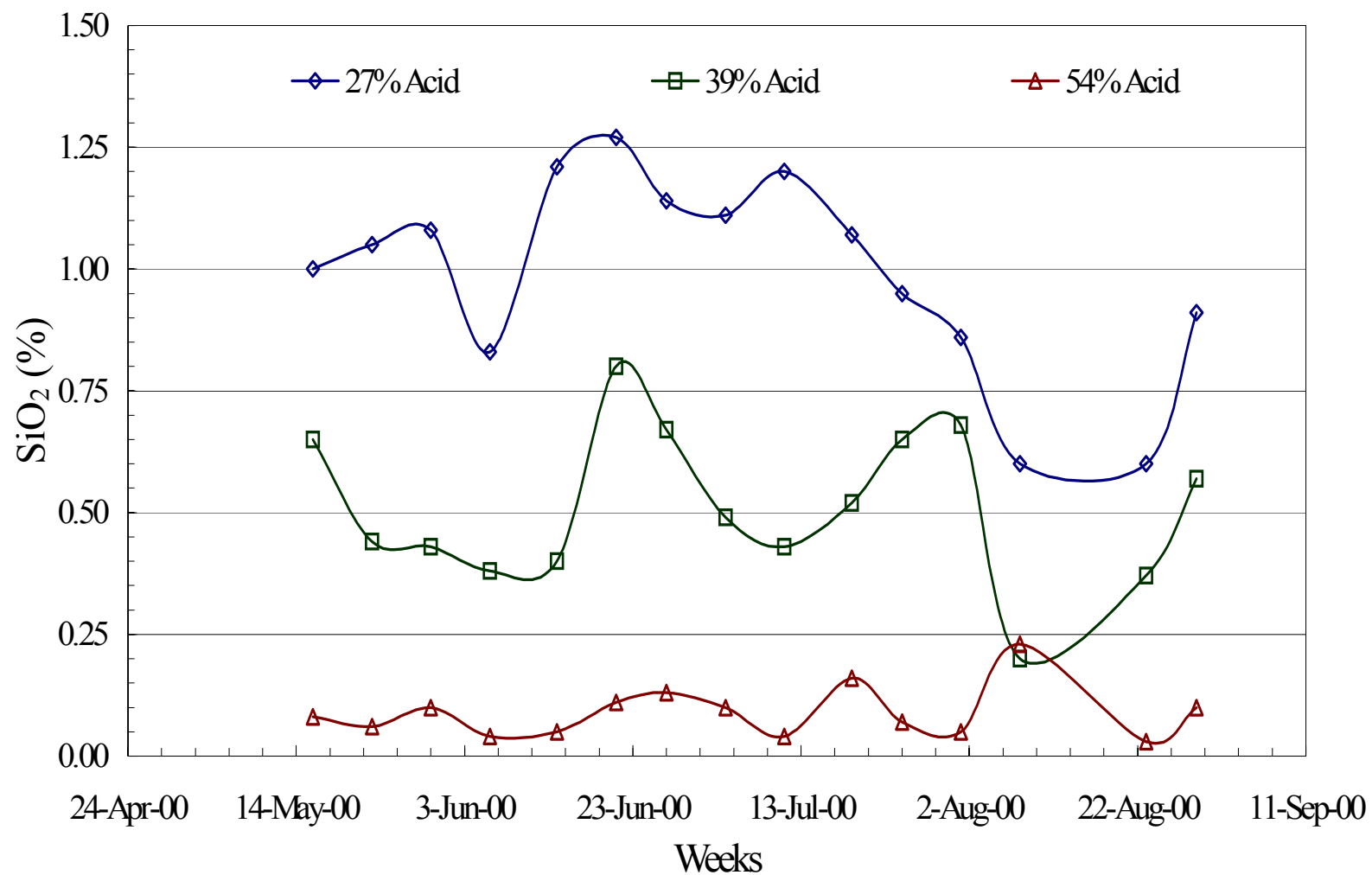
% K<sub>2</sub>O variations in the different acid concentrations over the time period investigated



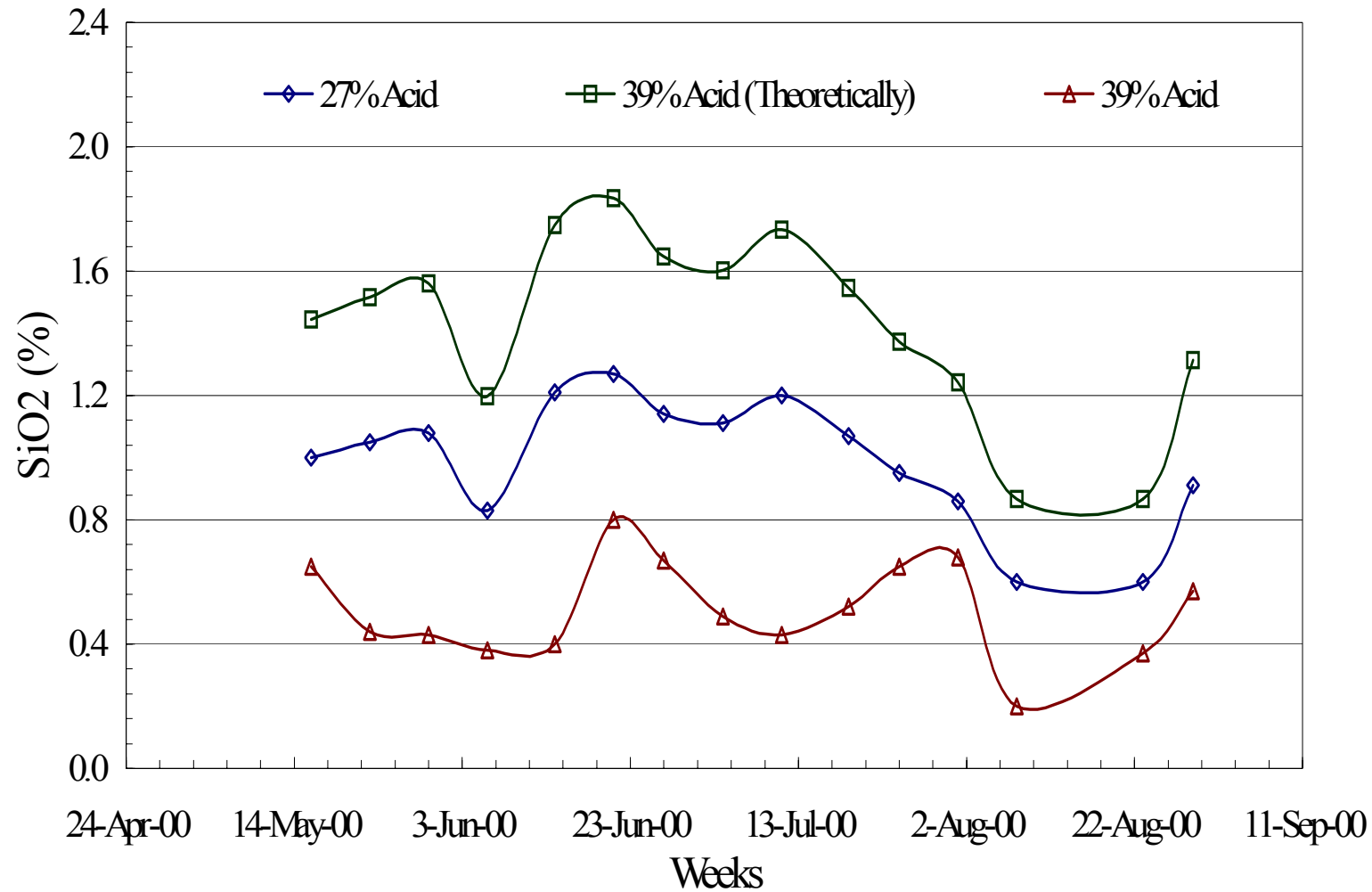
Comparing K<sub>2</sub>O concentration in 39 % P<sub>2</sub>O<sub>5</sub> theoretical and 39 % P<sub>2</sub>O<sub>5</sub> when 27 % P<sub>2</sub>O<sub>5</sub> is concentrated



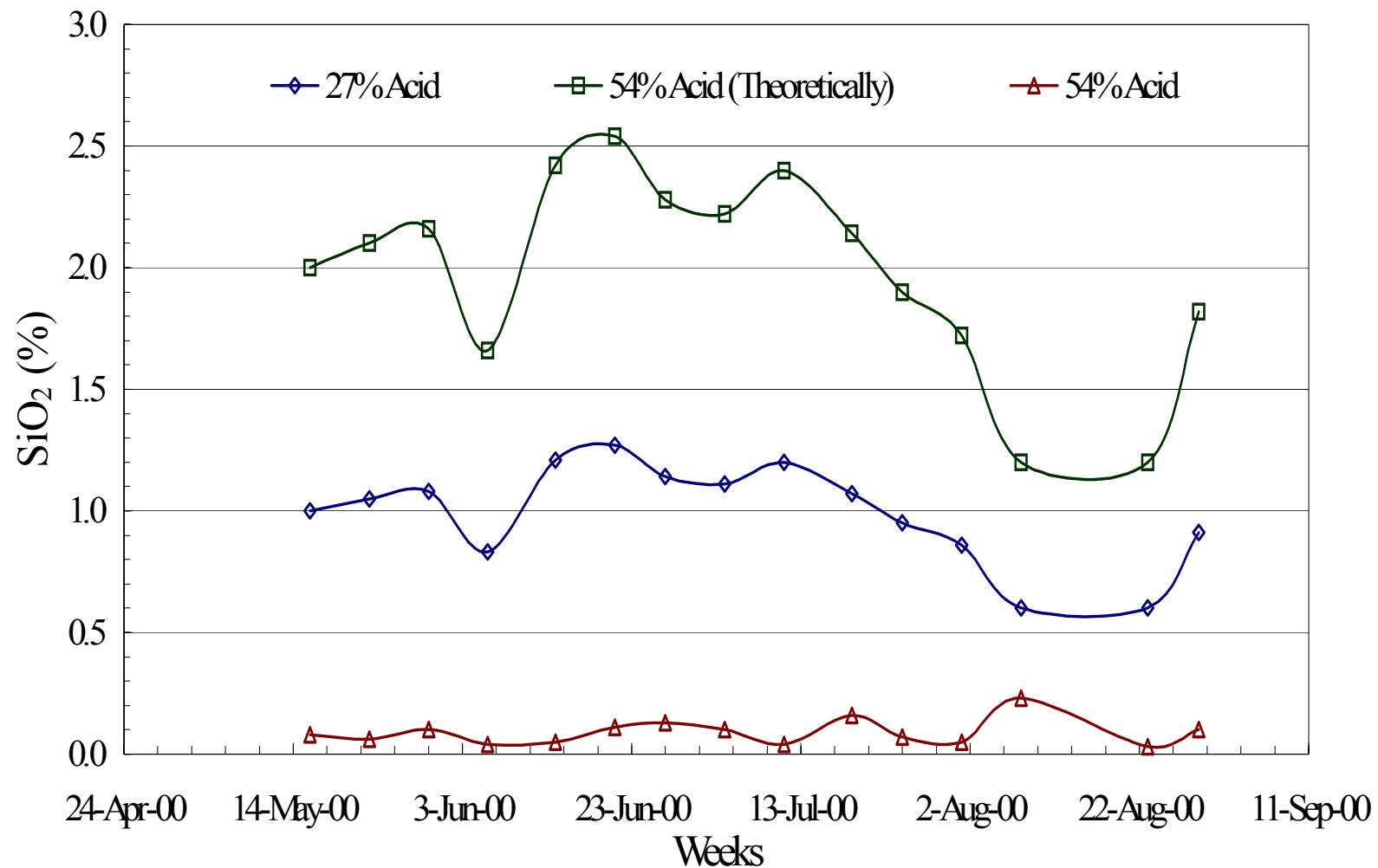
Comparing K<sub>2</sub>O concentration in 54 % P<sub>2</sub>O<sub>5</sub> theoretical and 54 % P<sub>2</sub>O<sub>5</sub> when 27 % P<sub>2</sub>O<sub>5</sub> is concentrated



% SiO<sub>2</sub> variations in the different acid concentrations over the time period investigated



Comparing SiO<sub>2</sub> concentration in 39 % P<sub>2</sub>O<sub>5</sub> theoretical and 39 % P<sub>2</sub>O<sub>5</sub> when 27 % P<sub>2</sub>O<sub>5</sub> is concentrated



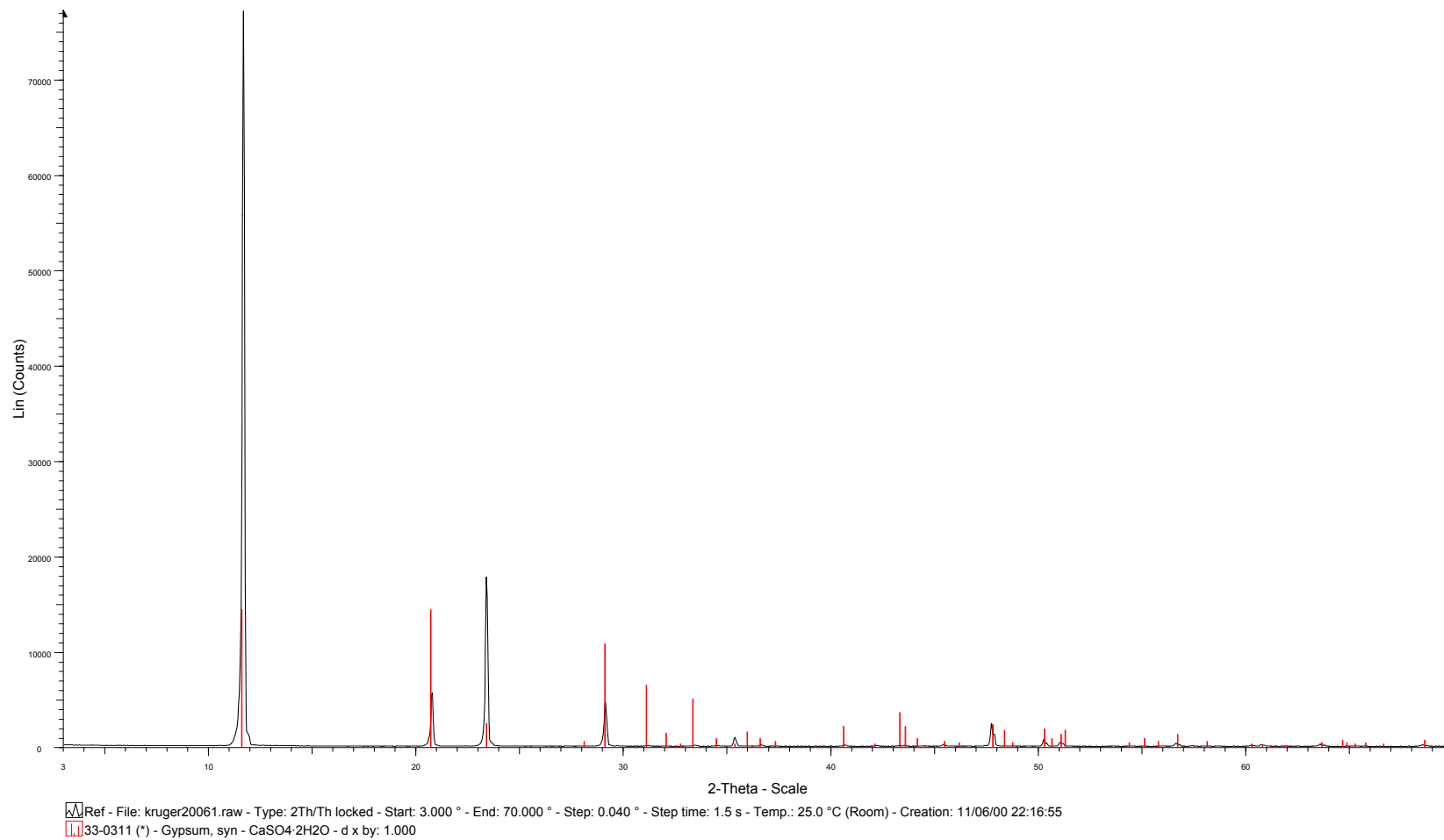
Comparing SiO<sub>2</sub> concentration in 54 % P<sub>2</sub>O<sub>5</sub> theoretical and 54 % P<sub>2</sub>O<sub>5</sub> when 27 % P<sub>2</sub>O<sub>5</sub> is concentrated



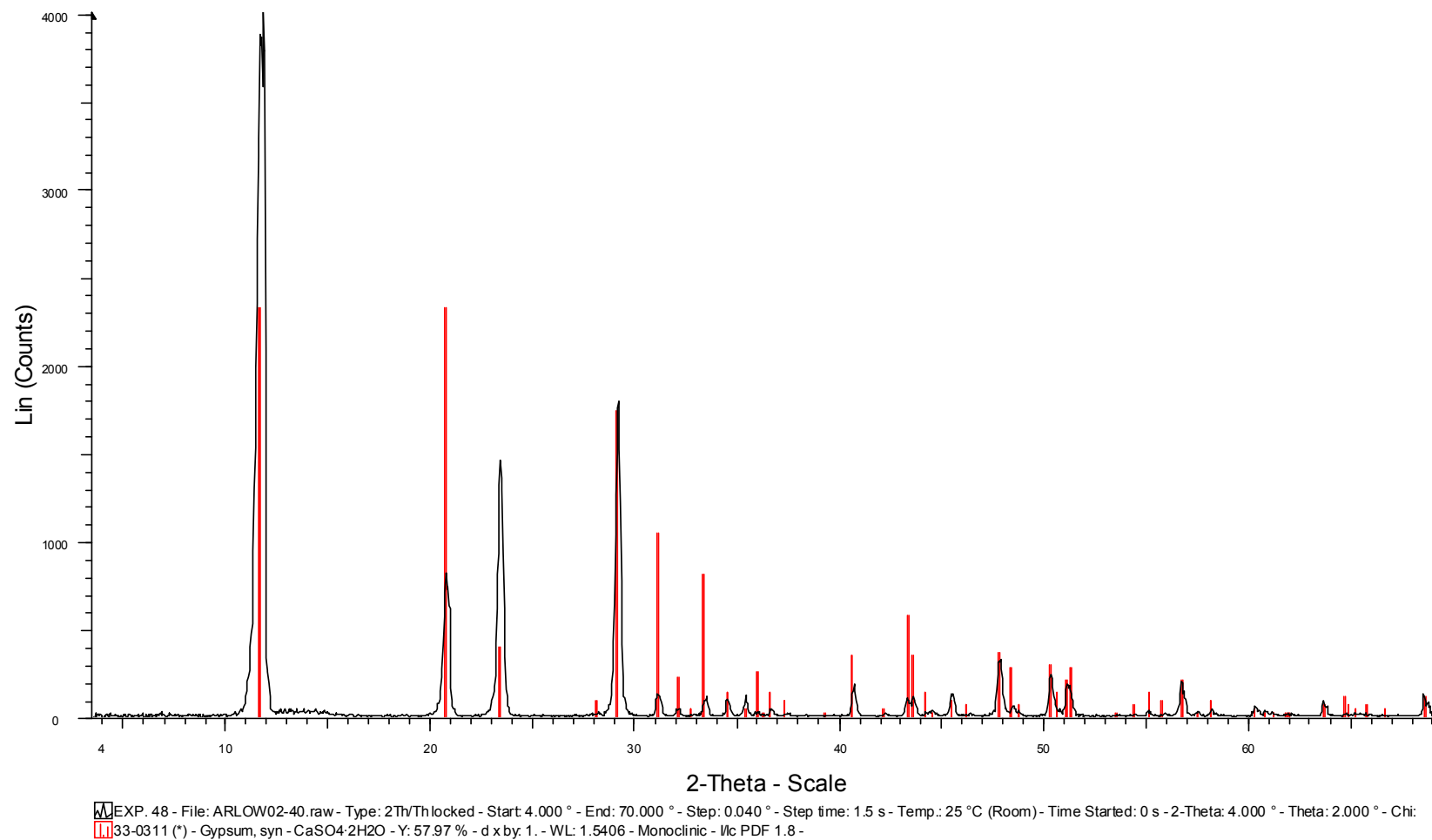
## **APPENDIX E**

### **XRD results for gypsum experiments**

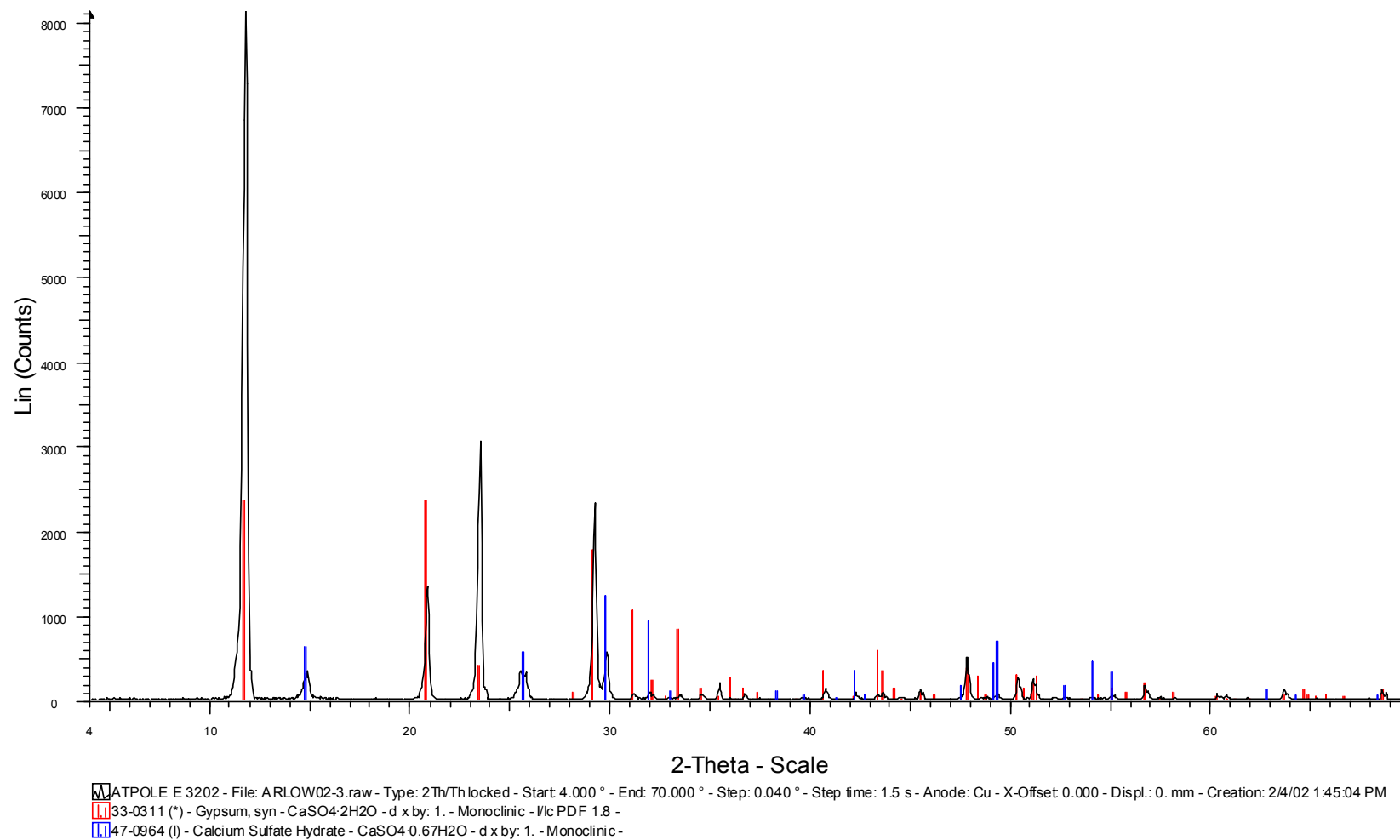
(see Chapter 4.3.3, p 4-10)



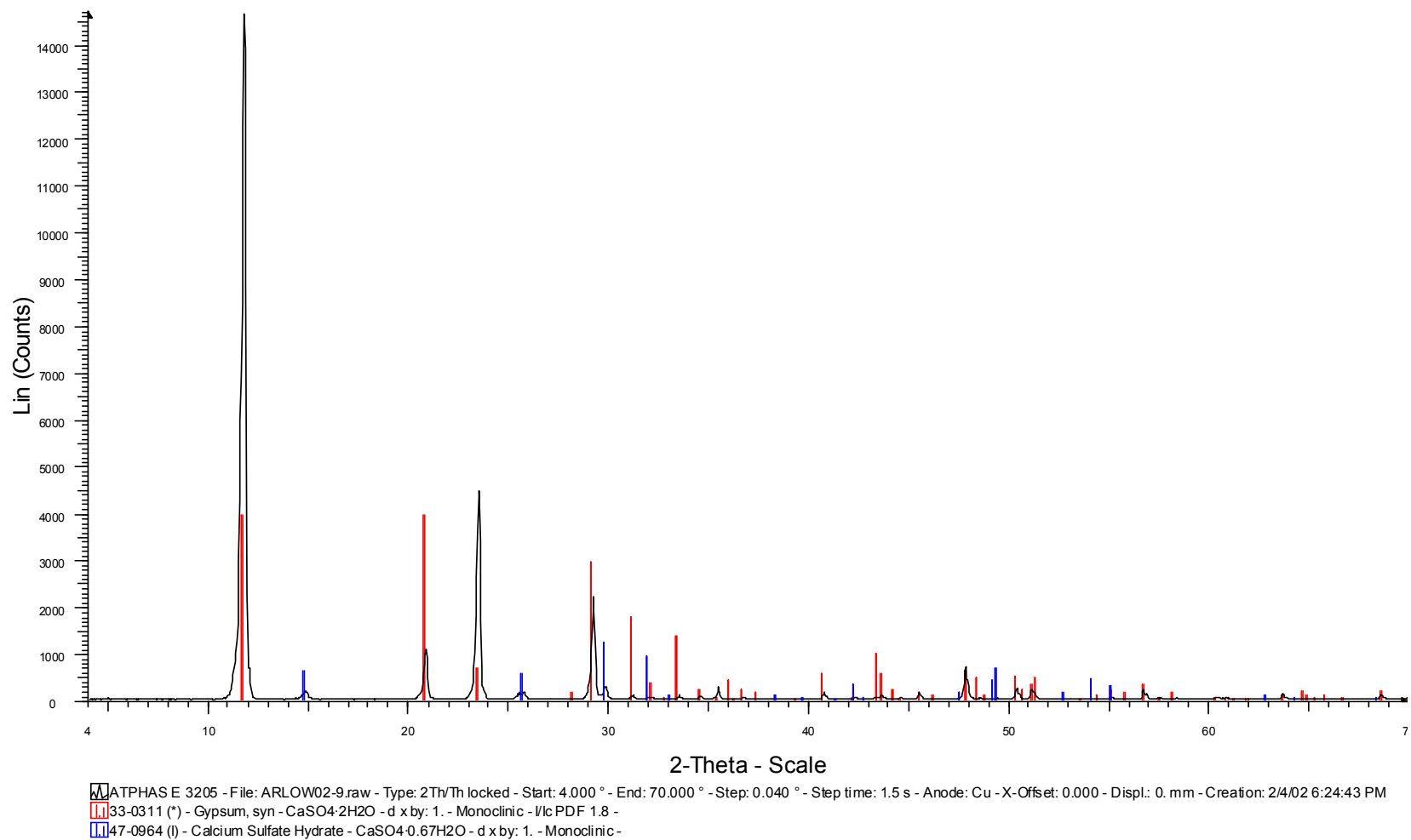
**XRD result of Method B reference experiment with 30 minute stirring**



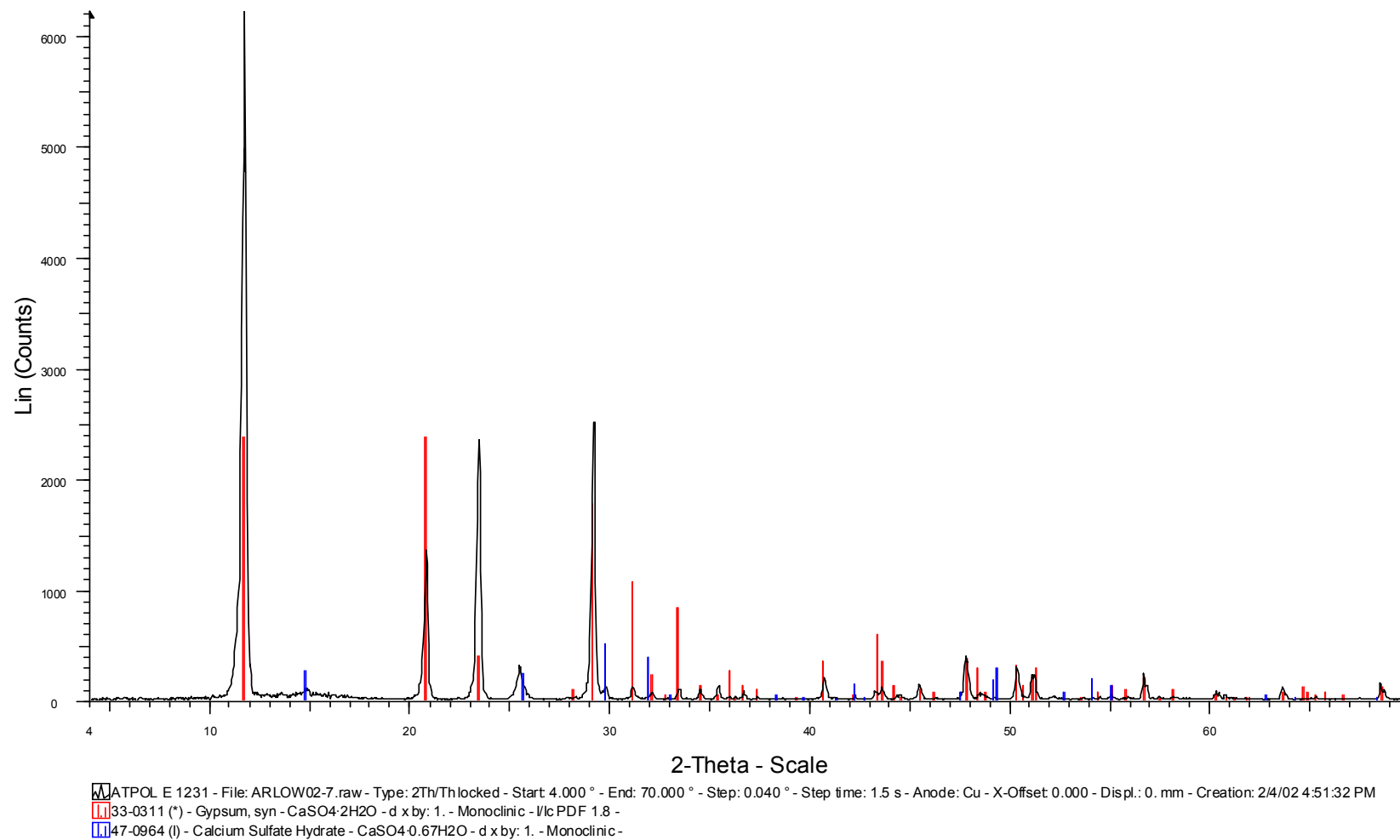
**XRD results of Method B reference experiments with 24 hour stirring**



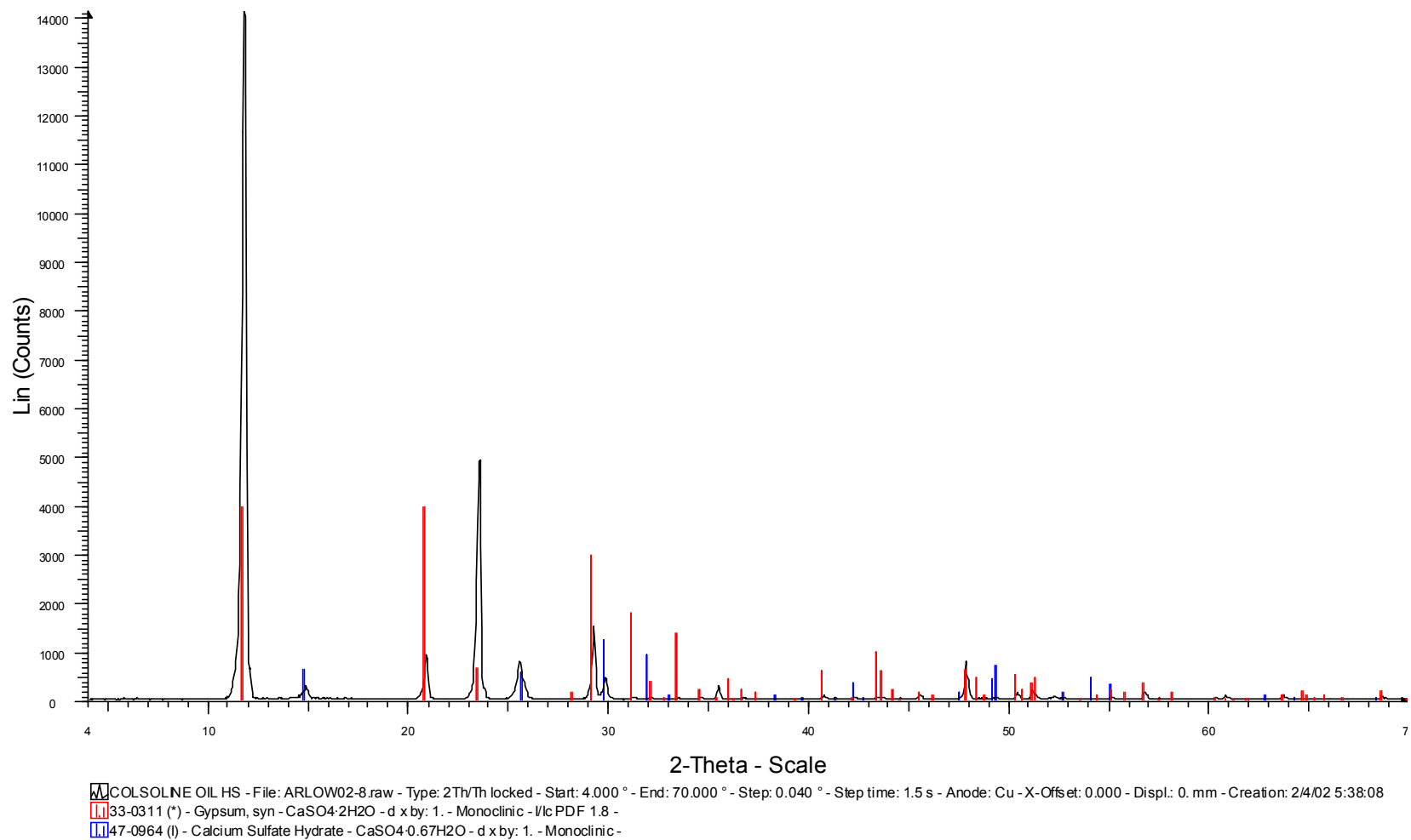
**XRD result of surfactant experiment using Atpol E3202**



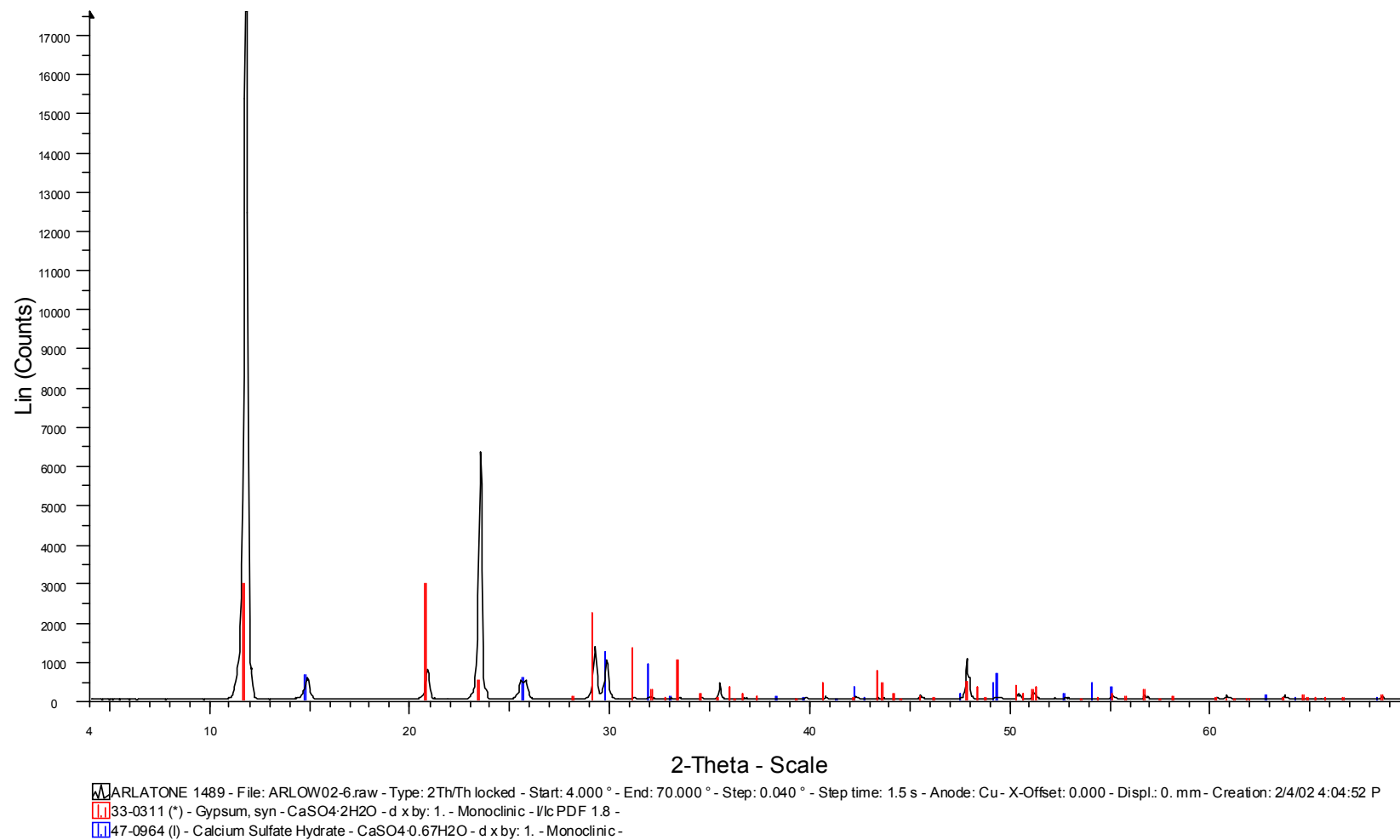
**XRD result of surfactant experiment using Atphos E3205**



**XRD result of surfactant experiment using Atpol E1231**

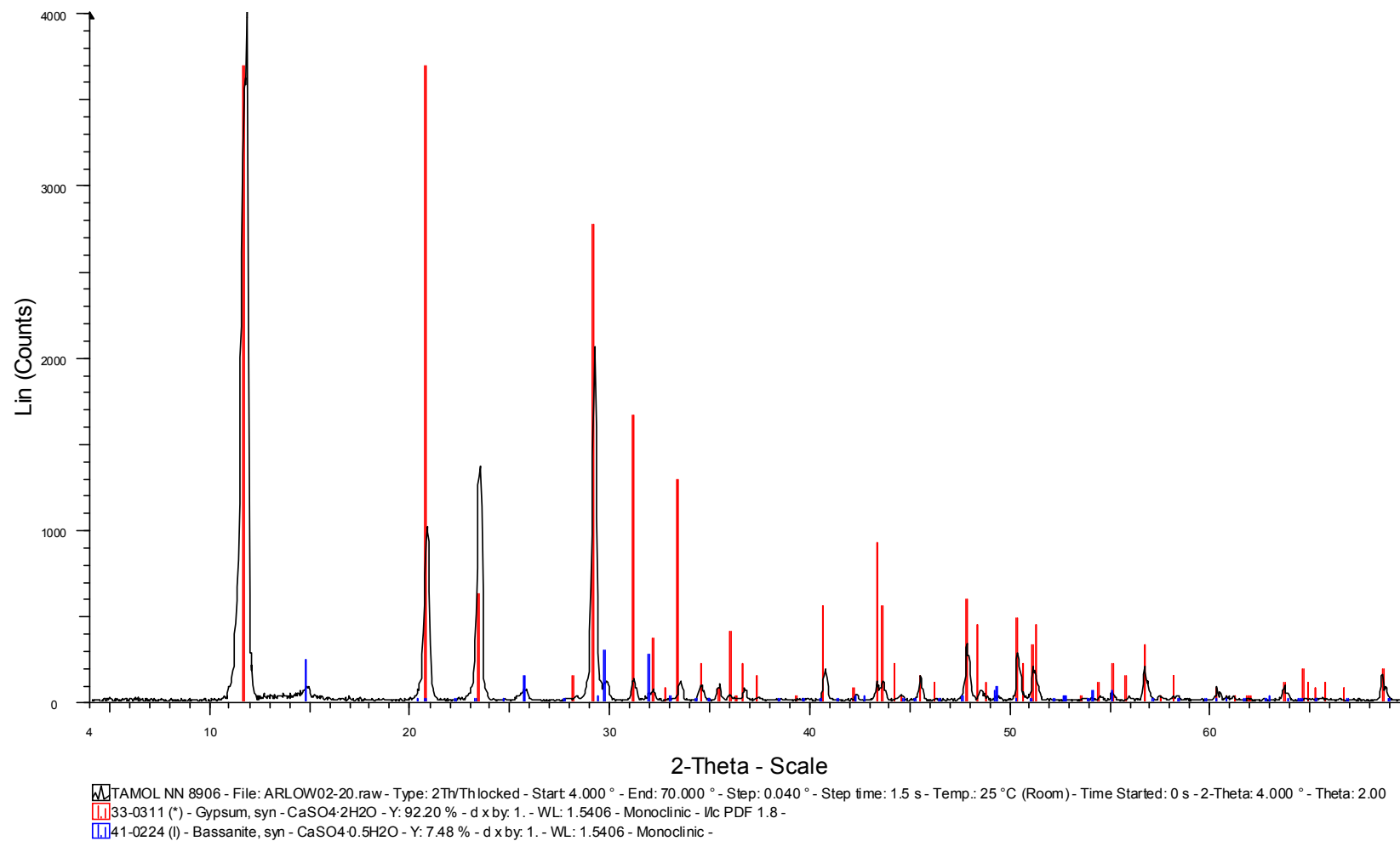


### XRD analysis of surfactant experiment using Calsoline oil

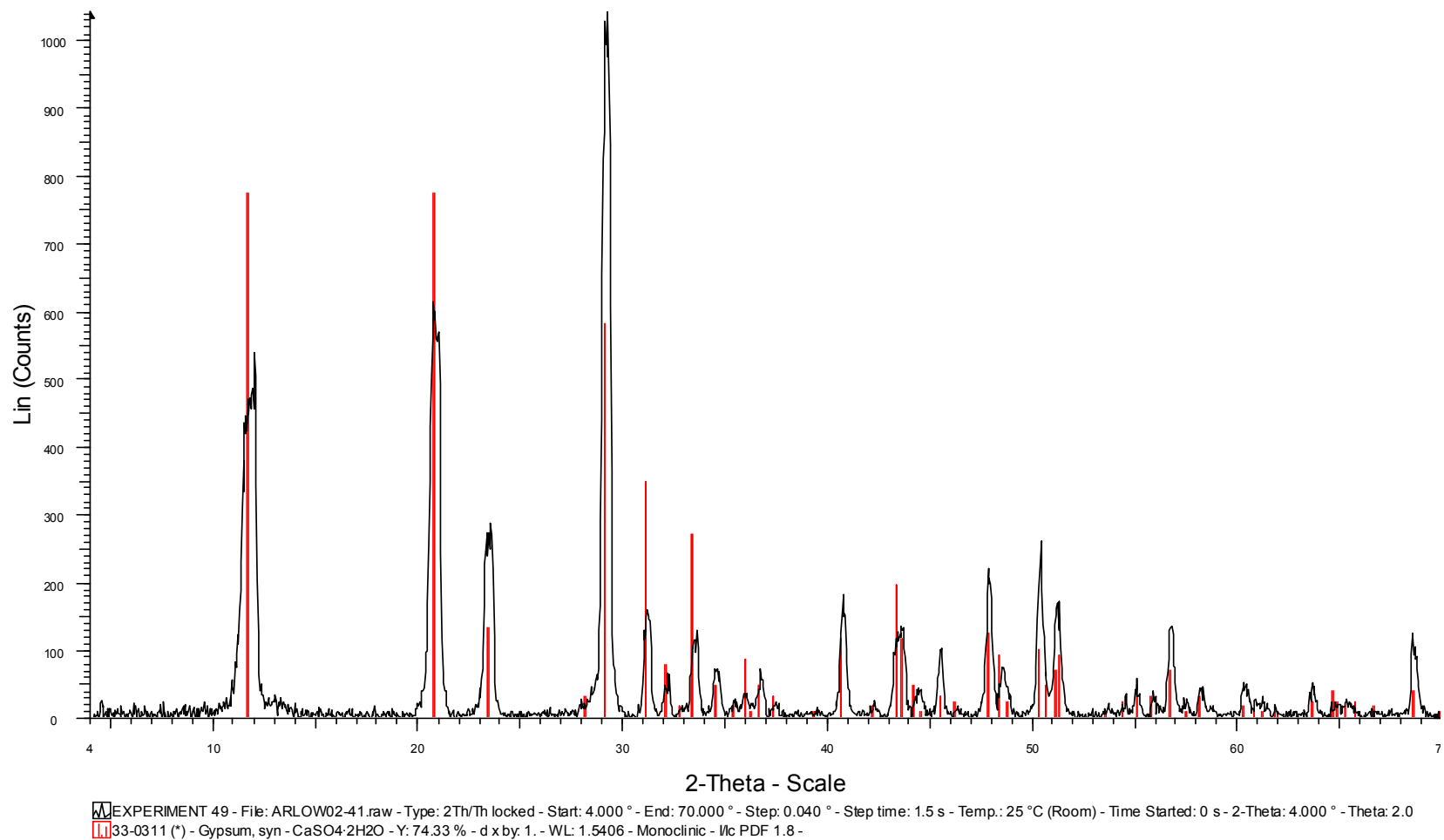


**XRD result of surfactant experiment using Arlatone 1489**

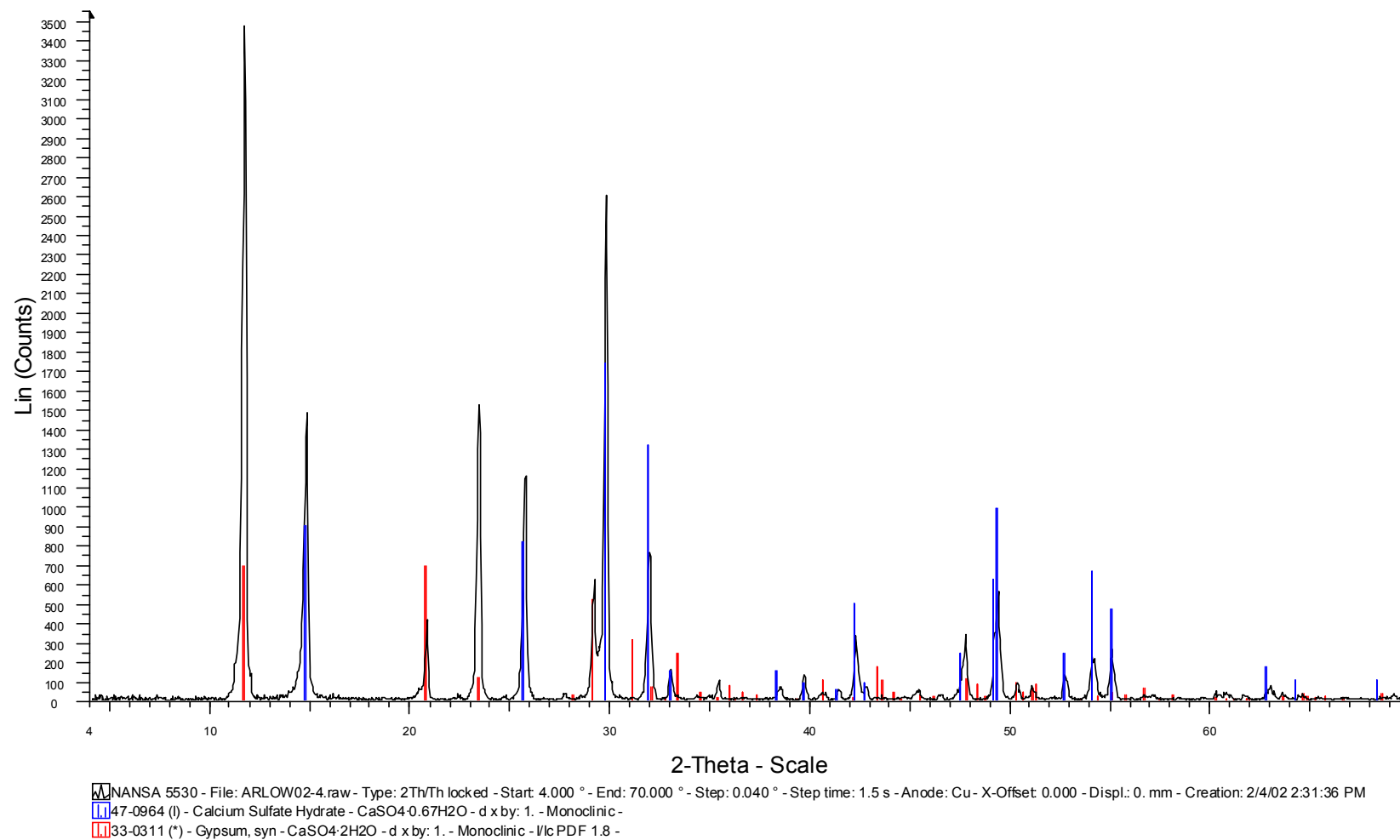




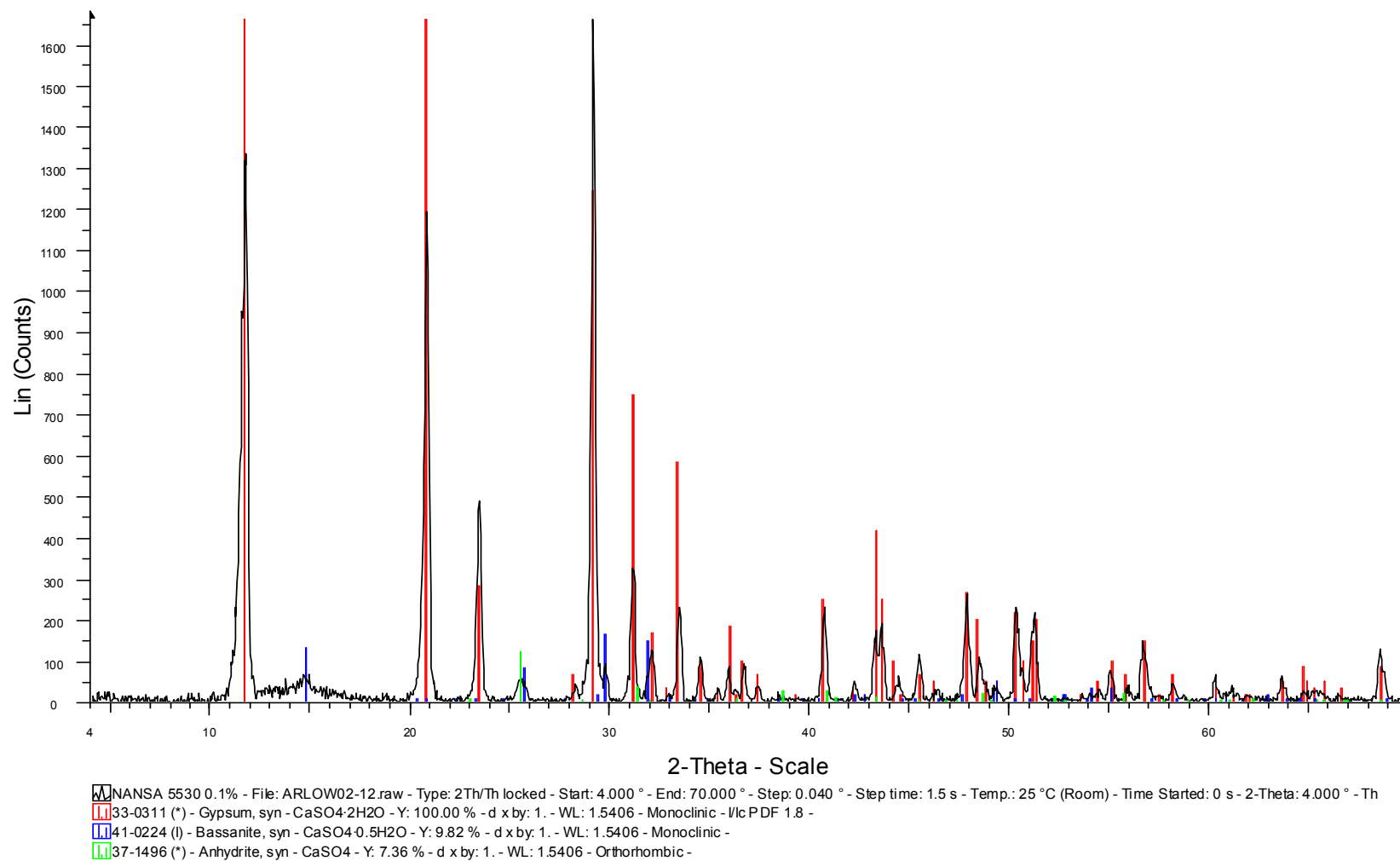
**XRD results of surfactant experiment using Tamol NN 8906**



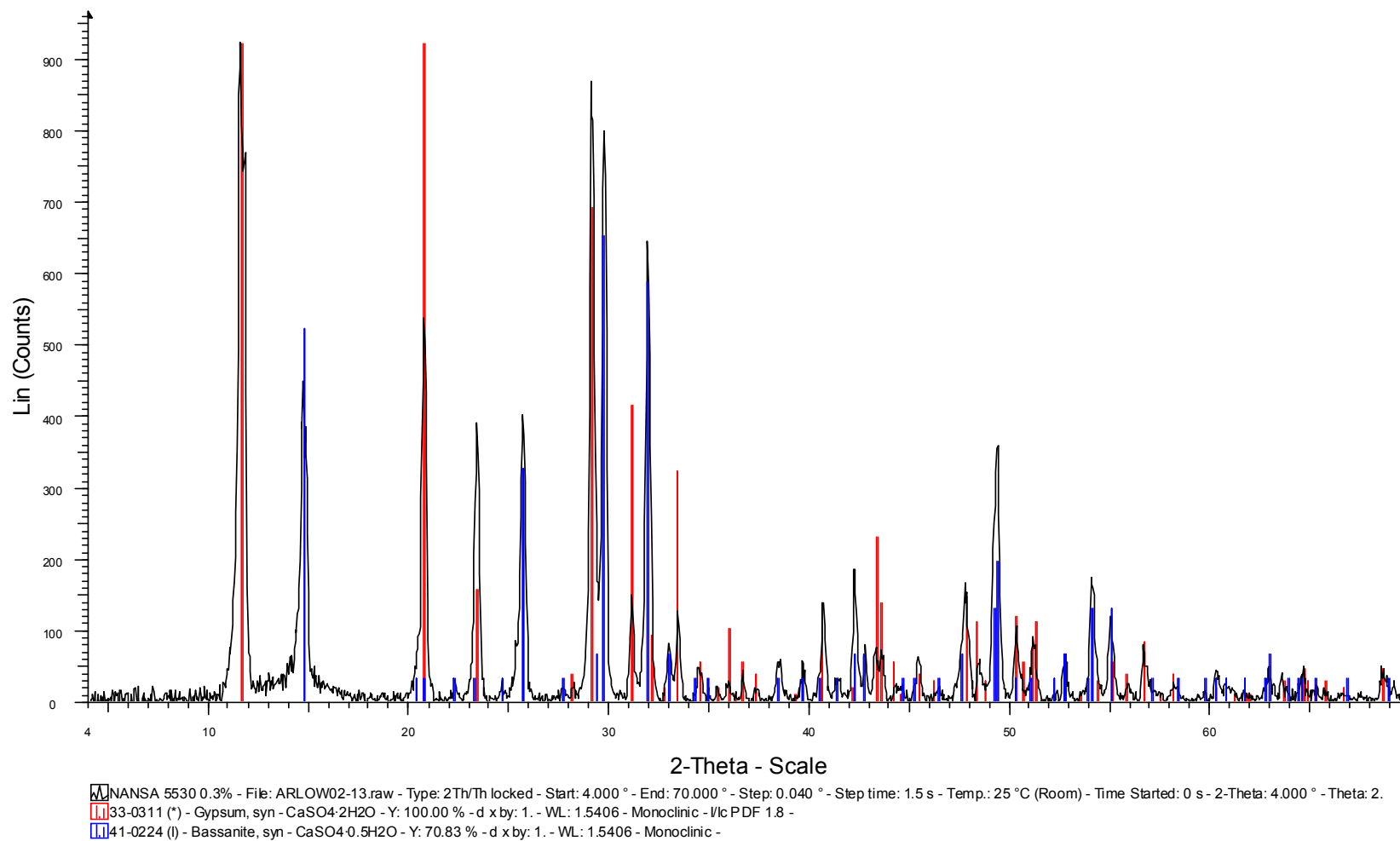
**XRD results of surfactant experiment using Calcium gluconate monohydrate**



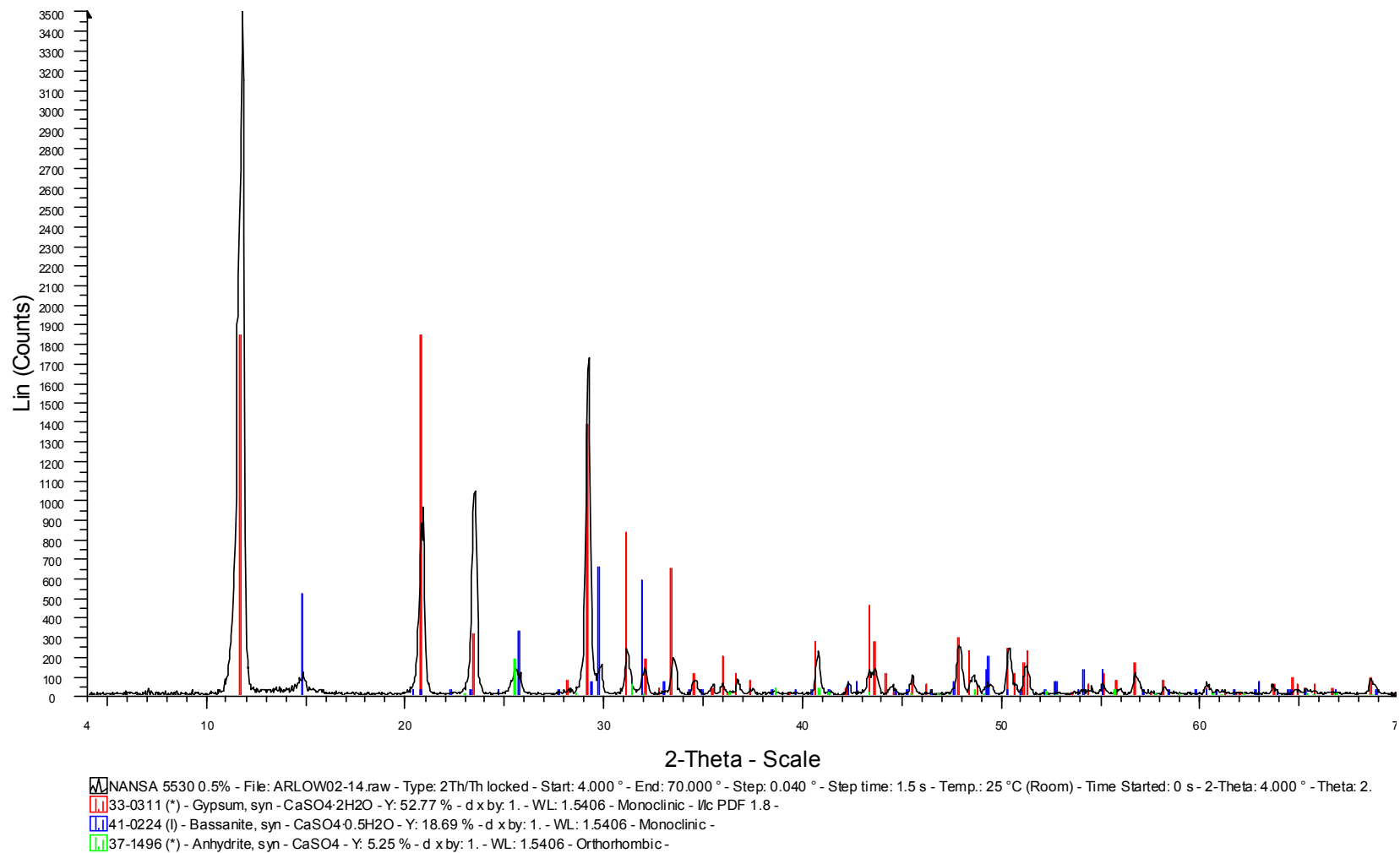
**XRD results of surfactant experiment using 0.20 % Nansa SS30 (30 minute stirring)**



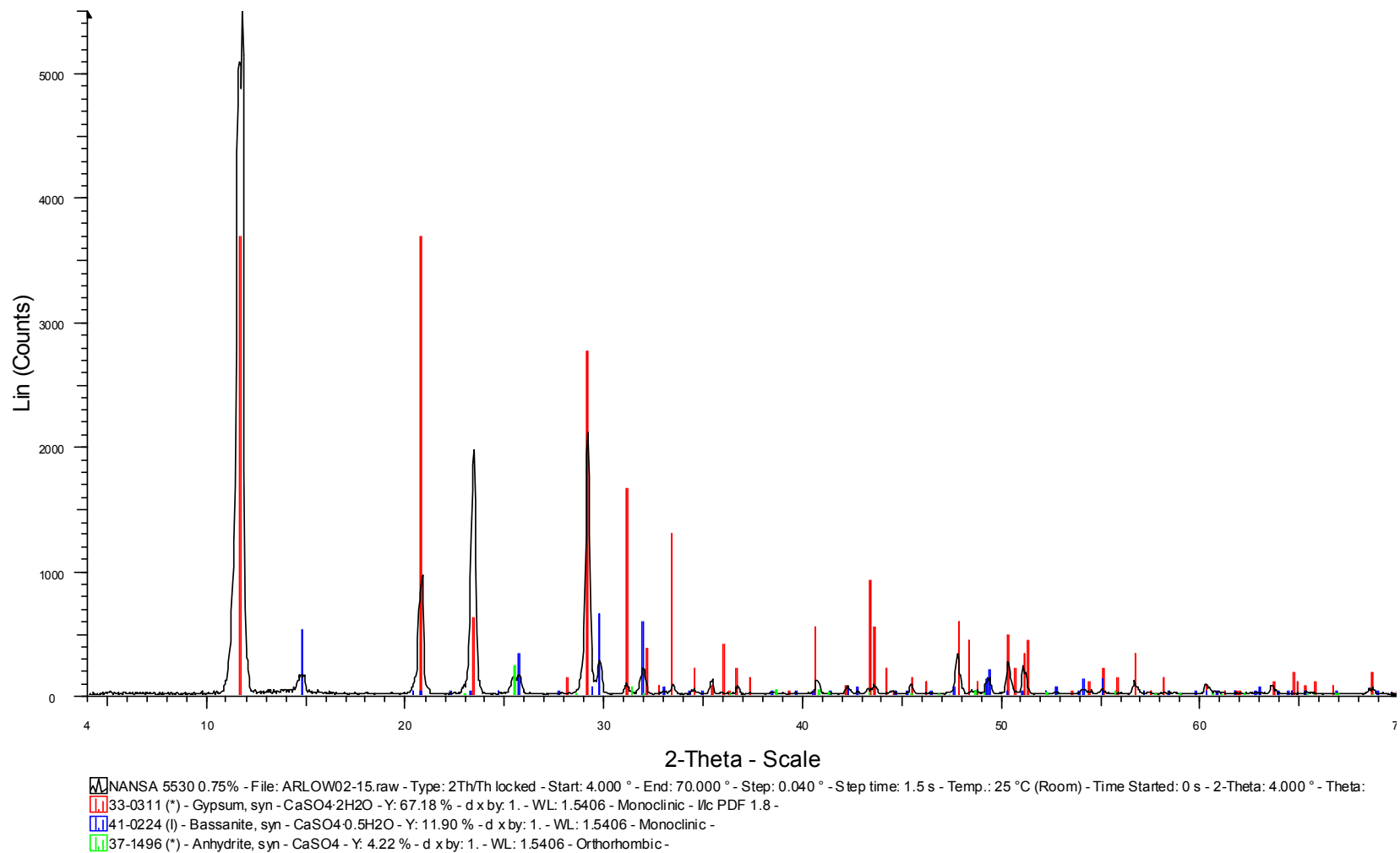
**XRD results of surfactant experiment using 0.10 % Nansa SS30 (30 minute stirring)**



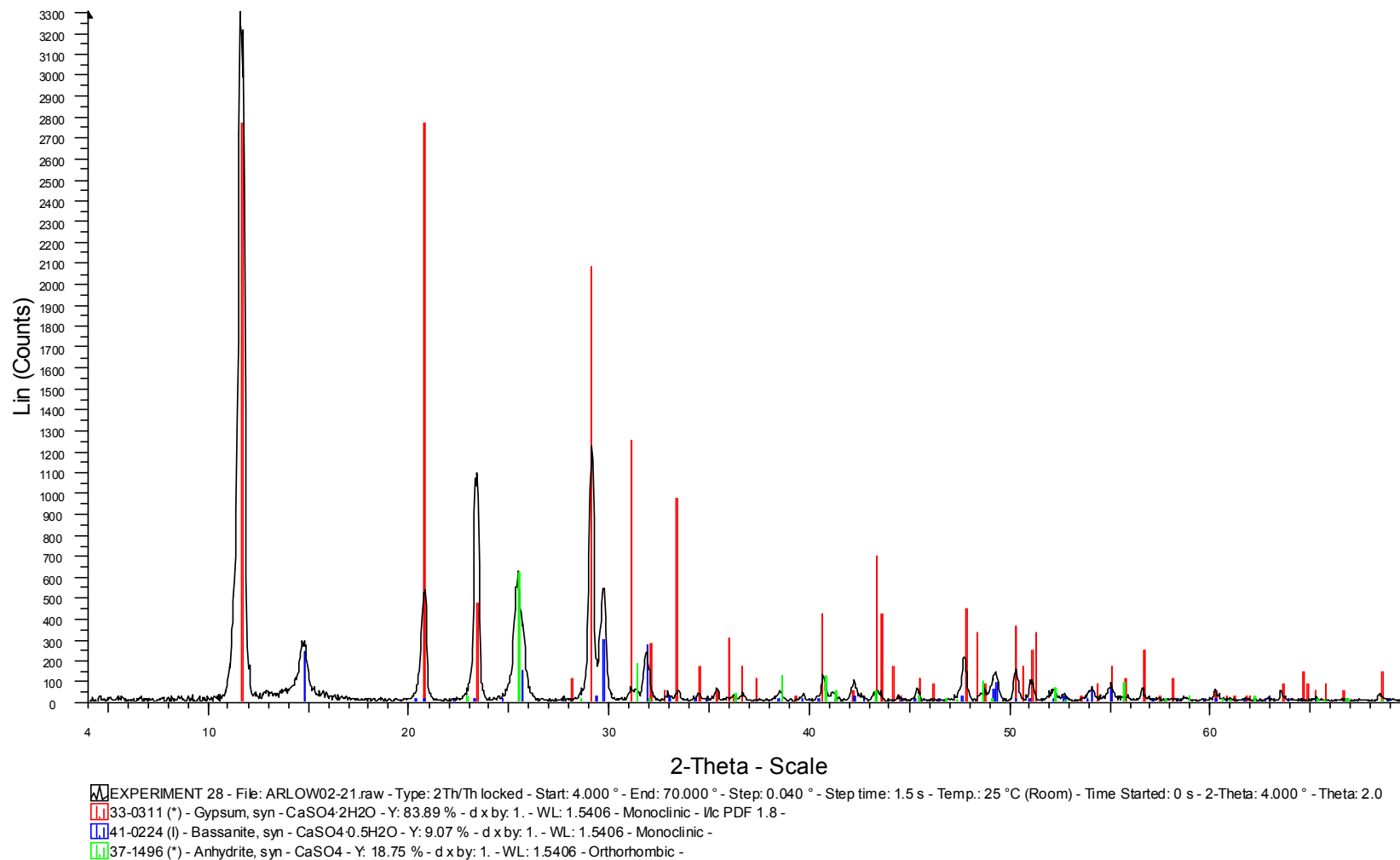
**XRD results of surfactant experiment using 0.30 % Nansa SS30 (30 minute stirring)**



**XRD results of surfactant experiment using 0.50 % Nansa SS30 (30 minute stirring)**

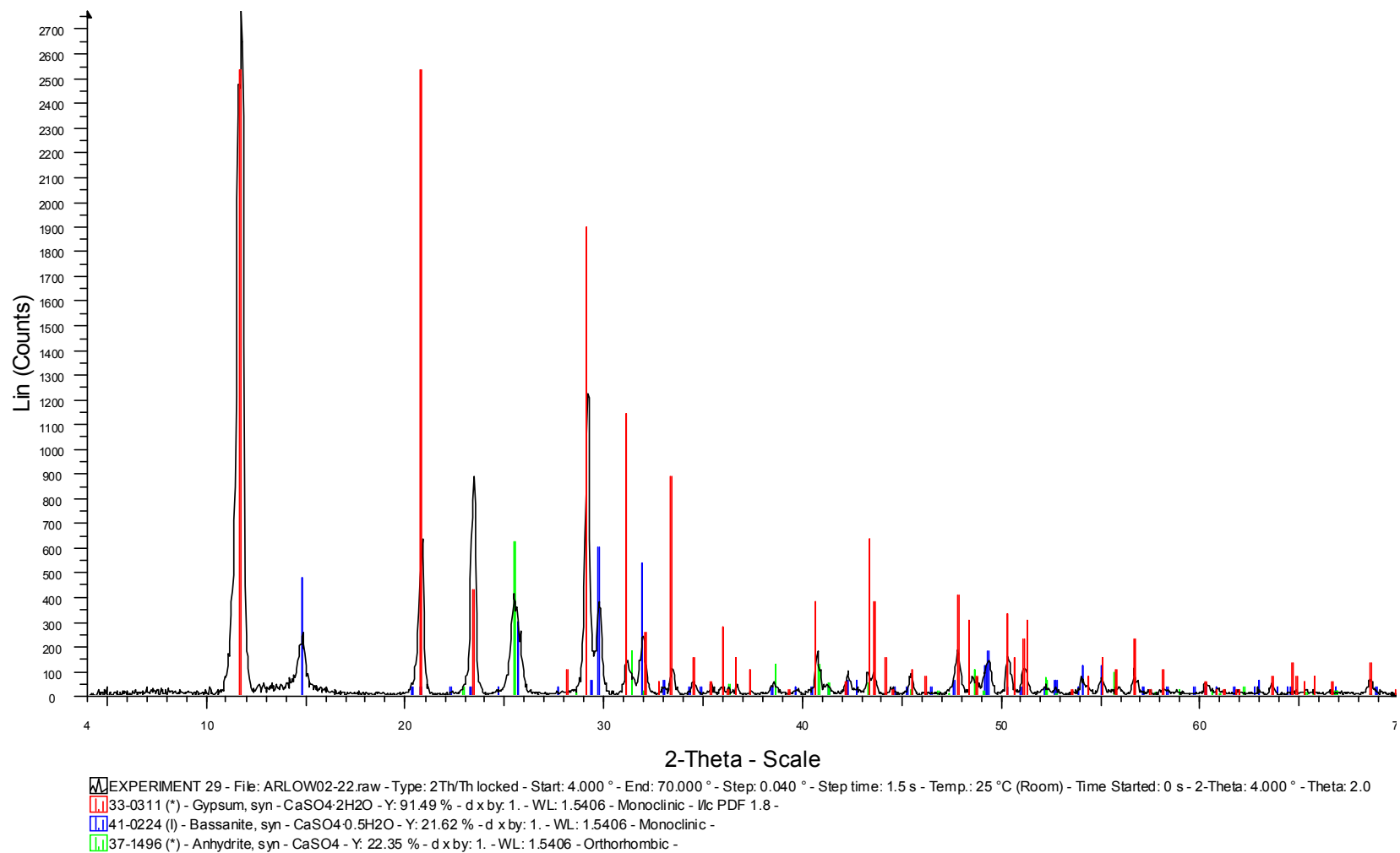


**XRD results of surfactant experiment using 0.75 % Nansa SS30 (30 minute stirring)**

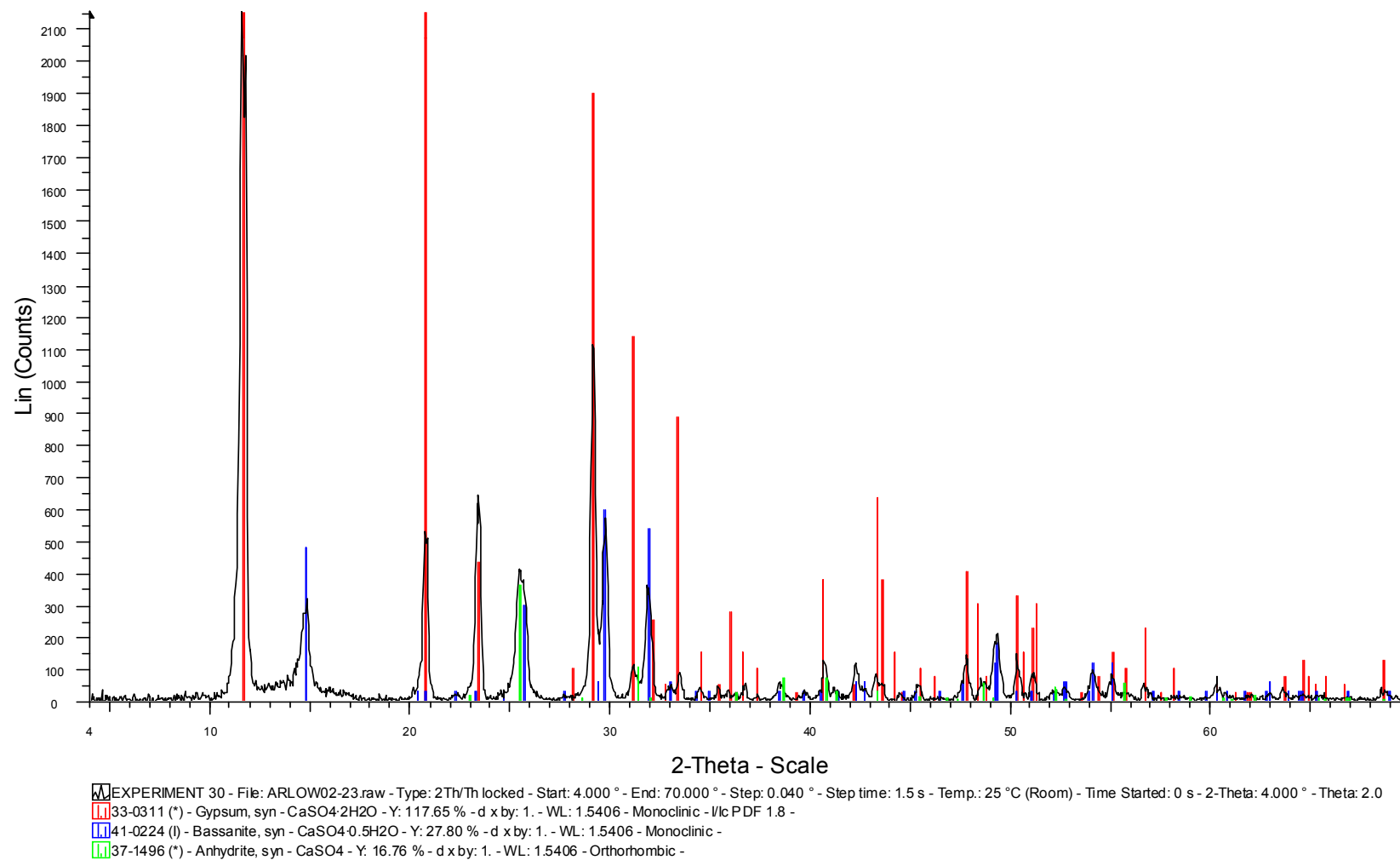


**XRD results of surfactant experiment using 1.00 % Nansa SS30 (30 minute stirring)**

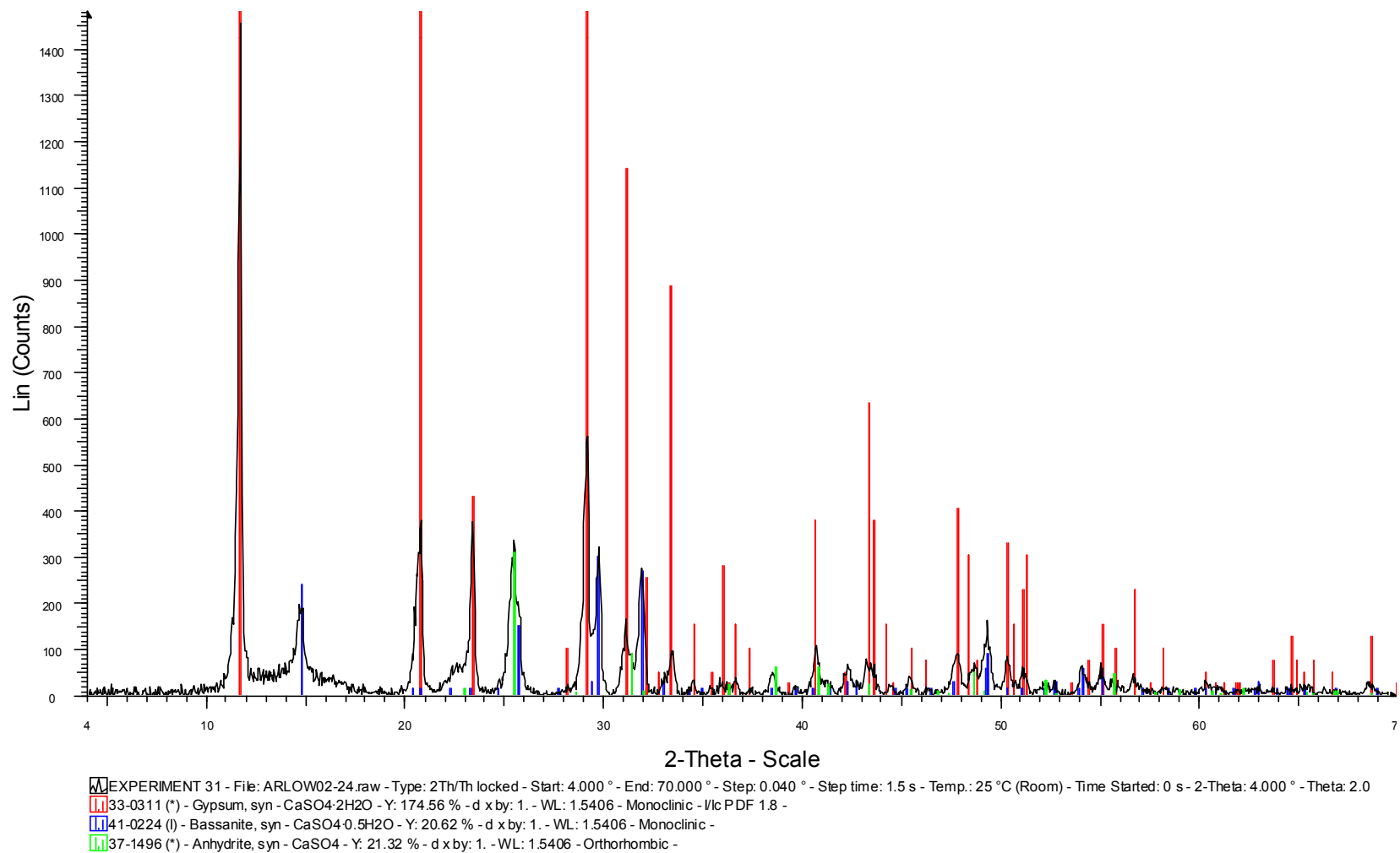




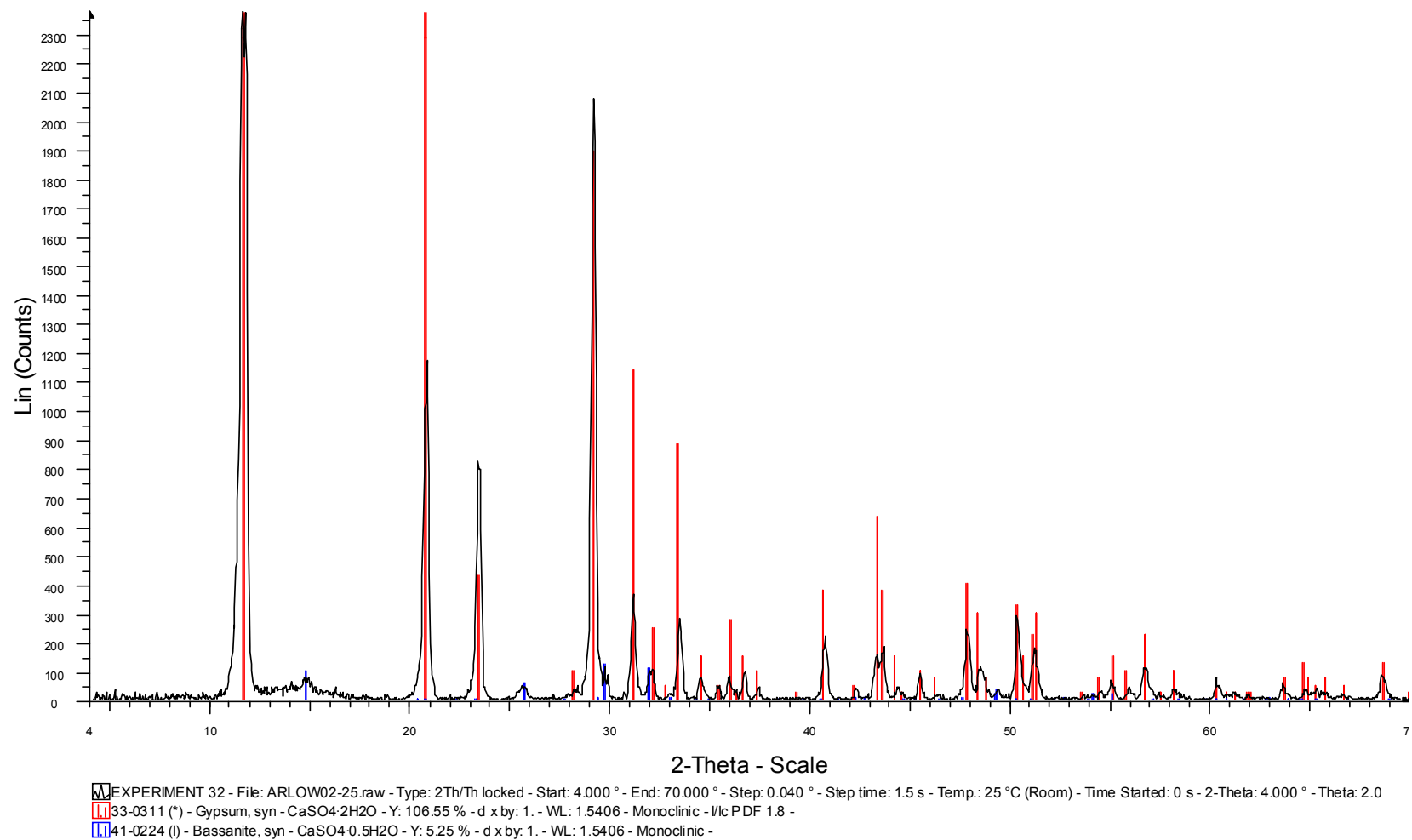
**XRD results of surfactant experiment using 1.50 % Nansa SS30 (30 minute stirring)**



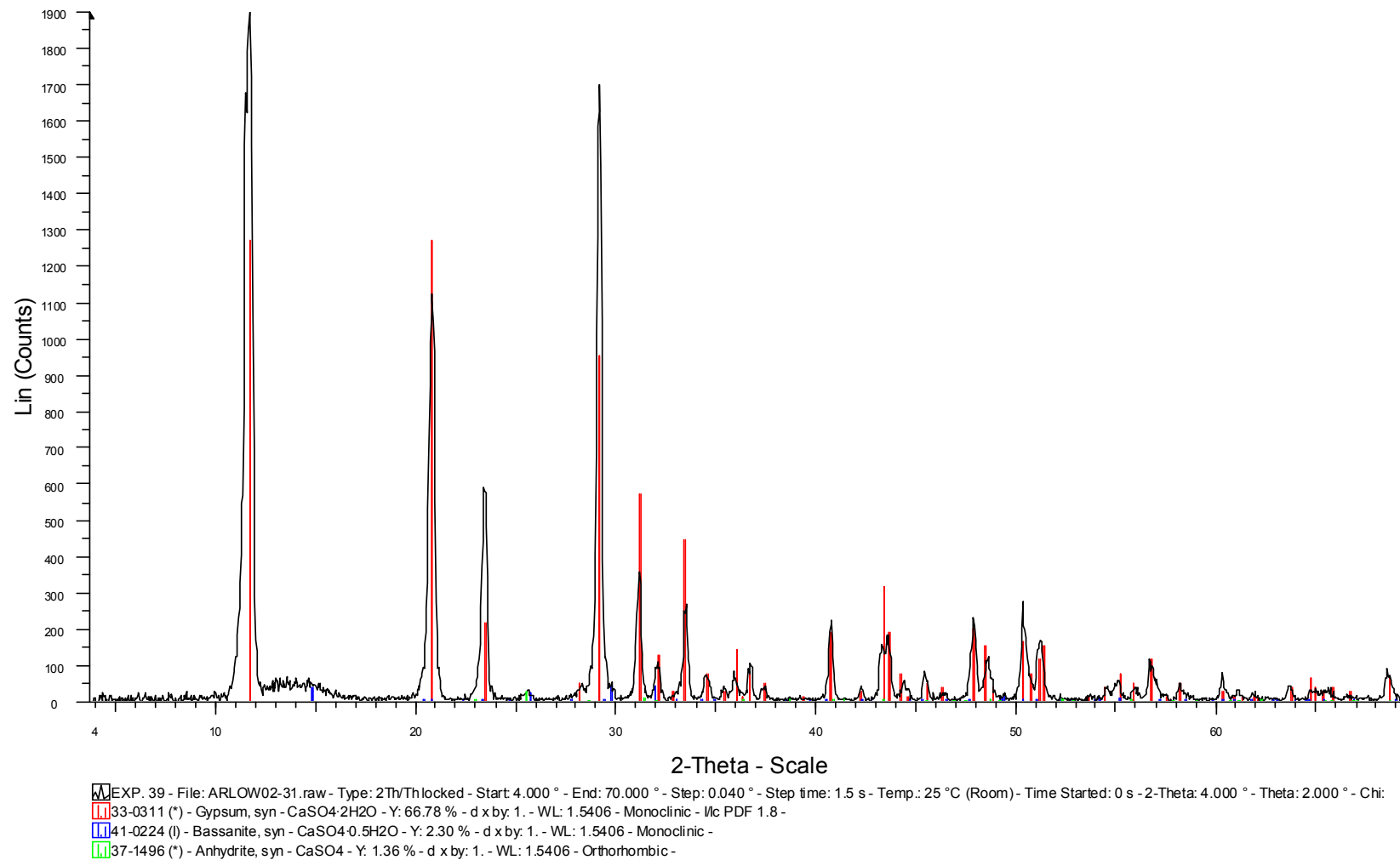
**XRD results of surfactant experiment using 2.00 % Nansa SS30 (30 minute stirring)**



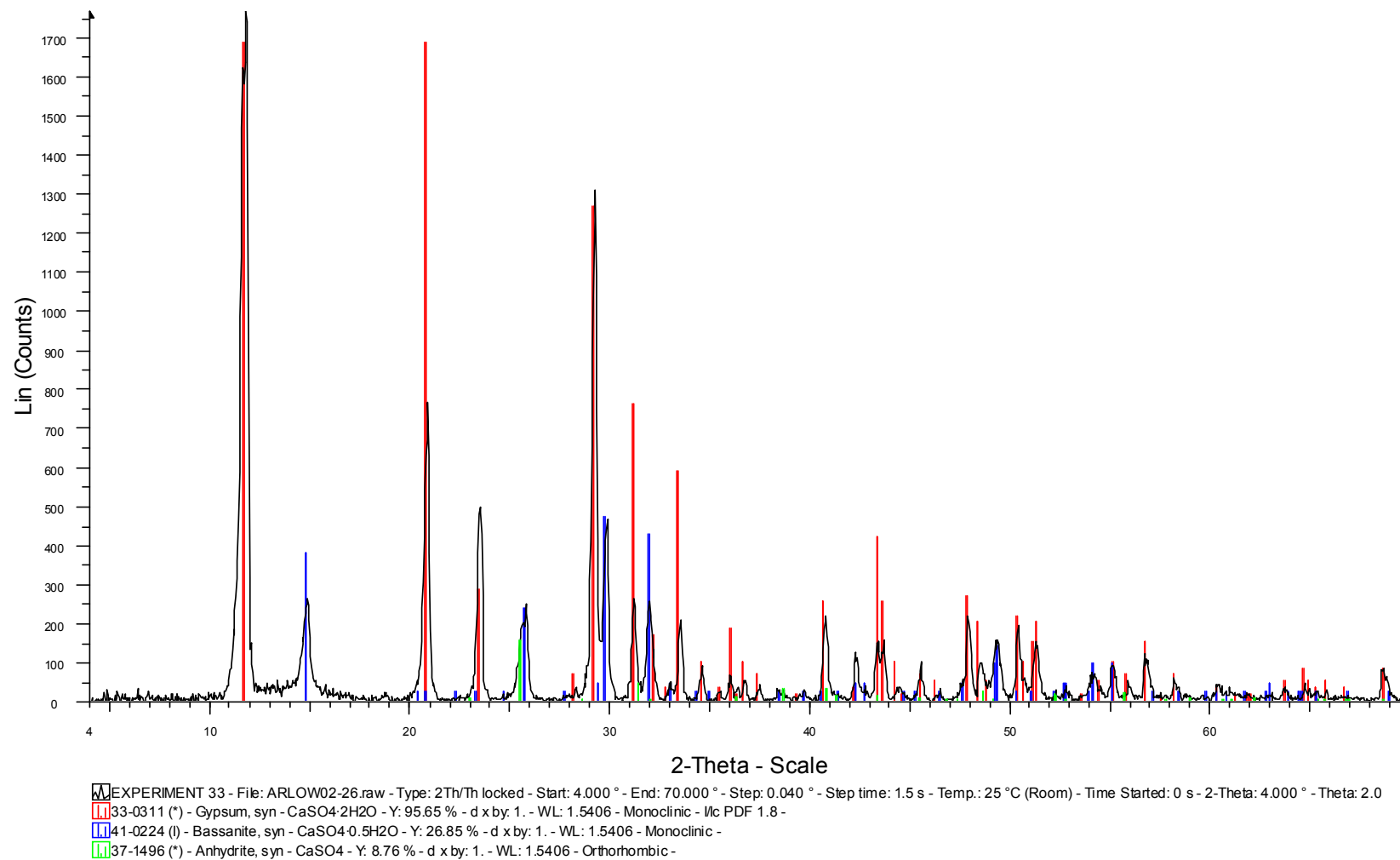
**XRD results of surfactant experiment using 2.50 % Nansa SS30 (30 minute stirring)**



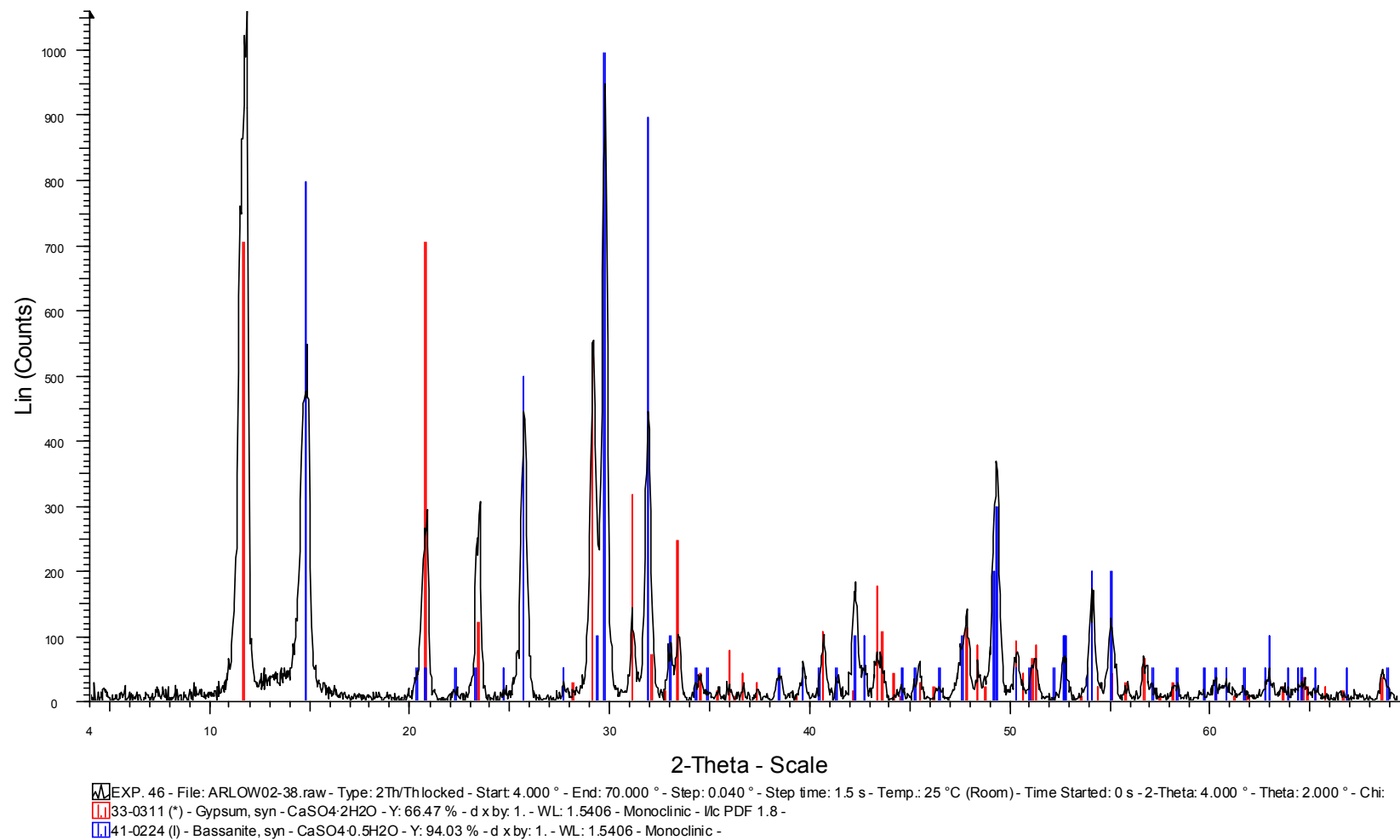
**XRD results of surfactant experiment using 3.00 % Nansa SS30 (30 minute stirring)**



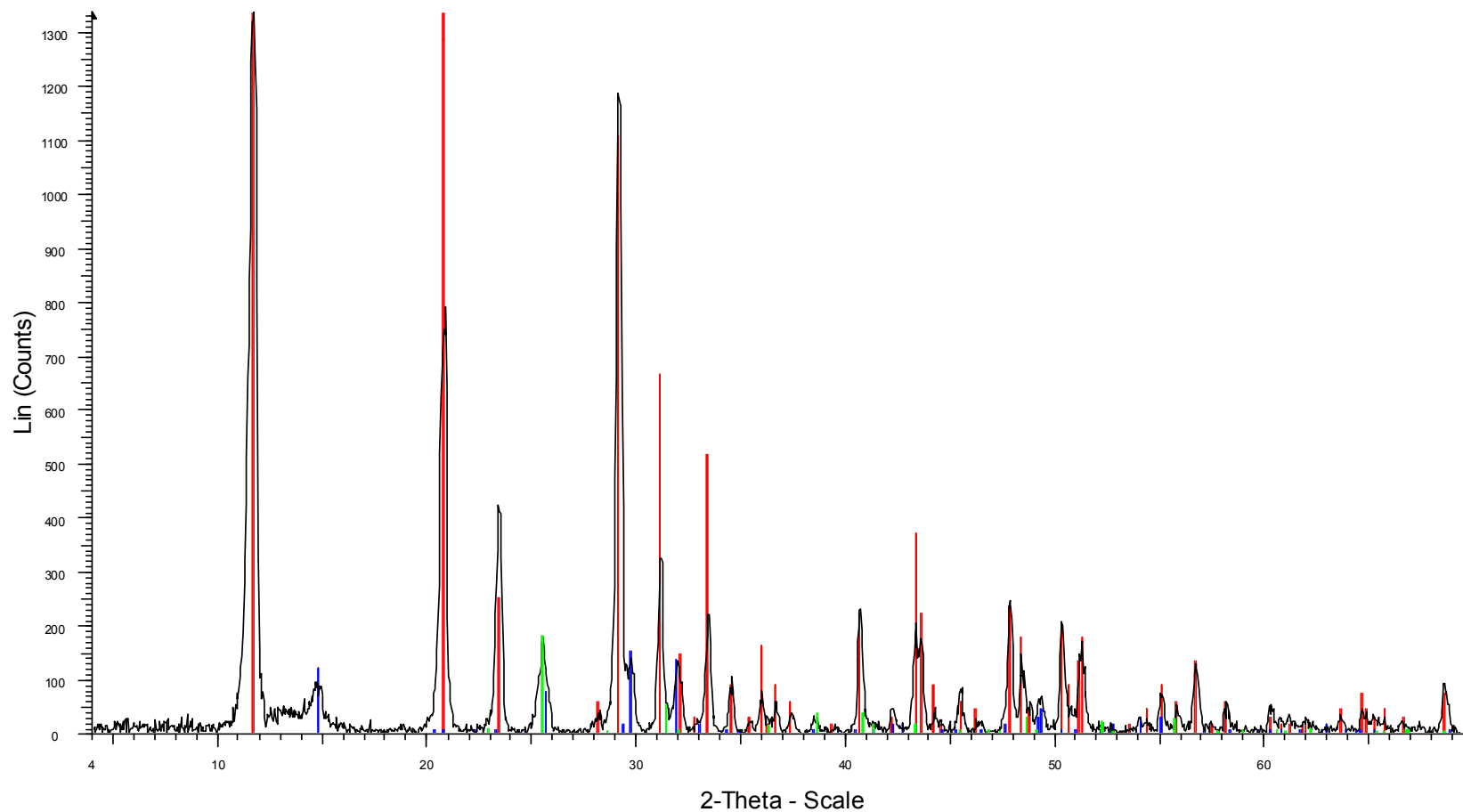
**XRD results of surfactant experiment using 5.00 % Nansa SS30 (30 minute stirring)**



**XRD results of surfactant experiment using 0.69 % Nansa SS30 (24 hour stirring)**



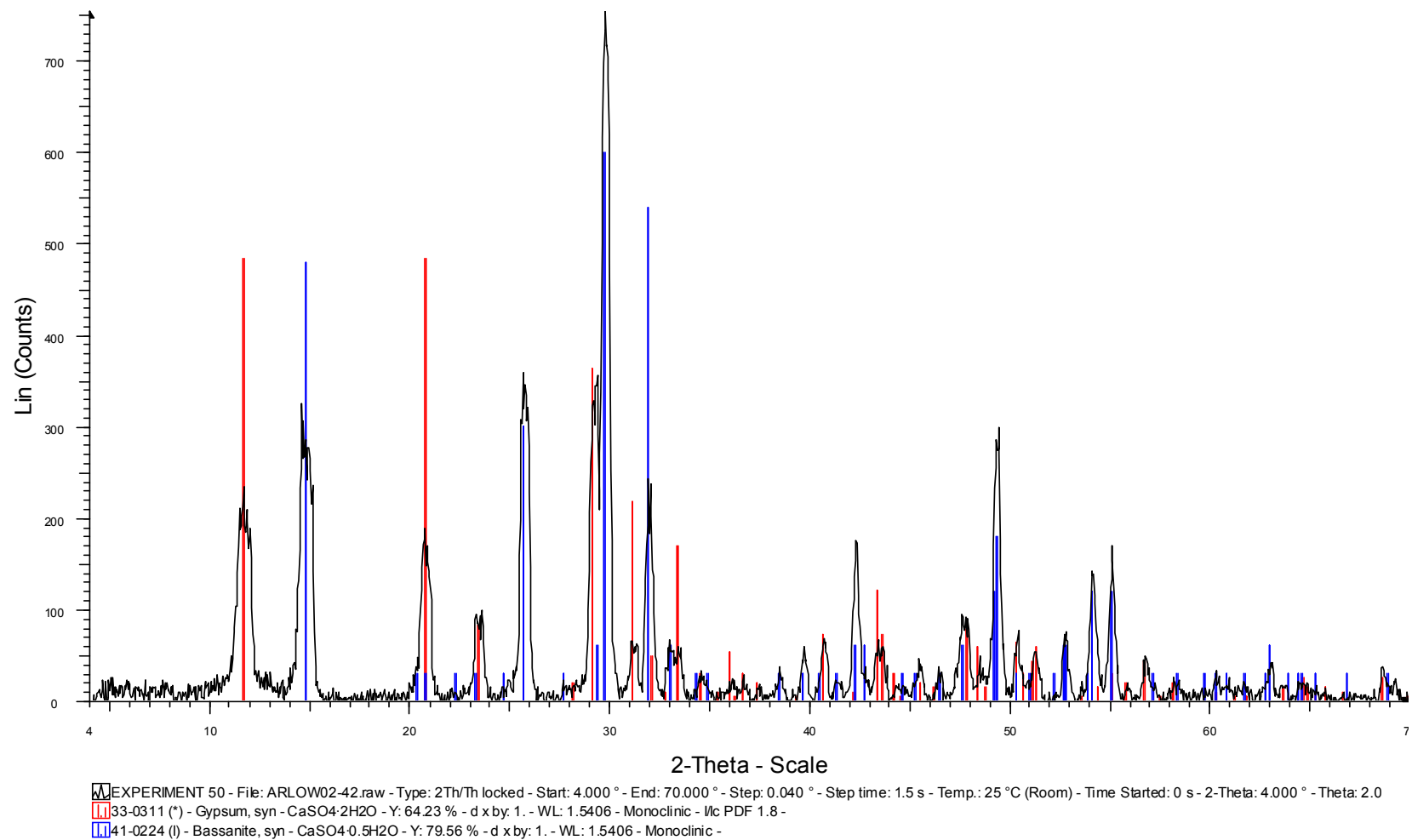
**XRD results of surfactant experiment using 1.40 % Nansa SS30 (24 hour stirring)**



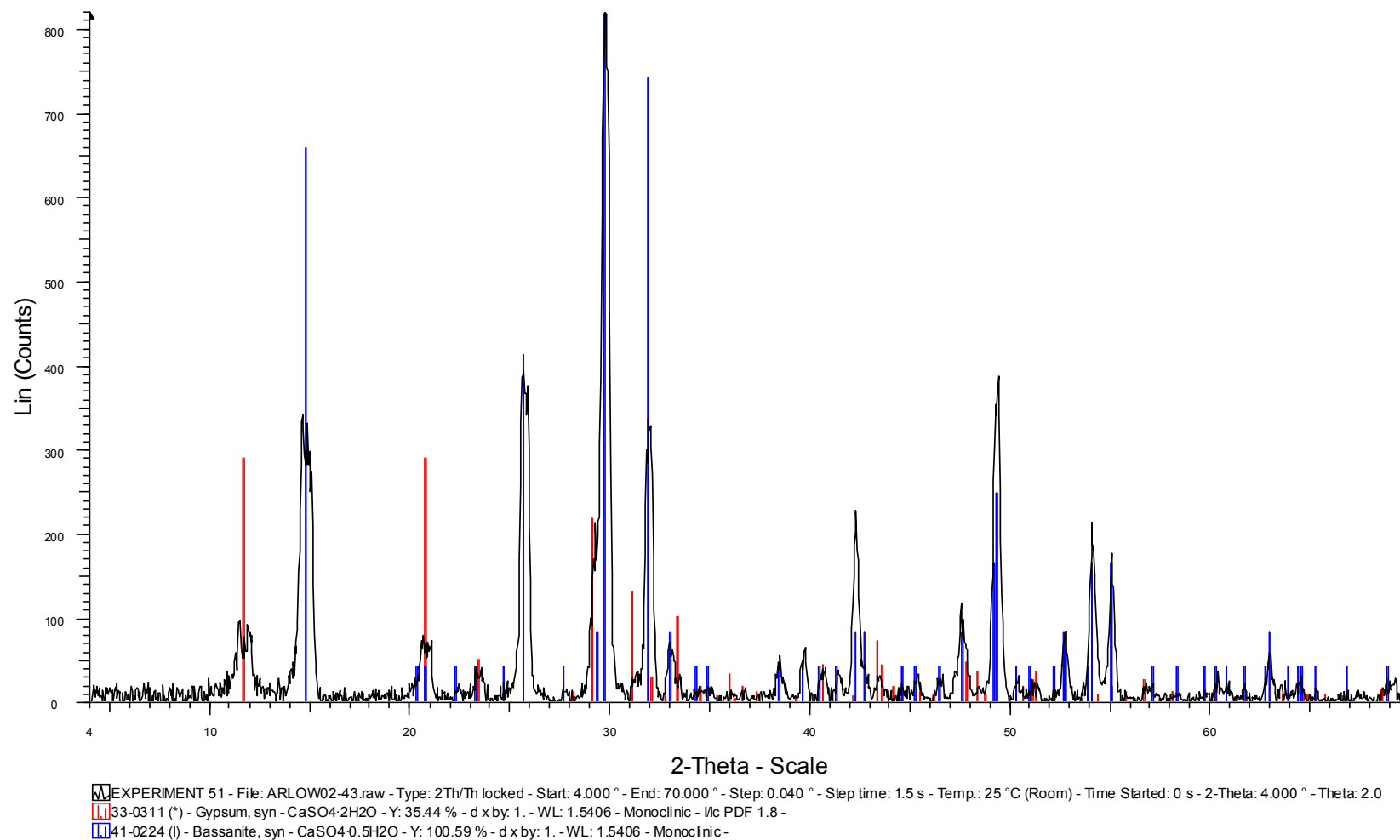
EXP. 47 - File: ARLOW02-39.raw - Type: 2Th/Thlocked - Start: 4.000 ° - End: 70.000 ° - Step: 0.040 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Time Started: 0 s - 2-Theta: 4.000 ° - Theta: 2.000 ° - Chi:  
33-0311 (\*) - Gypsum, syn - CaSO<sub>4</sub>·2H<sub>2</sub>O - Y: 110.56 % - d x by: 1. - WL: 1.5406 - Monoclinic - I/c PDF 1.8 -  
41-0224 (I) - Bassanite, syn - CaSO<sub>4</sub>·0.5H<sub>2</sub>O - Y: 11.21 % - d x by: 1. - WL: 1.5406 - Monoclinic -  
37-1496 (\*) - Anhydrite, syn - CaSO<sub>4</sub> - Y: 13.51 % - d x by: 1. - WL: 1.5406 - Orthorhombic -

**XRD results of surfactant experiment using 2.78 % Nansa SS30 (24 hour stirring)**

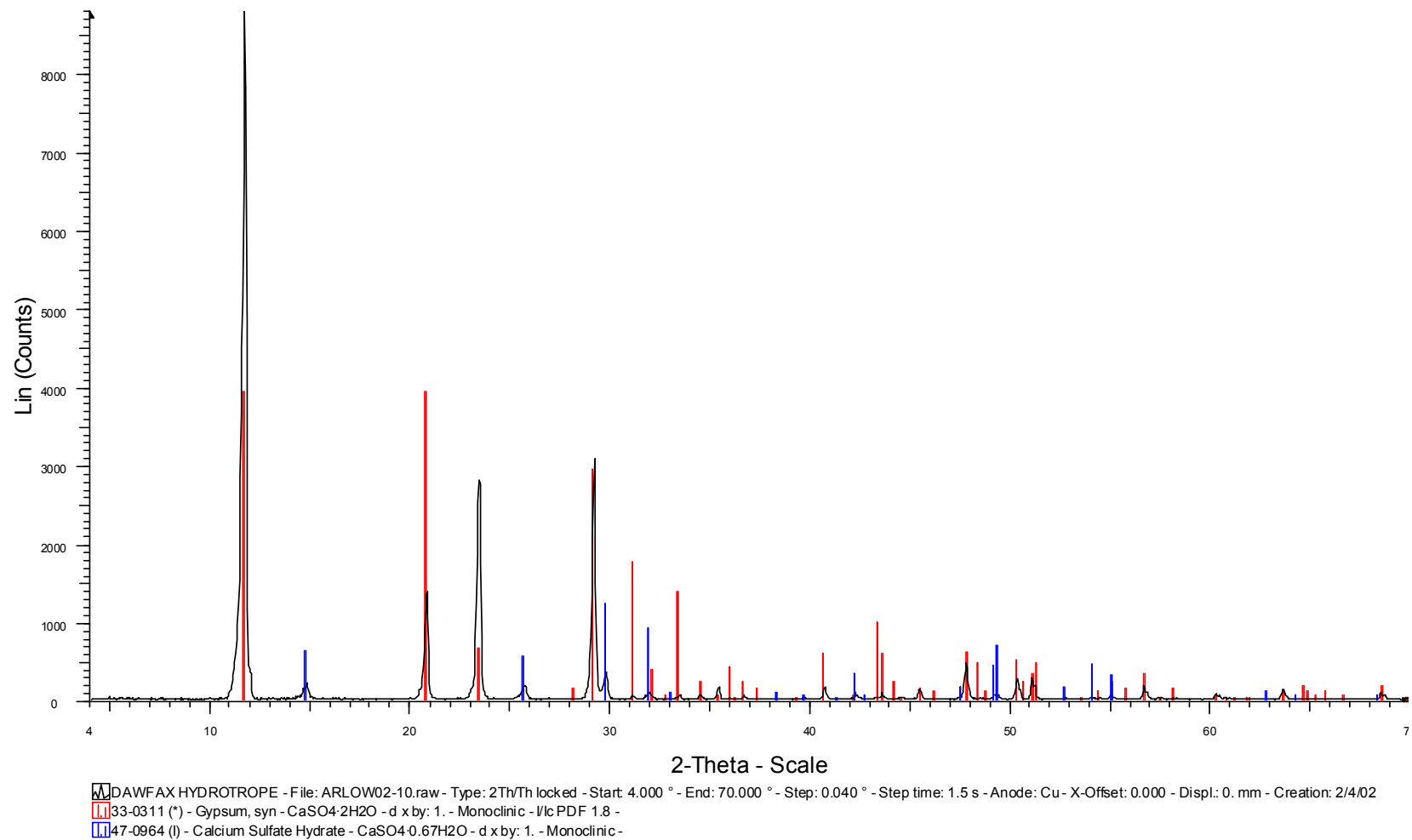




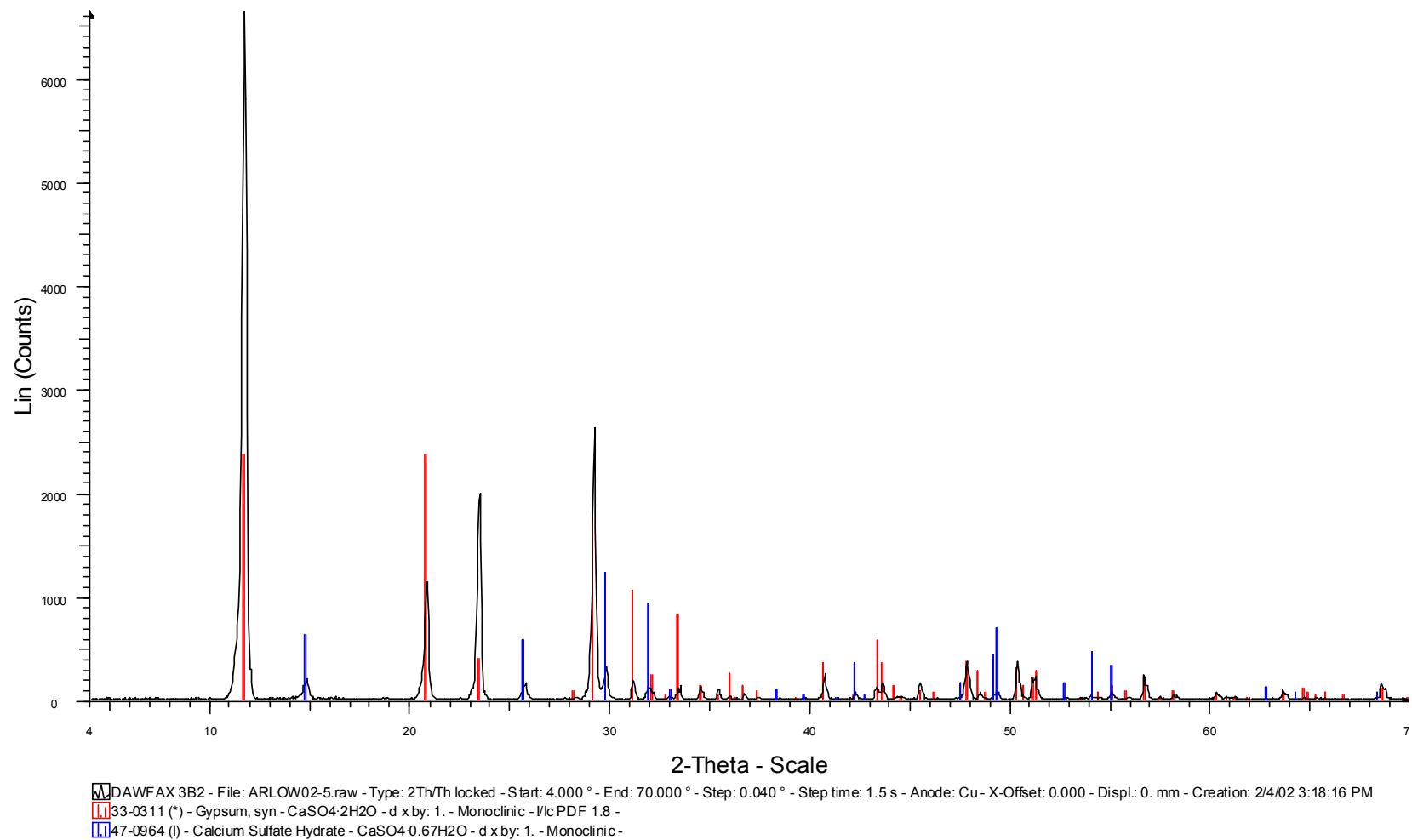
**XRD results of surfactant experiment using 6.94 % Nansa SS30 (24 hour stirring)**



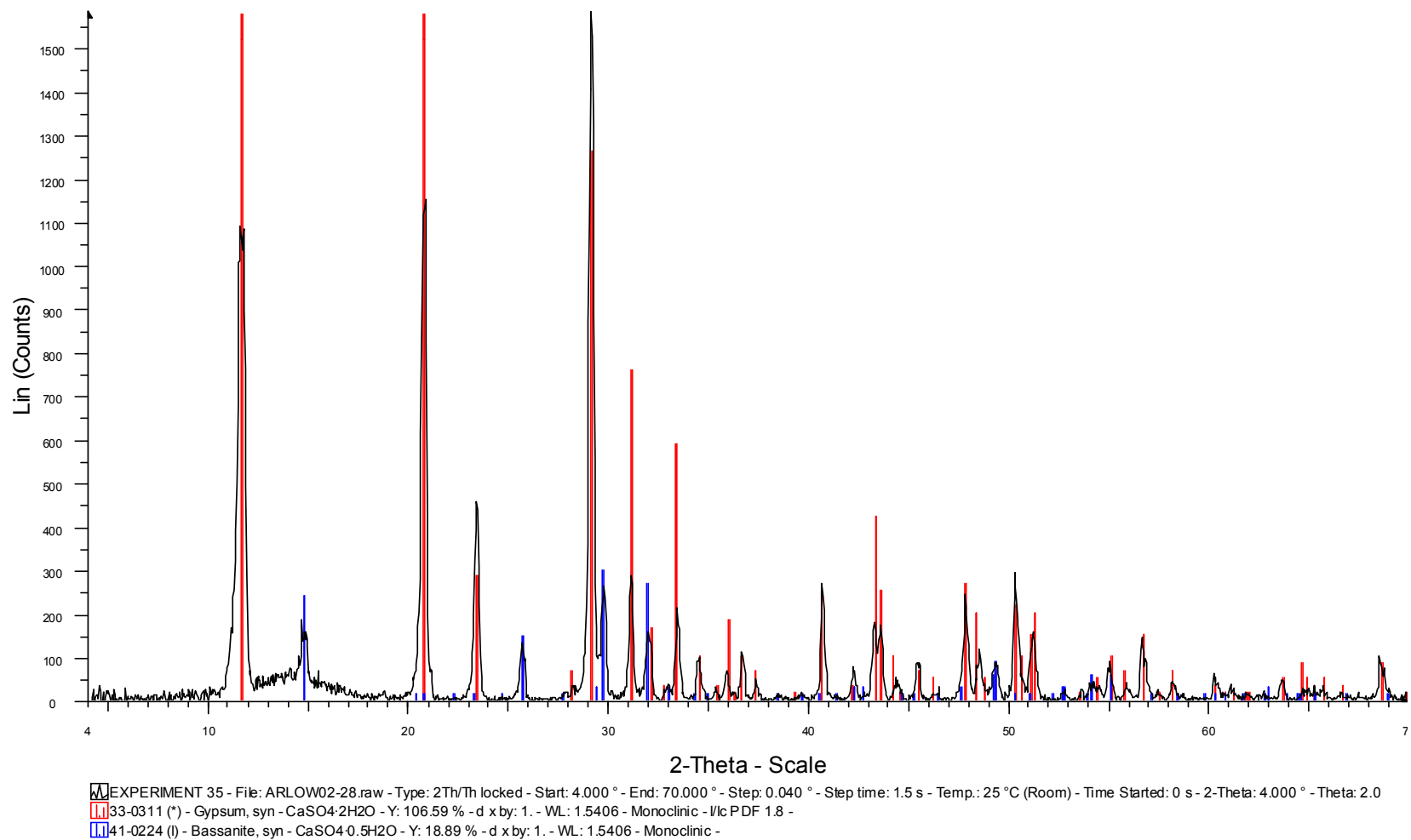
**XRD results of surfactant experiment using 14.09 % Nansa SS30 (24 hour stirring)**



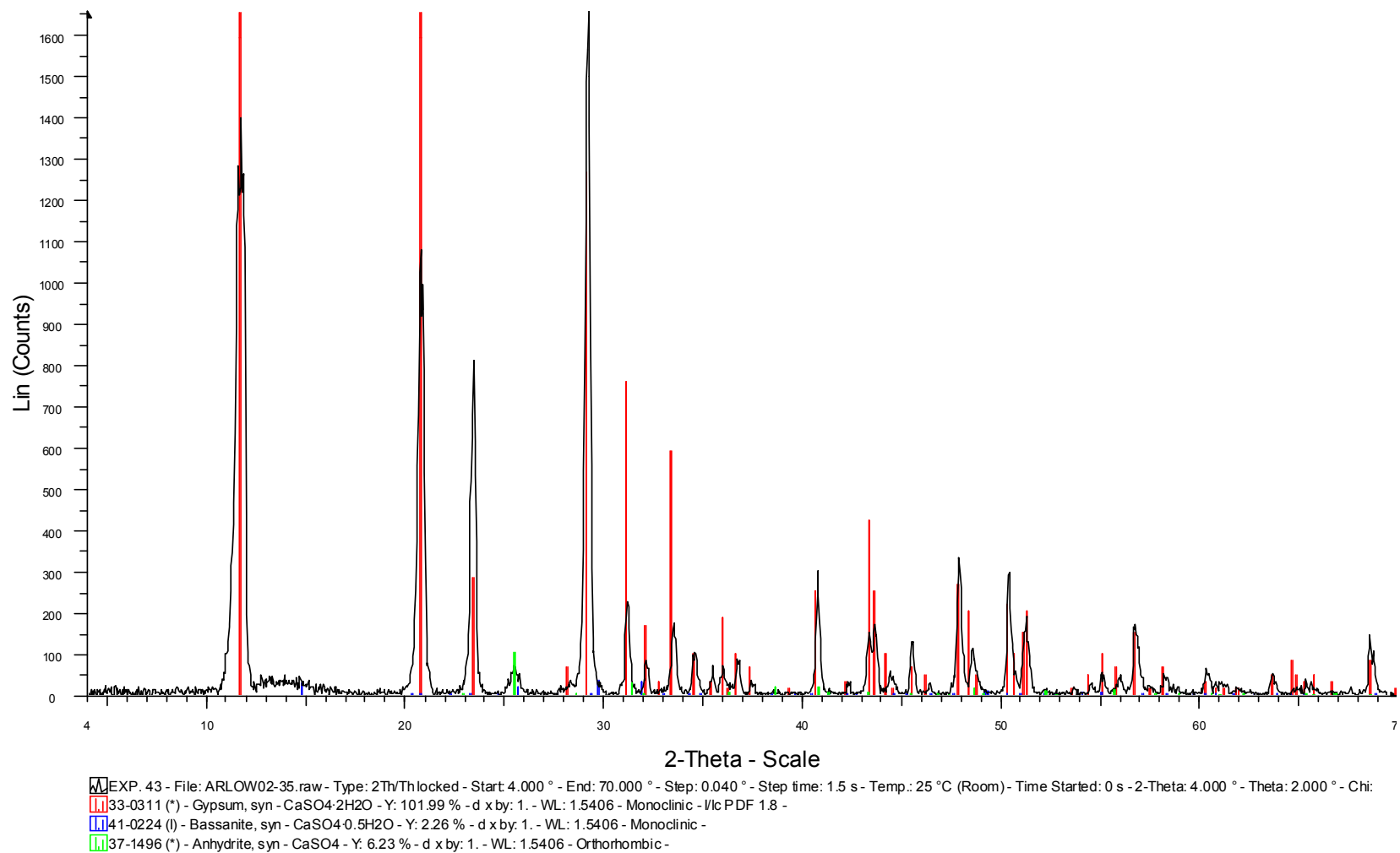
**XRD results of surfactant experiment using Dowfax Hydrotrope**



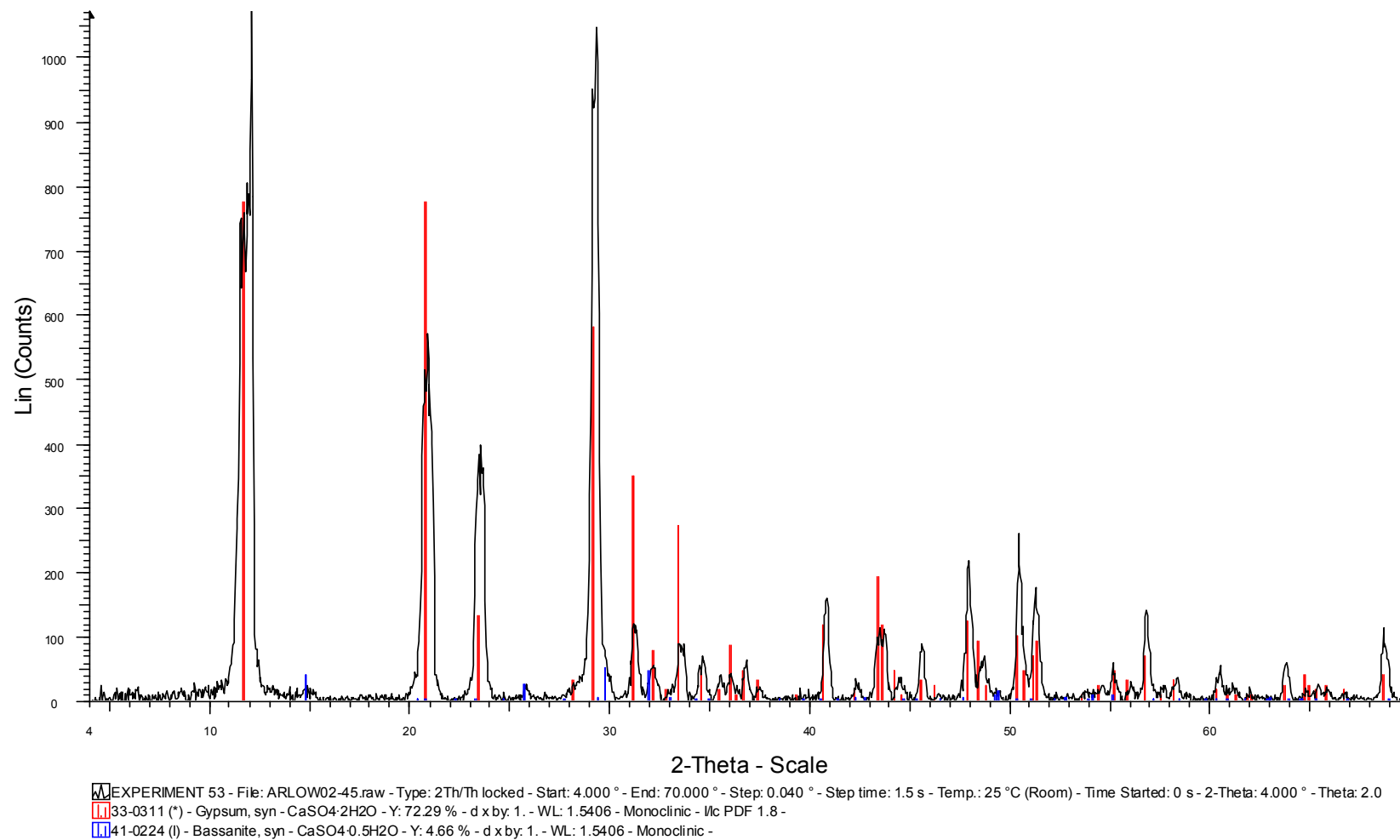
**XRD result of surfactant experiment using 0.20 % Dowfax 3B2**



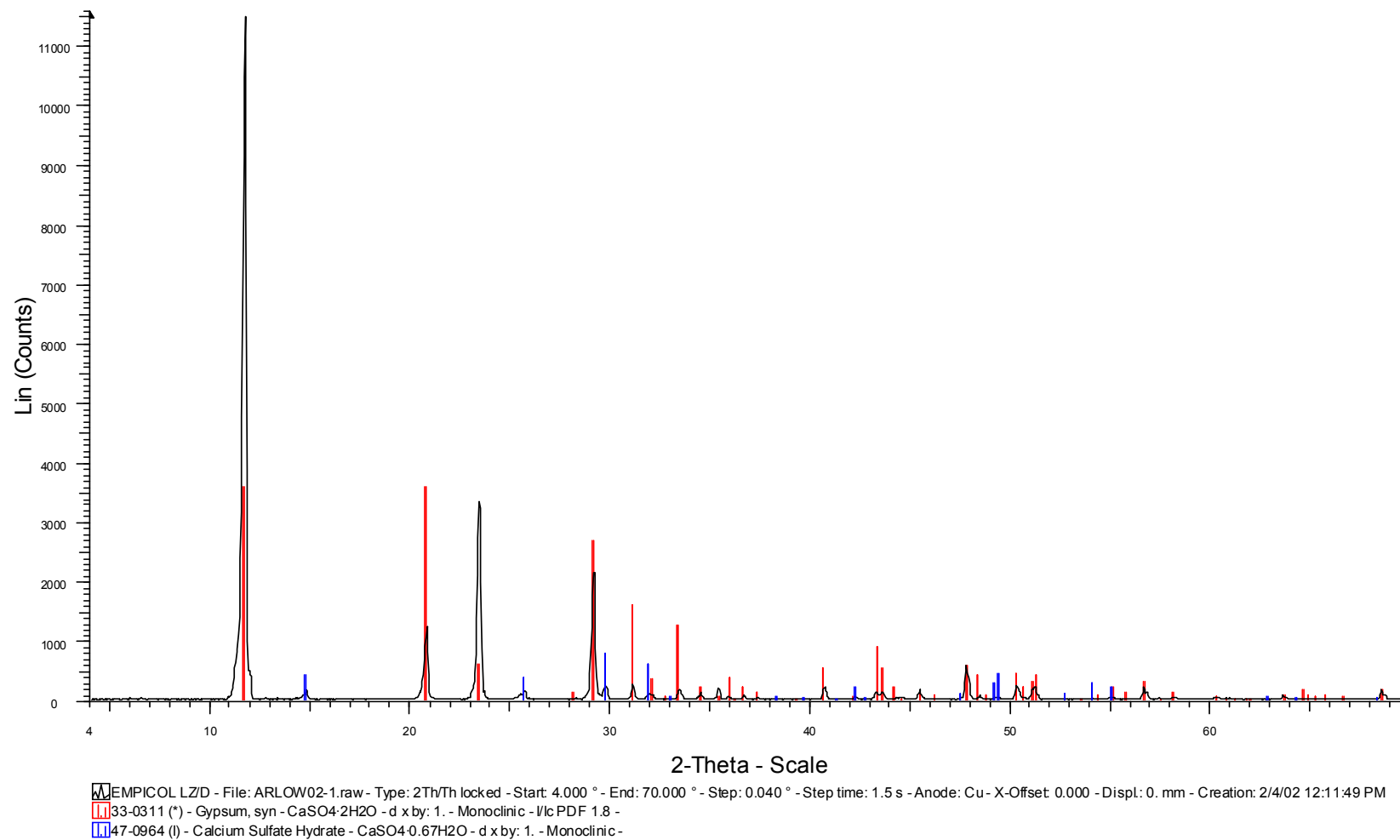
**XRD result of surfactant experiment using 1.00 % Dowfax 3B2**



**XRD result of surfactant experiment using 3.00 % Dowfax 3B2**

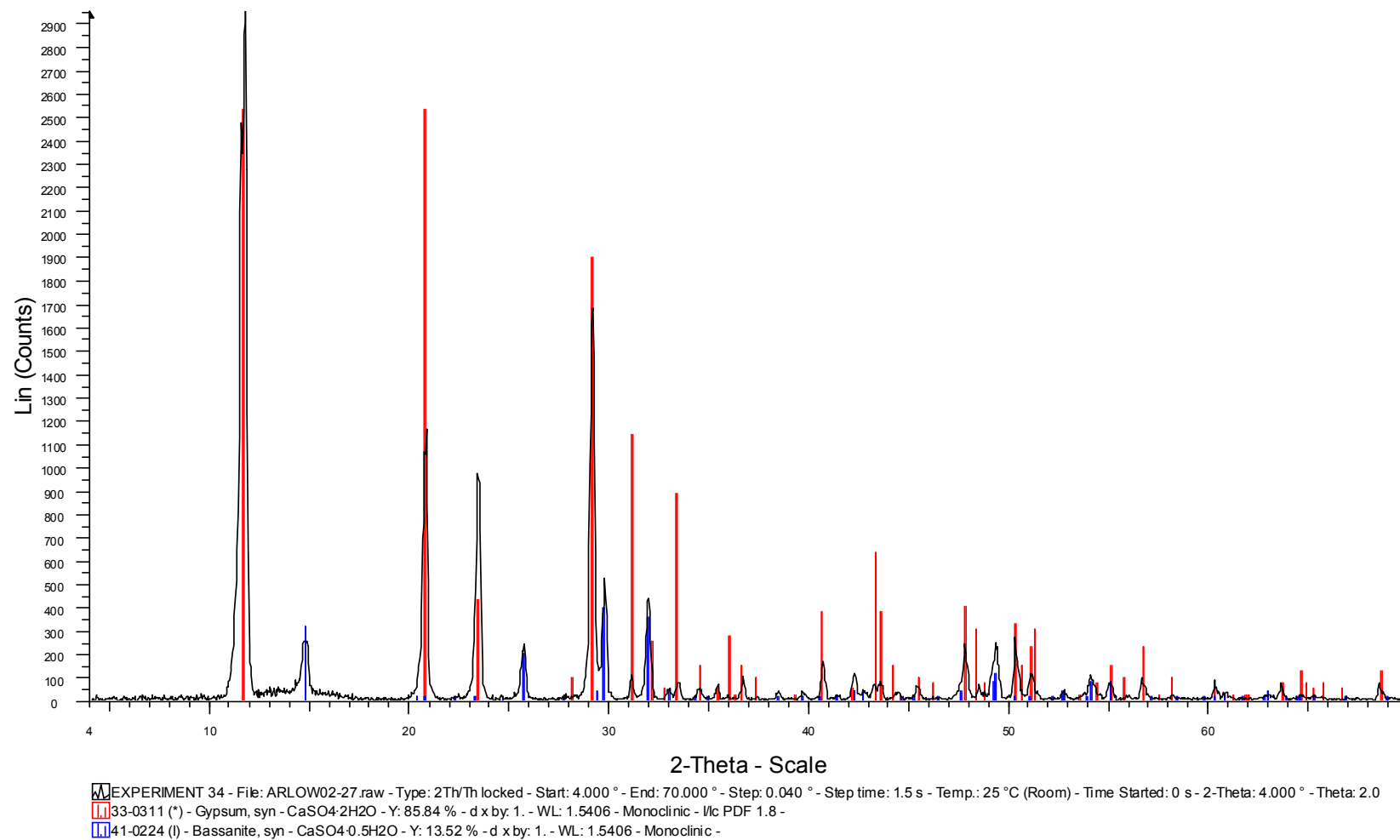


**XRD result of surfactant experiment using 5.00 % Dowfax 3B2**

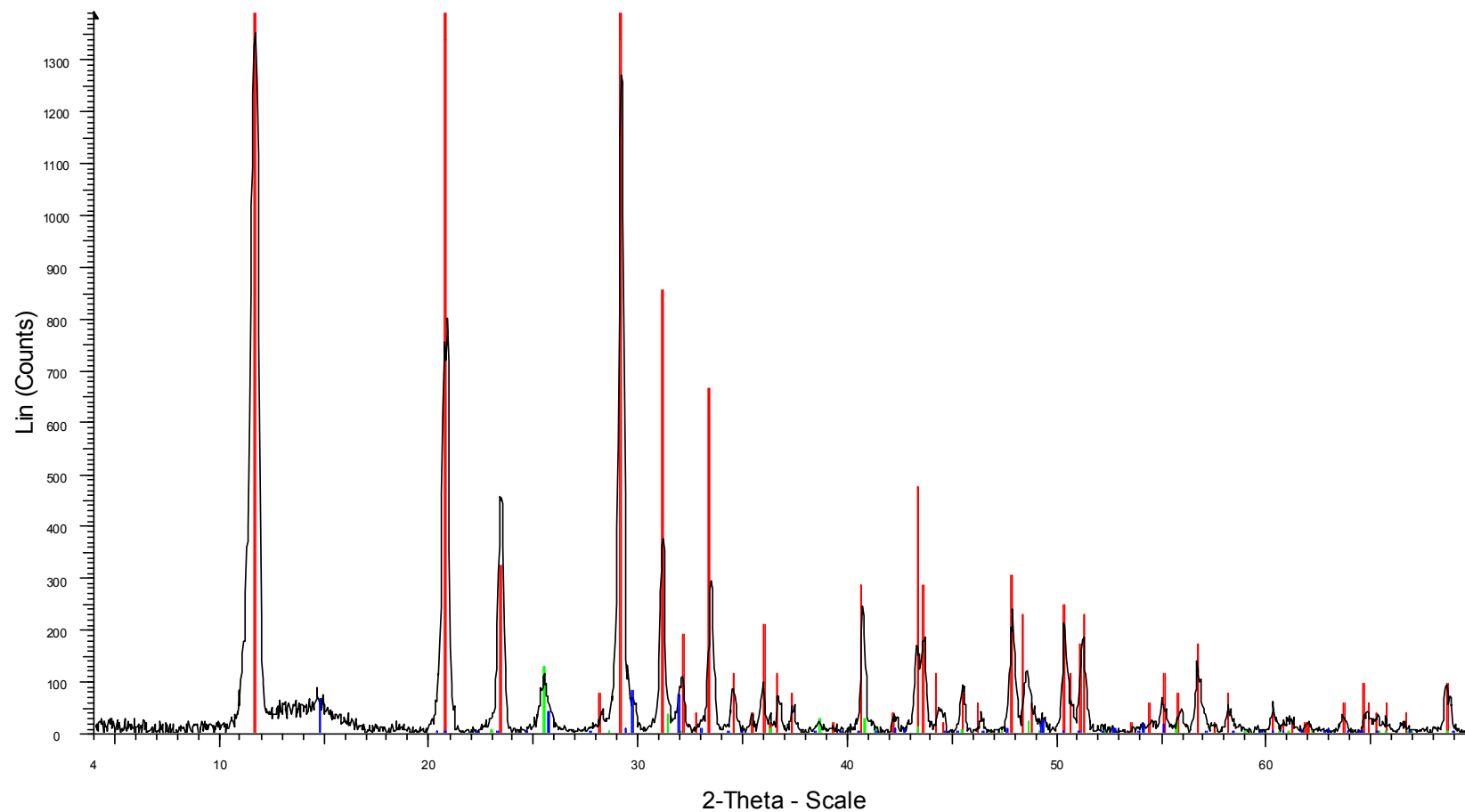


**XRD result of surfactant experiment using 0.20 % Empicol LZ/D**





**XRD result of surfactant experiment using 1.00 % Empicol LZ/D**



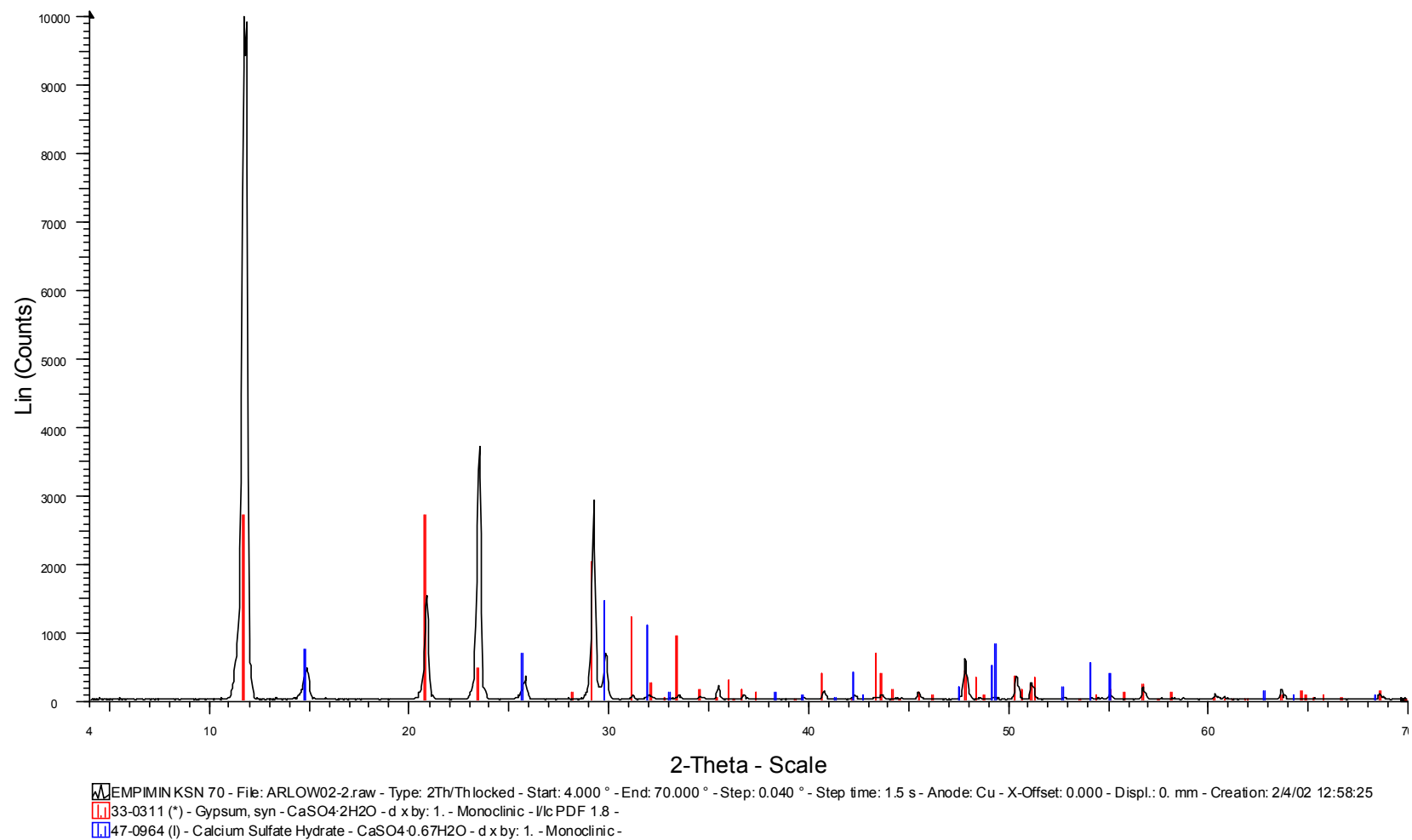
EXP. 40 - File: ARLOW02-32.raw - Type: 2Th/Thlocked - Start: 4.000 ° - End: 70.000 ° - Step: 0.040 ° - Step time: 1.5 s - Temp.: 25 °C (Room) - Time Started: 0 s - 2-Theta: 4.000 ° - Theta: 2.000 ° - Chi:

33-0311 (\*) - Gypsum, syn - CaSO<sub>4</sub>·2H<sub>2</sub>O - Y: 140.67 % - d x by: 1. - WL: 1.5406 - Monoclinic - I/Ic PDF 1.8 -

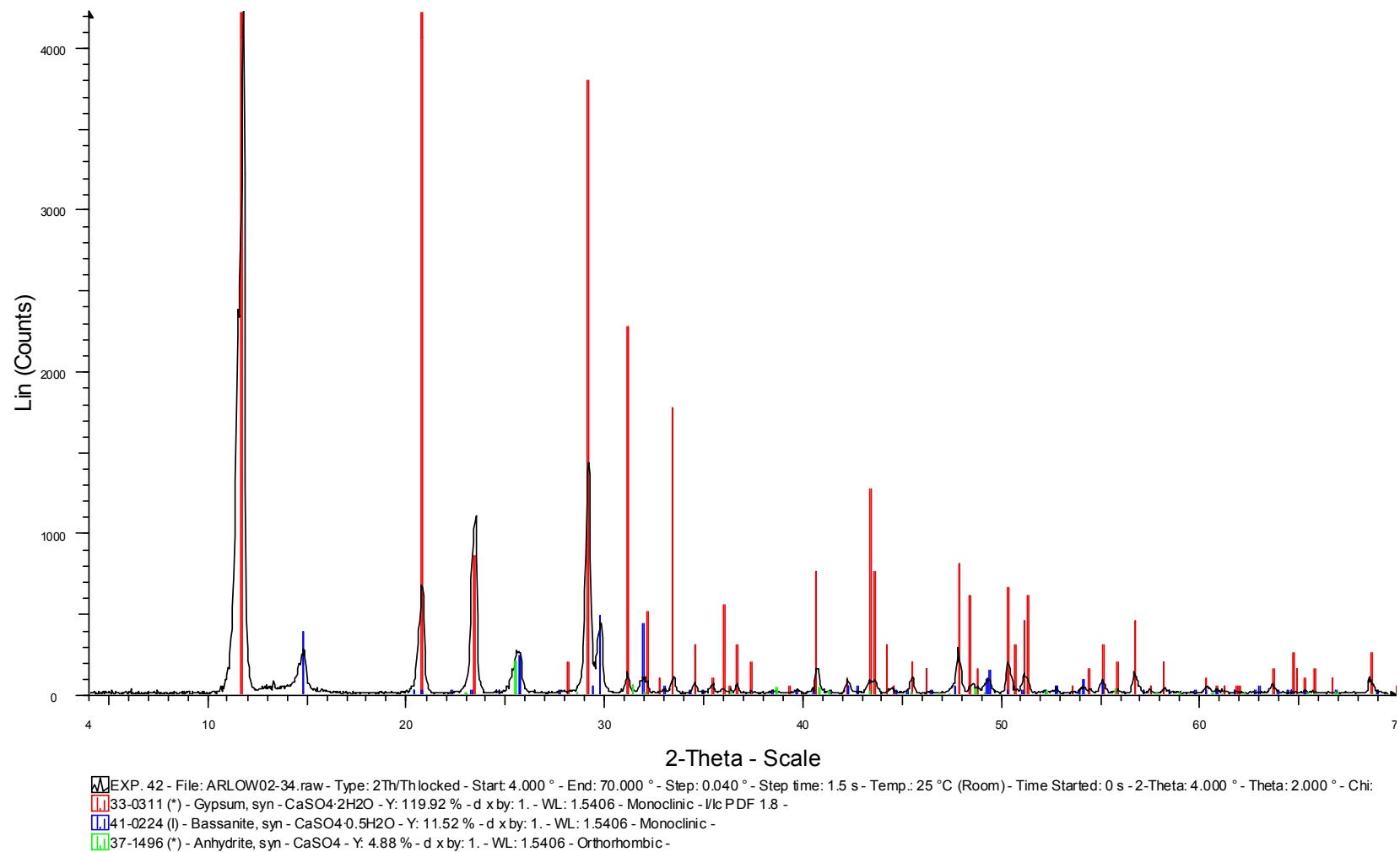
41-0224 (I) - Bassanite, syn - CaSO<sub>4</sub>·0.5H<sub>2</sub>O - Y: 6.01 % - d x by: 1. - WL: 1.5406 - Monoclinic -

37-1496 (\*) - Anhydrite, syn - CaSO<sub>4</sub> - Y: 9.55 % - d x by: 1. - WL: 1.5406 - Orthorhombic -

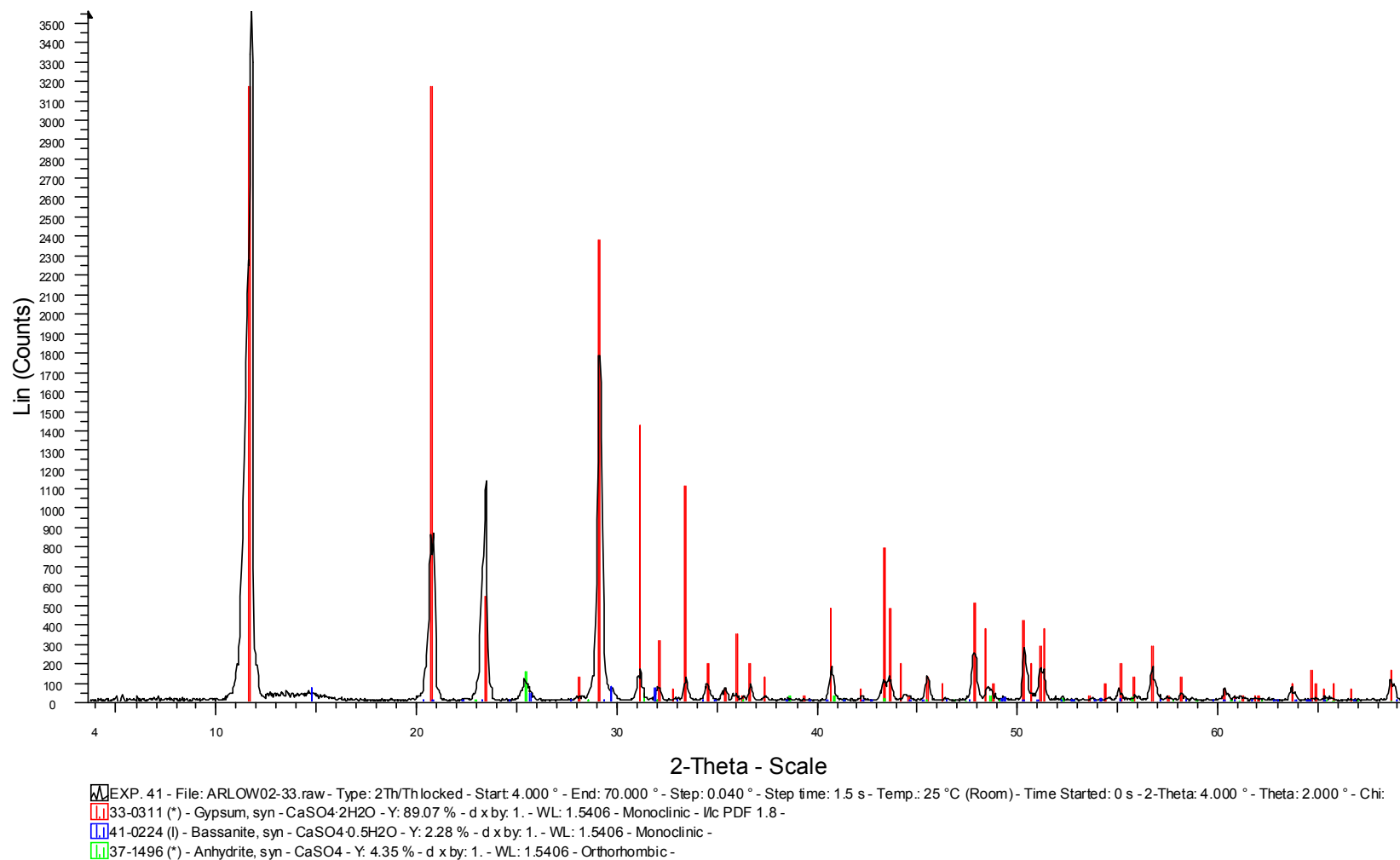
**XRD result of surfactant experiment using 3.00 % Empicol LZ/D**



**XRD result of surfactant experiment using 0.20 % Empimin KSN70**



**XRD result of surfactant experiment using 1.00 % Empimin KSN70**



**XRD result of surfactant experiment using 3.00 % Empimin KSN70**