CHAPTER 3: METHODOLOGY

3.1 Delimitations

The original focus of the investigation was to determine the alteration characteristics of the lithological units hosting the economic mineralization in the Uitkomst complex. For this purpose the study concentrated on those (or adjacent) boreholes that returned poor metallurgical recoveries during the feasibility test work conducted by Anglovaal Minerals (Ltd.) (L.Bradford, internal company report, 1996).

This project does not attempt to describe the entire succession of the Uitkomst Complex. Only representative samples from those affected lithological units encountered along a broadly defined profile and cross-section through the area of interest were sampled and analysed.

This thesis does not attempt to describe the metamorphic aureole surrounding the intrusion. Only a brief overview of the calc-silicate xenoliths is given as it is suggested that these inclusions may have a bearing on the mineralization in the complex. The metamorphic aureole is the subject of a project undertaken by Jens Kriste under the supervision of Dr. C.D.K. Gauert (University of the Free State).

It is assumed that all of the borehole descriptions by various company geologists contained in the logs are complete and correct.

3.2 Description of Methods

3.2.1 Sample selection

A map indicating the so-called talc-rich area on Uitkomst was used to select boreholes for this investigation (pers. comm. L. Bradford, 2005). The range of boreholes was chosen in such a way that it provides an approximate cross section through the lithlogies in the area designated for the future open cast mine. The boreholes selected are; UK3, UK12, UK20,

UK32, UK44, UK48, UK57, UK61 and UK68. Most of the boreholes are situated near the inferred edge of the Uitkomst Complex in the study area (Figure 1.6).

Representative samples of quartered core were taken at two to three meter intervals, where the Basal Gabbro, Lower Harzburgite and Chromatitic Harzburgite Units were intersected. Each sample was approximately twenty centimeters in length. Of these samples a selected few were broken into three pieces, as indicated in Appendix 1. The first of these was used to make thin sections and later polished thin sections, the second was crushed for whole rock analyses and the third kept for control purposes, should a duplicate be required.

3.2.2 Utilization of borehole logs

Borehole logs of the selected boreholes were obtained from African Rainbow Minerals Ltd. The borehole logs were utilized in part to aid in the selection of samples. The logs were also used in calculations, including the determination of the ratio of xenolith content to igneous rock mass, unit thickness, and thickness of the underlying host rock and in determining the proximity of samples to shear zones and diabase sills.

3.2.3 Thin sections

Thin sections for microscopic investigation were made at the Sectioning Laboratory of the Geology Department, University of Pretoria. The initial thin sections were used to describe the petrography, determine the mineral assemblages and establish the degree of alteration. This information was used to select samples for which polished thin sections were prepared for microprobe analyses. Samples that contained large amounts of minerals that may expand during normal polishing procedures were identified and submitted to the De Beers GeoScience Centre for making polished thin sections, using the paraffin method.

3.2.4 Sample preparation for X-Ray Diffraction (XRD) and X-Ray Florescence (XRF) analyses

In an effort to minimize contamination, the quartered core piece selected for analyses by XRD and XRF were wrapped in sample bags and broken into pea-sized fragments. The fragments were then milled in a carbon steel pot in the refurbished Dickie and Stockler

swing mill to the consistency of baby powder (<70 µm). A more comprehensive discussion of the sample preparation is given in Appendix 3. XRD and XRF analyses were preformed on 5 samples from the BGAB Unit, 23 from the LHZBG Unit, 6 xenoliths from the LHZBG Unit, 21 from the PCR Unit and 3 from the LrPRD Unit.

3.2.5 XRD Analyses

Samples were prepared according to the standard method applied in the XRD laboratory of the University of Pretoria. The samples were analysed in a PANalytical X'Pert PRO X-Ray Powder Diffractometer fitted with X'Celerator detector. Semi-quantitative mineral phase analyses of the samples were obtained with the aid of the Rietveld method and by using Autoquant software. The minerals analysed for are: actinolite, amesite, calcite, chlorite, chalcopyrite, goethite, quartz, muscovite, plagioclase, talc, biotite, chromite, diopside, dolomite, fosterite, hornblende, lizarite, pyrite, pyrrhotite, phlogopite, enstatite, cordierite, epidote and grossular.

Samples were crushed before being milled in a carbon steel mill pot. The mill pot was cleaned by milling pure quartz sand and washing with acetone and drying. The pot was then pre-contaminated with a small piece of the sample material to be milled. The milled material was then hand-pressed into the holder, using the in house back-fill method. The sample material is heaped on the holder, held in place by a spring loaded support holder. The material is then distributed evenly in the opening of the holder. Using a stainless steel, solid cylinder the material is manually compressed into the back opening of the holder. The excess material is then cleared of around the cylinder before the back support is placed on the holder (Loubser and Verryn, 2008; Steenkamp, 2009). X-Ray Diffraction (XRD) was used to determine the phases (minerals) present in the sample. Quantitative determination is done using the Rietveld method.

3.2.6 XRF Analyses

Samples were prepared according to the standard procedures of the XRF laboratory of the University of Pretoria. This is described in Appendix 3. The samples were analysed in an

ARL9400XP+ X-Ray Fluorescence Spectrometer. The instrument uses a sequential wavelength dispersive spectrometer to perform quantitative major and trace element analysis. The instrument uses UniQuant software to produce results. The elements analysed for consisted of the major oxides, SiO_2, TiO_2, Al_2O_3, Fe_2O_3, MnO, MgO, CaO, Na_2O, K_2O, P_2O_5, Cr_2O_3, NiO and SO_3. The trace elements analysed for consisted of As, Cu, Ga, Mo, Nb, Ni, Pb, Rb, Sr, Th, U, W, Y, Zn, Zr, Cl, Co, Cr, F, S, Sc, V, Cs, Ba, La and Ce.

Sample material was crushed in preparation for milling in a carbon-steel mill pot. In order to minimize cross-contamination the mill is cleaned after every sample by milling quartz sand and washing with acetone. A small sample was then milled to pre-contaminate the mill.

Major and Trace element analysis were executed on a pressed powder briquette using the ARL9400XP+ spectrometer. Elements are analysed, using an adaptation of the method described by Watson (1996) using a saturated Mowiol 40-88 solution as binder. The milled sample material was placed in aluminium cups to increase stability and strength before being pressed at ± 7 tons/in^2 (Loubser and Verryn, 2008; Steenkamp, 2009).

The XRF Spectrometer is calibrated with certified reference materials. The NBSGSC fundamental parameter program was used for Cl, Co, Cr, V, Ba and Sc. The Compton peak ratio method was used for the other trace elements. W, Cl, F and S should be considered semi-quantitative. Only pressed powder briquettes were used as most of the samples contained significantly large amounts of sulphides. Loss on ignition (LOI) is determined by weighing three (3) grams of powdered material into a pre-weighted aluminum-silicate crucible. The material is dried overnight at 100 °C and weighed again before being roasted overnight at 1000 °C and weighed (Loubser and Verryn; 2008). The method for determining LOI is described by Loubser and Verryn (2008).

The CIPW (Cross, Iddings, Pirrson, Washington) normative mineral calculation was applied to the XRF results, using *MinPet* software. The CIPW norm values are presented at the bottom of the tables.

3.2.7 Electron Microprobe (EMP) Analyses

Polished thin sections were made of the samples that were selected for mineral analyses. The grains of interest were identified with the aid of an optical microscope using both transmitted and reflected light. The minerals were photographed with a Leica microscope fitted with digital imaging equipment and connected to a computer for capturing and saving images for subsequent orientation in the microprobe.

The points selected for analyses were marked using a diamond-scribe fitted optical microscope. This microscope is connected to a computer that uses *Cotrans* software for logging the points that could subsequently be located in the electron microprobe. The polished thin sections were then cleaned and carbon coated in an EMITECH K950X turbo pump driven Carbon Coater.

The Geology Department, University of Pretoria operates a CAMECA SX 100 Electron Probe Micro Analyser. This instrument is fitted with four wavelength dispersive spectrometers as well as the latest energy dispersive system from Röntec. This allows the determination of the mineral composition of individual grains at microscopic scale. This instrument was used for the analyses of the silicate-, oxide-, sulphide and platinum group minerals. The silicate and oxide minerals were analysed on the basis of the following elements: SiO_2, TiO_2, Al_2O_3, FeO, MnO, MgO, CaO, Na_2O, K_2O, F, Cl, Cr_2O_3 and NiO. The sulphides were analysed on the basis of the following elements: S, Fe, Co, Ni, Cu and As.

The counting times on peak positions are 20 seconds and 10 seconds on background positions. A ZAF corrections procedure is applied throughout (Pers. Comm. Gräser, 2005).

Certified standards are used for silicate analyses, using the following standards for each element:

1. A Wollastonite Standard is used for Si and Ca.
2. Pure Oxide Standards are used for Mg, Al, Mn, Cr, Ti, Ni and Fe.
3. An Orthoclase Standard is used for K.
4. An Albite Standard is used for Na.
5. A Tugtupite Standard is used for Cl.
6. A Fluorite Standard is used for F.

Certified standards are also used for sulphide and oxide analyses, the following standards were used for each element:

a. Cu FeS_2 for Cu, Fe and S

b. NiO for Ni

c. Pure arsenic and cobalt standards for As and Co

3.2.8 Energy Dispersive Spectrometer (EDS)

An Energy Dispersive Spectrometer (EDS), mounted on the Cameca SX100 Microprobe, was used to find the Platinum Group Minerals (PGM's). The backscatter image contrast was adjusted to reveal minerals consisting of heavy elements. In a backscatter image heavy elements will appear brighter and lighter elements darker. An analysis of the mineral was performed. If the mineral was smaller than 5 micrometers, some of the surrounding host minerals were also analyzed.

3.3. The isocon method

The quantification of changes in the rock volume and elemental concentrations during hydrothermal alteration is an important component of lithogeochemical investigations of alteration zones. It is a simple matter to calculate the relative gain or loss of different components when the altered rocks are distinguished as the least-altered equivalents or source rocks especially in relatively undeformed and unmetamorphosed rocks. Gresens (1967) presented equations for these calculations based on chemical analyses and specific

gravities of altered and unaltered rocks. Grant (1986) suggested a graphical solution to those equations, known as the isocon method. This contributes greatly to a more quantified approach in the study of these phenomena.

Gresens's equations are based on mass rather than the volume (Gresens, 1967). The mass of element after alteration (M_i^A) is defined by the original mass (M_i^O) plus any change in the mass (ΔM_i) of that element during the alteration (Grant, 1986):

$$M_i^A = M_i^O + \Delta M_i$$

Dividing throughout by M^O to get concentration units and multiplying by M^O/M^A to obtain the original concentration, the following equation is derived:

$$M_i^A / M^A = M^O / M^A \, (M_i^O / M^O + \Delta M_i / M^O)$$

M_i^A / M^A may be substituted for C_i^A and M_i^O / M^O for C_i^O, with C being the abbreviation for concentration and the equation (1) becomes:

$$C_i^A = M^O / M^A \, (C_i^O + \Delta C_i)$$

For an immobile element $\Delta C_i = 0$, and:

$$C_i^A = M^O / M^A . C_i^O$$

This is a linear equation that passes through the origin with the slope of the resulting line (M^O / M^A), equal to $(C_i^A / C_i^O)_{\text{immobile element}}$. Therefore the final equation for an element is defined as:

$$C_i^A = (C_i^A / C_i^O)_{\text{Isocon line}} [C_i^O + \Delta C_i]$$

This is the general equation for the isocon line. If constant mass is assumed, then $C_{\text{immobile element}}^{A}$ will be equal to C^{O} for an immobile element. If constant volume is assumed, then:

$$C_{\text{immobile element}}^{A} = (\rho^{O}/\rho^{A})\, C^{O}$$

Recognizing the mobile and immobile elements is an important aspect of the isocon method. If elements are immobile during the alteration process, the isocon line will pass through the origin. The concentration of these immobile elements will not vary between the original and altered rocks. The gain and loss of the mobile elements may then be calculated and any volume change can easily be deduced.

The isocon line passes through the origin and immobile elements and oxides i.e., TiO_2, Al_2O_3, Zr, U, Th, Nb, Y, La, and Sc. Those elements, which plot in the upper part of isocon line, represent gain and those elements that plot below the isocon line represent loss. The delta value for each element is calculated based on the following formulae:

$(\Delta \text{ value})_i = C^{A}_i - C^{O}_i$. (Slope value of the isocon line), $_i$: the element of interest

If the calculated delta value [$(\Delta \text{ value})_i$] of an element is positive it is indicative that the concentration of that element in the altered rock is greater than in the least altered rock (i.e. gain). Where the calculated delta value for an element is negative it means that the concentration of the element in the altered rock is less than that of the least altered rock (i.e. loss). The delta value can be converted to percentage based on the following formula:

$$\text{Percentage of gain or loss} = [(\Delta \text{ value})_i / C^{O}_i\ ] \cdot 100$$