



Process mineralogical comparison of the Vaal and Crystalkop Reefs

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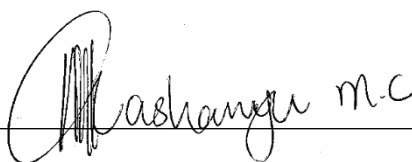
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Declaration of authorship

I, Mashanyu Makatu Casius, do hereby declare that this dissertation submitted to the Department of Chemical and Minerals Engineering, at the North-West University is my own original work that has not been previously submitted in fulfilment for any other degree, neither at this nor any other institution of higher education. I also declare that all the reference materials contained therein were duly acknowledged.

Student's signature:  Date: 01 July 2025

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*“To my late dearest grandmother, **Vho Tshinakaho Dorrah Phalanndwa-Mulaudzi**. Who never had the opportunity to read and write but always prayed for me to have a better future she could not fully see and would have been proud of me for being the first. This is for you.”*

[11 July 1938 – 02 June 2023]

“Ndo lwa nndwa tshidele, ndo vuledza mbambe”

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Abbreviations

AAS	: Atomic Absorption Spectroscopy
AGA	: AngloGold Ashanti
AMIS	: African Mineral Standards
BC	: Bushveld Complex
BSE	: Backscattered Electrons
CH	: Channel
CHL	: Chlorite
CIL	: Carbon in Leach
CIP	: Carbon in Pulp
CR	: Crystalkop Reef Sample
C-Reef	: Crystalkop Reef
CRG	: Central Rand Group
CV Reef	: Composite Sample for the Crystalkop and Vaal Reefs
ECD	: Equivalent Circular Diameter
FA	: Fire Assay
FNAA	: Fast Neutron Activation Analysis
FW	: Foot Wall
GSD	: Grain Size Distribution
HFSE	: High Field Strength Elements
HIRA	: Hazard Identification and Risk Assessment
HW	: Hanging Wall
ICDD	: International Centre for Diffraction Data
ICP-OES	: Inductively Coupled Plasma Optical Emission Spectrometry
LEM	: Low Emission Microscopy
LILE	: Large Ion Lithophile Elements
LOD	: Limit of Detection
LLD	: Lower Limit of Detection

MBA	: Main Bird (Carbon Seam)
MHM	: Malvern Hydro Mastersizer
MLA	: Mineral Liberation Analyser/Analysis
MSc	: Muscovite
MTx	: Matrix
MYA	: Million Years Ago
NWU	: North-West University
OPm	: Opaque Minerals
PAA	: Panalytical Activation Analysis
PGc	: Plagioclase
PPL	: Plane-Polarised Light
PPM	: Parts Per Million
PRp	: Pyrophyllite
PSD	: Particle Size Distribution
Py	: Pyrite
QTz	: Quartz
R. QTz	: Recrystallised Quartz Grains
REE	: Rare Earth Elements
ROM	: Run of mine
SACS	: South African Committee for Stratigraphy
SE	: Secondary Electrons
SEM	: Scanning Electron Microscopy
SPL-GX MAP	: Sparse Phase Liberation Grain X-ray Mapping
SPL-LT MAP	: Sparse Phase Liberation Lite Mapping
STDEV.S	: Standard Deviation of Samples
SW	: Stoping Width
UESM	: Unit for Environmental Sciences and Management
VMR	: Village Main Reef
VR	: Vaal Reef Sample

WDXRF	: Wavelength Dispersive X-ray Fluorescence
WRG	: West Rand Group
XPL	: Cross-Polarised Light
XRF	: X-Ray Fluorescence

Nomenclature

%	Percentage
aq	Aqueous
cm	Centimetres
k V	Kilovolt
L	Liquid
l	Litres
m	Metres
ml	Millilitres
mm	Millimetres
S	Solution
t	Time
v	Volume
wt. %	Weight Percentage
μ	Microns
μm	Micrometres

Papers and conference contributions

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Abstract

The mining of, and demand for gold has over the years gained traction ever since its discovery in alluvial deposits. As much as the chemical processes of refractory ores have improved since then, there is still some gold that gets lost to tailings. Kopanang Gold Mine is one of the underground Vaal River operations located on the boundary between the North-West and Free State Province. The mine is exploiting gold and associated uranium from two quartz-pebble conglomerates known as the Vaal and the Crystalkop Reefs.

Process mineralogical assessment of the Vaal Reef and the Crystalkop Reef at Kopanang Gold Mine can provide necessary information to assist with improving the gold recovery. This was done using a comparative interdisciplinary approach. In order to investigate this in more detail a series of channel samples were collected from two stope panels (59. DW14.R1. P.2 and 42.BW.26.RSE.P9. E). This study involved the mineralogical, chemical and metallurgical characterisation of the two ores through petrographical studies, automated mineralogical analyses and laboratory based extractive metallurgical test work.

Mineralogical characterisation revealed that both reefs were characterised by very fine gold particles, with about 80% of the gold measuring less than 50 µm. Furthermore, mineralogy showed that there is some percentage of gold that is locked within silicate minerals. Gold particles associated with pyrite are hosted within the matrix of the quartz-pebble conglomerating reefs. Chemical assay revealed that the Crystalkop Reef (156.4 g/t) is characterised by higher grades when compared to the Vaal Reef (77.0 g/t).

Laboratory based leaching investigation conducted on the Composite Reef (with a grade of 85.8 g/t) using AGA plant conditions at 4, 8 16 and 24-hours leach residence time intervals, revealed significant difference between forward and reverse leaching methods. With reverse leaching (76.0 to 84.3%) yielding higher gold recoveries than forward leaching (28.3 to 55.7%). Diagnostic leach test work results demonstrate that 87.0% (74.6 g/t) of gold can be recovered through direct cyanidation and 2.5% (2.2 g/t) of the gold remains occluded within silicate minerals, which is susceptible to loss.

The study concluded that although the two reefs are approximately 270 m apart, they are mineralogically and chemically distinguishable. The study further concluded that mineralogy has broader implications for gold recovery. It was also concluded that the Vaal and Crystalkop Reefs can be reclassified as locally refractory at the Kopanang Gold Mine.

Keywords: *Crystalkop Reef; Klerksdorp Goldfield; Process Mineralogy; Vaal Reef; Witwatersrand Basin.*

Chapter 1: Introduction

This chapter will introduce the study by giving a detailed background and rationale of the study, and problem statement that includes the gap in the knowledge that will be investigated. The research main purpose, specific objectives and research hypothesis will also be detailed in this chapter. The study location will be given here as well.

1.1. Background and rationale

Kopanang Gold Mine is an underground operation which was previously owned by AngloGold Ashanti and now owned by Village Main Reef (AngloGold Ashanti Annual Report, 2011). The mine started production in 1984, although the sinking of the shaft started in 1977 and came to completion in 1981 (Cloete, 2009). Geographically, Kopanang Gold Mine is located on the farms Pretoriuskraal 53, Edom 277 and Grootdraai 468, on a lease area that covers approximately 35km². Along with the Great Noligwa and Moab Khotsong Gold Mines, Kopanang Gold Mine is located on the Vaal River boundary between the North-West and Free-State provinces, South Africa (Figure 1).

The Witwatersrand Basin is globally renowned as the most prolific gold producing region in history, contributing significantly to South Africa's economic development (Tucker *et al.*, 2016; Frimmel & Nwaila, 2020). Traditionally, the basin has been broadly characterised as hosting predominantly free-milling gold ores (Swash, 1988; Harris & Watkins, 1990; Frimmel, 2005; Youlton *et al.*, 2021; Youlton, 2021). This classification overlooked the presence of localised refractory ores where the gold is encapsulated in sulphides, silicates, or other minerals where additional processing steps beyond cyanidation are needed (Youlton *et al.*, 2021). This generalised classification of gold reefs within the Witwatersrand Basin often leads to suboptimal recovery strategies and a limited understanding of their processing challenges (Mutele & Carranza, 2024).

Extensive studies on the Witwatersrand Basin emphasise cyanidation's success in recovering gold from native and free-milling ores. For instance, studies by Frimmel (2005) and Harris (1990) highlight the basin's suitability for traditional processing methods. However, localised studies reveal that some reefs contain refractory gold associated with sulphides, carbonaceous matter, or locked within silicate minerals (Pienaar *et al.*, 2015).

Bowell *et al.* (2011) have defined geometallurgy as a study of metallurgical characteristics of ore materials using geological properties and it may include concepts of process mineralogy. The application of process mineralogy and geometallurgy in refractory gold ore processing has gained

more attention over the years in gathering precise and accurate mineralogical data which informs the processing methods that can be tailored to specific localised ores (Zhou *et al.*, 2004). This is due to high demand of gold, depletion of free-milling ores, and the discovery of more refractory ores. From the discussions with Kopanang Gold Mine geologists the comminution circuit does not always adequately liberate the fine gold grains prior to cyanidation which also limits the recovery.

Consequently, this study revisits process mineralogical characterisation of the Vaal Reef and Crystalkop Reef, with emphasis to gold, demonstrating this will refine the understanding of refractory gold ore for recovery variability within the Klerksdorp goldfield of the Witwatersrand Basin.

1.2. Problem statement

According to Frimmel & Nwaila, (2020) about 40% of the earth's produced gold is from the Witwatersrand deposits. The mining of these deposits is responsible for the massive development of the city of Johannesburg and other small mining towns in South Africa (Tucker *et al.*, 2016). This clearly shows that the mining of these goldfields has had a great impact in the development of South Africa as a country (Tucker *et al.*, 2016). However, over the recent years the mining sector has become increasingly less profitable (Mkhatshwa, 2021). This is due to the decline in gold and uranium productions, the unstable market prices and increased labour costs, which poses operational and financial problems in the sector (Mkhatshwa *et al.*, 2020; Mkhatshwa, 2021)

The mine is currently exploiting gold and associated uranium in the Vaal Reef and Crystalkop Reef, hosted in the Klerksdorp goldfield within the Central Rand Group of the Witwatersrand Supergroup. The two gold-mineralised reefs are characterised by different mineralogical properties as well as different gold and uranium grades. The Vaal Reef is known to be a thick reef, with approximately 85% gold ore reserve tonnage, comprising oligomictic conglomerates. The Crystalkop Reef, located approximately 270 m above the Vaal Reef, is a thin small quartz-pebbled conglomeratic reef contributing 10% to the mine's estimated ore reserve (AngloGold Ashanti Annual Report, 2011). Due to this difference in mineralogy and grade each reef would require a different metallurgical process to recover the gold and associated uranium, however the mine is currently processing the two reefs together as a composite.

According to Pienaar *et al.*, (2015) recognising the ore type as either free-milling, refractory or complex ores and selecting the correct process is vital in maximising metal recovery. This is

achieved by gathering accurate mineralogical data to understand the probable impact it may have on ore processing and metal recovery when extracting the ore body. The detailed mineralogical and textural characteristics of gold and uranium such as phase type, mineral association, grain size distribution, mineral locking and liberation, and gangue mineralogy all have an impact on efficient recovery of metals (Pienaar *et al.*, 2015), which is not really understood at Kopanang Gold Mine, as compared to the other two mines (Moab Khotsong and Great Noligwa Gold Mine).

1.3. Research questions

This study will answer the following research questions:

- What are the primary mineralogical and geochemical characteristics of the Vaal and Crystalkop Reefs that contribute to their classification as refractory ores?
- How do observed gold mineral department, and associations inform the understanding of the physical properties of these ores?
- How does the mineralogy and geochemistry of the Vaal and Crystalkop Reefs impact the recovery of gold during leaching?

1.4. Hypothesis

The Vaal and Crystalkop Reefs at the Kopanang Gold Mine are hypothesised to exhibit refractory characteristics due to their mineralogical compositions, including a significant association of gold with silicate and sulphide minerals. It is further hypothesised that a fraction of fine gold is locked within silicate minerals, which hinder their accessibility during conventional leaching process (cyanidation) which may result in lower recovery than expected.

1.5. Overarching aim and objectives

The overarching aim of this study is to investigate and compare process mineralogical characteristics of the Vaal and the Crystalkop Reefs in order to re-classify the Klerksdorp goldfield as a refractory ore zone which will aid in understand implications to gold recovery processes thereof.

The overarching aim will be achieved through the following proposed objectives:

- To investigate physical and optical properties of the Vaal and Crystalkop Reef at Kopanang Gold Mine using descriptive macroscopic observations and optical microscopy.

- To compare mineralogy and geochemistry characteristics of the Vaal and Crystalkop Reefs and understand their implications to the recovery processes of gold.
- To document the behaviour of the Vaal and Crystalkop Reefs at Kopanang Gold Mine using AngloGold Ashanti's Great Noligwa plant standards at a laboratory scale.
- To understand gold deportment and how much gold is lost to tailings during gold recovery using a diagnostic leach test.

1.6. Location of the study area

Geographically, Kopanang Gold Mine is located on the farms Pretoriuskraal 53, Edom 277 and Grootdraai 468, on a lease area that covers approximately 35 km², within the Vaal River boundary between the North-West and Free-State provinces, South Africa (Figure 1.1). The mine is roughly 10 km south-eastern of a small mining town called Orkney and approximately 170 km from the city of Johannesburg (AngloGold Ashanti Annual Report, 2011).

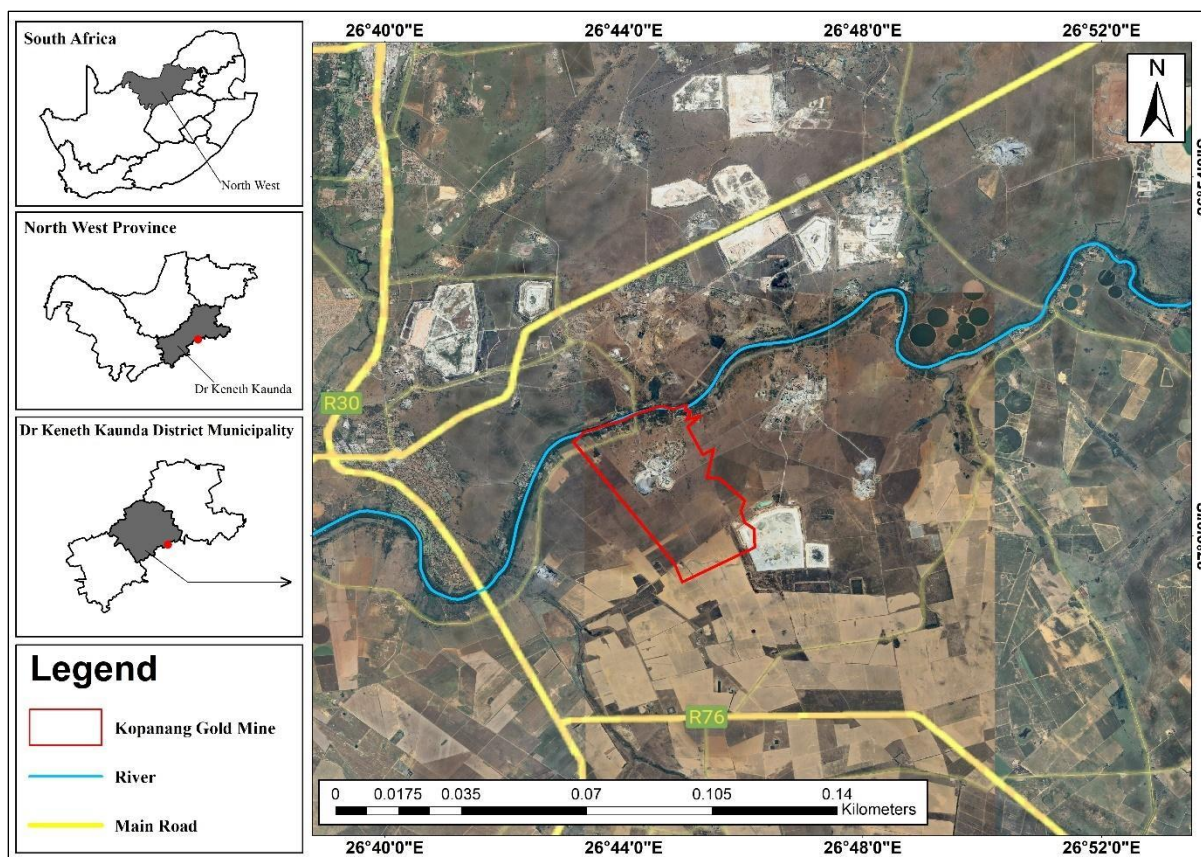


Figure 1. 1: Geographical location of VMR Kopanang gold mine (created by ArcGIS).

1.7 Study outline

This part serves to give insights on what each chapter will entail and references for each chapter will be given at the end of the chapter (Figure 1.2).

Chapter 1: Introduction

This chapter will focus on introducing the study as well as giving the background, problem statement and substantiation of the research project. The main aim, objectives and the hypothesis of the study will also be detailed in this chapter. This chapter will focus on detailing the study area as well as outlining the structure of the dissertation.

Chapter 2: Literature Review

Literature will be reviewed throughout the study. This chapter will only focus on the literature reviewed during the study. This literature will include current research on the mineralisation and processing of the gold.

Chapter 3: Research methods and materials

This chapter will address the methodology employed in this research study together with the materials used to obtain the objectives of the study. It will encompass the procedure utilized to collect and gather pertinent information, the processing methods that will be applied to derive useful information from the data collected in the field, and the tools used to fulfil the study's goal.

Chapter 4: Mineralogical and geochemical characterisation

This chapter focuses on the analysis and interpretation of mineralogical data obtained from collected samples as well as the laboratory analysis. The analysis will be done to compare and characterise the two reefs in question, based on their mineralogical composition and characteristics. Optical microscopy, SEM and MLA will be used to analyse the samples. Mineralogical characterisation results and findings will also be discussed in this chapter.

This chapter will also focus on the geochemical analysis and interpretation of the collected and feed samples (Vaal Reef, Crystalkop Reef, and Composite feed sample that will be prepared for metallurgical characterisation. The chemical analysis will be done using X-ray fluorescence, and ICP-OES. Geochemical characterisation results and findings will also be discussed in this chapter.

Chapter 5: Metallurgical Characterisation

This chapter will focus on extractive metallurgical test work and interpretation of results obtained from forward and reverse cyanide leaching tests of the Composite feed sample. The metallurgical analysis will be done to highlight how the feed samples responded to the leach tests using the plant standards. Diagnostic leaching results and findings will also be discussed in this chapter.

Chapter 6: Discussion, conclusions and recommendations

Chapter 6 will be the last chapter to discuss the overarching findings of the study based on the findings from chapter 1 to chapter 5, and from those findings, conclusions will be drawn, and recommendations will be considered for possible future work. The results and recommendations will also be presented to the mining company for processing alterations.

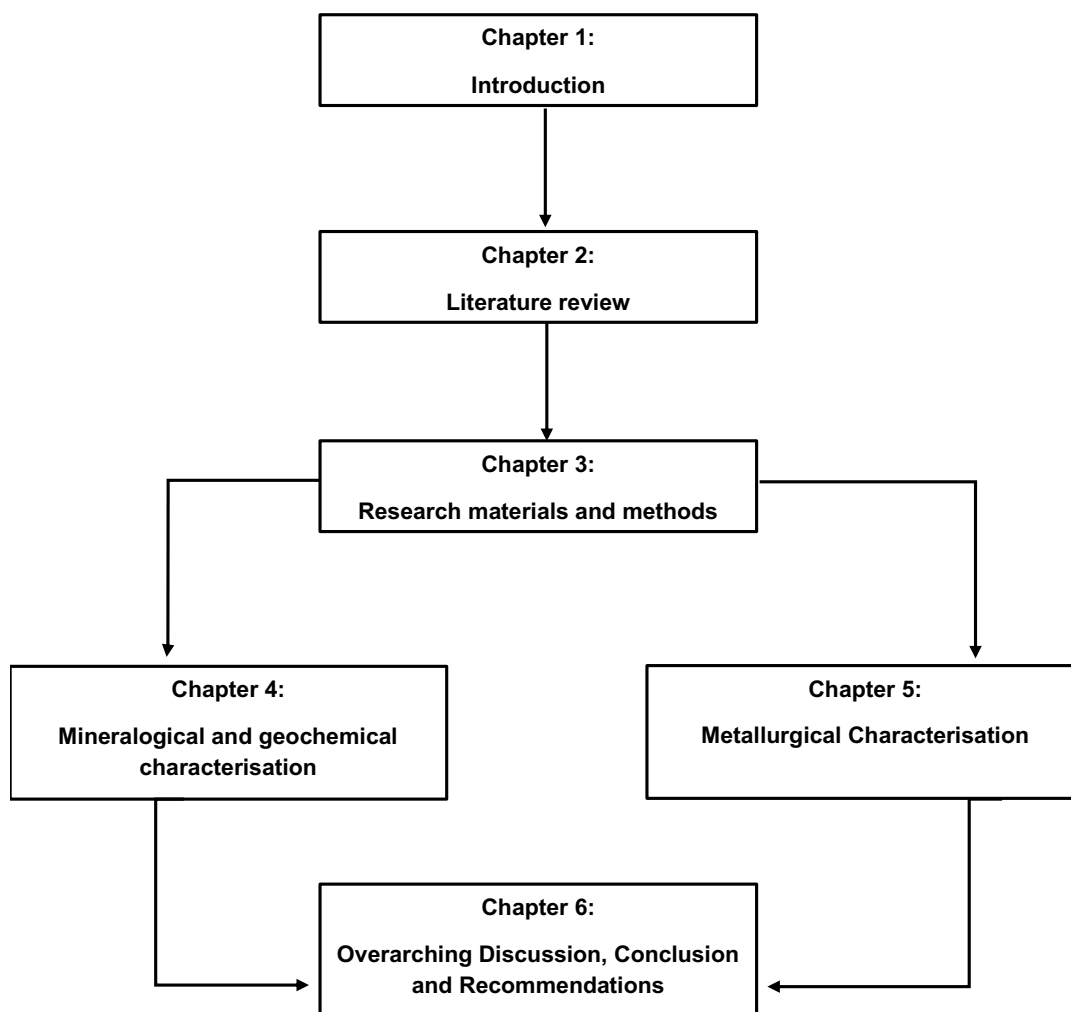


Figure 1. 2: Structure of the dissertation.

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Chapter 2: Literature review

This chapter reviews current literature on the processing of gold and uranium in the Witwatersrand Basin, with particular emphasis on gold leaching processes. The current literature on the regional and local geology will also be detailed in this chapter to understand the mineralisation and properties of the pebble conglomerates and the ore.

2.1. Regional and local geology

2.1.1. Kaapvaal craton

The Kaapvaal Craton is known to be one of Earth's oldest and most stable cratons (Kirk *et al.*, 2002), with an estimated area of approximately 1.2 million km² (Good and De Wit, 1997; Poujol *et al.*, 2003). It comprises Archean granitoid and greenstone belts overlain by volcano-sedimentary successions. This craton forms the basement of the Witwatersrand Basin and has provided a stable foundation for over 3 billion years (Eglington and Armstrong, 2004; Simon *et al.*, 2007). This has allowed the deposition and preservation of sedimentary as well as igneous sequences hosting valuable mineral resources (Good and De Wit, 1997).

2.1.2. Witwatersrand basin

The Witwatersrand Basin (Figure 2.1) is globally recognized as the world's largest gold province (Kirk *et al.*, 2001; Tucker *et al.*, 2016). Positioned near the centre of the Kaapvaal Craton, the basin covers an area approximately 300 km long and 160 km wide, with a stratigraphic thickness of around 7 km (Figure 2.2) (Robb and Meyer, 1995).

The basin is predominantly composed of meta-sedimentary rocks, including quartzite, shale, and auriferous meta-conglomerates (Coward *et al.*, 1995; Pienaar *et al.*, 2015). These strata are classified into the West Rand Group (WRG) and the auriferous Central Rand Group (CRG) (Figure 2.1) (SACS, 1980; Robb and Meyer, 1995). The sediments of the basin were deposited in fluvial and alluvial environments during the Mesoarchaeon to Neoarchaeon periods (Therriault *et al.*, 1997), with notable post-depositional modifications induced by the Vredefort meteorite impact event (Stevens *et al.*, 1997). The impact has also contributed to faulting and folding, shock metamorphism, and the remobilization and concentration of gold and other minerals within the strata of the basin (Stevens *et al.* 1997, Therriault *et al.*, 1997; Hayward *et al.*, 2005).

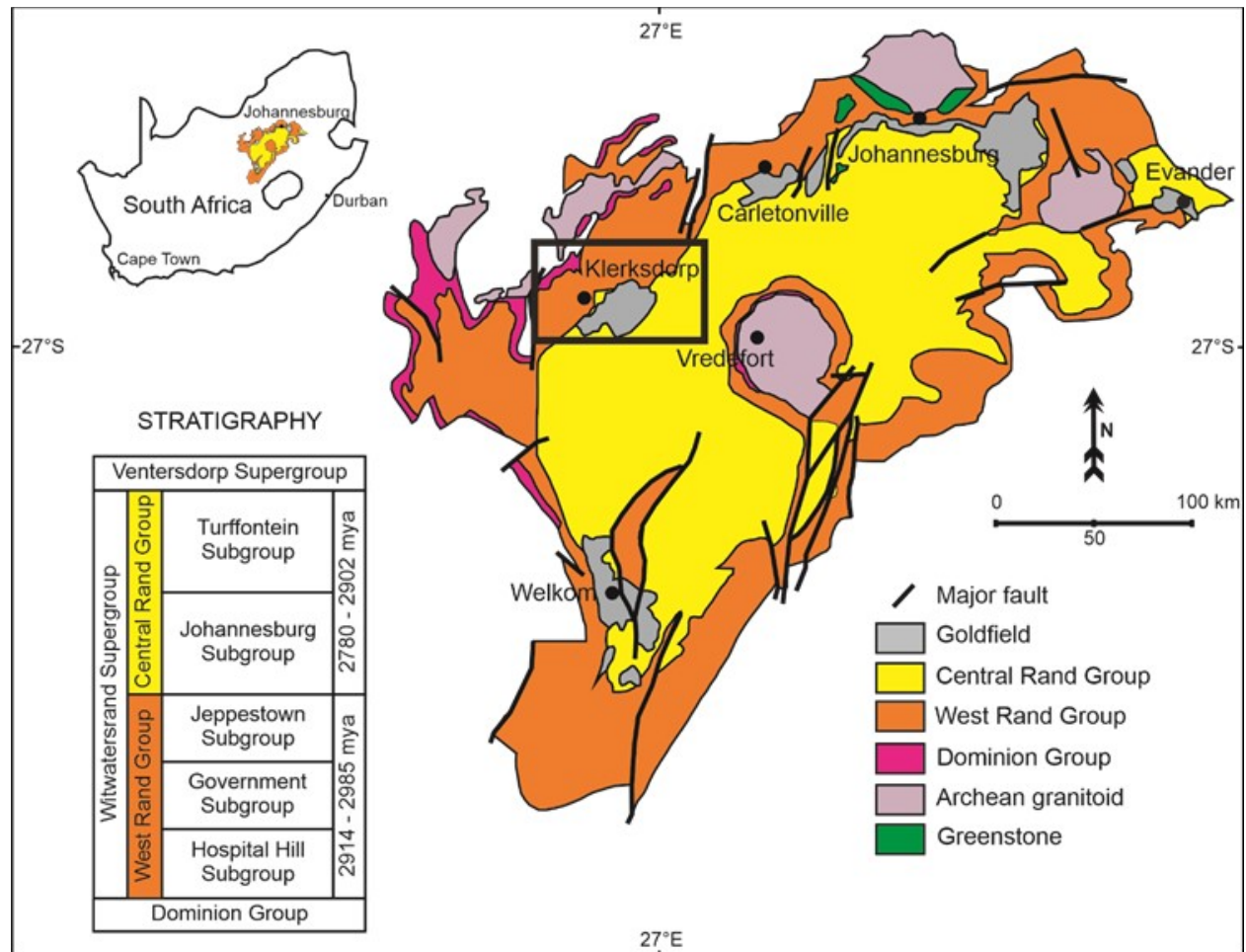


Figure 2. 1: General outline of the Witwatersrand Basin, general stratigraphy, and the location of the gold fields, where the surface rocks are stripped out (Adapted and modified from Frimmel *et al.*, 2005, Nwaila *et al.*, 2017).

2.1.3 Central Rand Group

The CRG forms the upper portion of the Witwatersrand Supergroup and is characterized by conglomerates, quartzites and shales (Coward *et al.*, 1995). The CRG is divided into the Johannesburg and Turffontein Subgroups (Figure 2.2), representing multiple depositional cycles interspersed with unconformities (Beukes and Nelson, 1995). These deposits reflect sedimentation in braided fluvial systems and exhibit auriferous conglomerate gold reef layers (Coward *et al.*, 1995, Pienaar *et al.*, 2017). Amongst others, the reef layers in the CRG include the Vaal and the Crystalkop and are critical for gold mining (Hayward *et al.*, 2005).

The gold-bearing Vaal and Crystalkop Reefs are found within the Klerksdorp goldfield in the Johannesburg Subgroup (Pienaar *et al.*, 2017). The Vaal Reef positioned between the

Krugersdorp and Luipaardsvlei Formations and the Crystalkop Reef lying between the Kimberly and Booyens Formations (Figure 2.2) (Beukes and Nelson, 1995; Smart *et al.*, 2016). These reefs are separated by quartzitic sandstones, and barren conglomerates (Pienaar *et al.*, 2017).

2.2.2 West Rand Group

The WRG primarily consists of fine-grained material with similar quantities of quartzite and shale (Taylor and Anderson, 2018). These sediments were predominately deposited in a marine environment. The present distribution of this layer in the stratigraphy outlines the preserved shape of the basin. The WRG is subdivided into three subgroups, namely Hospital Hill, Government and Jeppestown (Figure 2.2), which are separated by basin-wide unconformities. This succession alternates between quartzite and shale, reaching a thickness of about 4300 m in the Central Rand and 5150 m around Klerksdorp (Taylor and Anderson, 2018).

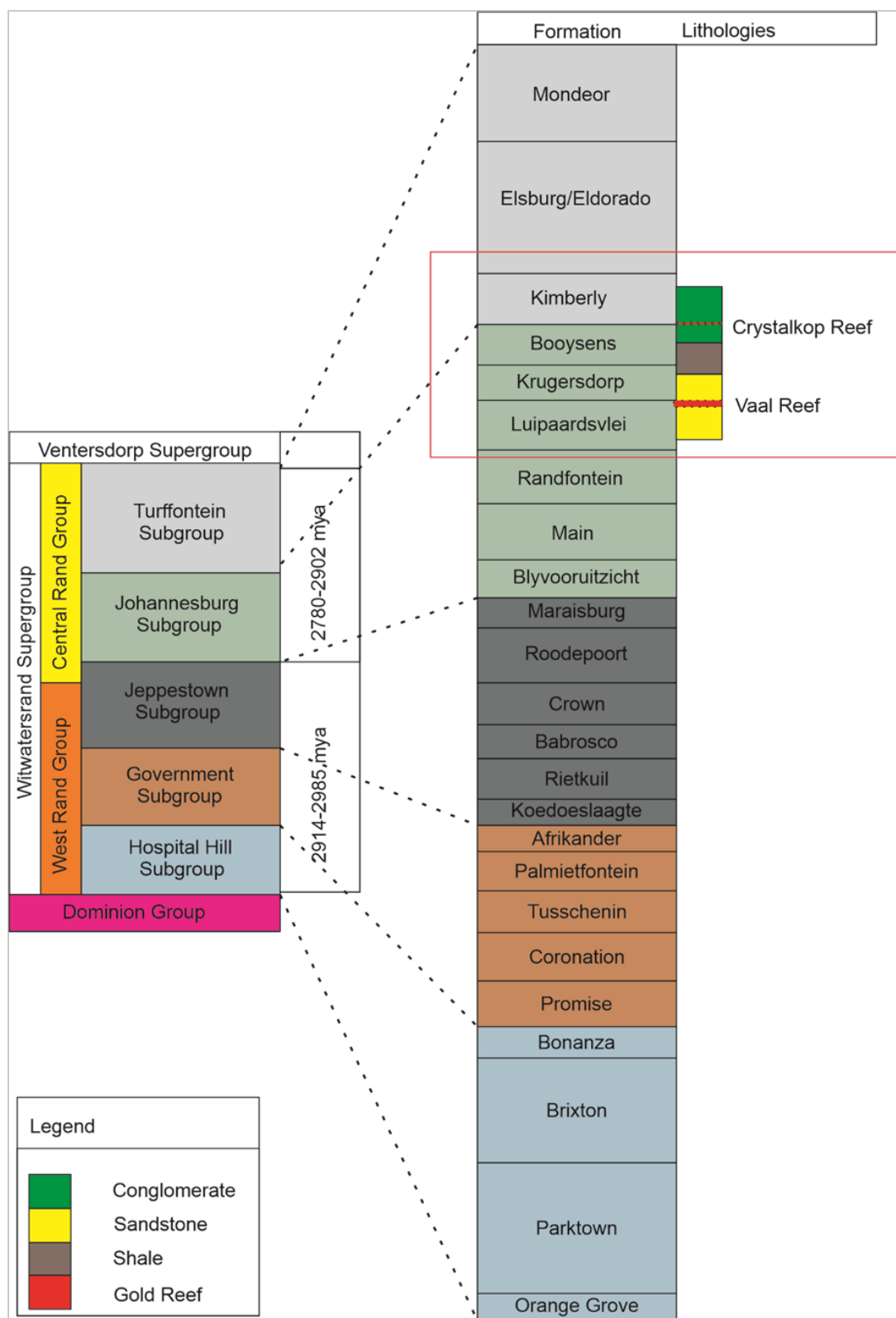


Figure 2. 2: General stratigraphy of the Witwatersrand Supergroup (Modified from Frimmel, 2005 and Smart et al., 2016).

2.3 Klerksdorp goldfield

Situated along the north-western margin of the Witwatersrand Basin (Figure 2.1), the Klerksdorp goldfield has been a significant contributor to South Africa's gold production (Tucker *et al.*, 2016), yielding approximately 4,900 tonnes of gold by 1992 (Tucker *et al.*, 2016). The goldfield is centred around the towns of Klerksdorp, Stilfontein, and Orkney in North-West province (Pienaar *et al.*, 2017), and exploit primarily the Vaal Reef, with minor production from the Crystalkop Reef and Ventersdorp Contact Reef (VCR) (Smart *et al.*, 2016).

The Vaal Reef contributes approximately 90% of the ore mined, while the Crystalkop Reef, a thinner and more complex deposit, accounts for less than 10% (AngloGold Ashanti Annual Report, 2008; Pienaar *et al.*, 2017). The gold mineralisation is predominantly explained by the hypothesis of the placer model (Kirk *et al.*, 2002), the hydrothermal model (Safonov, and Prokof'ev., 2006) of deposition or a mixture of the two models. The mixed model explains that gold was first concentrated by sedimentary processes in ancient river systems and later remobilized by hydrothermal activities, with post-depositional modifications in some areas, such as carbon-bearing veins (Tucker *et al.*, 2016, Handley 2023). Hydrothermal events coincided with key geological milestones: the deposition of the lower (ca. 2,500 Ma) and upper (ca. 2,300 Ma) portions of the Transvaal Supergroup (Robb and Meyer, 1995), the Bushveld Complex's (BC) emplacement, and the Vredefort meteorite impact (ca. 2,050–2,000 Ma; Robb and Meyer, 1995).

2.4 Structural geology

Structurally, the Klerksdorp goldfield is located on the margin of what has been proposed as a possible third outer remnant ring (Brink *et al.*, 2000). These rings are thought to have formed as a result of the shockwave generated by the Vredefort meteorite impact (Therriault *et al.*, 1997). The possible third ring, noted by Brink *et al.*, (2000), is seen extending through to Johannesburg and may include the Carletonville and Krugersdorp goldfields. Although most of the Witwatersrand Supergroup geological structures were an effect of the shockwave, Brink *et al.*, (1997) reported extensional structures such as faults that existed before the Vredefort event (Brink *et al.*, 1997; Waanders *et al.*, 2005). One of the pieces of evidence of the pre-Vredefort movement is the extensional Fakawi Fault system as reported by Antrobus *et al.*, (1986) and Brink *et al.*, (1997).

Three of the major faults that cut through the CRG and the WRG of the Witwatersrand Supergroup are the Jersey fault, the Zuiping fault and the De Hoek fault (Brink *et al.*, 1997; AngloGold Ashanti Annual Report, 2012) (Figure 2.4). At local and small scale, Kopanang Gold Mine shows small scale faults (Figure 2.4) that pose difficulties in mining. This is due to the complexity of the ore

body, which is disturbed, forming gaps in the reef (AngloGold Ashanti Annual Report, 2012) (Figure 2.3). Some of these minor faults include shaft flat faults, which are low angle inclined faults, and shaft steep faults, which are vertical or almost vertical faults. The BW fault and Pillar fault (also steeply inclined faults) seem to be associated with shaft steep faults and are exposed in underground workings of Kopanang Gold Mine (Figure 2.3 and Figure 2.4

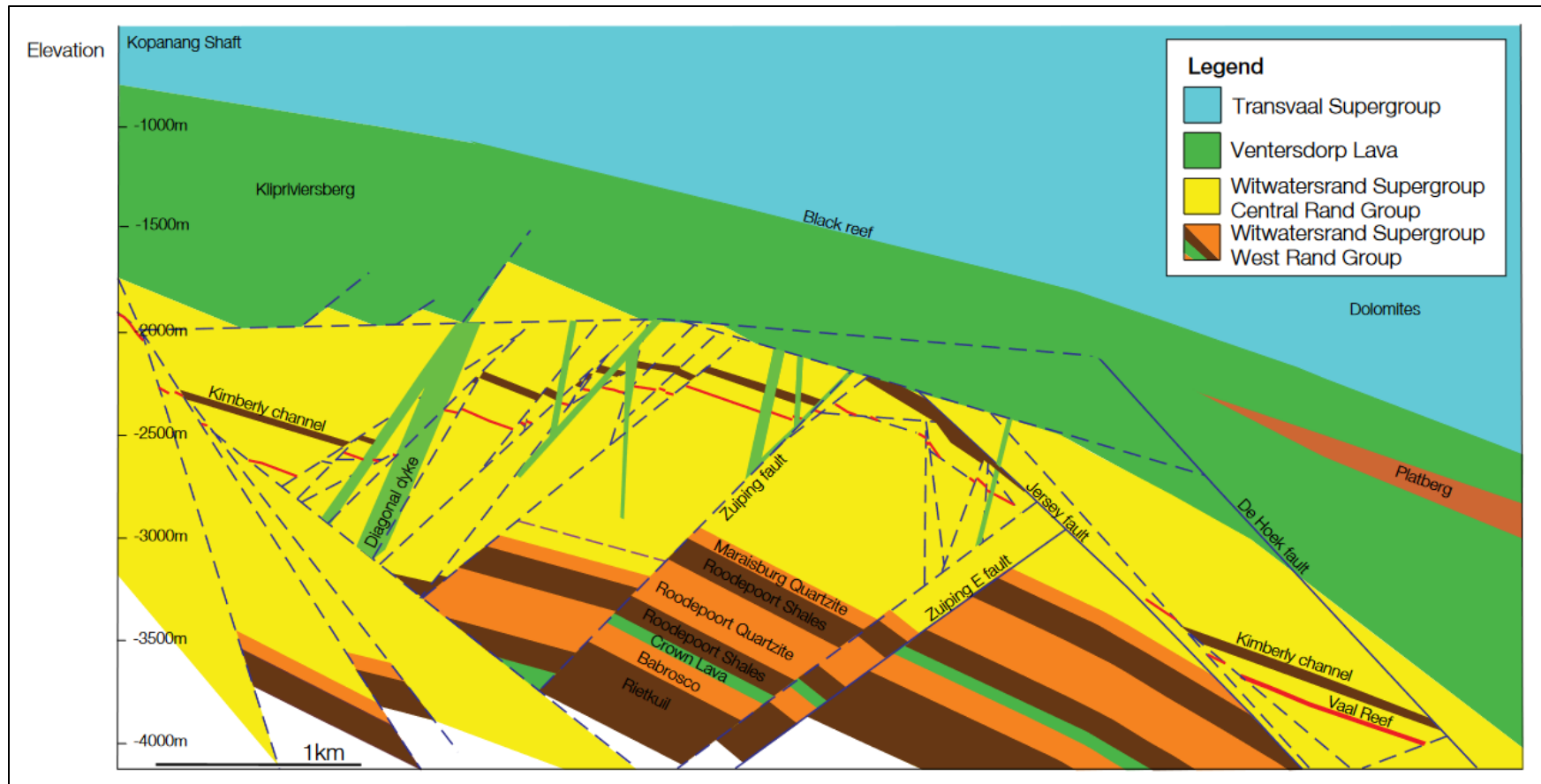


Figure 2. 3: Geological cross-section from north-west to south-east (adapted and modified from AngloGold Ashanti Annual Report, 2012).

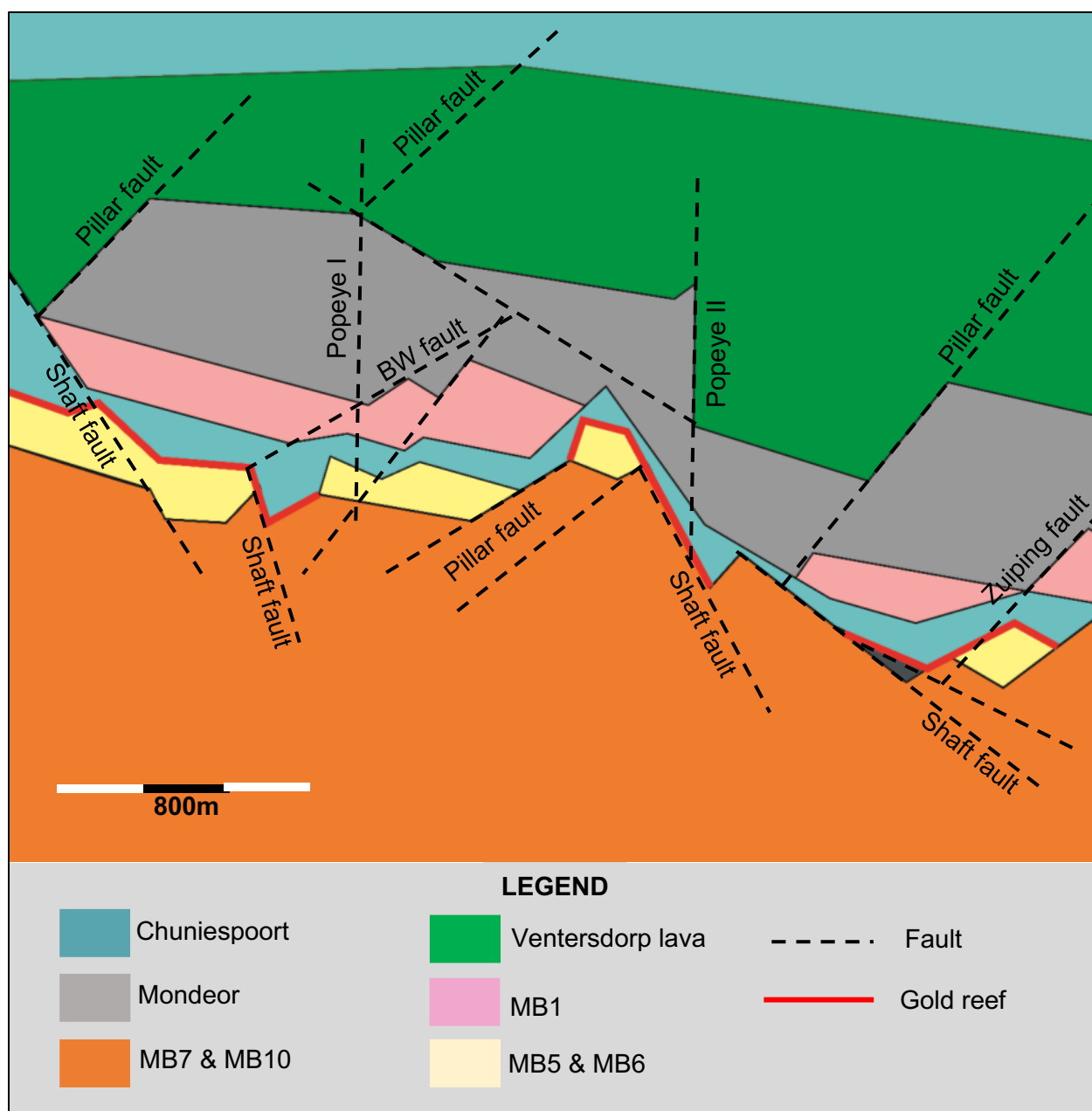


Figure 2. 4: Localised north-west to south-east cross-section showing structural geology and mining levels at VMR Kopanang gold mine (adopted and modified from Smart *et al.*, 2016).

2.5 Ore classification

The Witwatersrand Basin hosts a spectrum of ore types, from free milling to refractory and complex ores, each presenting unique processing challenges (Figure 2.5) (Dominy *et al.*, 2011; Youlton *et al.*, 2021). While free-milling ores historically dominated production, the increasing prevalence of refractory and complex deposits necessitates a shift towards advanced

metallurgical techniques (Youlton *et al.*, 2021). This section of literature review explores these classifications in the context of the basin's mineralogical and metallurgical characteristics.

2.5.1 Free-milling ores

Free-milling gold ores are characterised by their amenability to cyanidation (Figure 2.5), achieving high recoveries (typically >90%) without requiring pre-treatment. These ores predominantly contain gold as native metal, often associated with quartz or other gangue minerals, which can be liberated through standard comminution (Fleming, 1992; Wills and Finch, 2015). Different recovery methods were used in the early years. The cyanide process was introduced when the free-milling surface ore changed to a more pyritic ore type requiring finer grinding and cyanidation (the MacArthur–Forrest process). Historically, much of the gold mined from the Witwatersrand Basin was considered free-milling. This was due to its successful recovery using direct cyanide leaching (Harris, 1990; Frimmel, 2005).

The high recovery rates and low processing costs associated with free-milling ores contributed significantly to the initial economic success of the Witwatersrand Basin (Fleming, 1992). However, as mining progressed, the proportion of free-milling ores decreased, revealing more refractory and complex deposits (Tucker *et al.*, 2016).

2.5.2 Refractory ores

Gold ores in the Witwatersrand Basin are often refractory, meaning they resist standard cyanidation and typically yield less than 80% recovery without some form of pre-treatment (Youlton *et al.*, 2021). This refractory behaviour arises from the complex mineral makeup of the ore, which presents multiple barriers to liberating and dissolving gold (Alymore, 2016; Pienaar *et al.*, 2015). Understanding these barriers is essential for designing effective extraction strategies.

A significant portion of gold in these ores is locked within sulphide minerals such as pyrite and arsenopyrite. Because the gold is chemically bound within these minerals, it cannot be recovered efficiently through cyanidation alone. Investigations of the Vaal and Crystalkop Reefs at Kopanang Gold Mine show strong associations of gold with sulphides, highlighting the need for pre-treatment methods like ultrafine grinding, roasting, or bio-oxidation to liberate the gold and improve recovery (Pienaar *et al.*, 2017; Sebola, 2019; Alymore, 2016) (Figure 2.5).

In addition to sulphides, some gold occurs trapped within silicate minerals. This gold is physically shielded from chemical attack and requires fine grinding or other physical liberation methods to become accessible to cyanide leaching. Addressing this challenge is key to achieving higher

overall recovery rates (Alymore, 2016). Another challenge comes from preg-robbing materials, such as carbonaceous matter, which can adsorb gold cyanide complexes from solution and reduce leaching efficiency. Rees and van Deventer (2000) describe preg-robbing as a phenomenon where ore constituents remove dissolved gold from solution. Recognizing and managing these materials is crucial for ensuring that cyanidation is effective.

Beyond preg-robbing, certain minerals including iron- or copper-bearing phases and complex oxides consume cyanide without contributing to gold recovery. These reactions can interfere with leaching, underscoring the need to understand the full mineralogical composition of the ore before designing processing strategies. As deeper and older deposits in the Witwatersrand increasingly contain refractory ores, researchers and operators have developed innovative pre-treatment methods, such as pressure oxidation (POX) and bacterial oxidation (BIOX). These techniques break down the mineral matrices that encapsulate gold, enabling higher recovery rates and making the processing of refractory ores economically viable (Larrabure and Rodríguez-Reyes, 2021; Surimbayev et al., 2024).

2.5.3 Complex ores

Complex gold ores refer to deposits that exhibit a combination of free-milling, refractory, and additional metallurgical challenges due to their mineralogical and chemical diversity. These ores may contain gold, uranium, and base metals such as nickel and zinc, often in varying associations with sulphides, silicates, and oxides (Bowell *et al.*, 2011).

The Crystalkop Reef, for instance, demonstrates the challenges of complex ores, with high gold grades, but significant silicate-hosted gold and elevated levels of base metals that interfere with leaching processes (Nicol *et al.*, 1987; Pienaar *et al.*, 2015). Such ores often require a combination of metallurgical approaches, including gravity separation, flotation, and advanced chemical leaching, to optimize recovery (Dominy *et al.*, 2011; Fleming, 1992) Figure 2.5.

Understanding and managing the variability of complex ores is crucial for resource evaluation and long-term planning in the Witwatersrand Basin. Geometallurgical studies, integrating mineralogical, geochemical, and metallurgical data, are increasingly employed to address these challenges (Zhou *et al.*, 2004; Wang *et al.*, 2019).

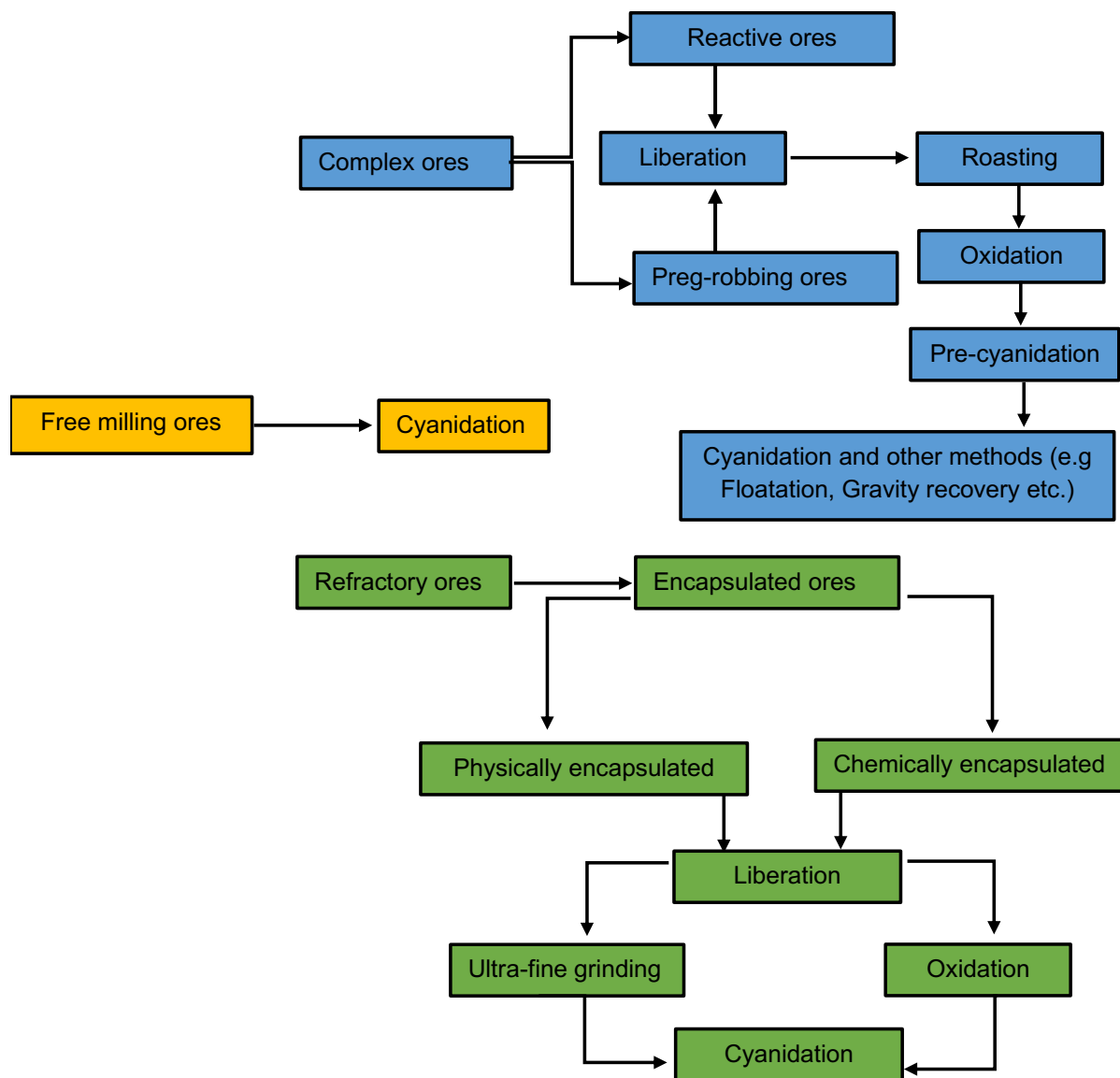


Figure 2. 5: A diagram depicting three different classifications of gold ore deposits; (a) free milling ores, (b) Refractory ores, and (c) Complex ores. (Adapted and modified from Youlton et al., 2021).

2.6 Mineralisation and processing

2.6.1. Klerksdorp gold mineralisation

Two reef horizons namely the Vaal Reef and the Crystalkop Reef are currently the only two reefs exploited by the mines situated along the Vaal River Operation within the Klerksdorp goldfield. As already highlighted, over 90% of the ore has been produced from the Vaal Reef and less than

10% of the bulk ore produced from the Crystalline Reef (Pienaar *et al.*, 2017) which is a relatively thin reef compared to the Vaal Reef (AngloGold Ashanti Annual Report, 2008).

Tucker *et al.*, (2016) stated that sedimentological research that commenced around the 1960s led to a better understanding of the depositional conditions and controls for gold mineralisation within the Witwatersrand Basin. This research supports the idea that alluvial gold mineralisation took place within the basin, although there has been a debate about it over the years. In several cases, post-depositional gold mineralisation can be seen in carbon-bearing veins (Tucker *et al.*, 2016).

2.6.2. Extractive metallurgy

Because process mineralogy is an important discipline for studying ore distributions and behaviour (Cropp *et al.*, 2014), it has over the years grown and gained attention in South Africa in particular. Process mineralogy is basically the combination of ore mineralogical studies, process analysis, flowsheet design as well as optimisation of ore recovery through metallurgical test-based work such as floatation, leaching and concentration, to name a few (Cropp *et al.*, 2014). Therefore, gold and uranium processing plants have over the years characterised ore mineralisation and process mineralogical properties in order try and to maximise ore recovery.

Ore processing is generally done in three phases, i.e. extraction of valuable ore from the ground, separation of the valuable metals from the bearing rock with chemical processes and refining and manufacturing simultaneously. There are diverse types of metallurgical processes that can be used to recover gold from the gold-bearing rock, with floatation, leaching and gravitational methods being some of the most used examples (Frimmel, 2019). The present study focuses more on leaching of the gold, using cyanide.

Fivaz (1988) defined leaching as a heterogenous process of using a solvent (chemical) to extract one mineral or metal by dissolving the other. Therefore, gold leaching is a process of extracting gold constituents by dissolving gold-bearing minerals from the ore using cyanide (Fivaz, 1988). Leaching is a complex procedure which always involves the use of toxic chemicals and three reaction phases, namely solvent diffusion, chemical reaction within the particle, and transportation of the dissolved substance within a solvent, being the major reactions taking place (Frimmel, 2019; Frimmel *et al.*, 2005).

Without the invention of the MacArthur-Forrest cyanidation process, South Africa's economic development might never have reached the point of significant growth (Fivaz, 1988). This is because the discovery of using cyanide as a leaching agent changed the processing of gold ores and made it possible to recover gold from ores that were not amenable to processing methods

such as amalgamation and gravitational concentration (Fivaz, 1988; Chryssoulis and Cabri, 1990). According to Pienaar *et al.* (2015), gold may be extracted effectively and economically using cyanide leaching from ores containing even as little as 0.5 g/t gold.

The cyanidation process is highly sensitive to various factors that influence its efficiency in extracting valuable metals like gold (Zhou and Cabri, 2004). Key determinants include the mineralogical composition of the ore and plant operating properties such as temperature, agitation rate, particle size, leaching duration, and pH. Each of these variables can either accelerate or hinder the dissolution of gold in cyanide solutions. For instance, temperature affects reaction kinetics, while agitation enhances mass transfers. Particle size impacts the surface area exposed to the leaching solution, which in turn affects the rate of gold recovery. Additionally, the leaching time and pH levels are critical for maintaining optimal reaction conditions, because an overly acidic or alkaline environment can interfere with the leaching efficiency and even increase reagent consumption (Zhou and Cabri, 2004).

These parameters significantly influence the optimal recovery of gold, as gold particles are often intricately bound within the mineral matrix of host minerals. It is in these matrices where the complex mineral structures can restrict cyanide access (Zhou and Cabri, 2004). Therefore, an in-depth understanding of these conditions is essential for optimizing cyanidation and ensuring maximum extraction of the desired metals (Kondos *et al.*, 1995). In this context, Srithammavut (2008) highlights that effective recovery depends not only on the chemical conditions but also on a strategic adjustment of the operational parameters to accommodate the unique mineralogical characteristics of each ore.

There are several stages that are involved during the gold-leaching process and these stages include material preparation, dissolution, recovery of metals from the solution and gold recovery (Pienaar *et al.*, 2015). To ensure minimal cyanide loss as hydrogen cyanide, the pH and temperature should be adjusted (Pienaar *et al.*, 2015).

2.6.4. Gold ore processing at the Witwatersrand Basin

During the early stages of gold production in the Witwatersrand Basin, shallow and oxidised ores were processed using mercury amalgamation (Taylor and Anderson, 2018). As mining progressed deeper, the processing method was switched to cyanide extraction for better recovery of the gold (Fleming, 1992). This transition was because more pyritic ores were discovered as mining progressed to the deeper levels. The processing of ore involves jaw crushing, ore pulverisation,

gravity separation for recovery of courser gold particles and the use of cyanidation for finer gold recovery (Fleming, 1992; Frimmel, 2005; Youlton *et al.*, 2021).

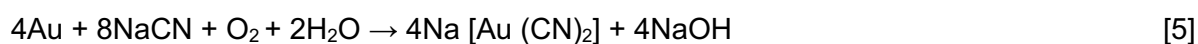
Gold cyanide dissolution reactions:

Yannopoulos (2012), explained the dissolution of gold in cyanide solution in the presence of oxygen to form cyanide complexes, depending on the type of cyanide used (Table 1), as the main reaction in the extraction of gold from its ore. This is because gold will only dissolve in a solution with an oxidizing agent and a ligand available (Yannopoulos, 2012).

Table 1: Table cyanide categories and gold-cyanide complexes they form.

Gold cyanide complexes	
Cyanide Category	Gold-Cyanide Complexes
Free Cyanide (CN⁻)	Au (CN) ₂ ⁻
Sodium Cyanide (NaCN)	Na [Au (CN) ₂]
Potassium Cyanide (KCN)	K [Au (CN) ₂]
Calcium Cyanide (Ca (CN)₂)	Ca [Au (CN) ₄] ₂

The sodium cyanide reaction, involves the reaction between Au and NaCN to form a water-soluble complex, known as sodium dicyanoaurate (I). This complex allows Au to dissolve into a solution that can further be processed to recover Au. The reaction can be expressed Equation [5] (Yannopoulos, 2012)



The potassium cyanide reaction is a reaction where the reaction between Au and KCN involves the formation of a water-soluble complex known as potassium dicyanoaurate. The leaching of Au with KCN can be expressed by the reaction given in Equation [6] (Bisceglie *et al.*, 2017)



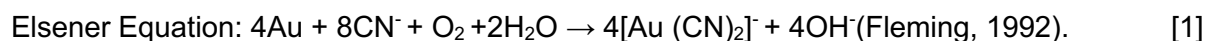
Finally, the calcium cyanide reaction involves the reaction of Ca (CN)₂ with Au and the formation of a water-soluble complex known as calcium dicyanoaurate, and Au leaching with Ca (CN)₂ can be expressed by the reaction given in Equation [7] (Aitimbetov, 2005).



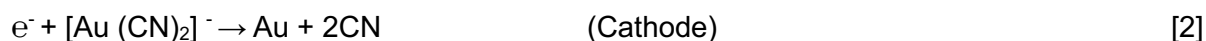
The cyanide solution needs to be alkaline with a basic pH of approximately 10 to ensure efficient extraction of the gold (Tutu *et. al.*, 2008). Gold can be extracted from the cyanide solution through

two methods, the first of which involves adding pulverised zinc powder to the solution and, through replacement reactions, resulting in gold precipitation (Tutu *et al.*, 2008).

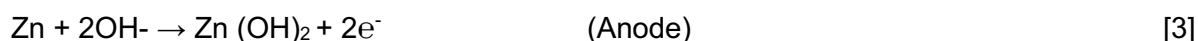
Taylor & Anderson (2018) stated that the cyanide extraction process involves fine crushing and pulverising of ore to liberate gold within the ore. A lower concentration of sodium cyanide is used to leach out gold from the ore by using the Elsener equation:



At the cathode, Au gets reduced by gaining electrons., as shown in Equation [2] (Nicol *et al.*, 1987)



At the anode, which is an intermediate step, $\text{Zn}(\text{OH})_2$ forms and gets oxidized by losing electrons. See Equation [3] and [4] (Nicol *et al.*, 1987)



Zinc dust cementation:

After dissolving gold in a dicyanoaurate soluble complex using cyanide, the recovery of gold is done by precipitation, following the Merrill-Crowe process, which involves the using of zinc dust or shavings, which act as a reducing agent (see Equation [8] (Kongolo and Mwema, 1998)).



Carbon in leach / carbon in pulp process

The second method involves using activated carbon for extraction and, due to the low cost and high efficiency, this method has replaced the use of zinc powder (Taylor and Anderson, 2018). The carbon leaching process is characterised by two variations, Carbon-In-Pulp (CIP) and Carbon-In-Leach (CIL). In CIL, activated carbon is added to the cyanide solution where gold cyanide complexes form and adsorb onto the carbon (Taylor and Anderson, 2018), whereas in CIP, carbon is added later to the downstream slurry tanks, allowing gold-cyanide complexes to occur first, followed by adsorption onto the carbon (Taylor and Anderson, 2018). Gold ore is firstly leached completely before the pulp is treated with activated carbon in a reactor to extract the gold cyanide (Fleming *et al.*, 2011).

2.7 Diagnostic leach test

Diagnostic leaching is a specialised metallurgical technique used to identify the deportment of gold in an ore sample, particularly in refractory ores where standard cyanidation is ineffective (Lorenzen, 1995). This sequential leaching process provides crucial insights into gold recovery potential and the mineralogical factors affecting extraction, helping to determine the necessity for pre-treatment methods (Celep *et al.*, 2009; Chingwaru *et al.*, 2025). By selectively dissolving different mineral phases before applying cyanidation, diagnostic leaching systematically reveals how gold is associated with various minerals, ensuring more efficient metallurgical processing (Kojonen and Johanson, 1999).

The process typically begins with mild lixiviants such as sodium cyanide to remove free gold, followed by stronger reagents like hydrochloric acid, nitric acid, or ferric chloride to dissolve sulphides, carbonates, and other gold-bearing minerals (Lorenzen and van Deventer, 1994). Several factors influence its effectiveness, including ore mineralogy, reagent selection, leaching sequence, particle size, temperature, and pH (Lorenzen and Tumilty, 1992; Celep *et al.*, 2009).

Diagnostic leaching has broad applications in mineral processing, including the optimization of cyanidation parameters, assessment of ore variability, and troubleshooting of gold recovery issues (Lorenzen and van Deventer, 1994). It is widely used in research and industrial settings to refine gold extraction strategies and improve processing efficiency (Kojonen and Johanson, 1999). However, despite its advantages, the process presents challenges such as procedural complexity, chemical interference, scale-up considerations, and ore heterogeneity, all of which can affect the accuracy and reliability of results (Lorenzen and Tumilty, 1992).

Despite these challenges, diagnostic leaching remains an invaluable tool for understanding gold deportment and optimising extraction techniques. The technique not only enhances recovery from refractory ores but also aids in selecting appropriate processing methods for maximising efficiency (Lorenzen and van Deventer, 1994). As advancements in mineralogical and analytical techniques continue, diagnostic leaching will further evolve, improving its application in gold processing and ensuring sustainable and economically viable metallurgical solutions (Celep *et al.*, 2009).

2.8 Grain size distribution

Grain size distribution (GSD) and particle size distribution (PSD) are fundamental concepts in geosciences, material sciences, and engineering as they provide insights into the textural and structural properties of materials. Although often used interchangeably, GSD and PSD describe different aspects, particularly in geological and mining contexts. Grain size distribution specifically

refers to the size distribution of crystalline grains within a rock or mineral matrix, considering only the structural grains of interest, such as gold grains in ore samples or sand grains in sedimentary rocks (Blott and Pye, 2001). Particle size distribution, on the other hand, generally applies to a broader range of particles in various contexts which may contain several unliberated grains. Particle size distribution measures the distribution of all particle sizes in each sample without necessarily differentiating between crystalline and non-crystalline components (Jha *et al.*, 2022). Grain size distribution is typically measured through microscopy, while particle size distribution can be determined using microscopy, physical screening, or laser diffraction techniques.

Grain and particle size distributions are both essential for understanding material properties. Grain size distribution in mineral deposits can reveal information about ore grade distribution, texture, and the genesis of mineralization, while PSD can offer insights into material processing, such as grinding efficiency and separation in mineral processing (Chen *et al.*, 2010). In mining, a more refined GSD can be crucial, particularly when studying deposits where coarser grains could lead to phenomena like the nugget effect, causing localized grade variability (Dominy, 2014).

The median grain size, or d50, is a key metric derived from GSD and PSD, representing the diameter at which 50% of the sample's mass or volume passes through a size fraction (Beuselinck *et al.*, 1998). This value is widely used as a single, representative parameter for comparing different samples and understanding textural changes. In geological contexts, d50 can help predict depositional environments, sediment transport, and, in mining, aid in assessing the likelihood of grade variability due to coarser particles (Dominy and Edgar, 2012). The d50 metric is particularly valuable for studying material heterogeneity, as it offers a central measure within a potentially skewed distribution, providing insights that mean or mode values might overlook (Sperazza *et al.*, 2004).

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Chapter 3: Research Materials and Methods

This chapter addresses the methodology employed in this research study. It encompasses the procedure used to collect and gather pertinent information, the processing methods applied to derive useful information from the data collected at the field, laboratory, and the tools used to fulfil the study's goal. To gather data used to achieve the aim and specific objectives of this study, mineralogical, chemical and metallurgical characterisation was conducted. Figure 3.1 provides a comprehensive overview of the sequential actions and analytical methods undertaken during this research study.

Preparation of feed materials is a major step in all analytical, macroscopic, and microscopy methods. This is because it helps with extracting, isolating, and concentrating the target analytes from complex matrices (Balaram and Subramanyam, 2022). Choosing the method of feed material preparation is dependent on the nature and type of the samples to be analysed, the analyte of interest, the analytical technique to be used, and the required outcome of the study. All feed materials were prepared using the methods outlined in Figure 3.1.

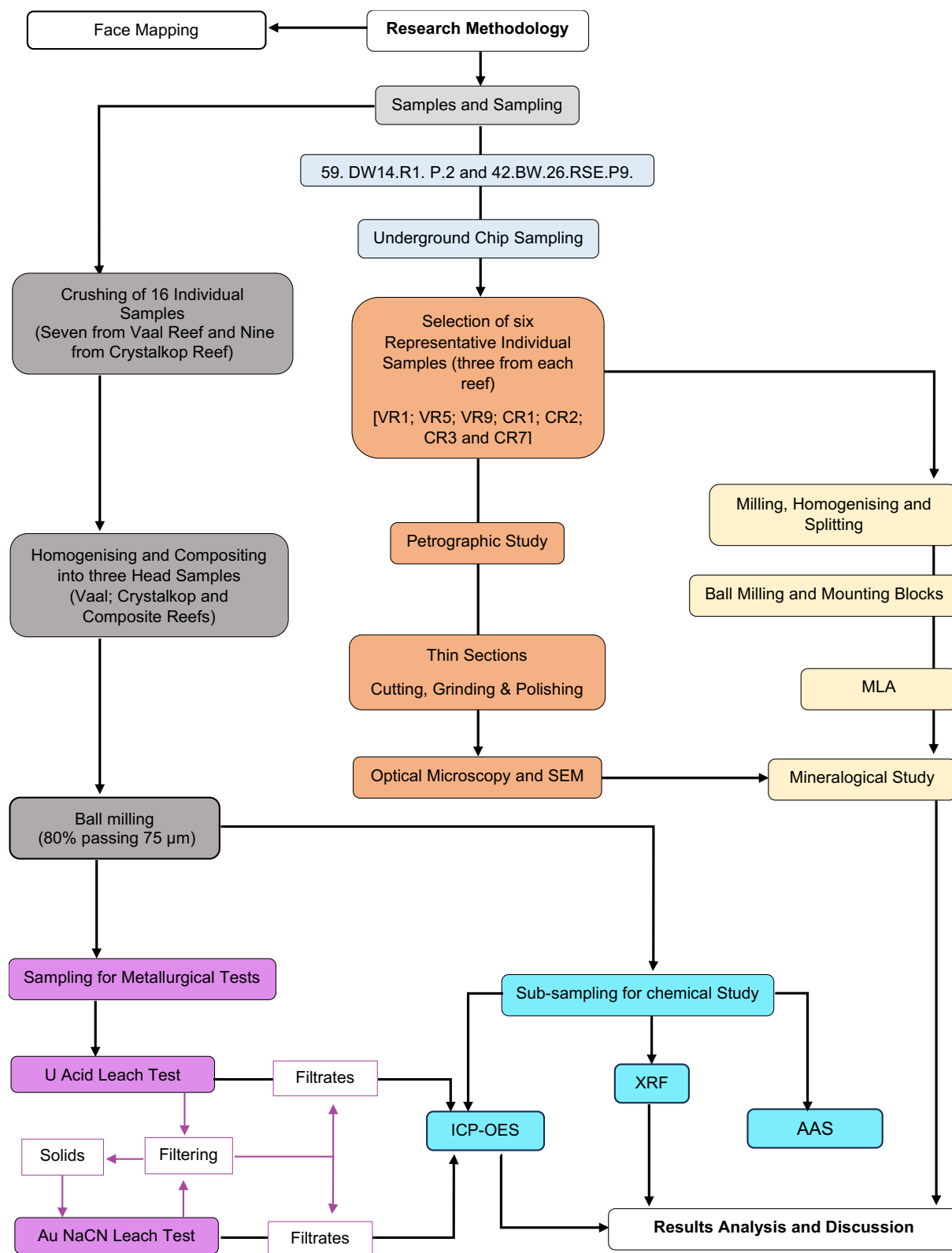


Figure 3. 1: Flow chart showing research methods, steps followed, and sample material used for this study.

3.1 Field work

Field work was conducted at Kopanang Gold Mine within the Klerksdorp goldfield. A visit to the underground mine operations was conducted with the aim of conducting face mapping and acquiring ore samples for further analysis. Face mapping and sampling was conducted at Level 42 (42.BW.26.RSE.P9. E) and Level 59 (59.D. West 14 Raise 2 Panel 2 (Peg No: B15086)) for the Vaal Reef and Crystalkop Reef, respectively (Figure 8 and Figure 9). All the field work was done following the standard procedures of the mine and with the guidance of mine professionals.

3.1.1 Face mapping

Face mapping was conducted on an actively exposed 17.5 m and 15.6 m long underground face of the Vaal Reef and Crystalkop Reef exposures, respectively (Figure 3.2, Figure 3.3, Figure 3.4, and Figure 3.5). The process of mapping the face was done to identify geological structures such as faults and physical properties of the reef, such as thickness and macroscopic properties. A tape measure was used to measure the length of the exposed face and the thickness of the reef, while a clinoruler was used to measure the dip angle of both the reefs and the faults. The widths of the two stope panels (59. DW14.R1. P.2 and 42.BW.26.RSE.P9. E) and the channel were also measured using a tape measure together with the widths of the hanging wall and the foot wall (Table 2).

Table 2: Field measurements of the underground face mapped sections at VMR Kopanang gold mine.

Field measurements		
	Vaal Reef face	Crystalkop Reef face
Face length (m)	15.6	17.5
Hanging wall width (m)	43.4	47.0
Food wall width (m)	31.2	33.1
Channel width (m)	14.4	8.8
Stopping width (m)	9.3	9.3

It is very important to note that the reefs mined at Kopanang Gold Mine are subdivided into facies (A facies, B facies and C facies). According to Cloete (2009), the A facies is characterised by small- to medium-pebbled conglomerate interbedded with quartzites. The A facies is pyrite mineralised and associated with a low Au and U grade when compared to the C facies. The B facies is a layer of barren quartzite, and the C facies is a quartz-pebbled conglomerate characterised by a carbon seam base (MBA) (Cloete, 2009). Pienaar *et al.*, (2017) stated that, of

all three facies, the C facies has higher grades than the others, and that the gold grade is mainly associated with the carbon present in the facies. This study focused on the Vaal Reef A facies and the Crystalkop Reef C facies. The A facies and C facies were sampled because the mine was currently mining a section where these facies were accessible and exposed on the faces where face mapping was being done.

During face mapping, it was noted that both faces were affected by faults (Figure 3.2, Figure 3.3, Figure 3.4, and Figure 3.5). The Vaal Reef was more oxidised on the parts along the faults and less oxidised far from the faults (Figure 3.2 and Figure 3.3). It was also noted that the Vaal Reef was characterised by small to medium pebbles and occurred between a barren hanging wall and a quartzite foot wall (Figure 3.2). whereas the Crystalkop Reef bottom is characterised by medium to large pebbles (Figure 3.4).

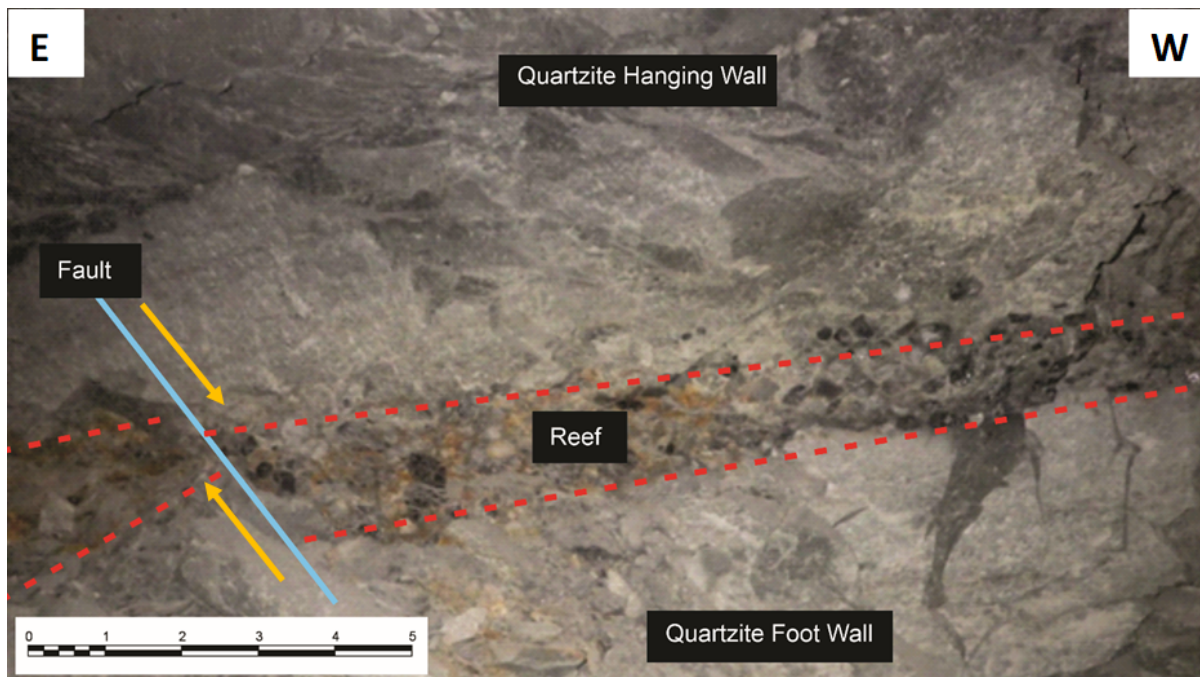


Figure 3. 2: A section photograph of an underground mapped and sampled face depicting the Vaal Reef.

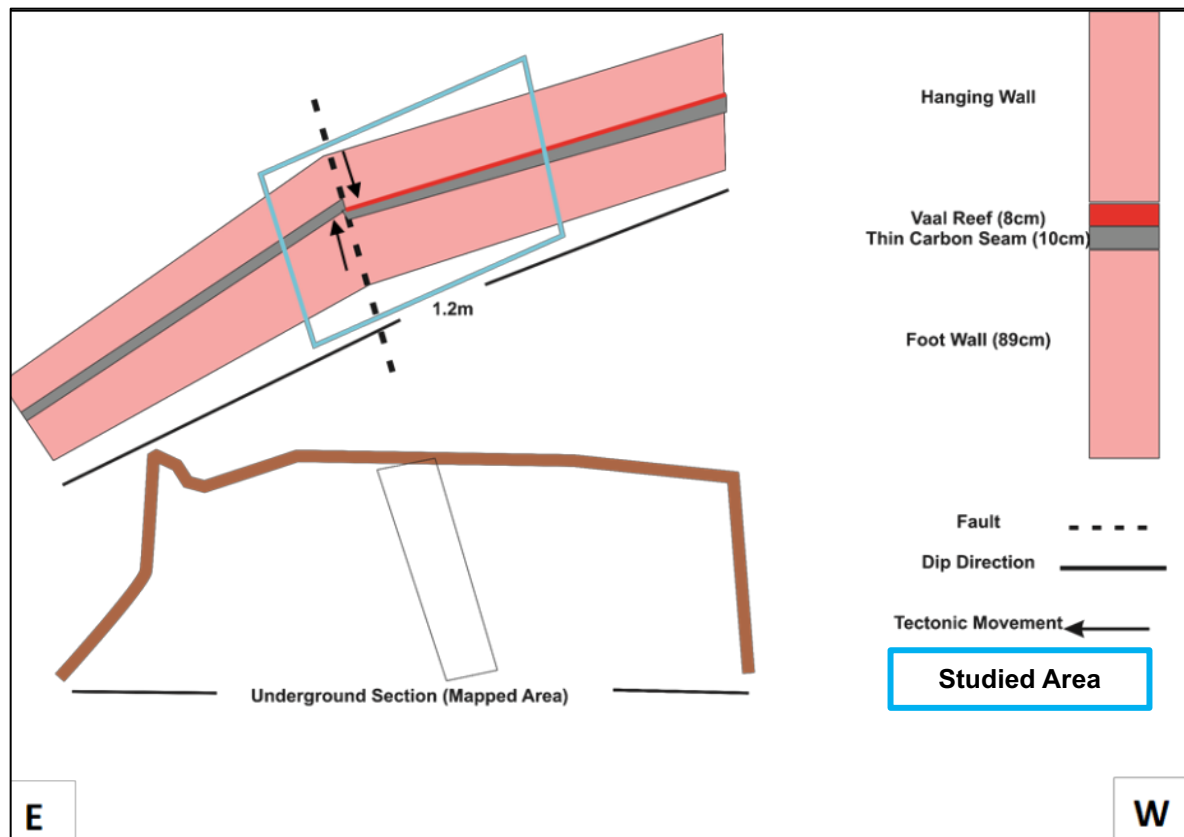


Figure 3. 3: Vaal Reef mapped face (underground workplace section: 59. DW14.R1. P.2).

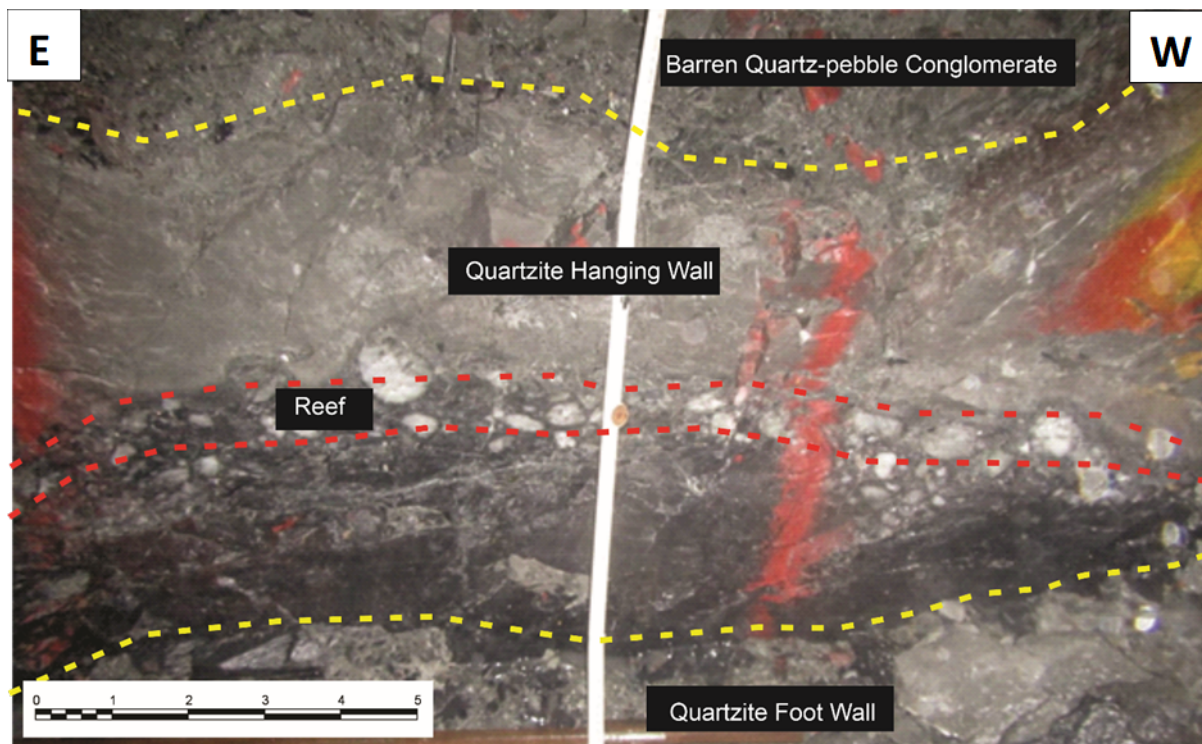


Figure 3. 4: A section photograph of an underground mapped and sampled face depicting the Crystalkop Reef.

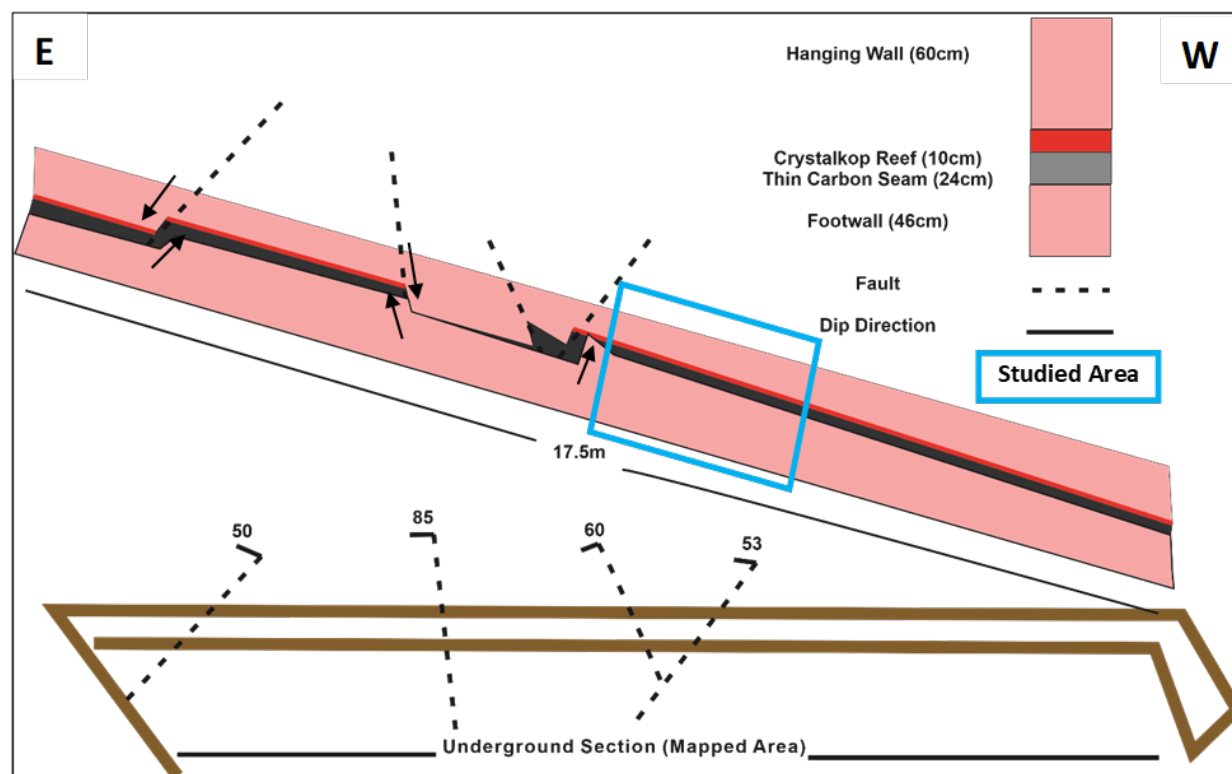


Figure 3. 5: Crystalkop Reef mapped face (underground workplace section: 42.BW.26.RSE.P9. E).

3.1.2 Sampling

The samples used for this study were obtained directly from an active underground mining operation. Chip sampling was done at an interval of 2.5 m along a 17.5 m and 15.6 m long exposed face of the Vaal Reef and Crystalkop Reef, respectively (Figure 3.3 and Figure 3.5). Sampling targeted the pyrite- and quartz-bearing conglomerates of both reefs.

Although some parts of the reefs were not accessible due to faulting where the reef was not exposed on the face, efforts were made to ensure that the mineralogy accurately reflected the two sections of focus and material that were later being sent to the plant for processing and extraction. To prevent cross contamination, and for further analysis and storage, each sample was placed in a separate sample bag. The sample bags were labelled with a name tag from CR1 to CR8 for the Crystalkop Reef and VR1 to VR8 for the Vaal Reef, where CR represented Crystalkop Reef and VR represented Vaal Reef.

3.2 Laboratory work

3.2.1 Polished thin sections and ore block

The procedure of preparing the ore blocks involved cutting and trimming, mounting, bonding, and polishing which was conducted at the North-West University geology laboratory. The criteria for selecting these samples were based on variations of physical characteristics of the ore samples. These characteristics included mineral composition, variations in matrix colour, macroscopic characteristics and texture. Thin sections were studied to compare and characterise the Vaal Reef and Crystalkop Reef based on their mineralogical composition and physical properties to shed some light on their effect on gold processing.

A total of six thin section and six polished ore blocks (three from each reef, respectively) were made and the mineral assemblages, gold particles, and mineral textures were analysed on a micro-scale using a Nikon Eclipse 50iPOL petrographic light microscope, under 10x, 30x and 50x magnifications. Thin sections were analysed under transmitted light (which will be discussed in detail later).

SEM-EDS

For both qualitative and quantitative investigations of any sample, the presence of EDS capabilities within an SEM instrument is critical (Mohammed and Abdullar, 2018). In the absence of EDS, SEM can only provide information on the specimen's surface topography (Mohammed and Abdullar, 2018). The SEM was operated at an acceleration voltage for capturing of secondary electron photomicrographs at a working distance from the surface of the polished sections. The EDS and images acquisition were accomplished through auto-beam and windows software, at a different magnification (Mohammed and Abdullar, 2018).

The polished thin section samples that were used for transmitted light microscopy were also analysed using a Zeiss EVO® MA15 SEM, which was equipped with a Bruker Energy Dispersive X-ray Spectrometer (EDS) and operated with Quantax software. The SEM functioned at an acceleration voltage of 20 kV, a working distance of 12 mm, and a stage tilt angle of 0.0°. The analysis relied on a bright phase search technique, which identifies uranium-bearing minerals by detecting their higher backscattered electron intensity in comparison to gangue minerals. A limitation of the method was that the equipment was calibrated only for Secondary Electrons (SE) imaging and not for backscattered electron (BSE) imaging, preventing the utilisation of BSE for analysis.

3.2.2 Comminution

A significant portion of the additional mineralogical testing, chemical assays and subsequent leaching required comminution at different levels. While size reduction is often emphasised for mineral liberation and preparation of material for cyanidation, it is equally important for obtaining representative samples for chemical assays. Proper comminution ensures that all mineral phases are proportionally represented, reducing sampling bias and improving the reliability of analytical results (Pitard, 1993; Gy, 1998). Following Gy's sampling theory, achieving a particle size below the safety line helps to minimize segregation and ensures that subsamples taken for assay accurately reflect the composition of the bulk material (Gy, 1998).

Comminution is simply a process of reducing the size of the ore samples and is essential in gold extraction as the recovery of gold from the ore is dependent on the liberation of gold mineral assemblages. Particle size distribution plays a vital role in the effectiveness and efficiency of mineral liberation within the ore. Comminution involves crushing, grinding and milling of the ore to the required sample size (Fuerstenau and Han, 2003; Tabatabaei *et al.*, 2014; Youlton, 2020). The optimal grind size for gold leaching is 80% passing 75 μm (Youlton *et al.*, 2021), though 50% passing 75 μm has been suggested for gold quantification (Coetzee *et al.*, 2011; Ofori-Sarpong and Amankwah, 2011). Although gold lost to tailings is associated with the presence of natural carbon within the ore (Melis and Rowson, 1989), sometimes not understanding gold grain shapes and size distribution influences the loss of gold as well (Mitchell *et al.*, 1997). To maintain consistency with standard leaching conditions at the plant, all samples in this study were milled to 80% passing 75 μm .

Crushing and milling

Three feed samples (Vaal Reef, Crystalkop Reef, and Composite Reef (a composite of the two reefs which was blended into one sample) were first crushed using a Keegor roll jaw crusher. After crushing, samples were split using a riffle splitter and then milled using a Siebtechnik rotating ball mill at North-West University, Department of chemical and minerals engineering, Biofuels laboratory. One kilogram of each sample was milled to 80% passing a 75 μm screen as per the standards used at AGA Great Noligwa Plant.

Following the standards of the mine where the Vaal Reef and Crystalkop Reef feed samples are mixed at the plant, a composite of the Vaal Reef and Crystalkop Reef was also done for this research by mixing and blending the two bulk reef aliquots at a 90%:10% ratio to obtain a combined homogenised composite aliquot (Composite Reef).

A particle size distribution test was conducted in triplicate on all three feed samples using a Malvern Hydro Mastersizer (MHM) to confirm the optimal mill size (80% passing 75 µm screen and 3% retained on the 150 µm screen). Milling curves produced for the milling masses are given in Appendix A. Milled samples were analysed for bulk mineralogy (MLA), chemical assay (XRF, Fire assay and ICP-OES). This was done to characterise the ore before metallurgical test work.

3.2.3 Mineralogy and assay

MLA

Quantitative mineralogical analysis was performed using an Auto-SEM, specifically a Mineral Liberation Analyser (MLA) QUANTA FEG 600 located at the University of Johannesburg, Department of Geology. MLA-based mineralogical analysis was conducted on six individual samples (three from each reef) which were the same samples that were used for optical microscopy. The method was used to determine textures, mineral associations, grain size distributions and abundance of gold mineral phases of milled sample material.

The primary modal mineral abundances were analysed using the rapid XMOD mapping technique (point count mapping), which is effective for identifying minerals present in major to minor quantities. For the finer-scale investigation of specific trace minerals, such as uranium-bearing phases, the slower SPL-GXMAP routine was used. This approach is more suitable for detecting and analysing trace components with concentrations well below 1 wt.% and captures chemical variations within individual mineral grains.

Analysis set-up was conducted with a SPL-LT-MAP which is specifically designed for bright phase search of in-situ material (SEM set-up conditions are reported in Annexure C). Furthermore, mineral chemical compositions were obtained by Energy Dispersive Spectrometry (EDS) using a Brucker energy dispersive spectrometer and Brucker Esprit software 1.9 which the MLA is coupled with.

XRF

Henderson (2013) stated that X-ray fluorescence spectrometry is capable of rapid determination of elements, and it is well suited for high throughput requirements. The method requires the samples to be milled into powder form so that about 10 g of the sample may be used to press a pellet prior to analysing using the XRF spectrometer. For this reason, the application of the technique in cases when the minerals are separated or when the samples are small, is limited. The technique is dependent on the quantitative measurements of the X-rays that are emitted

during 21 electronic transitions within the atoms excited by irradiation with primary X-rays (Henderson, 2013). The X-rays are released in the form of lines with discrete wavelengths in such a way that each element has a unique spectrum of lines.

Three feed samples (Vaal Reef, Crystalkop Reef, and Composite Reef) were used for both chemical assaying and leaching tests. Following the standards of the mine where the Vaal Reef and Crystalkop Reef feed samples are mixed at the plant.

The reef samples were crushed using a jaw crusher to reduce the size to a smaller manageable size that would fit into the mill rings for pulverising. After crushing individual samples, splitting was done using a riffle splitter to produce smaller representative samples which were later mixed to make three composite samples. Bulk samples were poured into the riffle splitter tray, which then allowed the material to flow down the chutes of the splitter. Subsamples were then collected at the bottom of each chute for further milling into smaller sizes. Samples were milled to powder using a ring milling machine. Each crushed sample was milled for approximately until 80% passed a 75 µm sieve as per the mine plant standard. To prevent cross-contamination between samples, mill rings were thoroughly washed with water and cleaned with acetone. Milled powder samples were prepared at the North-West university; Department of Geology and Science and the analysis was done at the Unit of Environmental Science and Management for X-ray fluorescence (XRF) analysis

ICP-OES

Houk *et al.* (1980) defined inductively coupled plasma optical emission spectrometry (ICP-OES) as an analytical method used for precisely analysing the concentration of trace elements and isotopic compositions. A typical ICP-OES instrument is composed of a pump, pneumatic nebuliser, spray chamber, ICP torch, and an optical emission spectrometer.

The entire process of analysing a sample within the instrument includes the introduction of a digested sample into the ICP torch (Beauchemin, 2008). Ionisation then happens inside the torch, where the sample is subjected to high temperatures that cause it to vaporise and form excited atoms and ions. These excited species emit characteristic wavelengths of light, which are then measured by the optical emission spectrometer. The intensity of the emitted light is directly proportional to the concentration of the elements in the sample (Kashani and Mostaghimi, 2010).

The ICP-OES analytical method has, over time, gained interest in accurately determining the concentration of gold and other trace elements in geological materials such as ores and soils due to its excellent detection capabilities (Khalil *et al.*, 2023). Although analysing digested solutions

using ICP-OES presents challenges, this method is known to have better throughput and be more sensitive in determining gold contents than other elemental analysis techniques (Tao *et al.*, 2017).

Tao *et al.* (2017) stated that a few factors can affect the sensitivity of the method, including the low ionisation efficiency of gold and the presence of oxides and hydroxides of matrix metals. Although this is the case, Gondonnmeau *et al.*, (2000) stated that to accurately determine and analyse gold quantities using ICP-OES, geological materials need to first be dissolved using high-purity acids. This ensures that trace elements present in very low concentrations can be measured accurately.

Accuracy can also be increased by either removing interferents using a collision cell procedure (Khalil *et al.*, 2023) or an aerosol dilution technique (Tao *et al.*, 2017). A study on the methodology of gold analysis by ICP-OES by Gondonnmeau *et al.*, (2000) has shown that ICP-OES provides better quantitative results of trace elements in gold ores compared to LA-ICP-MS, PAA, and FNAA methods.

ICP-OES was used on the leached samples to determine the gold grade in the ore samples. First, the samples were digested with aqua regia to dissolve the gold, followed by filtration to remove any residues. The ICP-OES system was calibrated using AMIS standard solutions of known gold concentrations, creating a calibration curve.

The prepared samples were introduced into the plasma, where gold atoms emitted light at specific wavelengths. The intensity of the emission at 242.795 nm was measured and correlated to the gold concentration using the calibration curve. Results were reported in parts per million (ppm) or grams per ton (g/t). Accuracy was ensured by running blank samples, duplicates, and certified reference materials alongside the test samples. The analytical accuracy was within $\pm 2\%$ and the method uncertainty was estimated at $\pm 5\%$ (1σ). ICP-OES was conducted at the NWU, School of Chemical and Mineral Engineering, Bio-fuels laboratory.

3.2.4 Leaching test work

Metallurgical test work was conducted on a composite sample to evaluate the leachability of gold and uranium, with a primary focus on gold recovery. The sample was milled to 80% passing 75 μm and divided into three 1 kg aliquots for the following tests forward leaching where gold was leached from raw material using sodium cyanide (safe handling HIRA is added on Annexure E), reverse leaching where gold was leached from the tailings of the uranium acid leach, and diagnostic leaching (Figure 3.6). Metallurgical work was conducted necessary laboratory standard

procedure for safe handling of cyanide. Hazard identification and risk assessment was also used (Annexure D and Annexure E).

Both forward and reverse leaching were performed under the conditions used at the Great Noligwa plant (Annexure A) to assess the impact of uranium acid leach as a pre-treatment on gold recovery. Additionally, a diagnostic leach was conducted following the methodology outlined by Lorenzen (1992).

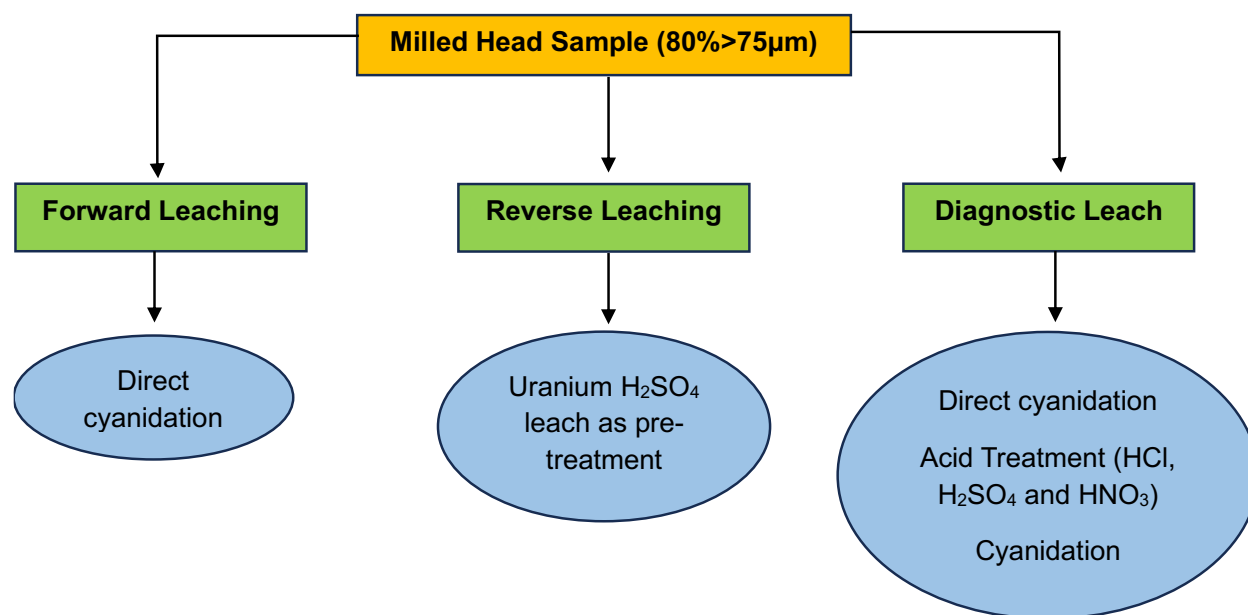


Figure 3. 6: Simplified metallurgical test workflow chat

Forward Leaching

Gold leaching was conducted at the NWU School of chemical and minerals engineering, Biofuels laboratory using a bottle roll testing method. A total of nine samples were run in sacrificial tests for time intervals (4, 8, 16 and 24 hours) with duplicate samples for 2, 8, 16 hours' time interval and triplicate samples for 24-hour time interval.

Milled solids of the Composite Reef were weighed on top of the pan balance and each mass of the 9 duplicated samples was recorded before transferring the sample into a 500 ml reagent bottle. A 1.45 relative density of each sample was prepared by adding a volume of distilled water into a reagent bottle with dried solid sample to make a pulp.

A densimeter was used to check if the density was within the required range of 1.45. The pH of each pulp sample was measured using a pH meter and the pH was subsequently increased to >10.5 using CaO. After increasing the pH, the sample was agitated using a magnetic stirrer for 1 hour before adding 360 g/t of NaCN into the pulp.

After adding the NaCN to the solution, the reagent bottle was closed with a lid that had a hole to allow oxygen into the sample. The reagent bottle was then placed on the roller bed inside a fume hood and agitated at 40 rpm for the following time slots: 4 hours, 8 hours, 16 hours and 24 hours. After each time elapsed, samples were filtered to obtain a filtrate sample and a solid sample. Solid samples were dried at 105 °C. Both filtrate and solid samples were transferred into sample containers for gold analysis on the ICP-OES. The solid samples were digested using aqua-regia (nitric and hydrochloric acid) to determine the gold content. The remainder of the solids were sent for XRF analysis and fire assay (FA) for comparative gold assaying.

The residual cyanide was titrated with silver nitrate. Titration was done to determine the concentration of the acid, in this case the concentration of the cyanide and this was done by preparing a solution of 0.01 mol.dm⁻³ silver nitrate and 0.2 g rhodanine indicator in 100 ml acetone before transferring the silver nitrate solution to a clean burette. Fifty ml of the solution sample was added to an Erlenmeyer flask while adding a few drops of rhodanine indicator. The solution was then titrated with silver nitrate until the colour was orange to salmon pink, after which the residual cyanide in the sample was calculated.

Reverse Leaching

For the metallurgical leach tests, 100 g of each milled sample was weighed using a pan balance and transferred into eight 500 ml reagent bottles. To simulate plant conditions, 66.7 ml of distilled water was added to each bottle, achieving a slurry density of 1.50 g/cm³.

The slurry (100 g of ore and 66.7 ml of distilled water) was heated to 50 °C in an oven before adding 0.99g of H₂SO₄. The mixture was stirred using a shaker inside the oven at 130 rpm for 1 hour and 40 minutes. Subsequently, 0.60 g of manganese dioxide (MnO₂) was introduced, and stirring continued for another 1 hour and 40 minutes at 50 °C. After 100 minutes, an additional 0.81 g of H₂SO₄ was added, and the slurry was stirred for a further 17 hours at 50 °C.

Following leaching, the slurry was filtered into a flask using filter paper, and the filtrate was transferred into sample bottles for uranium analysis via ICP-OES. The solid residue was dried overnight at 100 °C. A 50 g portion of the dried solids was sent to SGS Laboratories in Orkney for

XRF analysis, while approximately 80% of the remaining dried solids were retained for gold leaching.

Fire Assaying

Wall (1972) defined fire assay as a traditional technique for analysing gold concentrations and other precious metals in a sample. Although this process has a few disadvantages, it is highly valued for its effective ability to accurately separate and quantify precious metals (Alex, 2023).

After leaching gold with sodium cyanide, solid samples were sent to the SGS laboratories in Orkney for determination of Au, by fire assay. The samples were initially weighed and combined with a fluxing agent before undergoing smelting. During this process, 15–20 drops of lead were introduced as a collector. The mixture was then heated in a furnace at 1000 °C. Once fully molten, it was poured into an iron mould. As the material cooled, the silicate slag rose to the top while the lead now alloyed with any precious metals settled at the bottom, forming what is known as a 'button'. This lead button, containing the concentrated precious metals, was subsequently subjected to cupellation for further extraction and purification. After the sample was fire assayed, it was then subjected to ASS for quantification of gold in the solids.

Diagnostic leaching

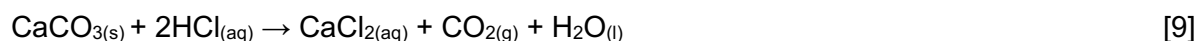
A diagnostic leaching procedure used for this study was adapted from Lorenzen and Tumilty (1992). A 300 g representative head sample, pulverized to 80% passing 75 µm (as confirmed by Malvern particle size analysis (Appendix A)), was used for the tests. The sample was slurried with 300 ml of deionized water, and the initial pH was measured. To ensure optimal cyanidation conditions, the pH was adjusted to above 10 using calcium oxide (CaO).

A sodium cyanide (NaCN) solution was added at a concentration of 5 kg/t, and the slurry was leached for 24 hours (Stage 1). The mixture was then filtered, and both the filtrate and the cyanide wash solution were collected for gold and cyanide analysis. This step targeted the easily recoverable free-milling gold. The remaining residue was subjected to a series of sequential leaching steps designed to dissolve specific mineral groups and release gold associated with them. Stage 1 of direct cyanidation was conducted using the Elsener Equation below:

Elsener Equation: $4\text{Au} + 8\text{CN}^- + \text{O}_2 + 2\text{H}_2\text{O} \rightarrow 4[\text{Au}(\text{CN})_2]^- + 4\text{OH}^-$ (Fleming, 1992). [1]

Stage 2 involved leaching the residue from stage 1 with hydrochloric acid (HCl) to dissolve acid-soluble minerals such as pyrrhotite, calcite, dolomite, and iron oxides. The acid leach residue was thoroughly washed with water before continuing.

The decomposition reaction with the soluble minerals and HCl is represented by equation 9 (Marsden and House, 2006; Chingwaru *et al.*, 2025):



This was followed by Stage 3, where another cyanide leach was performed (4 hours, 5 kg/t NaCN, pH >10), with both the leach and wash filtrates analysed for gold to assess recovery from minerals previously altered or dissolved by the HCl treatment.

Stage 4 involved leaching with sulfuric acid (H_2SO_4) to dissolve uranium-bearing minerals (e.g., uraninite, brannerite and coffinite) and base metal sulphides. This was again followed by thorough washing and gold analysis of the filtrates.

The oxidation reaction between sulphuric acid and soluble minerals is represented by equation 10 (Marsden and House, 2006; Chingwaru *et al.*, 2025):

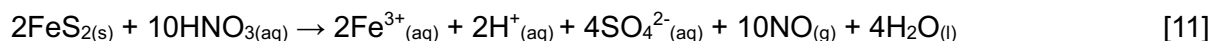


(where M represents Zinc (Zn), Copper (Cu), Uranium (U) and other metals present)

Stage 5 consisted of another cyanide leach under alkaline conditions, similar to the earlier ones, to extract any gold liberated by the sulfuric acid treatment.

In Stage 6, the residue was treated with nitric acid (HNO_3) to target refractory sulphides, especially pyrite and arsenopyrite, which are known to encapsulate gold.

The reaction between nitric acid and soluble minerals is represented by equation 11 (Celep *et al.*, 2009; Chingwaru *et al.*, 2025):



Finally, Stage 7 involved a last cyanide leach under alkaline conditions to recover any remaining liberated gold. At each stage, both leachates and wash solutions were collected and analysed for gold, and where relevant, for residual cyanide.

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Chapter 4: Mineralogy and chemical characterisation

This chapter focuses on the analysis and interpretation of the collected samples as well as the laboratory analysis done, as discussed in the previous chapter 3. Macroscopic description of the reef using physical properties was also conducted to characterise the two reefs. The mineralogical characterisation was done using optical microscopy under transmitted light, and SEM. This chapter also focuses on the characterisation of the gold bearing mineral phases, and gold grain size distribution using MLA. It's also in this chapter that the chemical analysis using XRF, ICP-OES, and Fire Assay (to compare and confirm the grade of the reefs) is reported.

4.1 Mineralogy

4.1.1 Physical properties

This section aims to characterise the physical properties of the Vaal Reef and Crystalkop Reef by giving a brief description of the macroscopic and microscopic properties of the two reefs. A total of six samples (three samples from each reef) were selected for petrographic characterisation, as examples of the reef, and not an entire representation of the whole reef. It is important to note that the samples used for macroscopic and microscopic descriptions were the same samples used for MLA analysis.

Macroscopic descriptions

Polished hand specimens were analysed by eye to identify physical properties and characterise them in terms of mineralogy, colour, matrix, packing and sorting, grain sizes and shapes and grain sphericity. Sphericity is the measure of how spherical the pebbles and grains are (Wadell H, 1932). Grain and pebble sizes were described using the convention in Appendix B. In Appendix C, a photographic representation and a description of hand specimen are given.

4.1.2 Petrography

This section aims to characterise the physical properties of the Vaal Reef and Crystalkop Reef by giving a brief description of microscopic properties of the two reefs. A total of six samples (three samples from each reef) were selected for petrographic characterisation as example representations of the reef, and not an entire representation of the whole reef. For the microscopic description, six polished thin section specimens were analysed under both cross polarised (XPL) and plane-polarised light (PPL). This was done to identify microscopic properties of both reefs and characterise them in terms of mineralogy, grain sizes and shapes, and matrix.

Sample VR1

In thin sections under transmitted light, Sample VR1 appears to be consisting of partly clast-supported and partly matrix-supported material. Mineralogically the reef conglomerate sample is dominated by sub-rounded to rounded quartz grains with fine-grained sub-rounded pyrite filling in the pore spaces. Recrystallised quartz grains are sub-angular, and they are bound with a matrix of sand, muscovite, and chlorite. Minor plagioclase grains were observed. The clasts are randomly orientated, poorly sorted, and moderately packed.

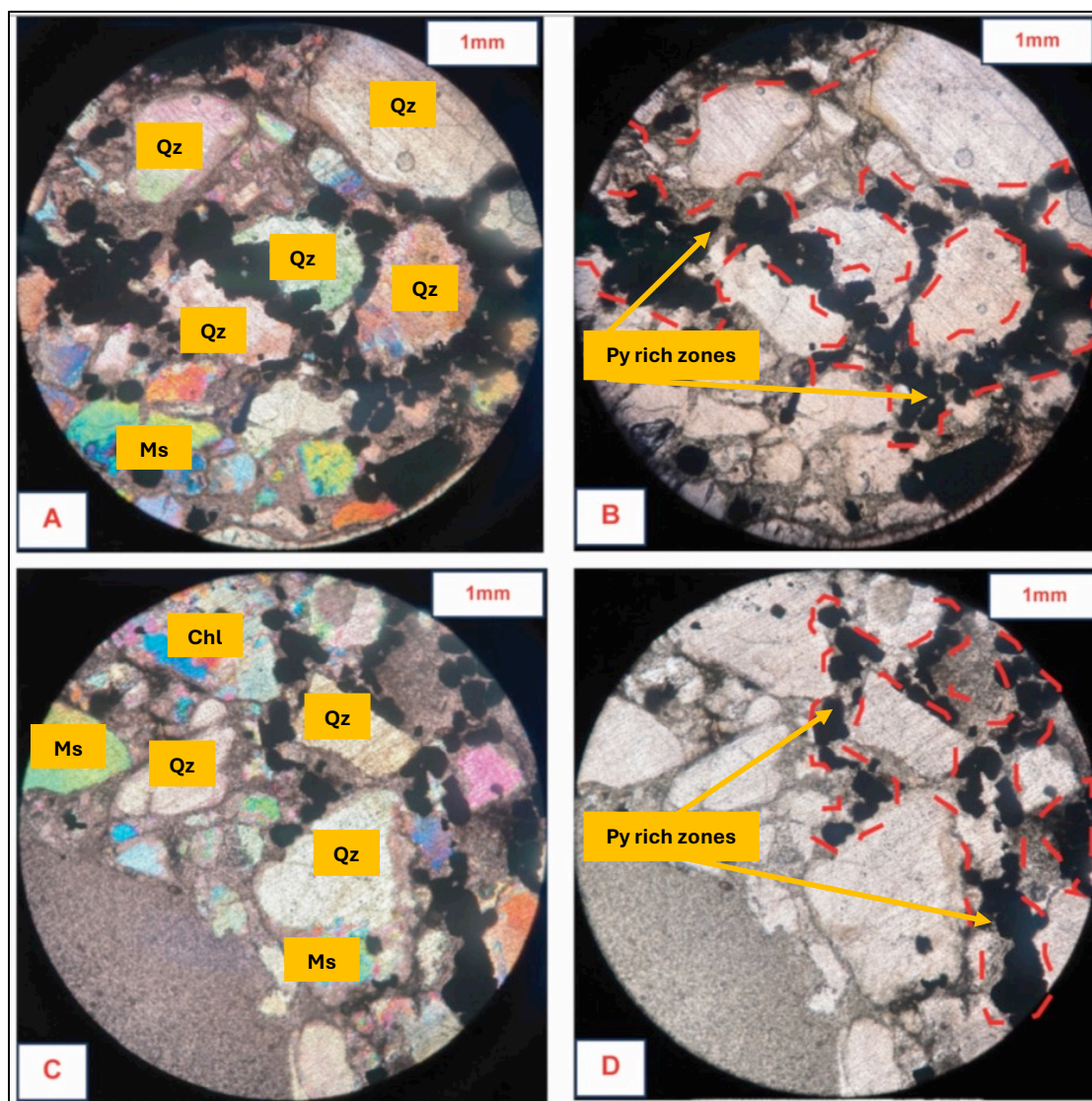


Figure 4. 1: XPL (left) and PPL (right) VR1 thin section photomicrographs indicating, in (A) and (C) quartz (Qz), muscovite (Ms) and chlorite (Chl), whilst in (B) and (D), pyrite (Py) rich zones between large quartz (Qz) grains.

Sample VR5

In thin sections under transmitted light, Sample VR5 appears to be partly clasts supported and partly matrix supported. Mineralogically, the reef conglomerate sample is dominated by sub-rounded to rounded quartz grains with sub-rounded pyrite finer grains filling in the pore spaces. Recrystallised quartz grains are sub-angular, and they are bound within a matrix of sand, muscovite, and chlorite. Minor plagioclase grains were observed. The clasts are randomly orientated, poorly sorted, and moderately packed.

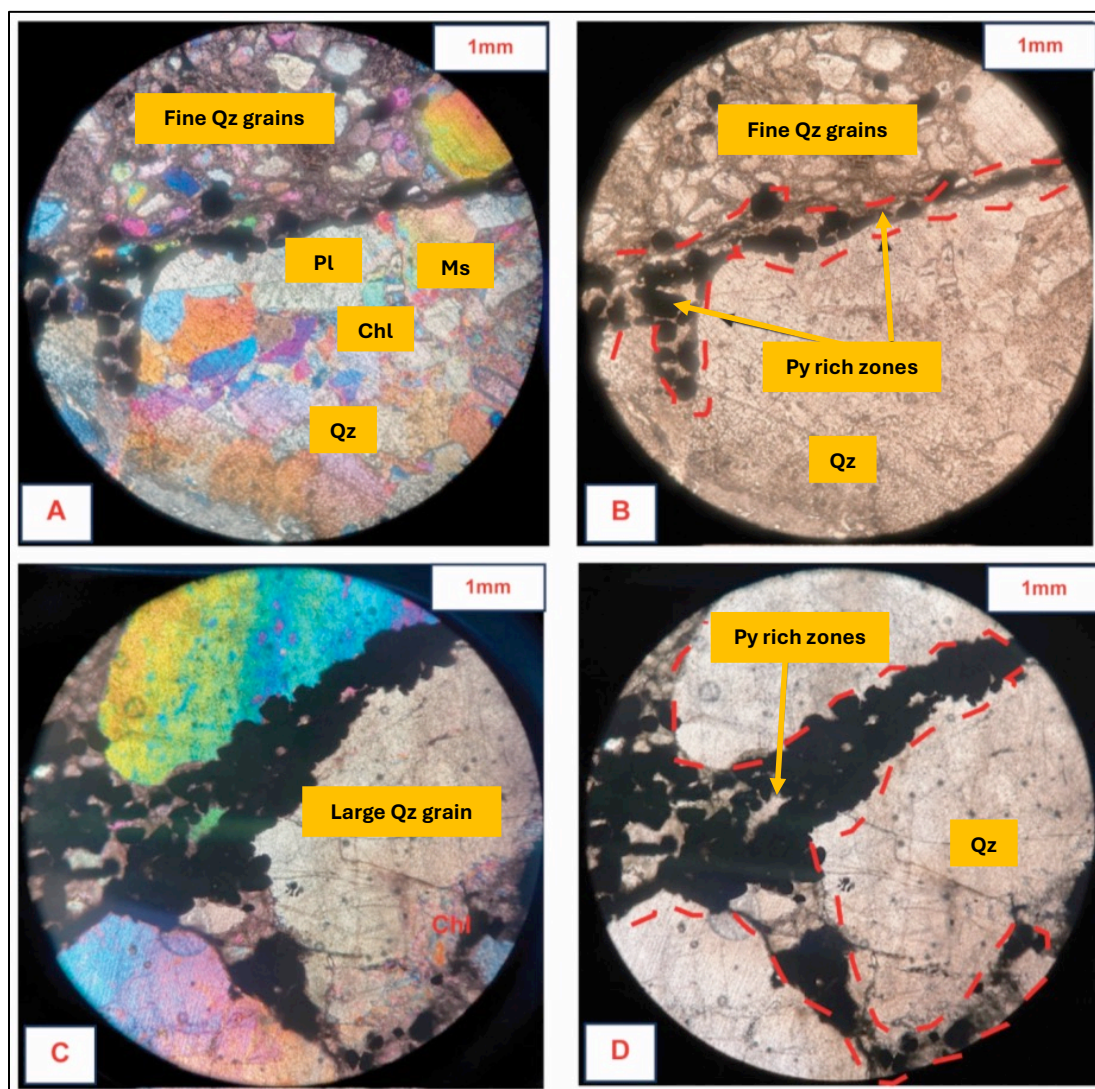


Figure 4. 2: XPL (left) and PPL (right) VR5 thin section photomicrographs indicating, in (A) and (C), fine quartz (Qz) grains, quartz (Qz), plagioclase (Pl), muscovite (Ms) and chlorite (Chl). (B) and (D) show pyrite (Py) rich zones between large quartz (Qz) grains.

Sample VR9

Under transmitted light, Sample VR9 appears to be a matrix-supported conglomerate. The quartz matrix sand is supporting sub-angular to sub-rounded quartz clasts. Sub-rounded pyrite grains occur as clustered grains along the larger quartz grains. The conglomerate is poorly sorted and loosely packed because it consists of quartz grains of different sizes and shapes, with quartz and muscovite sand filling in the porous spaces.

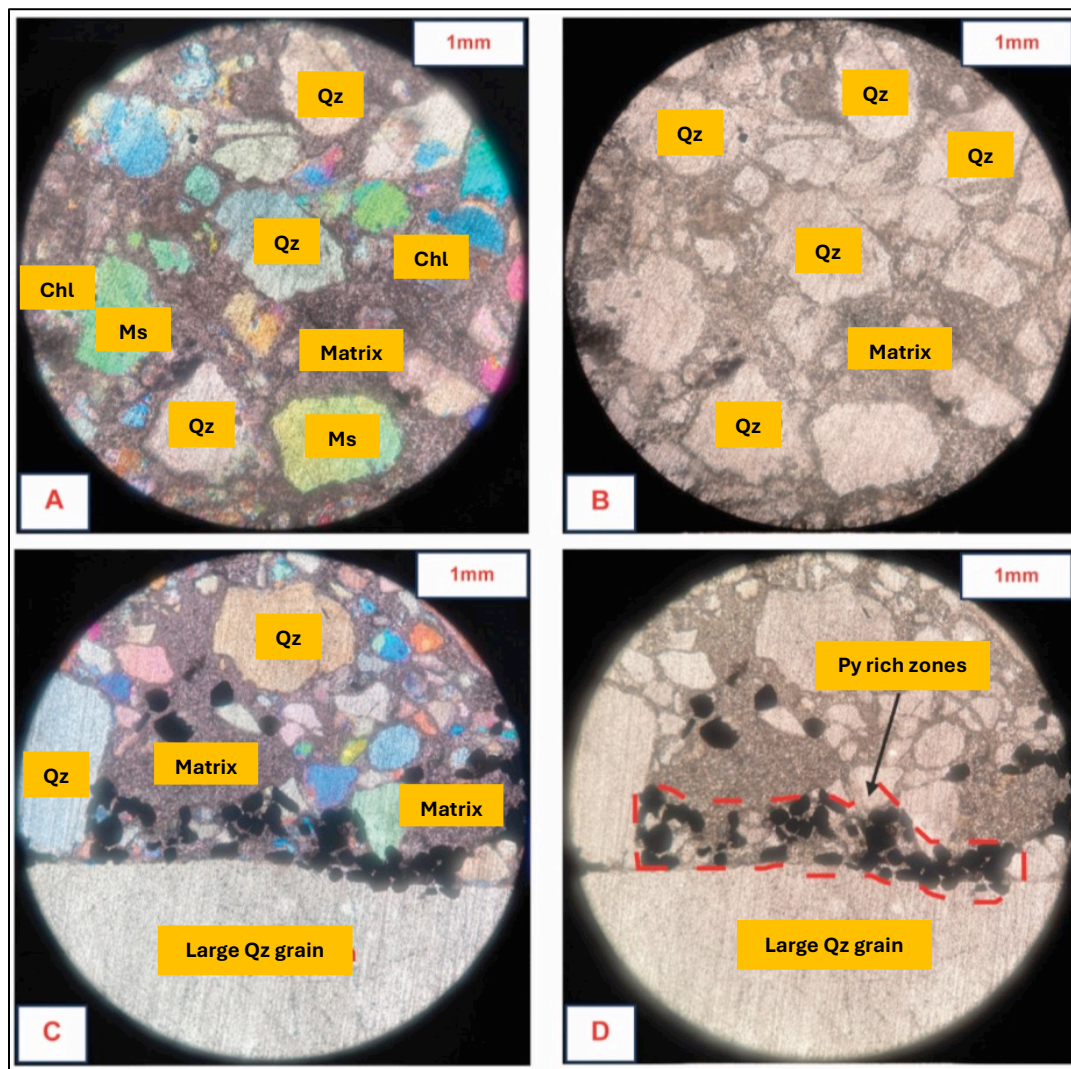


Figure 4. 3: XPL (left) and PPL (right) VR9 thin section photomicrographs indicating, in (A) and (C), quartz grains (Qz), muscovite (Ms) and chlorite (Chl) bound with fine quartz (Qz) grains. (B) and (D) pyrite (Py) rich zones between large quartz (Qz) grains within the matrix.

Sample CR1

Under transmitted light, Sample CR1 appears to be a clast-supported conglomerate with angular to sub angular quartz grains. The matrix is made up of fine to medium grains of quartz sand, muscovite, feldspars, and chlorite. Minor sub-angular to angular pyrite grains are randomly dispersed within the matrix. The sample is moderately sorted and moderately packed.

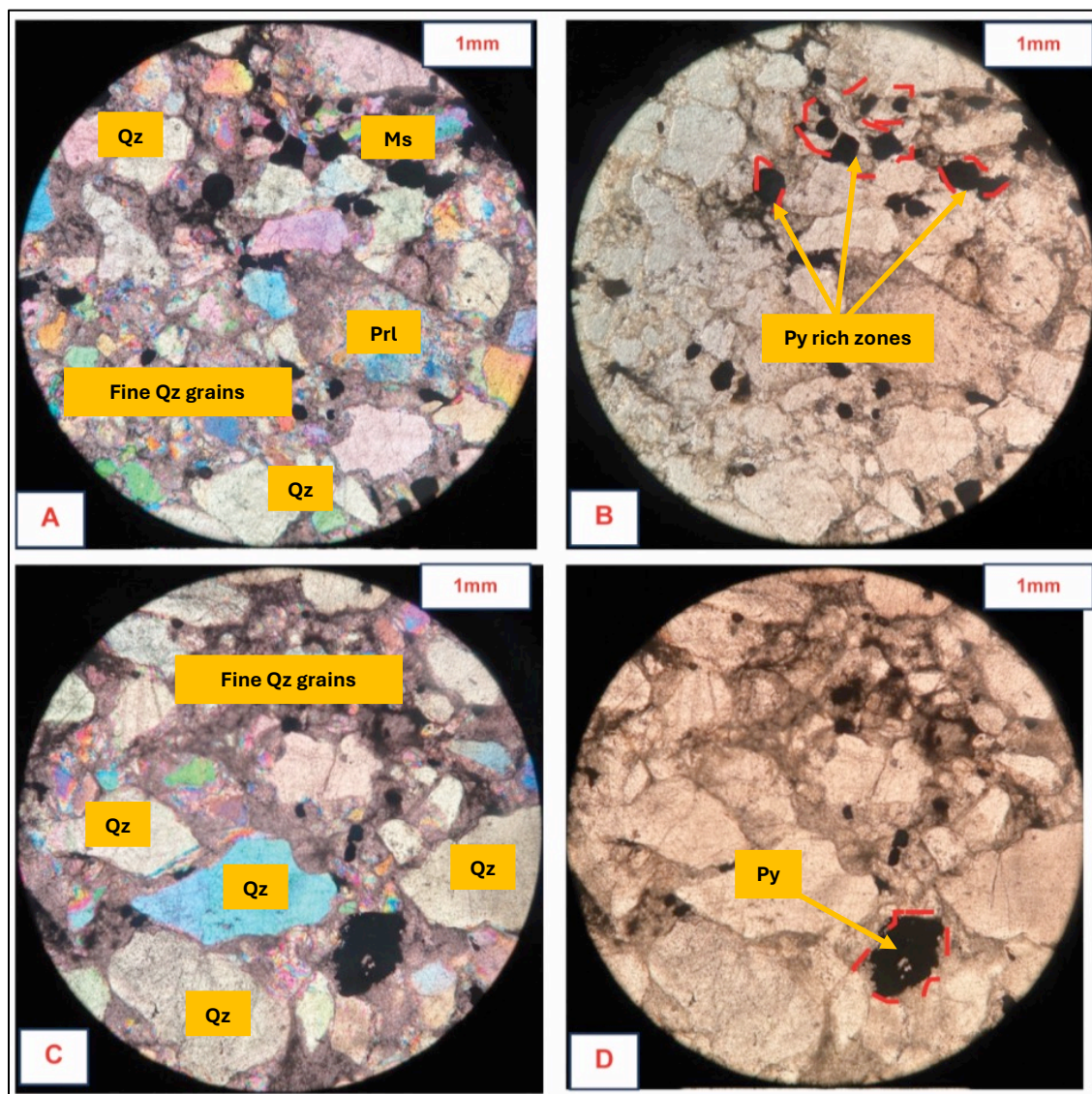


Figure 4. 4: XPL (left) and PPL (right) CR1 thin section photomicrographs indicating, in (A) and (C), re-crystallised quartz (Qz) grains, quartz (Qz), pyrophyllite (Prl), muscovite (Ms) and chlorite (Chl). (B) and (D) show pyrite (Py) rich zones sparsely distributed between large quartz (Qz) grains.

Sample CR2

Sample CR2 is a clast-supported conglomerate with angular to sub angular quartz grains. The matrix is made up of fine to medium grains of quartz sand, muscovite, feldspars, and chlorite. Minor sub-angular to angular pyrite grains are randomly dispersed within the matrix. The sample is moderately sorted and moderately packed.

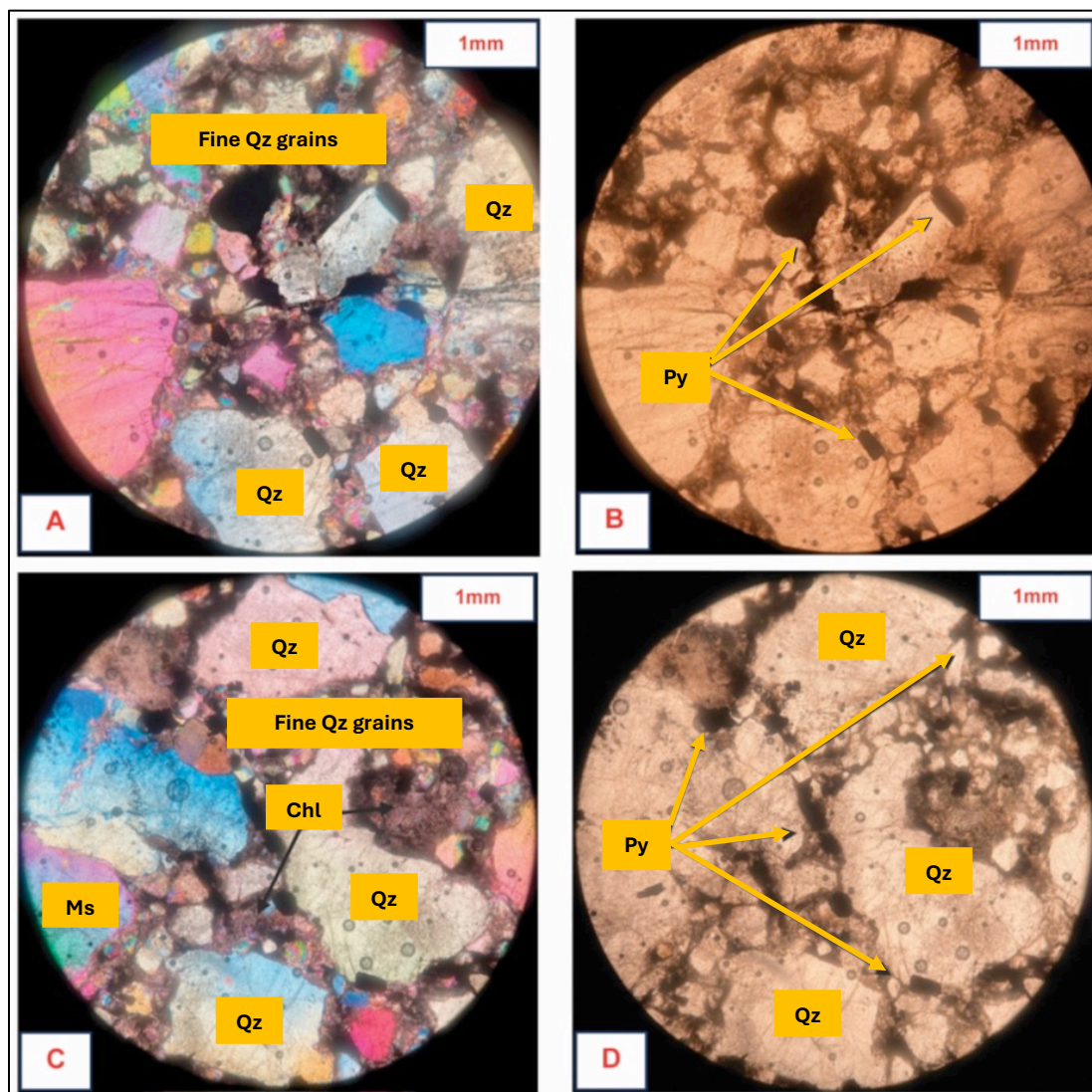


Figure 4. 5: XPL (left) and PPL (right) CR2 thin section photomicrographs indicating, in (A) and (C), re-crystallised quartz (Qz) occurring as fine quartz (Qz) grains, muscovite (Ms) and chlorite (Chl). (B) and (D) show randomly dispersed pyrite (Py) within a quartz (Qz) and muscovite-rich (Ms) matrix.

Sample CR7

Under transmitted light, Sample CR7 is a moderately clast-supported conglomerate with a matrix of coarse quartz, muscovite, and chlorite. Coarse quartz clasts are angular and finer quartz and muscovite grains are sub-angular. Minor pyrite is sparsely distributed within the matrix. The sample is moderately sorted and moderately packed with finer particle fillings of quartz sand, muscovite, pyrophyllite and chlorite in the pores between larger clasts.

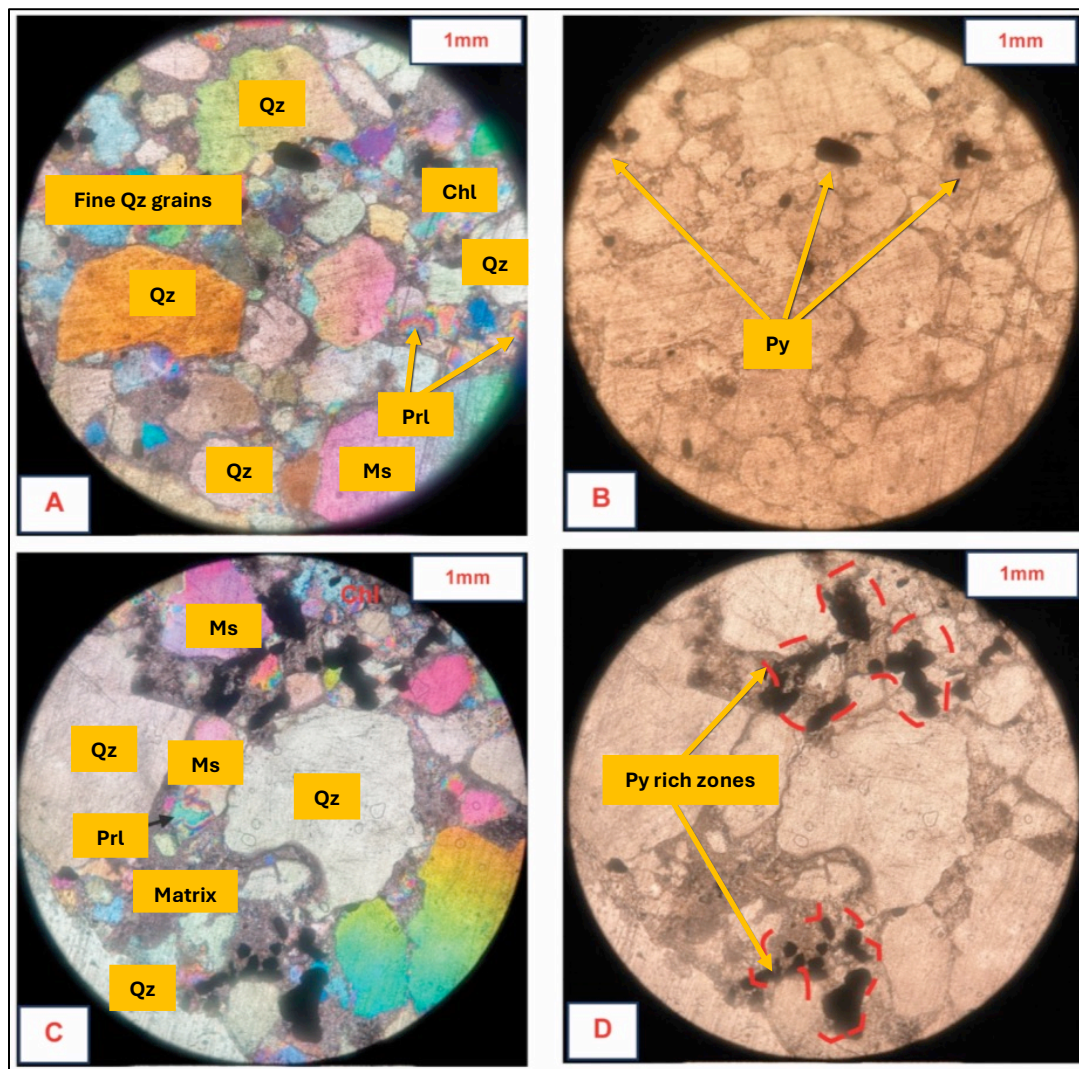


Figure 4. 6: XPL (left) and PPL (right) CR7 thin section photomicrograph indicating, in (A) and (C), quartz and muscovite grains (Ms) bound with finer particles of quartz sand (Qz), muscovite (Ms), pyrophyllite (Prl) and chlorite (Chl). (B) and (C) show pyrite (Py rich zone).

Microscopic descriptions under scanning electron microscopy (SEM)

Scanning Electron Microscopy was used to determine the spatial occurrence of gold within the ore, including whether any grains are locked within quartz. It was also employed to investigate the microscopic characteristics of the gold. This microscopic analysis was done on the same six samples that were used for transmitted light microscopy. It is important to note that the SEM technique used is limited to imaging discrete gold minerals, specifically those where Au is a primary component within the mineral's chemical formula. Furthermore, the SEM used, was not equipped with BSE and therefore only SE images were taken.

The microphotographs of the Vaal Reef samples (VR1, VR5, and VR9) show fine gold particles of less than 50 μm , some associated with sulphide minerals such as pyrite and others locked within quartz pebbles. Pyrite and pyrrhotite grains appear to be sub-angular to sub-rounded, with pyrrhotite occurring on the edges of pyrite mineral grains. Furthermore, within the matrix supporting quartz grains, pyrite grains are seen supported by fine quartz grains with other silicate minerals such as muscovite and pyrophyllite. Within sample VR5 (Figure 4.8 D), gold grains of less than 50 μm were noted. Sub-angular to sub-rounded pyrite grains appear as a matrix within quartz veins that are elongated and sub-angular. Silicate minerals such as chlorite and muscovite were also noted within the Vaal and Crystalkop Reefs samples (Figure 4.8, Figure 4.9, Figure 4.10, Figure 4.11 and 4.12).

The Crystalkop Reef microphotographs (CR1, CR2 and CR7) show that the samples are characterised by scattered and randomly sorted pyrite grains. Pyrrhotite appears along some edges of the pyrite grains (Figure 4.9 B). Under the SEM, the Crystalkop Reef samples are seen to have more gold grains locked within other minerals compared to gold grains that are along veins and within the matrix (Figure 4.10 C and D). Amongst other silicate minerals noted, the Crystalkop Reef C facies samples are also associated with an abundance of quartz, chlorite and muscovite and these gangue materials are occurring in larger grains of sub-angular to sub-rounded shapes.

Sample VR1

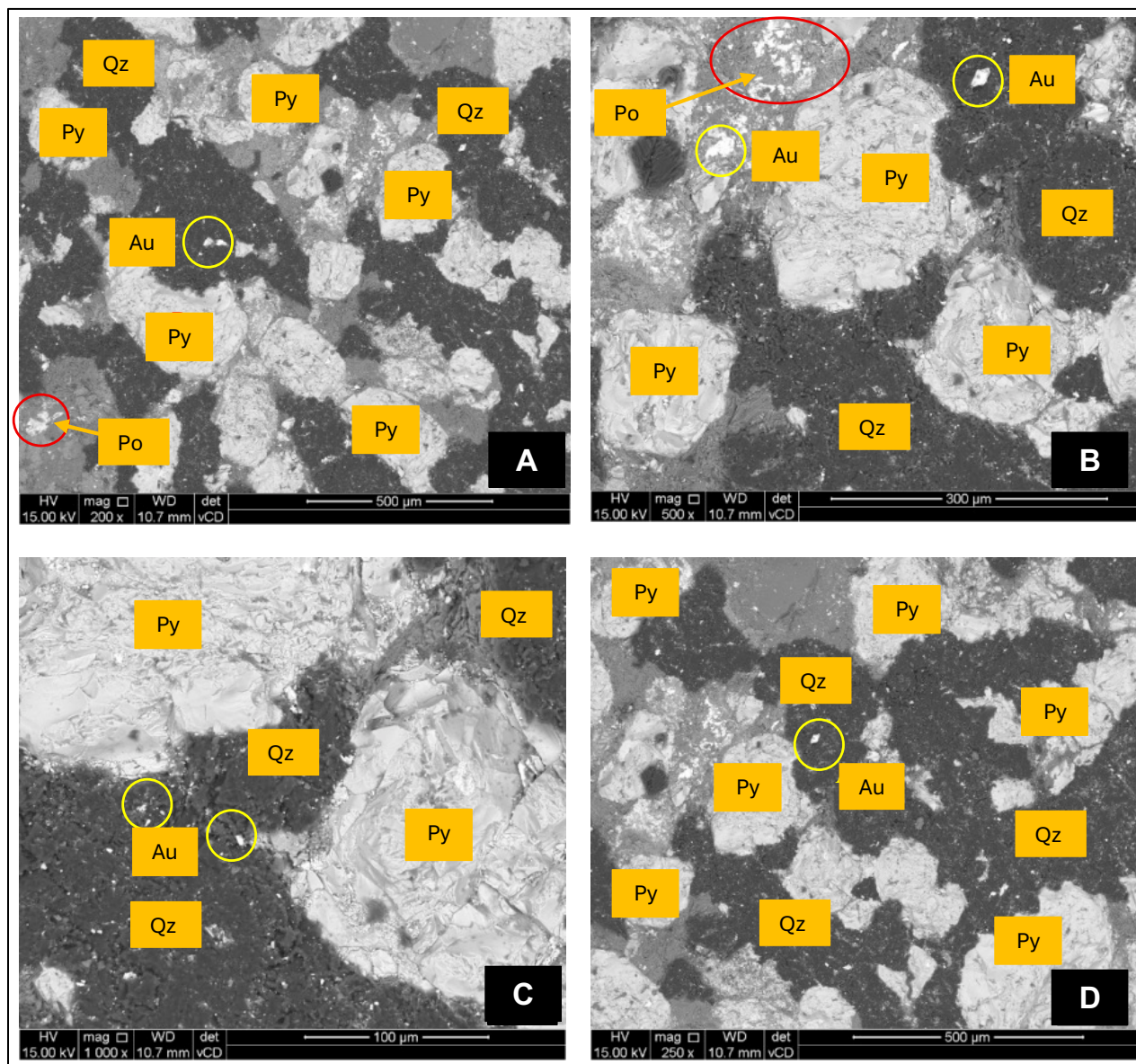


Figure 4. 7: Sample VR1 SEM SE image microphotograph. (A) contain quartz (Qz), pyrite (Py), pyrrhotite (Po) and fine particles of gold (Au); (B) contain quartz (Qz), pyrite (Py), pyrrhotite (Po) and fine particles of gold (Au); (C) contain quartz (Qz), pyrite and fine particles of gold (Au) and (D) contain quartz (Qz), pyrite (Py) and fine particles of gold (Au).

Sample VR5

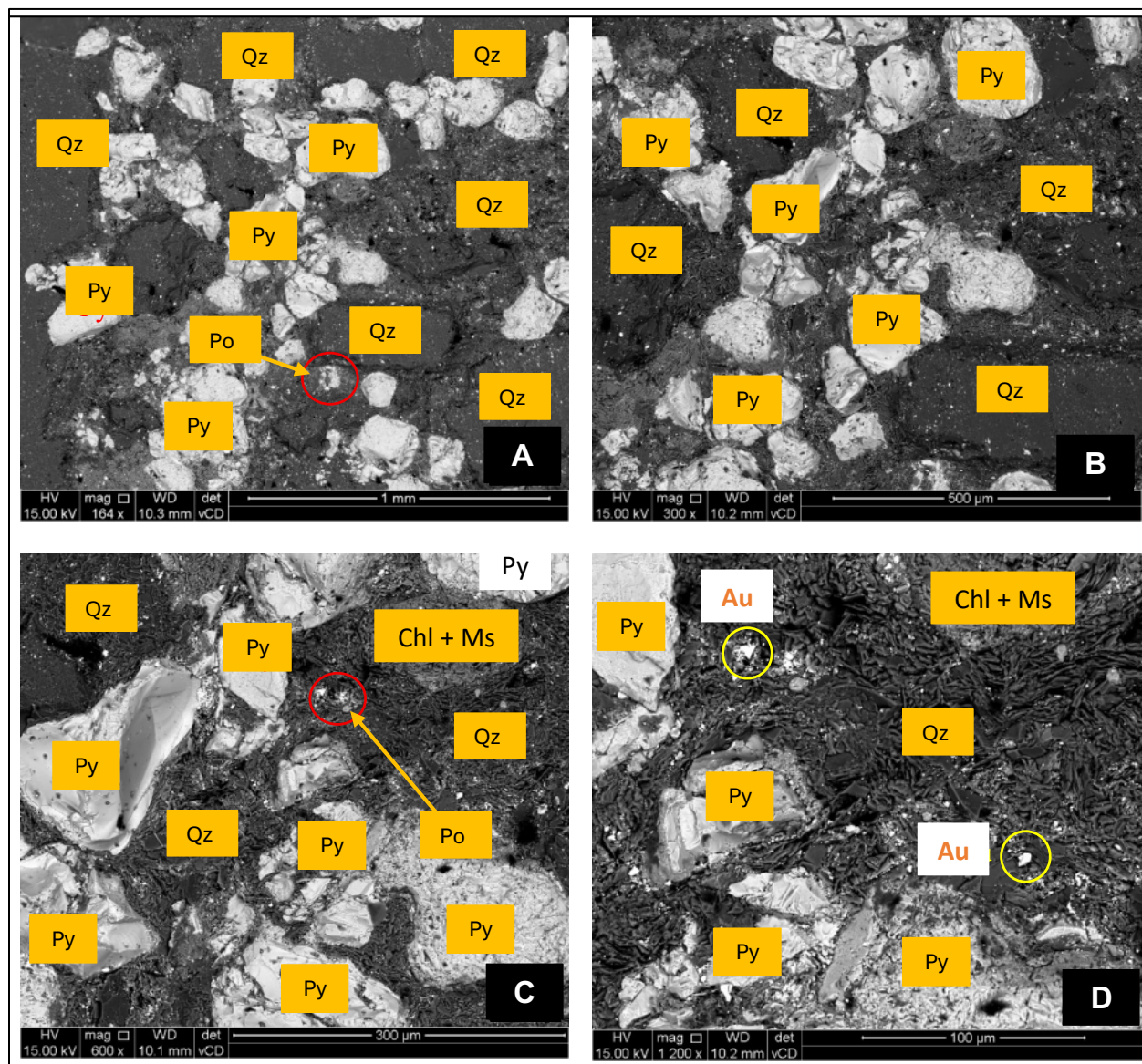


Figure 4. 8: Sample VR5 SEM SE image microphotograph. (A), contain quartz (Qz), pyrite (Py), pyrrhotite (Po); (B), contain quartz (Qz), pyrite (Py); (C) contain quartz (Qz), pyrite (Py), pyrrhotite (Po), chlorite and muscovite (Chl + Ms) and (D) contain quartz (Qz), pyrite (Py), fine particles of gold (Au), chlorite and muscovite (Chl + Ms).

Sample VR9

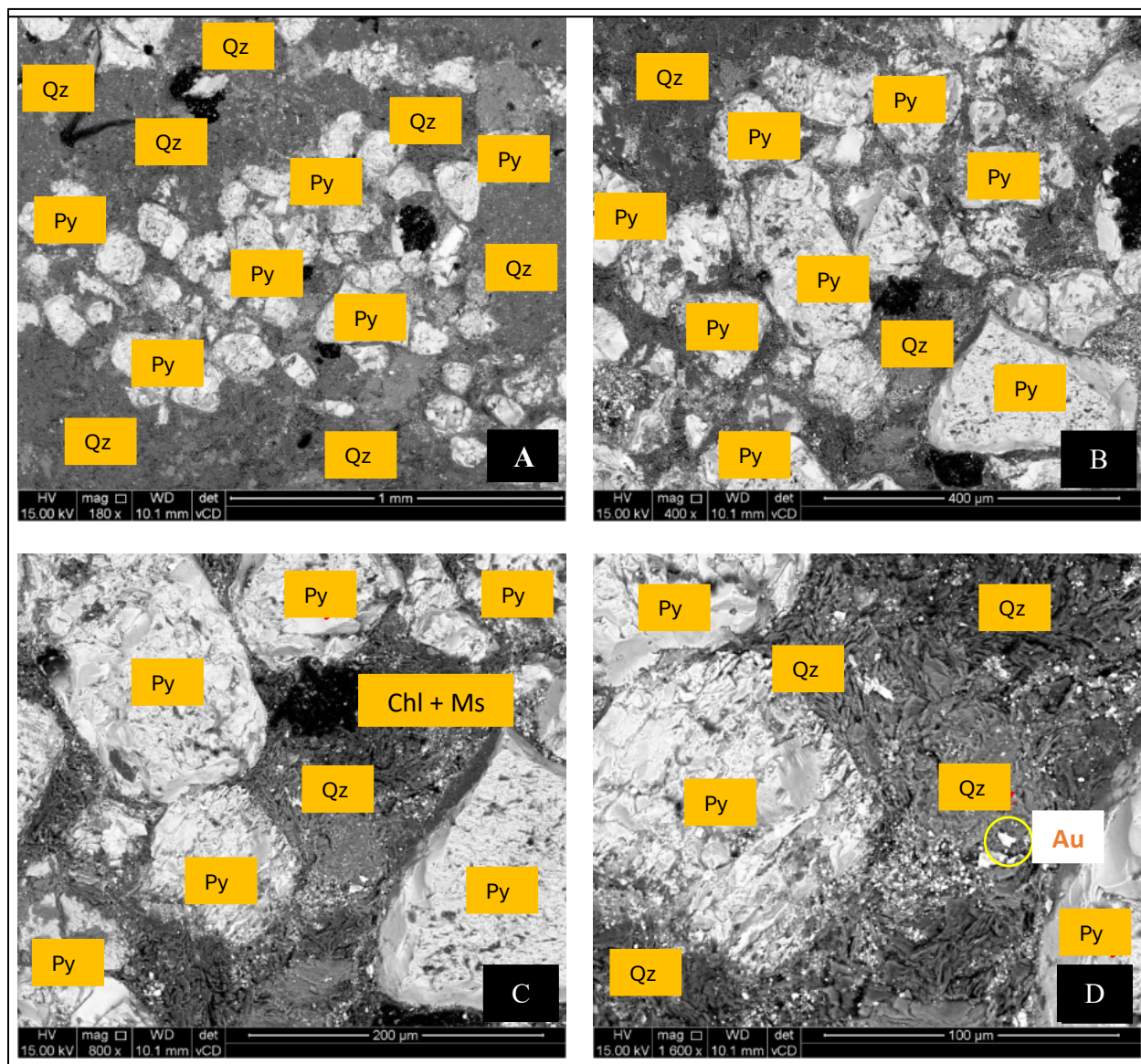


Figure 4. 9: Sample VR9 SEM SE image microphotograph. (A), contain quartz (Qz), pyrite (Py); (B) contain quartz (Qz), pyrite (Py); (C) contain quartz (Qz), pyrite (Py), chlorite and muscovite (Chl + Ms); (D) contain quartz (Qz), pyrite (Py), fine particles of gold (Au), chlorite and muscovite (Chl + Ms).

Sample CR1

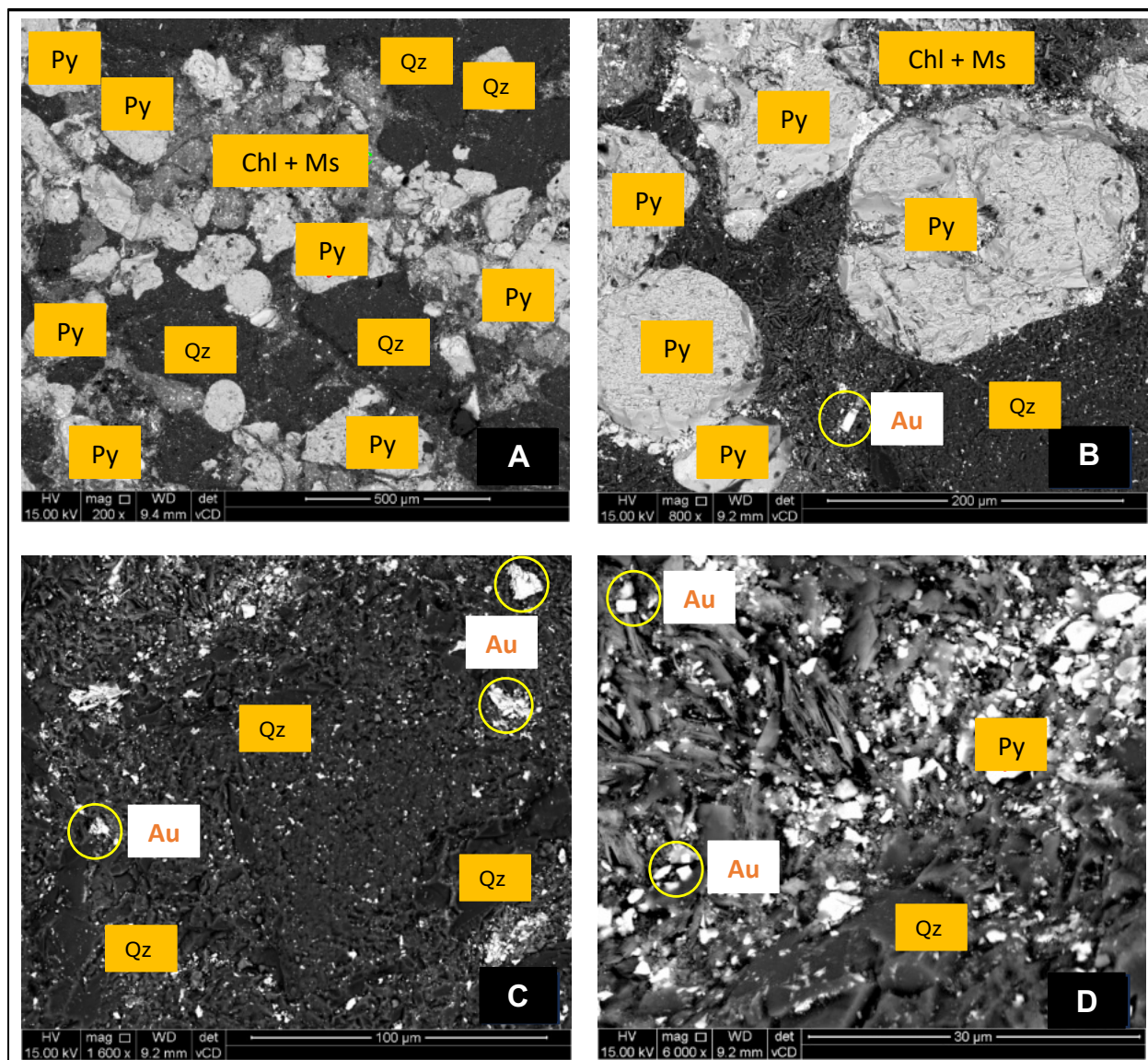


Figure 4. 10: Sample CR1 SEM SE image microphotograph (A), contain quartz (Qz), pyrite (Py), chlorite and muscovite (Chl + Ms); (B), contain quartz (Qz), pyrite (Py), pyrrhotite (Po); fine particles of gold (Au), chlorite and muscovite (Chl + Ms); (C) contain quartz (Qz), pyrite (Py), pyrrhotite (Po); fine particles of gold (Au); and (D) contain quartz (Qz), pyrite (Py), fine particles of gold (Au), chlorite and muscovite (Chl + Ms).

Sample CR2

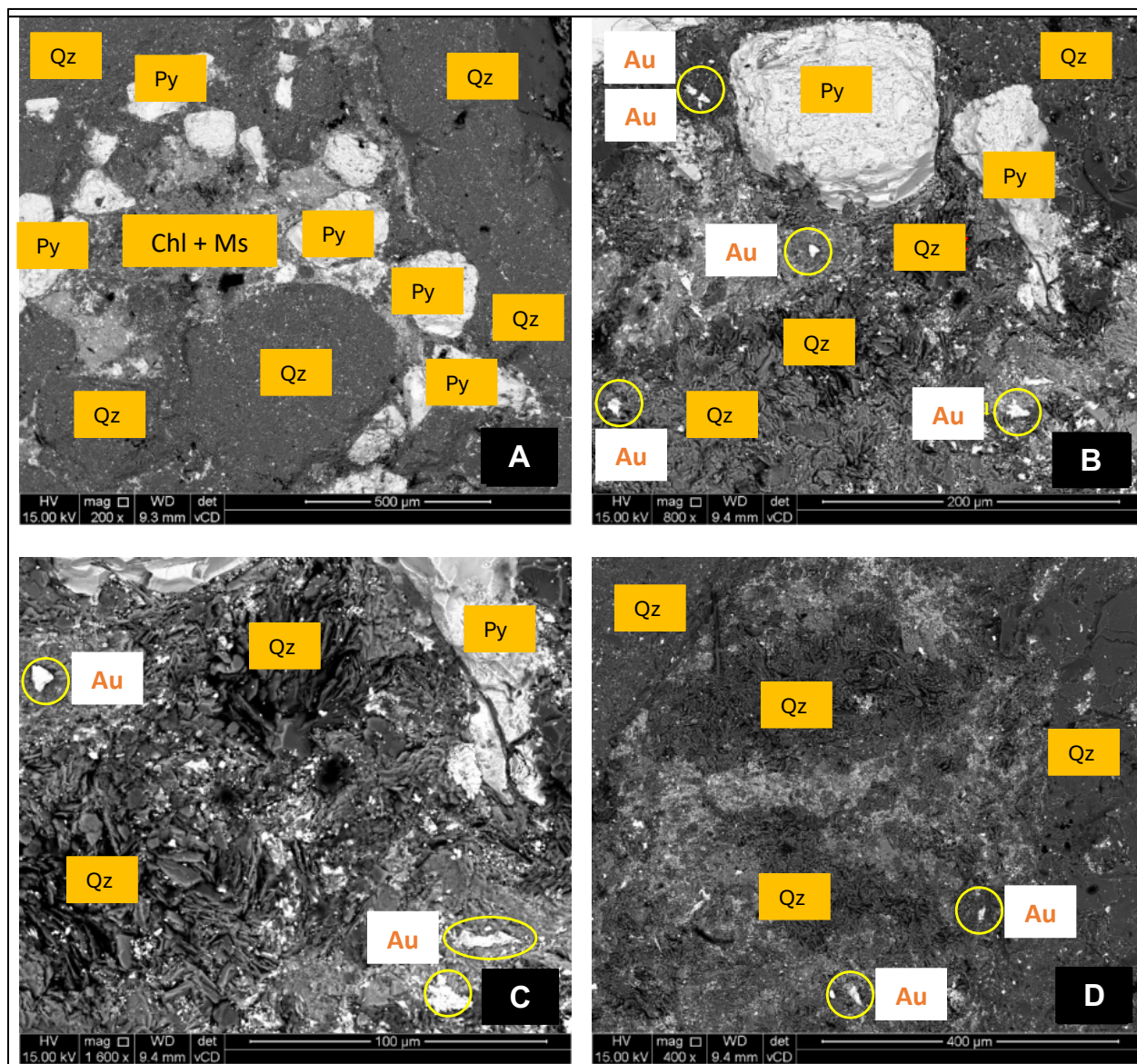


Figure 4. 11: Sample CR2 SEM SE image microphotograph. (A), contain quartz (Qz), pyrite (Py), pyrrhotite (Po); fine particles of gold (Au), chlorite and muscovite (Chl + Ms); (B), contain quartz (Qz), pyrite (Py), fine particles of gold (Au); (C) contain quartz (Qz), pyrite (Py), fine particles of gold (Au); and (D) contain quartz (Qz), pyrite (Py), fine particles of gold (Au), chlorite and muscovite (Chl + Ms).

Sample CR7

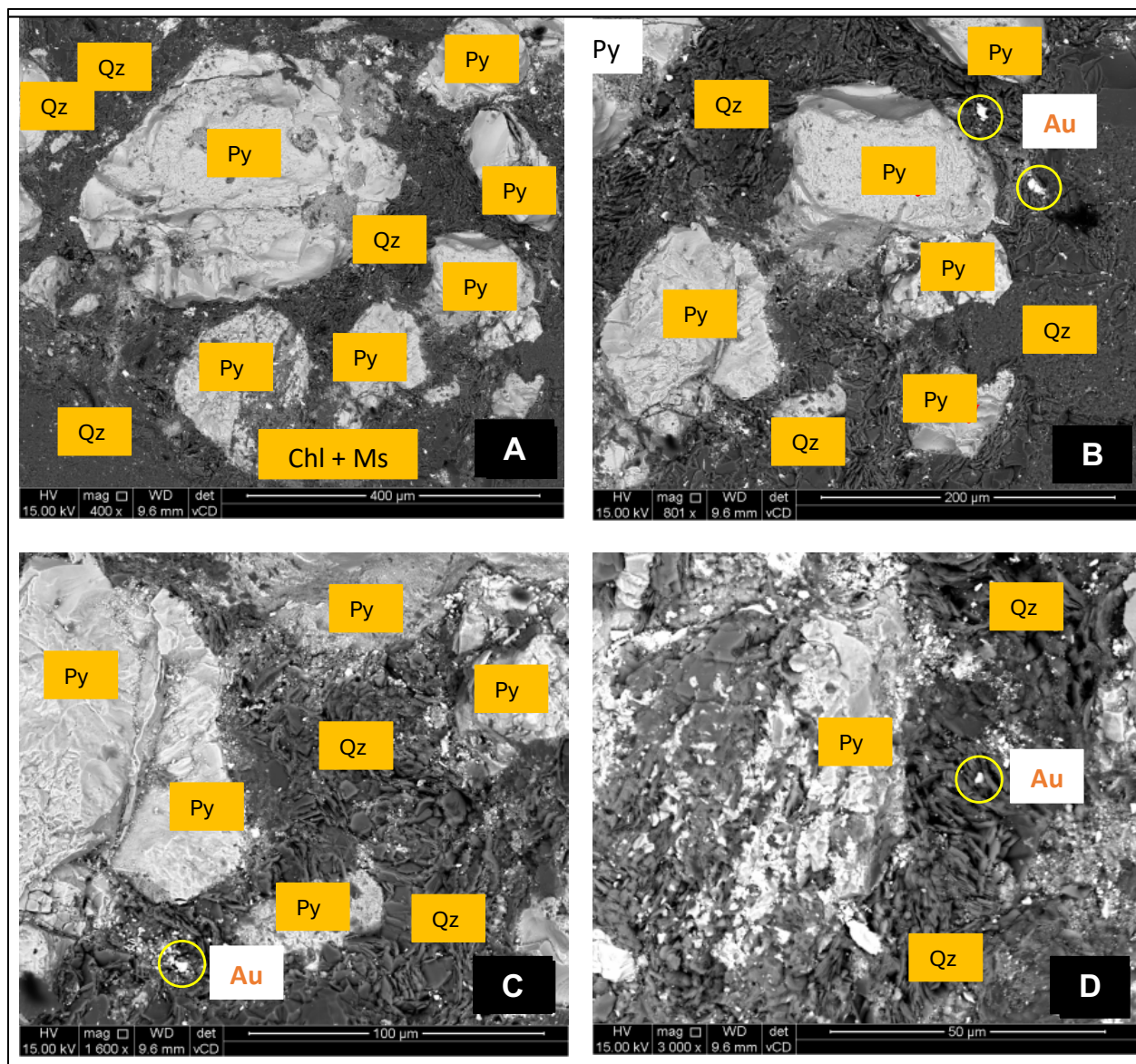


Figure 4. 12: Sample CR7 SEM SE image microphotographs. (A), contain quartz (Qz), pyrite (Py), chlorite and muscovite (Chl + Ms); (B), contain quartz (Qz), pyrite (Py), fine particles of gold (Au); (C) contain quartz (Qz), pyrite (Py), pyrrhotite (Po); fine particles of gold (Au); and (D) contain quartz (Qz), pyrrhotite (Po), pyrite (Py), fine particles of gold (Au).

4.2 Mineral deportment

This part focuses on the characterisation of the gold bearing mineral phases, and gold grain size distribution using MLA of material milled to the same grind that will be used in the leaching tests.

4.2.1 Gold particle count

The particle count was done using MLA with specific focus on native gold and three uranium bearing minerals, namely uraninite, coffinite and brannerite. This was done to understand the total number of specific mineral particles on the scanned surfaces of the samples. The results are recorded in Table 3 and Appendix D.

From the Vaal Reef, sample VR9 showed the highest Au particle count with 12 particle counts, followed by sample VR1 and VR5 both with 2 particle counts.

From the three primary uranium bearing minerals, brannerite showed a very high particle count with sample VR9 having the highest count of 5100 counts, followed by sample VR5 with a 2767 particle count of brannerite. VR1 showed the lowest brannerite particle counts of 155. After brannerite followed coffinite, where sample VR9 showed the highest count of coffinite particles with 175, followed by VR5 with 122. VR1 showed the lowest coffinite particle counts of 0. Uraninite within the Vaal Reef samples showed very low particle counts, with sample VR1 having no particles noted, followed by VR5 and VR9 returned with 17 and 28 uraninite particles, respectively (Table 3).

The Crystalkop facies samples showed a higher quantity of Au particle counts, with CR1 having the highest count of 104, followed by CR7 with a 31 Au particle count. CR2 showed lower quantities 28 Au particle counts.

Table 4 shows a summary of the gold particle count within the Vaal Reef and Crystalkop Reef samples, respectively. From the results it can be seen that all three Vaal Reef samples showed a very low Au particle count, compared to the Crystalkop Reef samples, which showed a considerable amount of Au particle counts.

The particle count data confirms a higher abundance of brannerite in the samples; however, the results also indicate that uraninite remains the dominant host of uranium. This distinction is important, as while brannerite contributes to the uranium inventory, its relative abundance does not outweigh the primary role of uraninite as the principal uranium-bearing phase. Brannerite is the major U_3O_8 -bearing mineral at Kopanang Gold Mine where uranium is mined as a secondary

mineral. The Crystalkop Reef revealed very high counts of these three minerals, suggesting that the Crystalkop Reef might have higher U_3O_8 .

Generally, both reef samples with higher Au particle counts showed relatively higher particle counts of the three uranium bearing minerals analysed. Brannerite is also seen to be the major uranium hosting mineral, followed by coffinite amongst all six samples analysed (Table 3).

Table 3: Selected minerals particle count.

Mineral	Vaal Reef samples		
	VR1	VR5	VR9
Gold	2	2	12
Uraninite	0	17	28
Coffinite	31	122	175
Brannerite	155	2767	5100
	Crystalkop Reef samples		
	CR1	CR2	CR7
Gold	104	28	31
Uraninite	98	29	58
Coffinite	779	446	688
Brannerite	8416	6355	6315

4.2.2 Gold mineral association

The mineral association was determined using MLA to understand the mineral phases that are associated with gold. Due to funding constraints and the scope of the study, only Au mineral phases association was conducted and not a U_3O_8 mineral phase association. A summary of mineral maps was also used to further understand prominent representation of mineral phases that are associated with Au within the Vaal Reef and Crystalkop Reef mined at Kopanang Gold Mine (Figure 4.13, Figure 4.14, Figure 4.15 and Figure 4.16). Detailed mineral maps for both reef samples are available in Appendix G.

Within the Vaal Reef samples, it was observed that gold is mostly associated with pyrite and pyrrhotite (Figure 4.13 and Figure 4.14). Sample VR1 showed that Au is also associated with chalcopyrite whilst it was observed in sample VR9 that Au also occurs in association with quartz, chamosite, other oxides and REE rich phosphate minerals. During leaching, the Au associated with silicates, oxides, REE rich phosphate minerals and carbonates is likely to be lost if the grains are not liberated, as the process at AGA plant is designed to flush gangue to tailings.

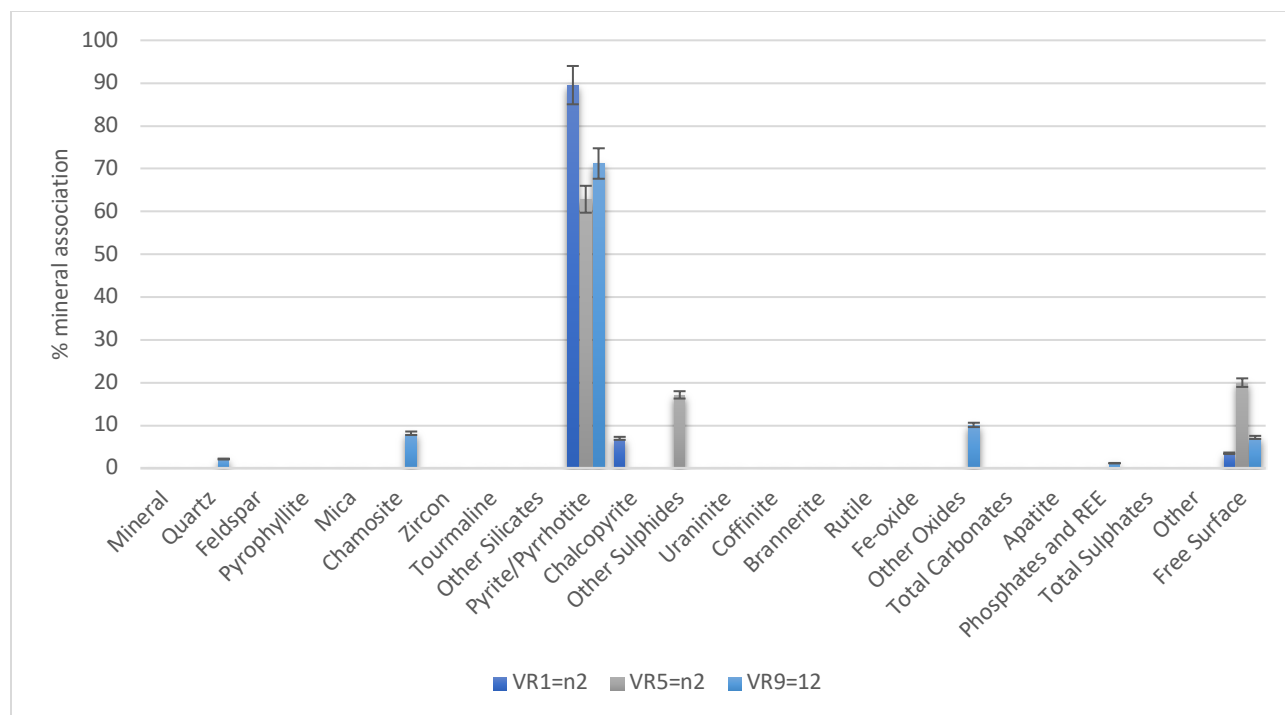


Figure 4. 13: A bar graph showing Vaal Reef Au mineral association (where n is the number of gold particles counted per sample).

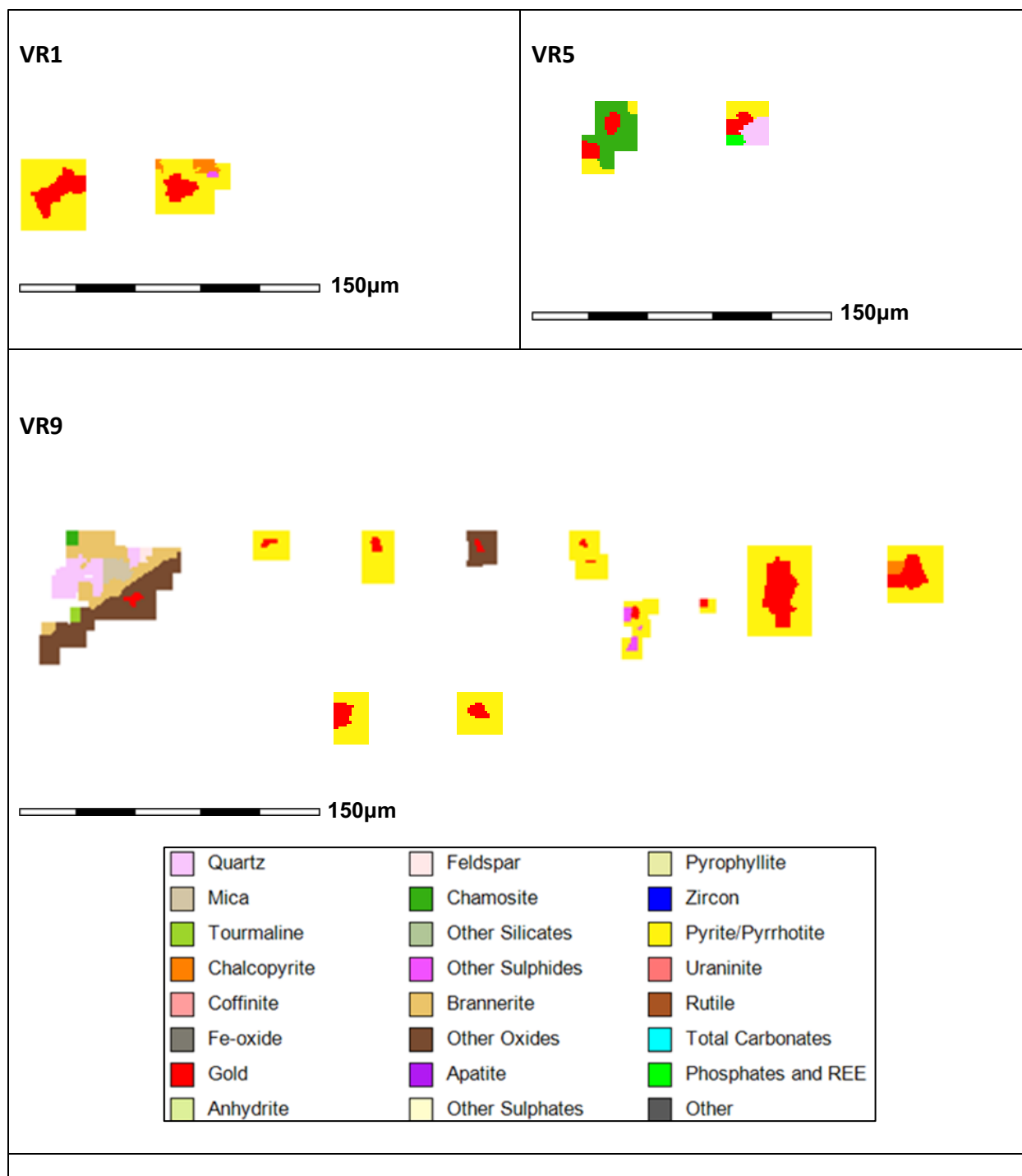


Figure 4. 14: False colour MLA images of the Vaal Reef showing Au association, grain size distribution and lower Au degree of liberation.

Within the Crystalkop Reef samples, it was observed that Au is mostly associated with pyrite and pyrrhotite (Figure 4.15 and Figure 4.16). Sample CR7 showed that Au is also associated with zircon (Figure 4.15 and Figure 4.16) and chamosite (Figure 4.15 and Figure 4.16). It was observed

in sample CR1 that there is also Au associated with silicate minerals such as quartz, feldspar, pyrophyllite and mica. However, most of the Au grains are 80 – 100% locked within pyrite.

From Figure 4.15, it is evident that most Au grains are locked within other minerals although there is some gold that occurs as free surface native gold. Figure 4.15 also shows that within the three Crystallkop Reef samples (samples CR1, CR2, and CR7) Au is associated with sulphides such as chalcopyrite, and primary uranium bearing minerals such as brannerite and coffinite.

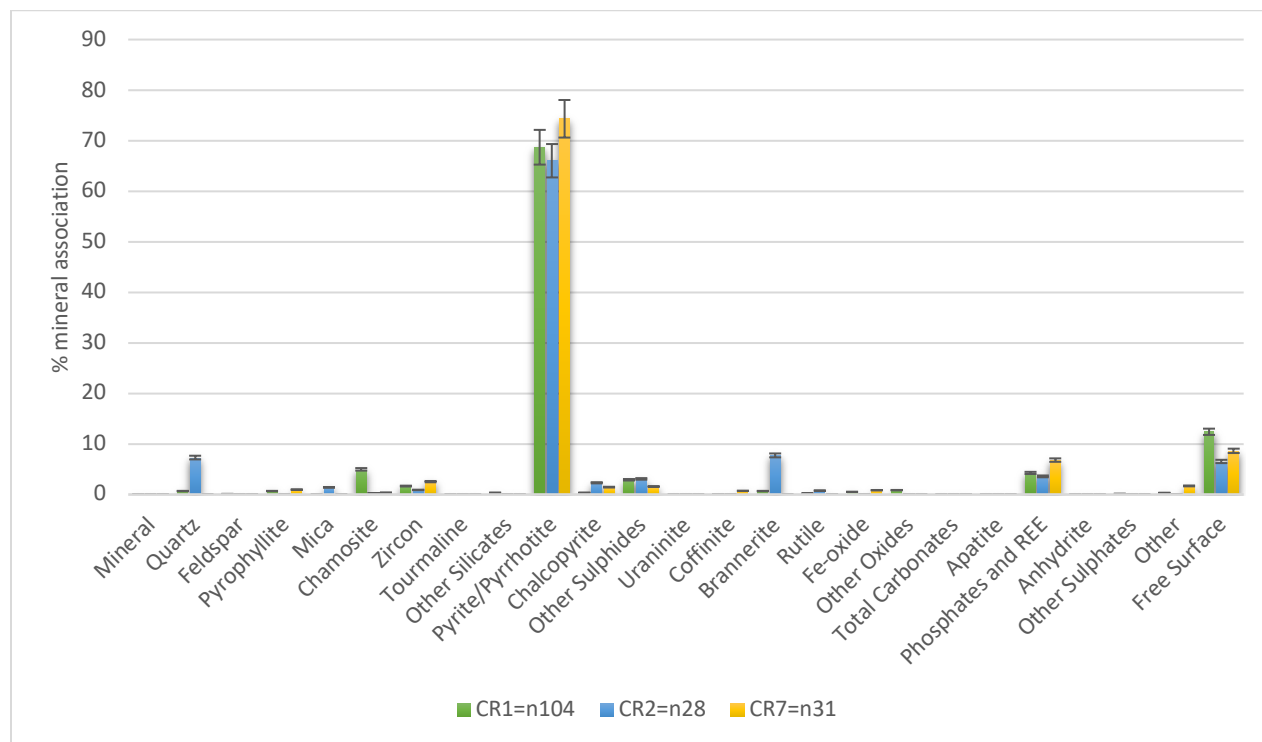


Figure 4. 15: A bar graph showing Crystallkop Reef Au mineral association (where n is the number of gold particles counted per sample).

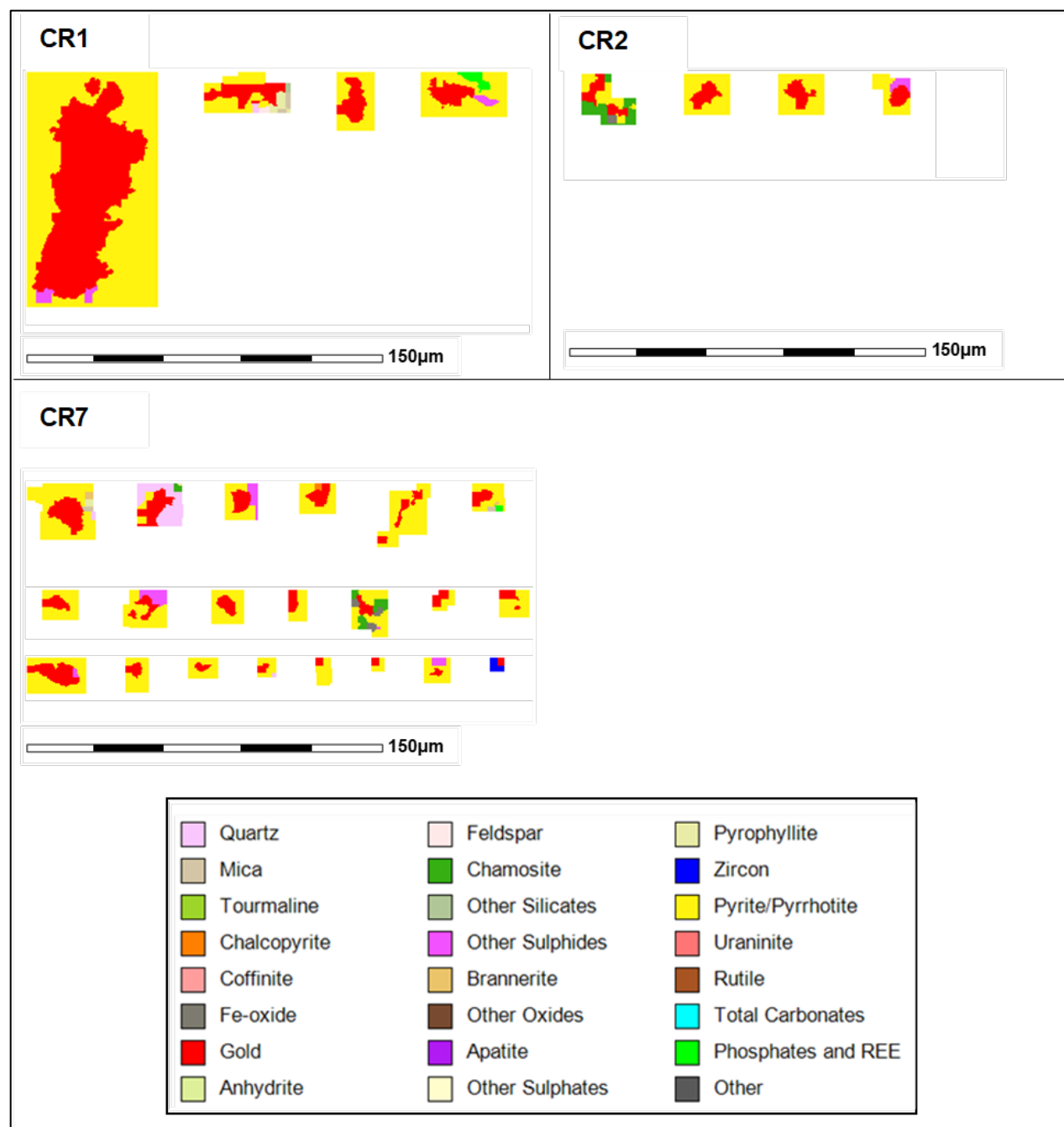


Figure 4. 16: False colour MLA images of the Crystalkop Reef showing Au association, grain size distribution and lower Au degree of liberation.

The false colour MLA images for both the Vaal Reef (Figure 4.14) and the Crystalkop Reef (Figure 4.16) reveal a generally low degree of gold liberation. In both cases, a significant proportion of gold grains are either fully locked within gangue phases or partially exposed, indicating that the gold is not readily accessible for leaching.

4.3 Au Grain size distribution

The distribution of Au grain sizes was also conducted using MLA. This was conducted to understand Au particle sizes and gain insights on how they are distributed within both the Vaal Reef and the Crystalkop Reef. Cumulative, retained and percentage passing were calculated and measured in wt.% for specific Au particles size fractions. Size fractions and cumulative retained (wt.%) were used to draw grain size distribution graphs. Detailed GSD results were recorded in Appendix H.

Grains were categorised into different size fractions in order to understand the range where gold is mostly distributed. The size fractions intervals used are recorded with the summary of grain size distribution results for the Vaal Reef (Table 4 and Figure 4.17) and Crystalkop Reef (Table 4 and Figure 4.18).

Table 4: Summary of Vaal Reef Au grain size distribution.

Size fractions (μm)	Cumulative (wt.%) Au grains		
	VR1	VR5	VR9
25 to 30	100	100	100
20 to 25	100	100	100
15 to 20	100	100	100
10 to 15	65.7	100	34.3
5 to 10	32.1	26.2	0
1 to 5	2.7	0	0
1<	0	0	0

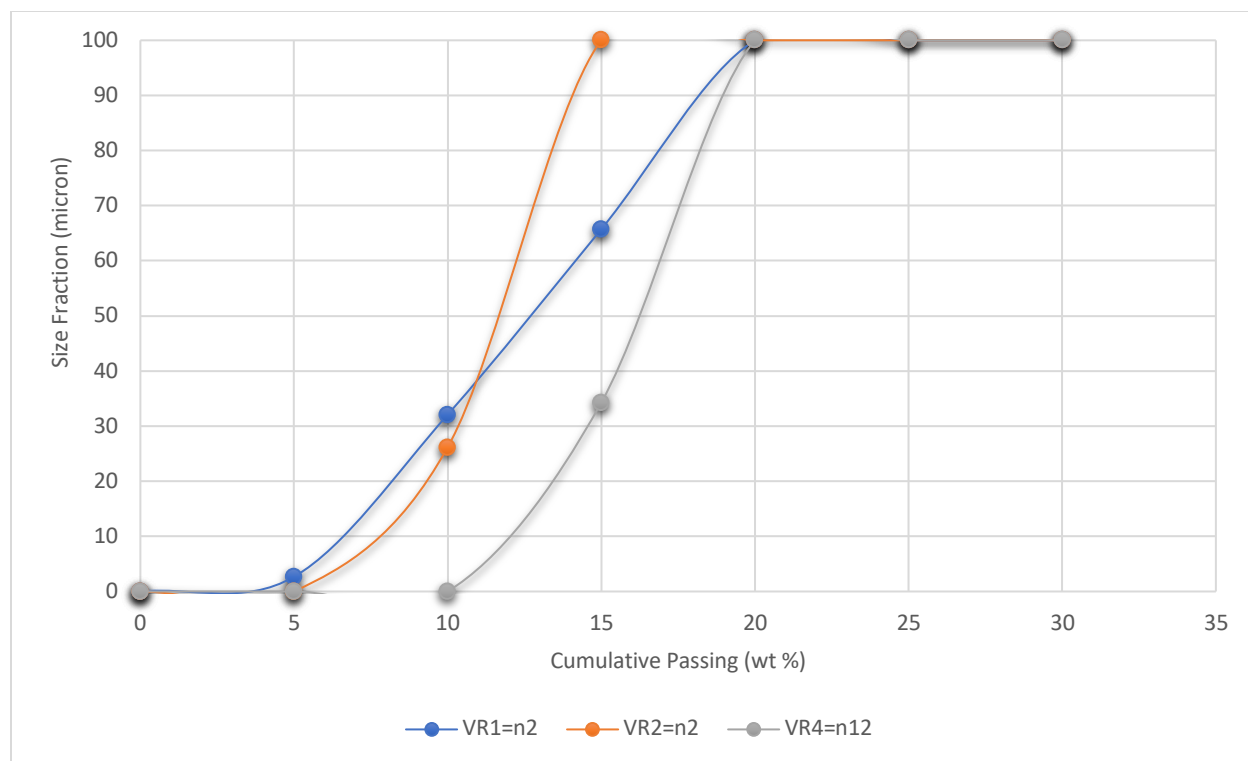


Figure 4. 17: Graph representing the gold grain size distribution within Vaal Reef samples (VR1=n2, VR5=n2 and VR9=12, where n is the number of gold particles analysed per sample).

Within the Vaal Reef samples (VR1, VR5, and VR9) it was observed that Au grain sizes were distributed between 1,75 and 16 microns (Figure 4.17). Generally, the results showed that gold grain sizes were mostly distributed from 1 to 5 and 15 to 20 μm . Sample VR1 showed that about 60% of the Au was retained in the 15 to 20 μm size fraction and about 70% Au grains were distributed between the 5 to 10 μm sieve sizes in sample VR5. Sample VR9 revealed that Au grains were distributed between 1 to 5 and 10 to 15 μm . All three samples did not show any Au grains that are less than 1 μm . Locked gold grains finer than the target grind size are unlikely to be liberated during milling and therefore are more likely to be unrecovered during cyanidation.

Within the Crystalkop Reef samples (CR1, CR2, and CR7) it was observed that the Au grain sizes were well distributed between 1 and 50 μm (Table 5 and Figure 4.18). Generally, the results showed that gold grain sizes were mostly distributed between 1 to 5 and 20 to 25 μm . Sample CR1 showed that about 50% of Au was retained in the 30 to 75 μm size fraction, with about 22% Au grains distributed between the 20 to 25 μm size fraction in sample CR2. Samples CR7 revealed that 45% Au grains were distributed between 15 and 20 μm . All three samples did not show any Au grains of less than 1 μm .

Table 5: Summary of Crystalkop Reef Au grain size distribution.

Size fractions (μm)	Cumulative passing (wt. %) Au grains		
	CR1	CR2	CR7
75 to 100	100	100	100
30 to 75	48,4	100	100
25 to 30	48,4	100	100
20 to 25	43,9	77,5	100
15 to 20	36,6	60,9	100
10 to 15	30,6	40,7	53,9
5 to 10	14,1	23,9	44,1
1 to 5	0,9	1,7	6,2
1<	0	0	0

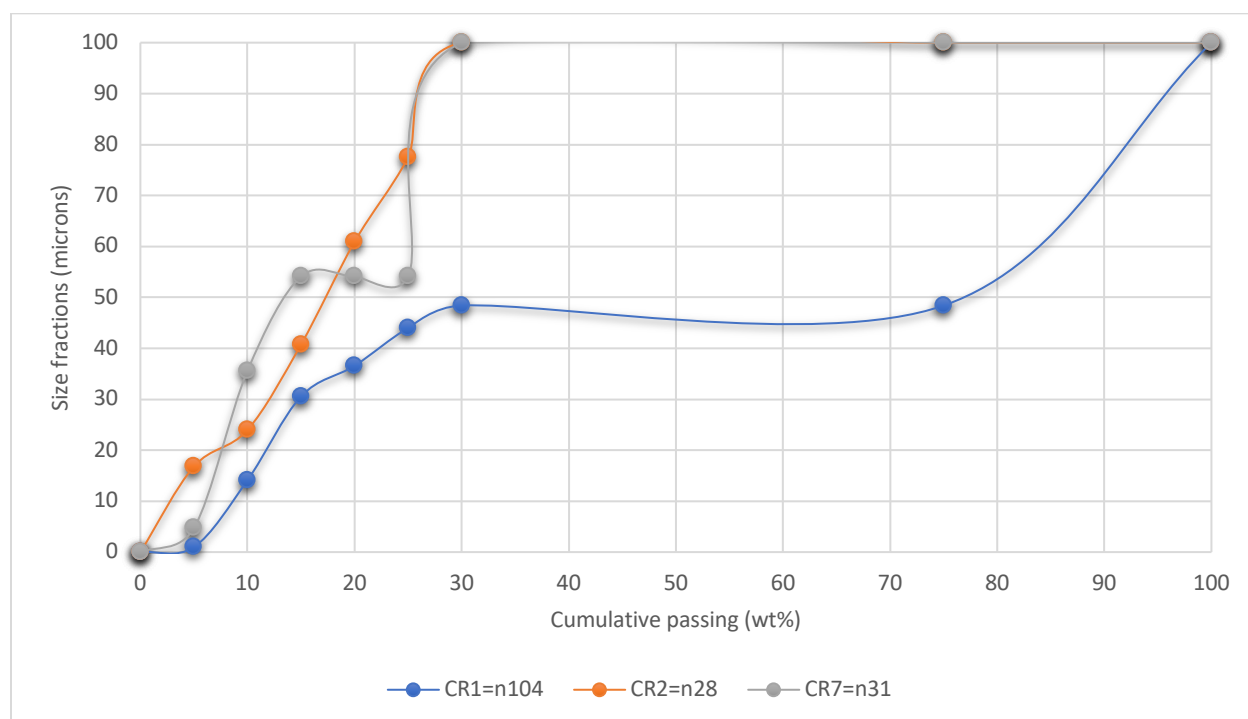


Figure 4. 18: Graph representing the gold grain size distribution within Vaal Reef samples (CR1=n104, CR2=28 and CR7=n31, where n is the number of gold particles analysed per sample).

Median grain size (d50) variability

The grain size distribution (GSD) analysis revealed significant insights into the differences between the Vaal Reef and the Crystalkop Reef samples, with implications for understanding the

nugget effect and its influence on gold grades in these two reefs. The median grain size calculated from Figures 4.17 and 4.18 are recorded in Table 6.

Table 6: Median grain size (d50) variability of the Vaal Reef and Crystalkop Reef.

	(d50) in microns		
Vaal Reef	VR1	VR5	VR9
	12.7	11.6	16.2
Crystalkop Reef	CR1	CR2	CR7
	32.7	17.3	13.9

The d50 values for Crystalkop Reef samples range from 13.0 to 32.7 μm with CR1 showing a notably larger d50 of 32.7 μm (Table 6). This higher d50 in CR1, along with moderate d50 values in the other Crystalkop Reef samples, points to an overall coarser grain size distribution in this reef. In contrast, the Vaal Reef samples have a more consistent range of d50 values, from 11.6 to 16.2 μm (Figure 4.17). The relatively smaller and narrower range suggests a more uniform gold particle size distribution, which might result in more consistent grades and a reduced nugget effect.

4.4 Bulk modal mineralogy

The bulk modal mineralogy reveals the difference in the mineralogy of the Vaal Reef and Crystalkop Reef. Mineral components of the former reef include quartz, mica, pyrite/pyrrhotite, feldspar, pyrophyllite, chamosite, rutile, zircon, tourmaline, and other minerals (less than 0.3 %) (in descending order) (Table 7). The mineral components of the latter include quartz, pyrite/pyrrhotite, chamosite, feldspar, mica, rutile, zircon, pyrophyllite, brannerite, and other minerals (less than 0.3%) (in descending order) (Table 7).

Table 7: Bulk modal mineral composition of the Vaal Reef (VR1; VR5 and VR9) and Crystalkop Reef (CR1; CR2; and CR7) samples conducted using MLA.

Mineral	Vaal Reef			Crystalkop Reef		
	VR1	VR5	VR9	CR1	CR2	CR7
Silicates						
Quartz	66.7	71.4	71.4	46.1	56	41.8
Feldspar	3.3	4.0	4.2	3.9	4.4	4.9
Pyrophyllite	1.2	0.3	1.8	1.1	2	1.5
Mica	12.7	6.9	15.5	3.6	7.4	4.4
Chamosite	2.1	1.4	2	6.5	3.3	6.8
Zircon	0.4	0.6	0.3	3.3	3.5	4.3
Tourmaline	0.3	0.3	0.3	<0.1	<0.1	<0.1
Other Silicates	<0.1	<0.1	<0.1	0.6	0.8	0.6
Sulphides						
Pyrite/Pyrrhotite	11.8	13.6	3.6	26.4	15.3	27.2
Chalcopyrite	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Other Sulphides	<0.1	<0.1	<0.1	0.3	0.5	0.3
Uranium bearing phases						
Uraninite	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Coffinite	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Brannerite	<0.1	0.3	0.1	1.6	1.3	0.9
Oxides						
Rutile	0.9	0.4	0.5	4.4	3.8	4.8
Fe-oxide	0.1	0.1	0.1	0.3	0.2	0.3
Other Oxides	0.1	0.3	0.01	0.5	0.4	0.8
Carbonates	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Gold	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Phosphates	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Apatite	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
REE rich phosphate minerals	0.1	0.3	0.1	1.1	1.2	1.4
Sulphates	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Other	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total	100.00	100.00	100.00	100.00	100.00	100.00

4.5 Chemical assay results

Grade analysis revealed that the Crystalkop Reef has higher grades of gold and uranium with 156.4 g/t and 8.0 g/t, respectively as compared with the Vaal Reef with 77.0 g/t and 4.6 g/t gold and uranium, respectively (Table 8). The gold grade of the Composite Reef will be reported in chapter 5 together with the leaching results.

Table 8: Head grade of the Vaal Reef and the Crystalkop Reef.

Vaal Reef and Crystalkop Reef grade results			
	Vaal Reef	Crystalkop Reef	STD.DEV
Au (g/t)	77.0	156.4	56.14
U₃O₈ (g/t)	4.6	8.0	2.40

The base metal analysis of the Vaal Reef and Crystalkop Reef samples was comparable to the base metal analysis of the Composite Reef (Figure 4.19).

The analysis revealed that both reefs have the highest contents of Pb compared to other base metals, with the Vaal Reef, Crystalkop Reef and Composite Reef having 2815 ppm, 3739 ppm and 3638 ppm, respectively. This was followed by Ni, with 896 ppm and 1108 ppm in the Vaal Reef and Crystalkop Reef samples, respectively. All three reef samples showed that Cu was below the limit of detection (LLD) from the XRF analyses (Figure 4.19).

All three feed samples are characterised by a similar Co content, with the Vaal Reef having 363 ppm and Crystalkop Reef with 302.6 ppm. Higher contents of Zn were noted in the Crystalkop Reef (493 ppm) compared to the Vaal Reef (171 ppm).

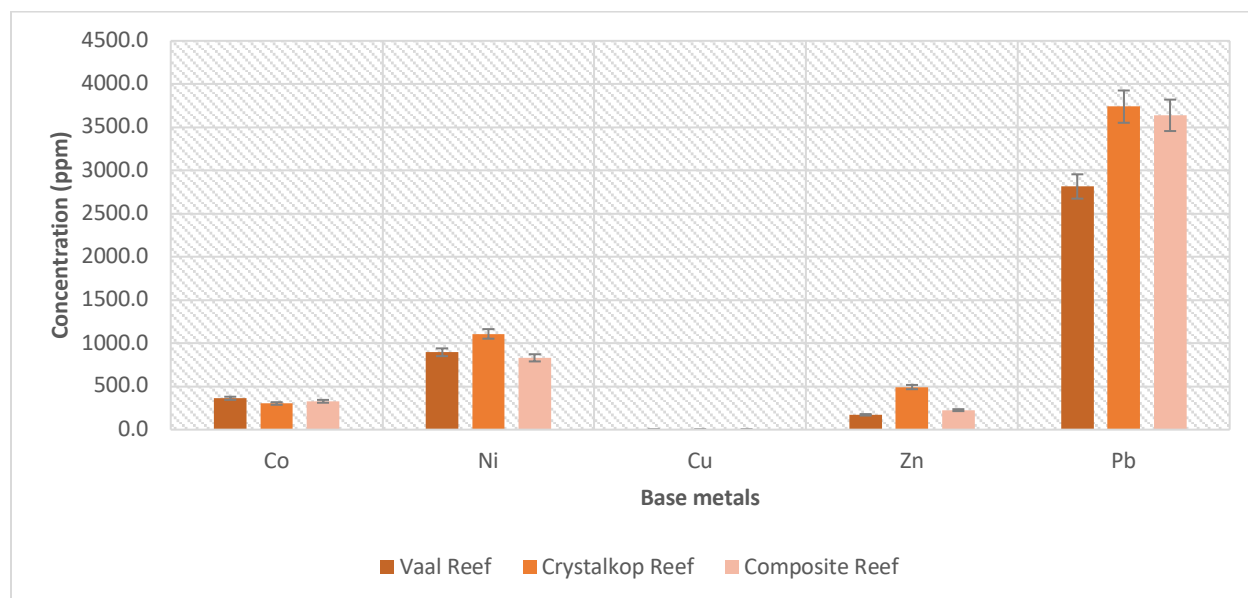


Figure 4. 19: Base metals concentration for the Vaal Reef, Crystalkop Reef and Composite Reef (ppm).

The minor elements results (Figure 4.20) showed that Bi had the highest contents in both the Vaal Reef and Crystalkop Reef with 1080 ppm and 1268 ppm, respectively, followed by As and Ce, with concentrations ranging from 406.6 ppm to 471 ppm. The Crystalkop Reef has the highest contents of Ba (1369 ppm) compared to the Vaal Reef with 398 ppm. There was no Rb detected in samples from both reefs. Tin (Sn) was not detected in the Vaal Reef but is present in the Crystalkop Reef with 65 ppm. W has the lowest concentrations of <4 ppm in both reefs. The Crystalkop Reef has a higher content of Th (696 ppm) compared to the Vaal Reef with 334 ppm.

The Composite Reef showed comparable trace element results with the Vaal and Crystalkop Reef. The contents of the Composite Reef were ranging between the contents of the other two reef samples, which was to be expected.

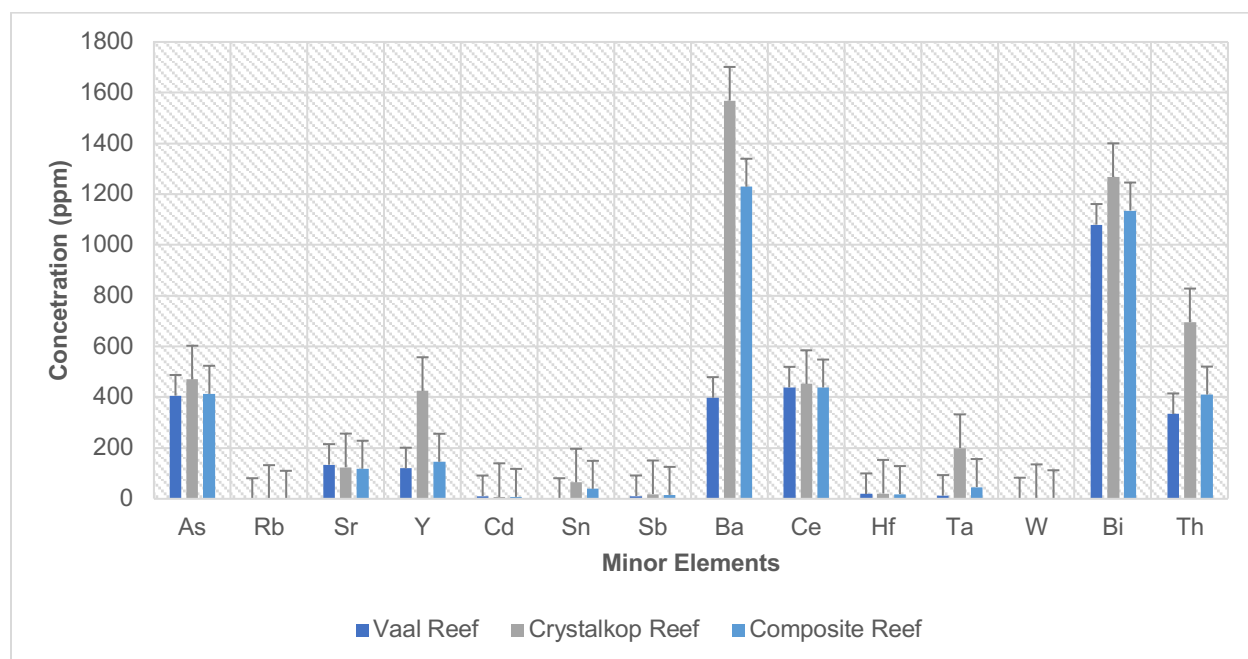


Figure 4. 20: A bar graph showing minor elements concentration for the Vaal Reef, Crystalkop Reef and Composite Reef (ppm).

4.6 Chapter Summary

Mineralogical, petrographic, and chemical assay analyses of the Vaal Reef and Crystalkop Reef from Kopanang Gold Mine revealed significant contrasts in their grade, and gold mineral distributions. Both reefs are dominated by silicate minerals, with quartz and muscovite as primary gangue phases. Texturally, the Crystalkop Reef is clast-supported, whereas the Vaal Reef is predominantly matrix-supported which influences mineral liberation during processing (Reid *et al.*, 1986; Philips and Law 2000).

Optical microscopy and bulk modal mineralogy confirmed that gold is predominantly associated with sulphide minerals, particularly pyrite and pyrrhotite, with limited occurrences as free gold. MLA further revealed that approximately 80% of the gold in both reefs is associated with pyrite. Sebola (2015) stated that attrition during sediment movement gave rise to the compact pyrite found in the Witwatersrand Basin and its sub-rounded shape. Due to reworking that took place because of remobilisation (Genis, 1990, Large *et al.*, 2013), allogenic pyrite frequently appears rounded (Sebola, 2015) and these observations are similar to those of this study. The occurrence of these opaque minerals adds to what makes the Vaal Reef and Crystalkop Reef slightly distinguishable.

The strong gold-pyrite association restricts the usefulness of direct cyanidation, as the gold might remain inaccessible without pre-treatment (Nwagboso, 2022; Surimbayev *et al.*, 2024). However, the presence of free gold suggests that a portion of the gold may be amenable to gravity recovery (Clarke, 2005; Dominy *et al.*, 2011), providing opportunities to maximise the initial stages of processing.

Previous studies by Pienaar *et al.*, 2015 and Pienaar *et al.*, 2017 at Great Noligwa and Moab Khotsonq mines noted that, petrologically, the Vaal Reef is characterised mostly by moderately packed, sub-rounded quartz and pyrite, and the gold grains are not visible. The term moderately packed describes the arrangement of clasts within the matrix, where larger grains are interspersed with finer material (Kleynhans, 1970; Ulmer-Scholle *et al.*, 2014) such as quartz sand, muscovite, pyrophyllite, and chlorite. Gold found in the samples was entirely native, which was mostly associated with and locked in sulphides (pyrite and pyrrhotite), silicates (quartz, chamosite), uranium-bearing minerals (brannerite) and REE rich phosphate minerals in descending order of abundance. This is similar to the observation of this study.

Best (2013) described trace elements as those elements that are available in rocks in very low concentrations. Trace elements are categorised into compatible and incompatible elements (Arth, 1976). For the longest of time, trace and minor elements have been studied and considered mineral impurities during ore processing (Kyle *et al.*, 2011). Generally, trace elements are divided into large ion lithophiles (LILE), high field strength elements (HFSE), rare earth elements (REE) and transition elements (Mason, 1952). This part of the study only focuses on the significance of selected trace elements and their implication during gold and uranium processing.

Although trace elements in gold and uranium ores are perceived as impurities (Kyle *et al.*, 2011), they sometimes assist by increasing the rate of leaching during gold cyanidation (Kyle *et al.*,

2011). The Vaal Reef and Crystalkop Reef at Kopanang Gold Mine are characterised by trace quantities of elements such as Rb, Th, Cd, and Bi. Marsden and House (2006) noted that, although these elements are present, their concentrations are generally too low to significantly diminish or enhance the passivation effect on gold during cyanidation. However, although the Crystalkop Reef is known to have a higher grade than the Vaal Reef, the presence of considerable quantities of Rb, Th, Cd, and Bi (ranging from 471 ppm to 1267 ppm) may still pose metallurgical challenges, with potential implications for gold recovery efficiency and downstream processing performance.

Reference

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Chapter 5: Metallurgical characterisation

This chapter focuses on the metallurgical test work, characterisation, and interpretation of results obtained from gold cyanide leaching of the Composite Reef sample. The test work was conducted in two parts: bottle roll tests were performed in duplicates, while diagnostic leach tests were conducted in triplicate. The metallurgical analysis evaluated how the feed sample responded to the leach tests, using AGA plant standards as a benchmark at North-West University's School of Chemical and Minerals Engineering. Filtrate samples and solid residues were analysed for gold, providing insight into gold recovery efficiency. Additionally, cyanide consumption was assessed to determine the amount of sodium cyanide used for each leach test.

Sacrificial tests were conducted for all duplicate samples which were leached and rolled for 4, 8, 16 and 24 hours using the bottle roll method (Annexure A). After leaching, the filtrate and residue samples were obtained and analysed for their respective Au content. For quality control and quality assurance of the leach test results, self-prepared standards using silica and AMIS gold standards were used. Detailed leaching laboratory measurements and conversions are recorded in Appendix E1, E2, and E4.

Prior to conducting metallurgical tests, the Composite Reef sample was divided into three equal portions. Each portion was assayed for gold using fire assay and subsequently analysed by AAS, while for ICP-OES analysis the samples were first subjected to microwave digestion. Results indicate that the average gold grade, using fire assay, is 85.81 g/t and 85.54 g/t using ICP-OES (Table 9). The two methods yielded very similar gold grades, indicating consistency and reliability in the assay results.

All error values in this chapter were calculated using a standard deviation.

Table 9: Average head grade of the Composite Reef sample determined in triplicates by AAS and ICP-OES.

Average head grade of the Composite Reef			
	AAS (g/t)	ICP-OES (g/t)	STDEV.S
Run 1	86.01	87.90	1.34
Run 2	84.04	85.15	0.84
Run 3	87.60	83.56	2.82
Average Au grade	85.81	85.54	0.21

5.1 Forward leaching results

The forward leaching tests were conducted on the Composite Reef sample to determine the amount of gold recoverable by direct cyanidation. The experiments were conducted over varying leaching times of 4, 8, 16 and 24 hours and were ran in duplicate. The average results are presented in Table 10 and Figure 5.1.

Table 10: Forward leaching average gold percentage recovery results of the Composite Reef sample determined in duplicates.

Average Au percentage recovery of the Composite Reef					
	4 hrs	8 hrs	16 hrs	24 hrs	STDEV.S
Au % recovery	28.3	31.2	46.4	55.7	12.9

The gold recovery results obtained from the leaching experiments indicate a clear relationship between leaching time and gold extraction efficiency. Gold recovery was observed to increase progressively with longer leaching durations, ranging from 28.3% at 4 hours to 55.7% at 24 hours (Table 10 and Figure 5.1). The lowest recovery recorded at 4 hours, suggests that the contact time between the leaching solution and the gold-bearing material was insufficient for significant gold dissolution to occur. As the leaching time increased, there was a corresponding improvement in gold recovery, with the highest percentage achieved at 24 hours.

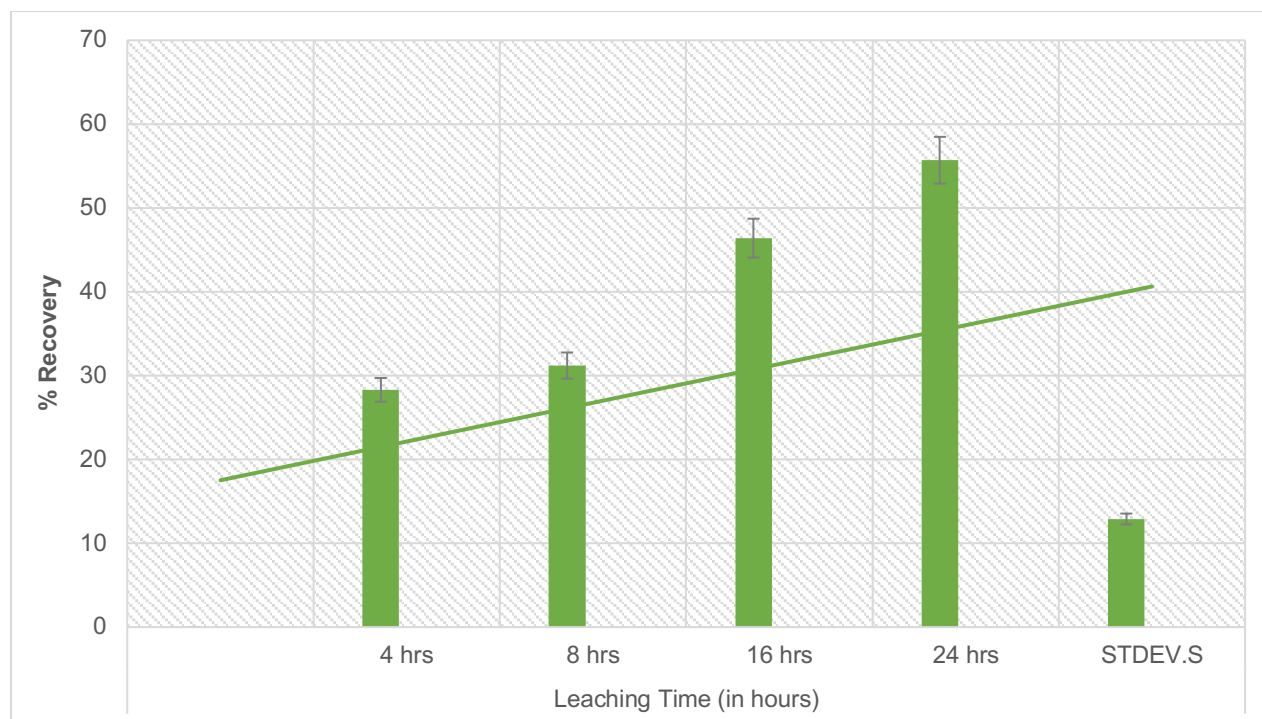


Figure 5. 1: Percentage recovery of gold at different leaching times.

5.2 Reverse leaching results

The reverse leaching experiments were conducted on a Composite Reef sample to evaluate gold and uranium recovery over varying leaching times of 4, 8, 16, and 24 hours. The results are presented in Table 11 and Figure 5.2 and show notable trends in the recovery efficiency of both gold and uranium.

Table 11: Reverse leaching average gold and uranium percentage recovery results of the Composite Reef sample determined in duplicates.

Average Au and U percentage recovery of the Composite Reef					
	4 hrs	8 hrs	16 hrs	24 hrs	STDEV.S
Au % recovery	76.0	78.3	83.7	84.3	4.07
U % recovery	48.9	51.5	51.2	51.6	1.27

Gold recovery ranged between 76.0% and 84.3% across the four leaching durations evaluated (Figure 5.2). The lowest recovery was recorded at 4 hours, while the highest was achieved at 24 hours. Overall, the gold extraction showed a positive correlation with increased residence time, suggesting that longer leaching periods were more favourable for gold dissolution under the

applied conditions. The values reported in Table 11 reflect a gradual improvement in recovery efficiency over time, consistent with time-dependent leaching behaviour.

Uranium recovery, on the other hand, remained relatively stable, ranging from 48.9% to 51.6% throughout the tested leaching intervals (Table 11 and Figure 5.2). The highest uranium recovery was obtained at 24 hours, while the lowest occurred at 4 hours.

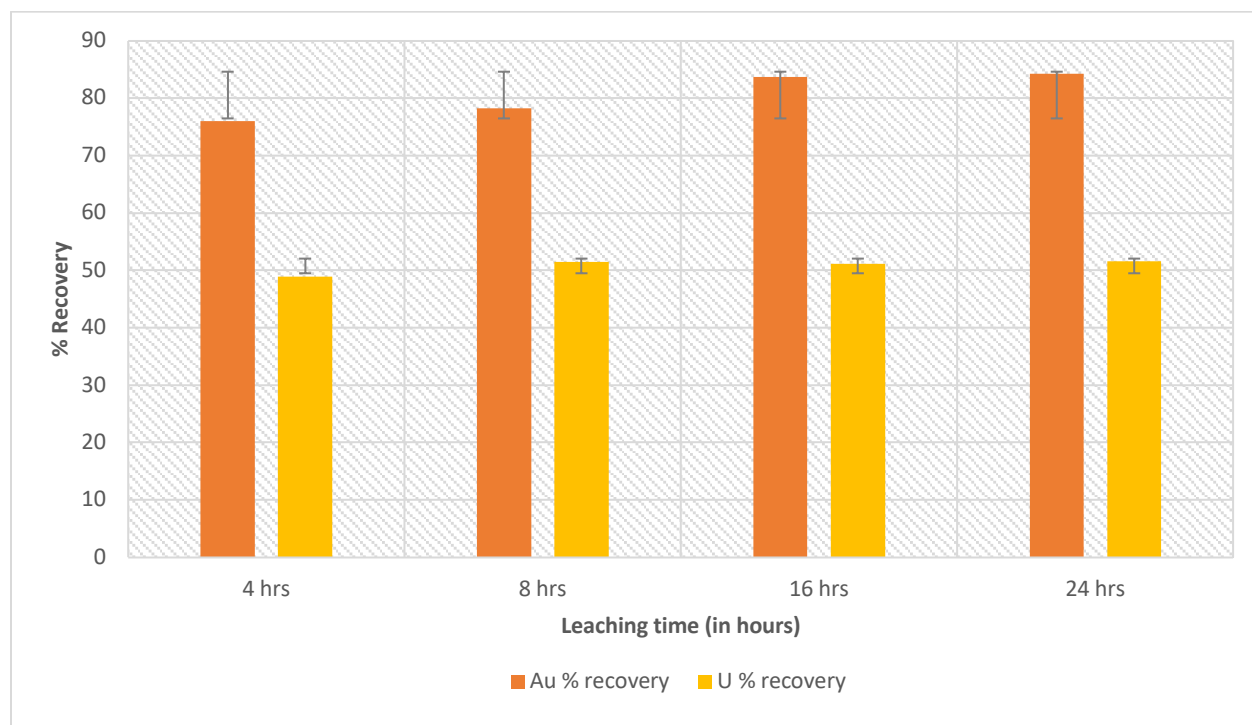


Figure 5. 2: Percentage recovery of gold and uranium at different leaching times

5.3 Cyanide consumption

The results presented in Figure 5.3 and Figure 5.4 illustrate the relationship between the amount of NaCN added and the amount consumed during the leaching process, using both forward leaching and reverse leaching methods, respectively. The error bars were calculated used standard deviations.

In the forward leaching process (Figure 5.3), it was observed that while a consistent amount of NaCN was added across the different leaching periods (4, 8, 16 and 24 hours), the actual amount consumed by the leach solution increased with longer leaching times. At shorter leaching durations (4 hours), less cyanide was consumed, likely due to the slower rate of gold dissolution.

Similarly, in the reverse leaching process (Figure 5.4), a comparable trend was noted. However, reverse leaching demonstrated slightly higher cyanide consumption over time compared to forward leaching.

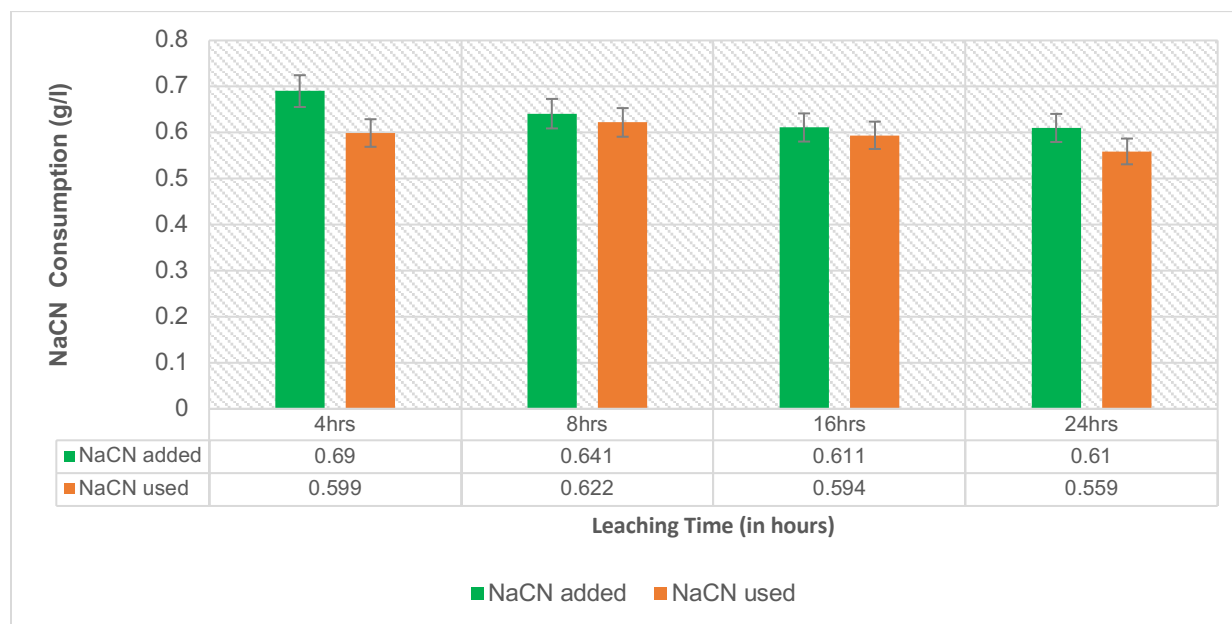


Figure 5. 3: Bar graph showing the amount of NaCN added for the leach and the amount used for the leach using forward leaching method.

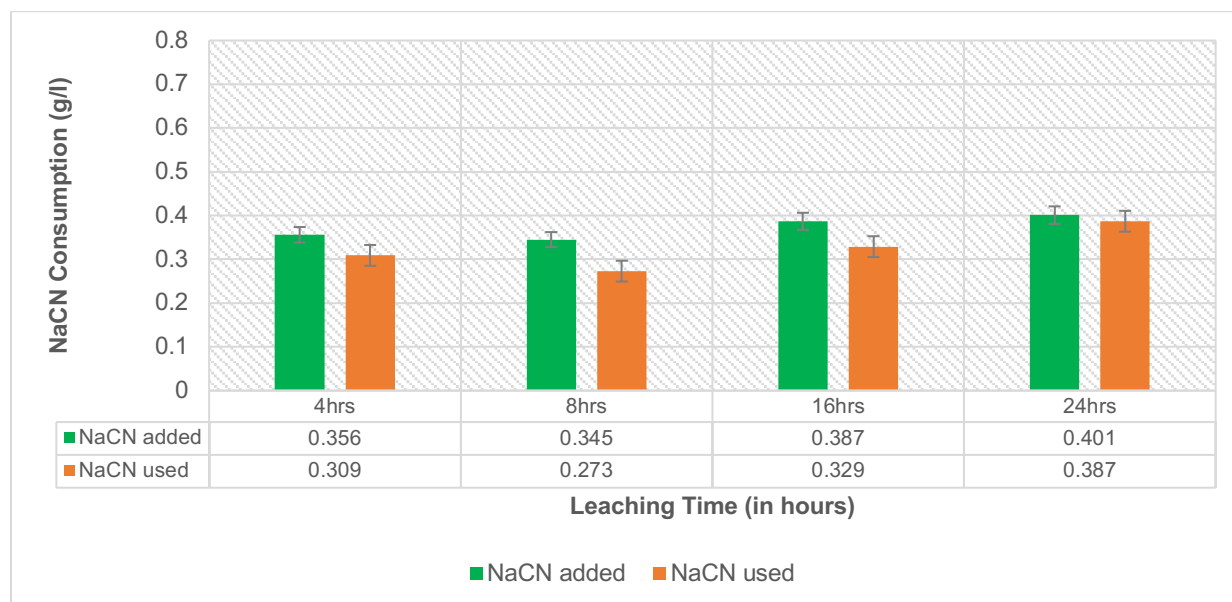


Figure 5. 4: Bar graph showing the amount of NaCN added for the leach and the amount used for the leach using for reverse leaching method.

5.4 Diagnostic leaching

The results of the diagnostic leaching tests performed on the run-of-mine (ROM) sample are summarized in Table 12, Figure 5.5 and detailed in Appendix E3. The initial stage involved direct cyanidation, which targeted the free-milling gold fraction. This stage yielded a significant gold recovery of 87.0%, equivalent to 74.6 g/t, indicating that the majority of the gold was present as free or easily accessible particles.

Subsequent leaching with hydrochloric acid aimed to dissolve acid-soluble phases such as pyrrhotite, calcite, calcium carbonates, and dolomite. This stage resulted in a gold recovery of 8.4% (7.22 g/t), suggesting a notable association of gold with these mineral groups. The third stage employed sulphuric acid to evaluate gold encapsulated in labile sulphides and uraninite, yielding a recovery of 1.4% (1.21 g/t).

Nitric acid treatment was then applied to assess gold associated with pyrite and other refractory sulphides. This stage showed a relatively low recovery of 0.6% (0.58 g/t), consistent with the known refractory nature of such associations. To evaluate the remaining unrecovered gold, the final leach residue was analysed and done in duplicate. The results indicated that approximately 2.5% (2.24 g/t) of the gold remained occluded within silicate minerals.

Overall, the diagnostic leaching sequence confirmed, that while the majority of gold in the composite sample (from the Vaal and Crystalkop Reefs at the Kopanang gold mine) is free-milling, a significant proportion remains locked within mineral phases that contribute to the ore's refractory nature. These findings are largely consistent with earlier work by Lorenzen and Tumilty (1992), who emphasized the utility of diagnostic leaching in differentiating between recoverable and refractory gold.

Table 12: Diagnostic leach test results.

<i>Diagnostic Leaching Sequence</i>						
	Head Grade	Direct Cyanidation	HCl	H₂SO₄	HNO₃	Residue
Au %		87	8.4	1.4	0.6	2.5
Au (g/t)	85.81	74.63	7.22	1.21	0.58	2.24

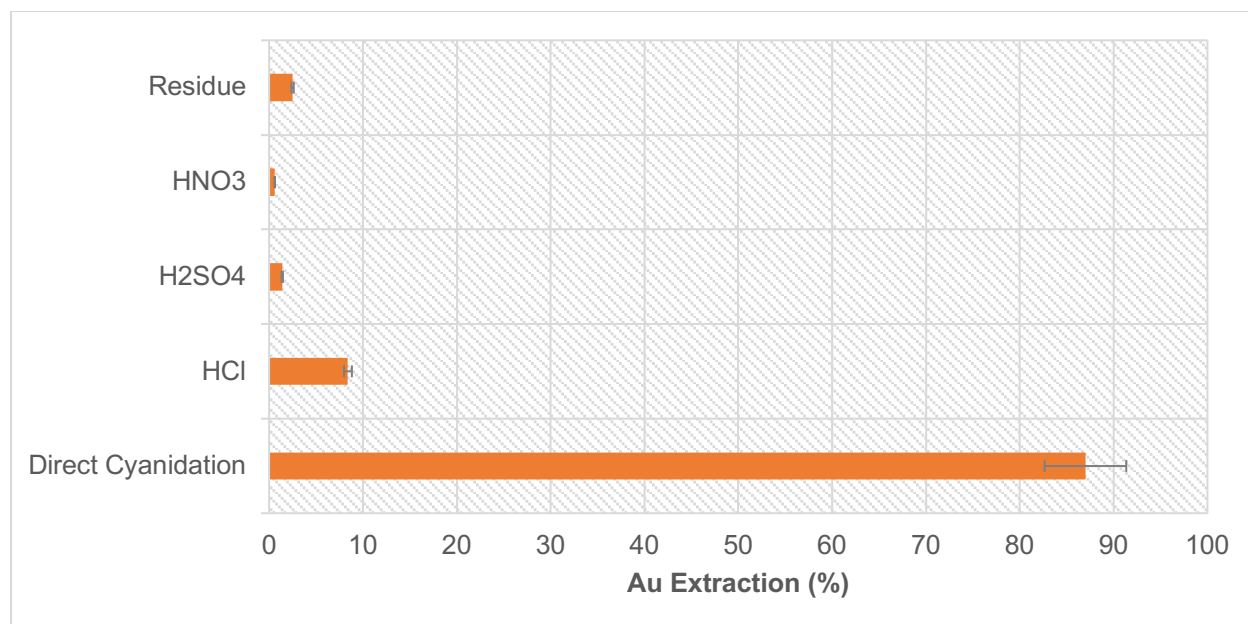


Figure 5. 5: Gold extraction from ROM sample material at different stages of the diagnostic leaching tests.

5.5 Chapter Summary

Gold assays, using fire assay and ICP-OES methods, showed a high consistency, with average head grades of 85.81 g/t and 85.54 g/t, respectively. The results of the gold cyanide leaching tests revealed that gold recoveries ranging from 28.3% to 55.71% were achieved through forward leaching, while higher recoveries of 76.0% to 84.3% were obtained through reverse leaching under plant conditions. Despite the improvement in recoveries using reverse leaching, the overall gold recoveries for both methods remain below 90%, which is the conventional threshold distinguishing free-milling ores from refractory ores. Consequently, the gold in this ore can be regarded as refractory, as less than 85% of the contained gold is recoverable through standard cyanidation techniques, even under optimised plant conditions.

The uranium acid leach tests conducted in this study indicated that uranium recoveries for the Composite Reef were significantly lower than anticipated, particularly when compared to the recoveries of up to 90% reported by Lottering *et al.*, (2008). However, the results obtained here showed closer alignment with those reported by Pienaar *et al.*, (2015) and Pienaar *et al.*, (2017), where uranium recoveries ranged between 30.2% and 64.2% at the Great Noligwa and Moab Khotsong mines. In the present study, uranium recoveries achieved under the plant conditions outlined in Annexure A ranged between 48.9% and 51.6%. These findings suggest that, while

uranium recovery from the Composite Reef is lower than the ideal target, it falls within a range that has been observed in other operations within the Klerksdorp Goldfield.

Cyanide consumption increased with leaching time in both forward and reverse leaching processes, reflecting a direct correlation between contact duration and reagent usage. Forward leaching showed lower cyanide use at shorter intervals, with gradual increases up to 24 hours. Reverse leaching followed the same pattern but exhibited slightly higher overall consumption, likely due to more intensive chemical interaction and improved gold recovery.

The diagnostic leaching results indicate that approximately 13% of the gold within the composite ROM sample from the Vaal and Crystalkop Reefs is potentially unrecoverable if only conventional milling and direct cyanidation are applied. This fraction of gold remains largely inaccessible due to its strong association or entrapment within specific mineral phases, despite extensive milling to 80% passing 75 µm. Of the refractory component, 7.22% of the gold is associated with minerals such as calcite, dolomite, pyrrhotite, and calcium carbonates, while 1.4% is hosted by labile sulphides (e.g., uraninite), and 0.6% is linked to pyrite and arsenopyrite. An additional 2.24% of the gold remains locked within silicate minerals, mainly quartz, and was not recovered despite hydrochloric, sulfuric, and nitric acid leaching suggesting robust encapsulation.

The gold recovery trends observed in the diagnostic leaching process are largely consistent with the findings of the mineralogical analysis, particularly in terms of mineral association. However, a notable deviation exists in the case of gold associated with sulphide minerals such as pyrrhotite and pyrite. While mineralogical data (e.g., MLA results) indicated a strong association between gold and these sulphides, the diagnostic leaching results reveal that most of this gold is, in fact, recoverable through direct cyanidation. This demonstrates that mineral association alone does not necessarily equate to gold being locked within the mineral matrix. This distinction between mineral association and mineral locking has been highlighted by (La Brooy *et al.*, 1994; Adams, 2005), who caution that mineralogical association alone does not predict leach behaviour or recovery efficiency. In this case, although gold may be spatially associated with sulphides, it is not encapsulated or bound in a way that inhibits cyanide access and thus behaves as free-milling gold.

In contrast, the silicate-bound gold represents a truly refractory fraction. The 2.24% of gold not recovered through acid leaching or cyanidation remains occluded within silicate phases such as quartz, as confirmed by both the final residue analysis and MLA data. This indicates that further liberation would require more aggressive or destructive techniques, such as hydrofluoric acid (HF)

treatment, to dissolve the silicate matrix and expose the gold (Corrans *et al.*, 1982; Lorenzen and Tumilty, 2000; Chingwaru *et al.*, 2023; Chingwaru *et al.*, 2025).

Overall, the combined use of diagnostic leaching and mineralogical analysis has provided a comprehensive understanding of gold deportment within the ore. While the majority of the gold is free-milling and amenable to cyanide leaching, the presence of refractory gold, particularly within silicates, underscores the need for more advanced processing strategies should higher recoveries be pursued.

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Chapter 6: Discussion, conclusion and recommendations

This chapter serves to give a summary of all previous chapters. It is also in this chapter that all the data acquired during the course of this study is summarised and conclusions of the implications of mineralogy, chemistry and metallurgical properties of the Vaal Reef A facies, Crystalkop and Composite Reefs are given. Moreover, recommendations are given based on the findings and shortcomings of this study to the mine and for future research purposes.

6.1 Discussion

The study presented an integrated analysis of the mineralogical, chemical, and metallurgical characteristics of the Vaal Reef, Crystalkop Reef and the Composite Reef at Kopanang Gold Mine, aiming to enhance the understanding of processing challenges and to propose strategies for improving the recovery of gold. Each aspect of the research was seen to contribute uniquely to a comprehensive understanding of how these reef facies behaved during the leaching tests.

Preg-robbing is a well-recognized issue in gold leaching processes, occurring when gold complexes are removed from solution by certain ore constituents. This phenomenon affects nearly all types of gold lixiviants (Ahtiainen *et al.*, 2018). Within the two studied reefs, sulphide minerals such as pyrite and pyrrhotite are present. While pyrite is a refractory mineral that can host gold, preg-robbing is typically associated with other phases such as carbonaceous matter (Alex, 2023). To counteract this, technologies like nitric acid pre-treatment have been developed to break down gold-hosting gangue minerals and enhance gold accessibility (Zhou *et al.*, 2004; Alex, 2023).

In addition to sulphides, non-sulphide minerals also contribute to preg-robbing. Quartz (Mohammadnejad *et al.*, 2011) and clay minerals such as kaolinite and muscovite (Goodall *et al.*, 2005a; Goodall *et al.*, 2005b) are known to trap gold during processing. Fine grinding and crushing of quartz can activate its surface, creating sites that facilitate gold precipitation from solution (Mohammadnejad *et al.*, 2013).

Mineralogical analysis revealed both the Vaal and Crystalkop Reefs as predominantly silicate based, containing substantial quantities of quartz and muscovite. The analysis also showed that the Vaal Reef as being matrix-supported, in contrast to the clast-supported Crystalkop Reef, which influenced the distribution and liberation of gold during milling. Gold grains across both reefs were noted to be frequently associated with pyrite, silicates such as quartz and chamosite, and uranium-bearing minerals like brannerite, indicating a requirement for intensive milling to achieve liberation. The study highlighted that this mineralogical distribution affected both the visibility of

gold particles and the efficiency of subsequent leaching, as gold locked within sulphides and silicates hindered complete liberation.

Trace elements such as lead (Pb), thorium (Th), cadmium (Cd), and bismuth (Bi) were identified in varying quantities across the reefs. The study explained that these trace elements, often regarded as impurities, could affect cyanidation as indicated by Kyle *et al.* (2012). It was also noted that high concentrations of arsenic-bearing minerals might facilitate gold recovery through cyanidation by altering the mineral matrix to increase gold accessibility (Nwaila and Frimmel, 2019). However, the concentration of trace elements at the Kopanang Gold Mine did not reach a level that significantly influenced this effect, although the presence of trace elements might have added complexity to the leaching process.

Analyses of the grain size distribution revealed that both reefs were characterized by very fine gold particles, with about 80% of the gold measuring less than 50 μm . Previous studies (Pienaar *et al.*, 2015; Pienaar *et al.*, 2017) had suggested that such smaller gold grains might present challenges in liberation, often requiring more intense grinding to separate them from the mineral matrix. The study observed that this fine distribution of gold grains, particularly within silicate minerals, made recovery more complicated, as standard grinding might not sufficiently expose these particles. Consequently, the study concluded that significant portions of gold were likely to remain locked with pyrite (as shown by mineral maps), potentially leading to losses during processing and disposal in tailings (Mkhatshwa *et al.*, 2020).

Reagent consumers are particularly problematic in gold leaching processes because they compete for critical reagents, most notably dissolved oxygen, which is essential for the effective dissolution of gold in cyanide solutions (Mohammadnejad *et al.*, 2013; Alex, 2023). In the cyanidation process, oxygen acts as an oxidant that facilitates the formation of the gold-cyanide complex (Ahtiainen *et al.*, 2018). However, the presence of certain minerals such as reactive pyrite and pyrrhotite can consume significant amounts of oxygen, thereby reducing the availability of this crucial reagent for gold dissolution (Becker *et al.*, 2010). This becomes especially detrimental in refractory ores where gold is already encapsulated within sulphide and silicate matrices.

The study reported that the presence of cyanide consuming minerals, such as muscovite and chlorite added an additional challenge to gold recovery at the Kopanang gold mine. These minerals are known for their reaction with cyanide during leaching, reducing the amount available to dissolve gold effectively. The study also noted that the locked nature of gold within silicate

minerals, particularly in the Vaal Reef, compounded the effect of cyanide consumption, as gold particles within silicates were less accessible for leaching. These findings aligned with Wang (2019), who had previously noted that cyanide consumption by silicate-hosted minerals could severely impact gold recovery.

A central finding of this study is the significant difference in gold recovery between the forward and reverse leaching methods. Forward leaching, involving direct cyanidation of raw ore, achieved relatively modest gold recoveries ranging between 28.3% and 55.7%. In contrast, reverse leaching in which gold was leached from the residue of prior uranium leaching using sulphuric acid yielded notably higher recoveries of 76.0% to 84.3% after 24 hours. This supports earlier work by Lottering *et al.* (2007), who reported improvements in gold recovery when uranium was leached prior to gold, due to the acid leach acting as an effective pre-treatment step. Since gold is insoluble in acid, this process dissolves interfering gangue minerals and enhances gold accessibility for subsequent cyanidation (Pienaar *et al.*, 2017). In addition to improving recovery, reverse leaching significantly reduced cyanide consumption compared to forward leaching. The lower cyanide usage in reverse leaching suggests that the preceding acid leach likely removed cyanide-consuming phases such as pyrrhotite (Chingwaru *et al.*, 2023). This observation corroborates Nwaila *et al.* (2020), who identified pyrrhotite as a major consumer of both cyanide and oxygen in Witwatersrand ores.

Another factor influencing recovery was ore blending. The composite nature of the Composite Reef sample (Vaal + Crystalkop Reefs) may have contributed to the suboptimal gold recovery observed with forward leaching. Nwaila *et al.*, (2020) showed that blended ores had lower gold recoveries (69%–70%) compared to unblended ores (90%–97%) in the Carletonville goldfield. Youlton *et al.*, (2021) also reported high gold recoveries (96%) in unblended Ventersdorp Contact Reef samples within 24-hour leach period. According to Pienaar *et al.* (2015) and Pienaar *et al.*, (2017), gold recoveries exceeding 92% were achieved from unblended Vaal and Crystalkop Reefs in the Klerksdorp Goldfield using the reverse leaching method. These findings emphasize that blending affects mineralogical interactions, potentially leading to complex matrices that may hinder efficient leaching.

Although reverse leaching showed improved gold recovery, compared to forward leaching, neither method reached the >90 % recovery benchmark typically associated with free-milling ores (Swash, 1988; Harris, 1990; Frimmel, 2005; Hartnady 2009; Youlton *et al.*, 2021). As a result, the composite ore is classified as refractory, given its inability to achieve high recoveries even under optimised leaching conditions. This classification is further reinforced by the outcomes of the

diagnostic leaching tests. The diagnostic leaching results strongly support the reclassification of the Vaal and Crystalkop Reefs as refractory gold ores. Although direct cyanidation recovered 87% of the gold, approximately 13% remained unrecovered.

The persistence of the silicate-bound fraction, accounting for approximately 2.24% of the total gold, represents a refractory component that cannot be treated by conventional cyanidation or acid leaching alone. The presence of this fraction, along with the significant mass of gold held within chemically resistant mineral matrices, supports the reclassification of the Vaal and Crystalkop Reefs as partially refractory gold ores. This has major implications for metallurgical possibilities, as it suggests that conventional free-milling flow sheets would underperform unless supported by pre-treatment technologies such as ultra-fine grinding.

6.2 Conclusions

The demand of gold has forever been increasing. Gold and uranium chemical processing methods of the Vaal River operations have been improving over the years; however, not all the gold is recovered during the leaching process and some of it still gets lost to tailings. The purpose of this study was to investigate and characterise process mineralogical, geochemical and metallurgical properties of the Vaal Reef and Crystalkop Reef at Kopanang Gold Mine with specific focus on gold.

The main methods used in the process of achieving the aims and objectives of this study included geochemical characterisation, mineralogical characterisation and metallurgical characterisation. For geochemical study, XRF, ICP-OES and Fire assay analytical methods were used, mineralogical study was done using optical ore microscopy, MLA and SEM, and lastly, metallurgical study was done using forward, reverse leaching tests as well as a diagnostic leach.

This study was conducted to address the following aim:

The overarching aim of this study is to investigate and compare process mineralogical characteristics of the Vaal and the Crystalkop Reefs to re-classify the Klerksdorp Gold fields as a refractory ore zone in order to understand their implications to gold recovery processes

The objectives were:

To investigate physical and optical properties of the Vaal and Crystalkop Reef at Kopanang Gold Mine using descriptive macroscopic observations and optical microscopy.

To compare mineralogy and geochemistry characteristics of the Vaal and Crystalkop Reefs and understand their implications to the recovery processes of gold.

To document the behaviour of the Vaal and Crystalkop Reefs at Kopanang Gold Mine using AngloGold Ashanti's Great Noligwa plant standards at a laboratory scale, to understand how much gold can be recovered during gold recovery.

Consequently, the study successfully and confidently met the objectives, and the following conclusions were drawn based on the field observations and laboratory results analysed:

The two reefs are approximately 270 m apart with different mineralogical aspects, but the mine process them in the same plant. Face mapping proved that the two reefs are physically different, with the 10 cm Vaal Reef A facies showing some traces of oxidation and being overlain and underlain by a barren quartzite layer (MB5), whilst the 8 cm thinner Crystalkop Reef is overlain by quartzite and underlain by a 24 cm carbon seam. The mineralogical study also proved the difference in the mineralogy of the Vaal Reef and Crystalkop Reef. Mineral components of the former reef include quartz, muscovite, pyrite, pyrrhotite and vermiculite (in descending order) whilst the mineral components of the latter include quartz, pyrophyllite, sericite, kaolinite, pyrite and vermiculite (in descending order).

Gold mineral association results indicated that the Vaal and Crystalkop Reefs are distinguishable. While both reefs show major gold association with pyrite and pyrrhotite, their associations with silicate, oxide, and uranium-bearing minerals differ. In the Crystalkop Reef, gold is associated with a wider variety of silicate minerals, including quartz, pyrophyllite, chamosite, and micas.

In contrast, the Vaal Reef shows gold association only with quartz and chamosite among the silicates. Additionally, the Crystalkop Reef exhibits gold association with uranium-bearing minerals such as coffinite and brannerite, as well as with Fe-rich oxides. These associations are notably absent in the Vaal Reef.

The study demonstrated that reverse leaching, where gold is leached from the residue of uranium acid leaching, yields significantly higher recoveries (76.0%–84.3%) compared to forward leaching (28.3%–55.7%). This supports its use as a more effective strategy for processing refractory gold ores within the Klerksdorp Goldfield. Despite process optimisation, gold recoveries for both forward and reverse leaching remained below the 90% benchmark typical of free-milling ores. Combined with diagnostic leaching results, this confirms the refractory nature of the Vaal and

Crystalkop Reef ores, requiring additional and alternative processing techniques for optimal gold recovery.

The diagnostic leaching results confirm that, while the majority of gold in the composite sample from the Vaal and Crystalkop Reefs is free-milling and recoverable through conventional cyanidation, approximately 23% remains refractory. This refractory fraction is primarily associated with calcite, dolomite, sulphides, and silicate minerals. Notably, although gold shows mineralogical association with sulphides like pyrite and pyrrhotite, most of it is locked within sulphide minerals and is recoverable. In contrast, gold occluded within silicate minerals remains unrecovered and would require more aggressive techniques such as HF treatment or ultra-fine grinding.

6.3 Limitations of the study

This study was limited to reef exposures at level 42 (42.BW.26.RSE.P9E) and level 59 (59.DW.14.R1.P2) and therefore does not represent the entire Kopanang Mine operation. As such, the findings may not fully capture the variability in gold and uranium grades across the broader mining area. Further studies covering additional stopes are recommended to provide a more comprehensive understanding of grade distribution and leaching performance throughout the VMR Kopanang Gold Mine.

The mineralogical dataset employed in this study is constrained by the limited number of sampling points, which were collected only from selected areas of the mine. Such spatial restriction introduces potential sampling bias, thereby limiting the representativeness of the dataset and reducing the overall confidence in the mineralogical interpretations and subsequent inferences.

Another limitation of this study is that the analytical methods employed, primarily SEM and MLA were only able to identify discrete, visible gold particles. These techniques are not sensitive enough to detect gold occurring in solid solution, as sub-microscopic inclusions, or as colloidal gold within host minerals. As a result, the proportion of gold hosted in refractory phases or incorporated within the crystal structure of minerals (e.g., pyrite or arsenopyrite) could not be quantified. This limitation may partially explain the lower-than-expected gold recoveries, as undetected gold in such forms would not be amenable to conventional cyanidation.

6.4 Recommendations and future work

Based on the outcome of this study, the following recommendations to the mine and for future research are presented:

Because the finer-grained gold in VMR Kopanang Mine is mostly locked in silicate minerals (silicates, oxides, etc.) it is thus susceptible to loss during the leaching process, whereas the larger-grained gold is mostly liberated and is likely to be recovered. Consequently, this study recommends further consideration of ultra-fine milling to further liberate even the finest gold particles.

This study only focused on reef exposures at level 42 (42.BW.26.RSE.P9E) and level 59 (59.DW.14.R1.P2) and is not a representation of the entire mine operation, therefore more detailed studies like this one are recommended to compare the results of this study with those of other stopes in order to get a representation of the entire VMR Kopanang gold mine for better understanding of gold and uranium grade fluctuations within the mining operation.

The Vaal Reef at VMR Kopanang Mine is characterised by lower gold grades and is to some extent a refractory ore, while the Crystalkop Reef is characterised by a nugget effect. Therefore, it is recommended to investigate ways of recovering the gold associated with silicates, oxides, phosphates, etc. and the fine-grained size locked in silicate minerals using aggressive leaching conditions to improve the recovery.

Since this study has proved that there is some gold that is locked within silicate minerals which is then susceptible to loss, a study to evaluate the VMR Kopanang tailings is recommended in order to understand the amount of gold lost and if the reprocessing of these tailings is not a viable option.

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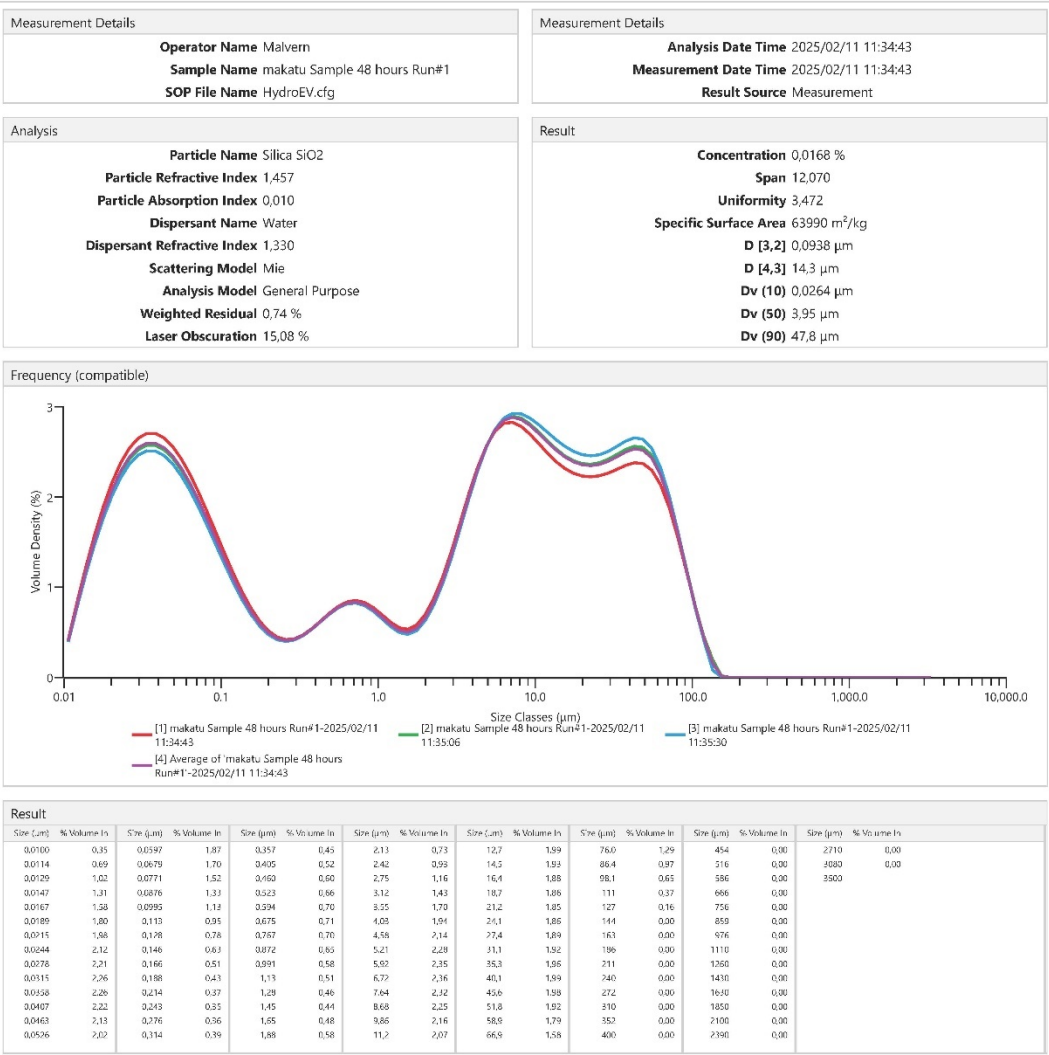
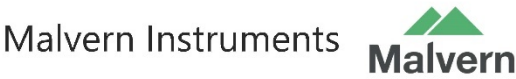
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Appendix A: Milling curves produced from feed samples milling

Appendix A1: Run 1

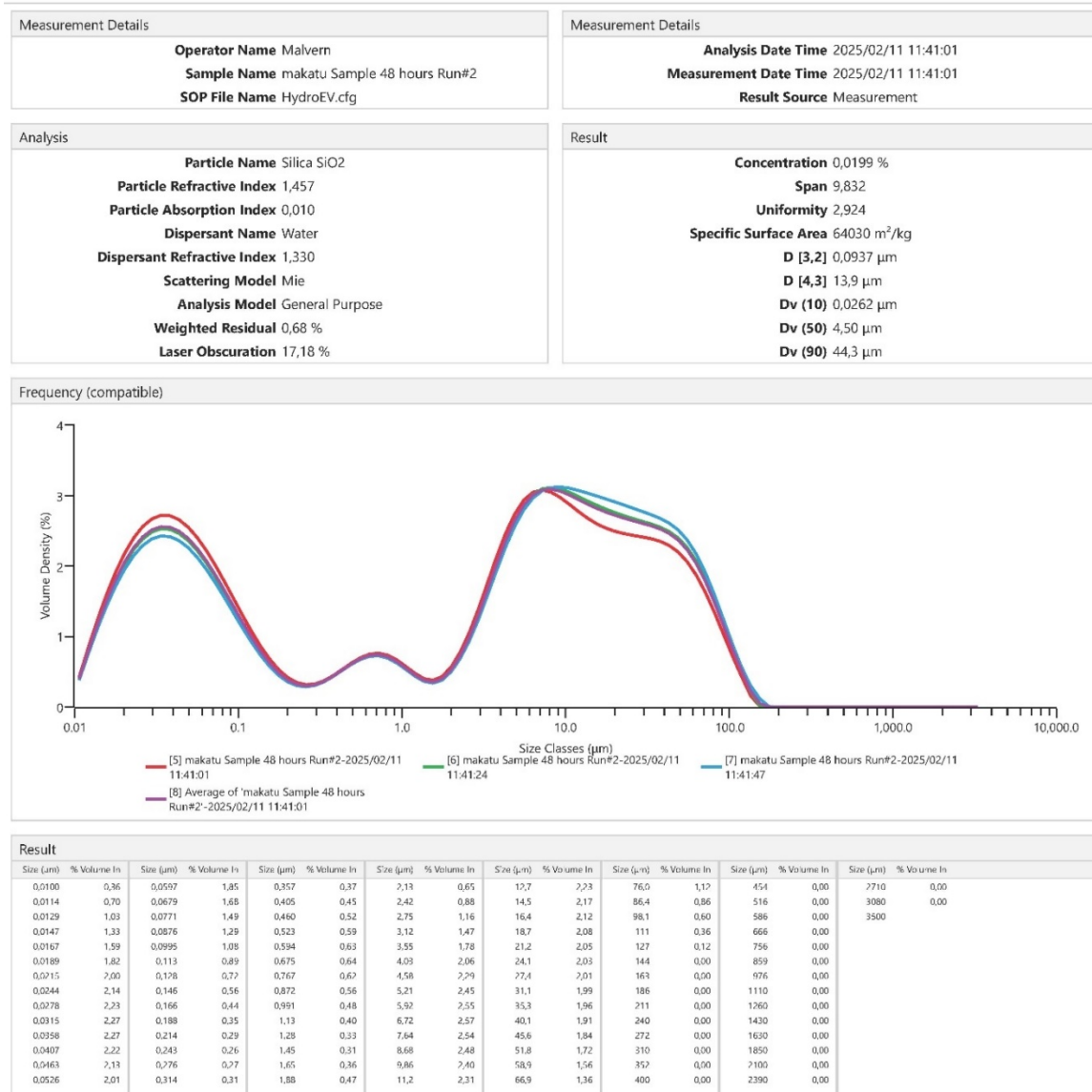
Analysis



Appendix A2: Run 2

Analysis

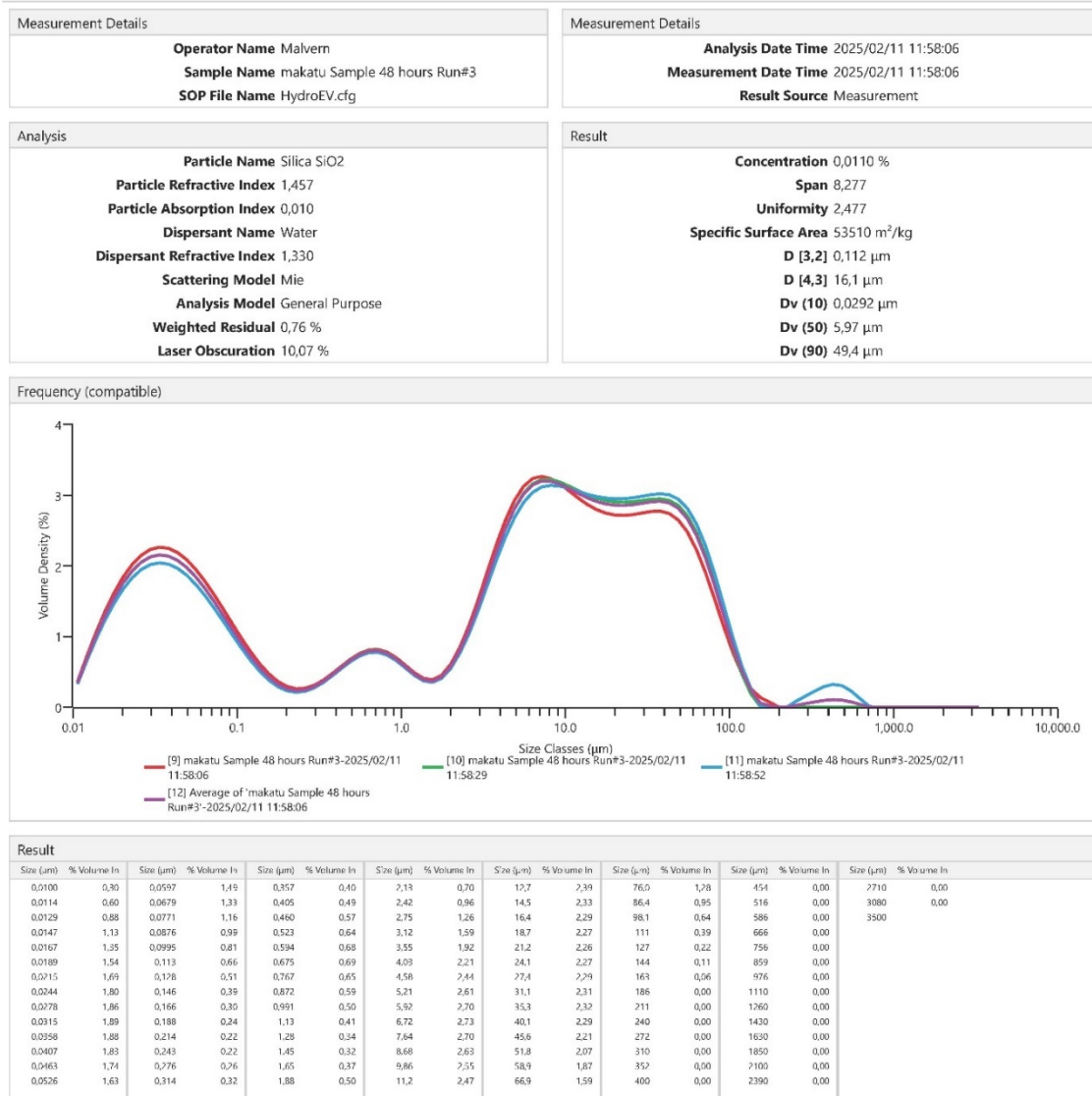
Malvern Instruments



Appendix A3: Run 3

Analysis

Malvern Instruments



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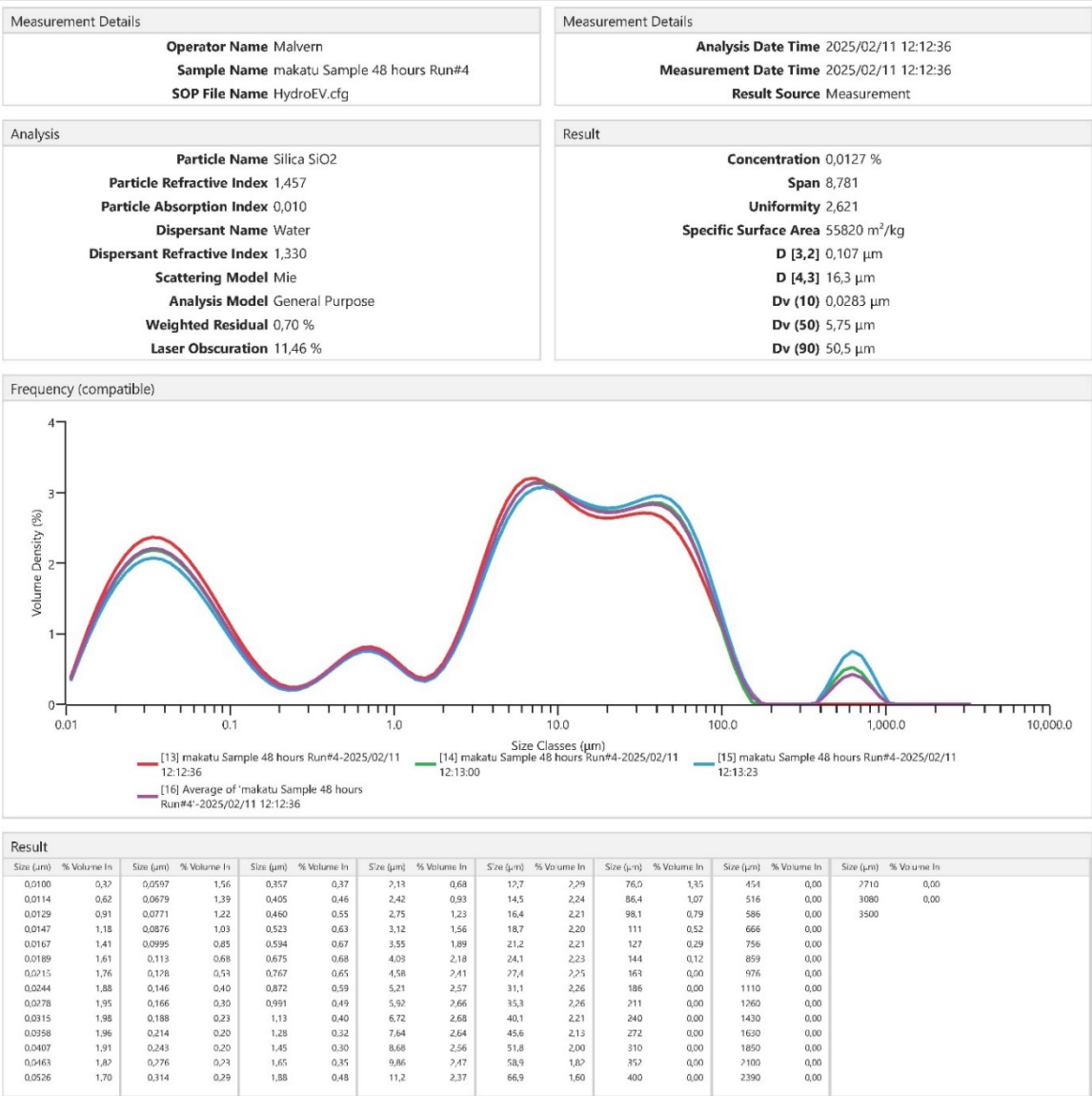
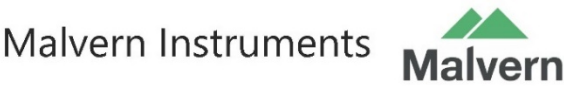
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Page 1 of 1

48hours Run#1

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Appendix A4: Run 4

Analysis



Appendix B: Guidelines for describing grain sizes.

GRAIN SIZE			
Pebble (mm)	Granule (mm)	Sand (mm)	Silt (mm)
64 – Large		1 - Course	0.125
32 - Medium	4-2 (Grit)	0.5 - Medium	0.063 – Clay
4 - Small		0.25 - Fine	

Appendix C: Photographs and descriptions of macroscopic properties of hand specimens.

Sample VR1



In the polished hand specimen, sample VR1 can be described as a quartz pebble oligomictic ortho- to paraconglomerate. The sample is characterised by a variety of smokey quartz granules with sizes ranging between 0.4mm and 1.5mm. Large quartz granules are well rounded with low sphericity whilst the majority of medium to small grains are sub-angular to sub-rounded.

The grains/pebbles in sample VR1 are randomly orientated with poor sorting and moderate packing. The matrix is characterised by an abundance of pyrite and fine to medium dark-greyish sand particle sizes.

Sample VR5



The polished hand specimen, sample VR5, can be described as a quartz pebble oligomictic paraconglomerate. The sample is characterised by a variety of smokey quartz granules with sizes ranging between 0.6mm and 1.5mm. The majority of quartz granules are well rounded with high sphericity.

Grains/pebbles in sample VR5 are randomly orientated with poor sorting and moderate packing. The matrix is characterised by an abundance of pyrite and fine to medium sized dark-greyish sand particles. Few pyrite veins are observed in the overlying fine grained quartzite.

Sample VR9



The hand specimen, Sample VR9, can be described as a quartz pebble oligomictic orthoconglomerate. The sample is characterised by well rounded, smokey and milky quartz grainules with sizes ranging from 0.4mm to 1mm.

Grains/pebbles in sample VR9 are randomly orientated with no imbrication. The grains are moderately sorted to poorly sorted with pyrite grains filling in the voids between the quartz grains. The sample is also characterised by a few pyrite veins occurring within the overlying fine grained quartzite.

Sample CR1



Hand specimen, sample CR1, can be described as a quartz grain oligomictic ortho- to paraconglomerate. Quartz clasts are moderate to well rounded with characterised by moderate sphericity with grain sizes ranging between 0.4mm to 1.2mm.

Grains and pebbles in sample CR1 are randomly orientated with poor sorting and moderate packing. The matrix is characterised by an abundance of pyrite, black carbon around the quartz and fine to medium dark-greyish sand particle sizes. The sample has an MBA footwall which is a black carbon seam.

Sample CR2



Hand specimen, sample CR2, is characterised by large smokey and milky quartz clasts. The clasts are well rounded with very high sphericity and grain sizes ranging from 0.8mm to 2mm and can be described as a quartz pebble oligomictic orthoconglomerated as it is clast supported.

The matrix of sample CR2 is characterised by fine to medium sand size particles of dark-greyish colour with abundant pyrite particles filling in the spaces between the matrix and clasts. The sample is randomly orientated with no imbrication.

Sample CR7



The hand specimen, sample CR7, can be described as a quartz grain oligomictic ortho- to paraconglomerate. Quartz clasts are moderate to well rounded and characterised by moderate sphericity with grain sizes ranging between 0.4mm to 1.2mm.

Grains/pebbles in sample CR7 are randomly orientated with poor sorting and moderate packing. The matrix is characterised by an abundance of pyrite and fine to medium dark-greyish sand particle sizes. Few pyrite veins are observed in the overlying fine grained quartzite.

Appendix D: Particle Count

Mineral	VR1	VR5	VR9
Quartz	85	1884	2845
Feldspar	41	1154	1516
Pyrophyllite	9	334	677
Mica	206	2455	3912
Chamosite	48	689	2794
Zircon	172	287	469
Tourmaline	12	277	627
Other Silicates	3	57	41
Pyrite/Pyrrhotite	245	2741	6964
Chalcopyrite	1	30	56
Other Sulphides	48	258	1134
Uraninite	0	17	28
Coffinite	31	122	175
Brannerite	155	2767	5100
Rutile	110	386	363
Fe-oxide	4	85	50
Other Oxides	0	67	211
Total Carbonates	1	13	3
Gold	2	2	12
Apatite	0	1	23
Phosphates and REE	130	461	1333
Total Sulphates	4	44	207
Other	29	715	1200
Total	688	6641	12871

Appendix

Mineral	CR1	CR2	CR7
Quartz	3760	4591	5615
Feldspar	3197	4424	6756
Pyrophyllite	2490	2848	3000
Mica	5062	6907	7562
Chamosite	7635	3539	7890
Zircon	2802	2934	4673
Tourmaline	2	2	1
Other Silicates	2150	1664	1721
Pyrite/Pyrrhotite	9390	4675	11349
Chalcopyrite	332	189	370
Other Sulphides	3197	1221	3033
Uraninite	98	29	58
Coffinite	779	446	688
Brannerite	8416	6355	6315
Rutile	4065	2720	4159
Fe-oxide	896	216	636
Other Oxides	540	356	720
Total Carbonates	0	1	1
Gold	104	28	31
Apatite	0	0	0
Phosphates and REE	3972	3906	5010
Anhydrite	0	0	0
Other Sulphates	74	14	51
Other	2619	729	1627
Total	25950	20689	31104

Appendix E1: Forward Leaching

Composite Reef Uranium Acid Leach Test Results				
	Leaching Time (hrs)	Au in Leached Filtrate mg/l	Au in Solids after Leach (ICP-OES) g/t	% Leach Availability using ICP-OES
1	4	30.51	59.5	30.6
2	4	27.07	63.4	26.0
3	8	25.14	60.1	29.9
4	8	26.36	57.9	32.5
5	16	58.49	50.1	41.6
6	16	49.45	41.8	51.2
7	24	59.57	43.3	49.5
8	24	58.37	42.7	50.2
9	24	55.97	40.6	52.7

Appendix E2: Reverse Leaching

Composite Reef Uranium Acid Leach Test Results				
	Leaching Time (hrs)	U ₃ O ₈ in Leached Filtrate kg/t	Solids after Leach (XRF) kg/t	% Leach Availability using XRF
1	4	3.141	2.485	50.7
2	4	3.657	2.401	52.3
3	8	3.895	2.694	46.5
4	8	3.777	2.452	51.3
5	16	3.992	2.415	52.1
6	16	3.946	2.502	50.3
7	24	3.796	2.41	52.2
8	24	3.684	2.462	51.1

Composite Reef Gold Cyanide Leach Test Results				
	Leaching Time (hrs)	Au in Leached Filtrate mg/l	Au in Solids after Leach (ICP-OES) g/t	% Leach Availability using ICP-OES
1	4	49.9	19.4	82.2
2	4	78.8	14.8	86.4
3	8	92	20.2	81.5
4	8	75.2	26.1	76
5	16	83.8	17.1	84.2
6	16	82.1	18.4	83.1
7	24	81	17.9	83.5
8	24	70.6	34.2	68.5

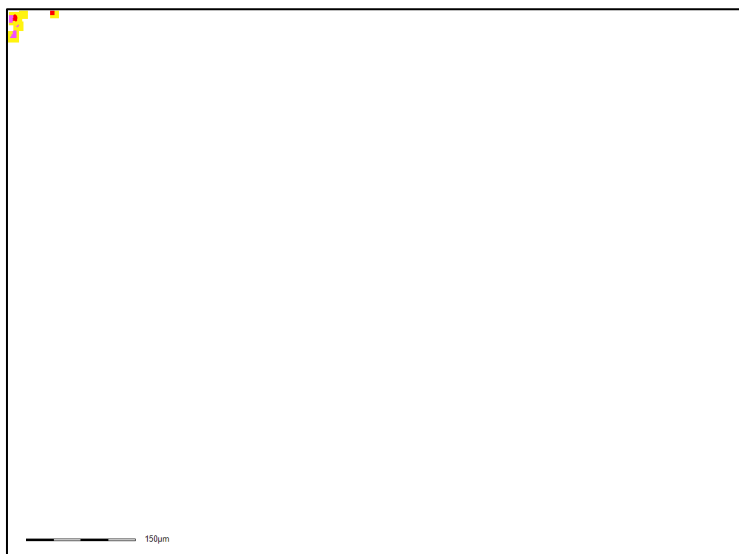
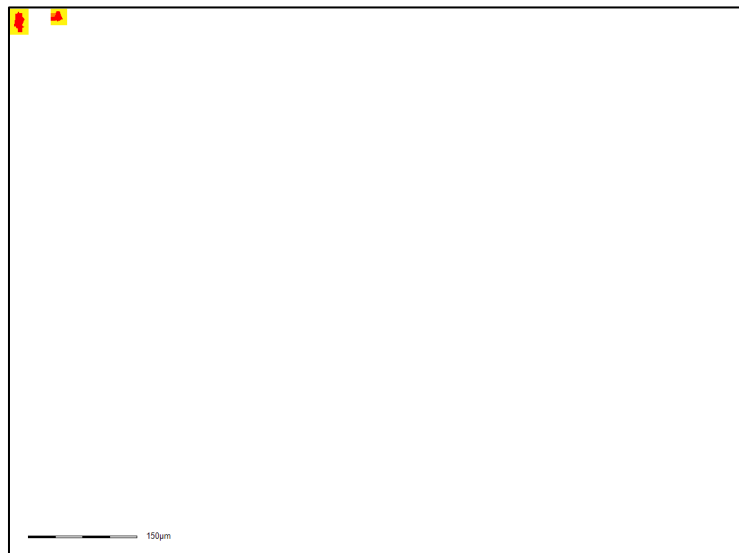
Appendix E3: Diagnostic Leach Results

Lixiviant Sequence	Aim	Au Extractions		Duplicates
		%	g/t (Average)	g/t
-	Head Grade		85.81	
NaCN Stage 1	Leachable Gold	87.0	74.63	71.35
NaCN Stage 1				77.92
HCL Stage 2		3.1	2.67	2.60
HCL Stage 2	Gold in Calcite / dolomite			2.74
NaCN Stage 3		5.3	4.55	5.96
NaCN Stage 3				3.15
H2SO4 Stage 4		0.6	0.55	1.03
H2SO4 Stage 4	Gold in labile Sulphides			0.06
NaCN Stage 5		0.8	0.66	0.82
NaCN Stage 5				0.50
Nitric Stage 6		0.2	0.21	0.22
Nitric Stage 6				0.20
NaCN Stage 7	Gold in Pyrite and other sulphides	0.4	0.37	0.39
NaCN Stage 7				0.34
Final Residue 1	Final Residue	1.9	1.64	1.15
Final Residue 2				2.12
	Total	99.4	85.3	

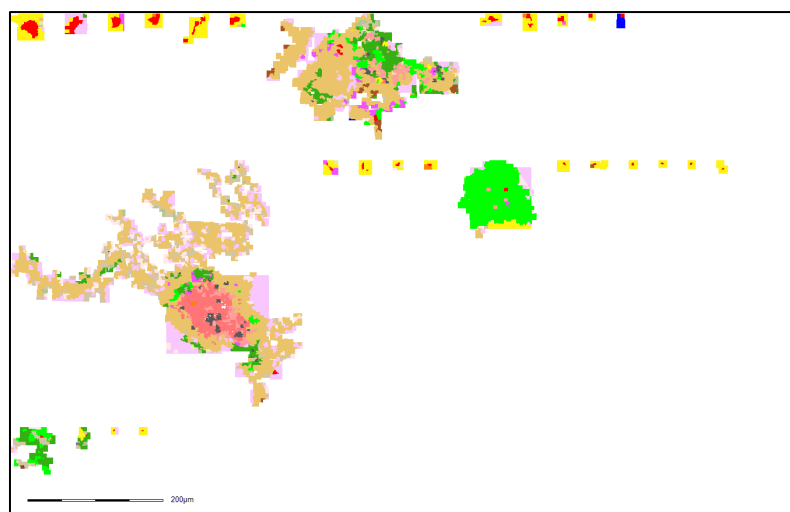
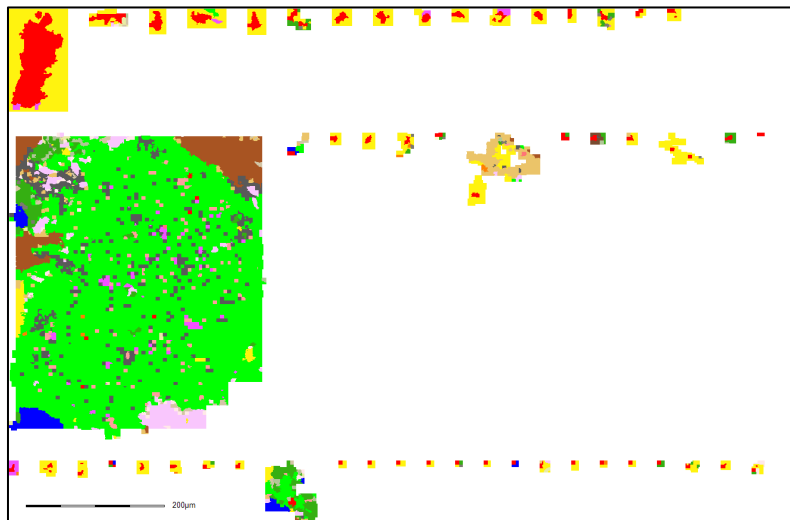
Appendix F: XRF Results for all three feed samples

Sample Name	SI Unit	Vaal Reef	Crystalkop Reef	Composite Reef
Sum of Concentration		100.453	99.651	100.136
Na ₂ O	(wt.%)	0.094	0.111	0.100
MgO	(wt.%)	0.045	0.016	0.000
Al ₂ O ₃	(wt.%)	7.527	8.181	7.950
SiO ₂	(wt.%)	69.083	76.982	73.009
P ₂ O ₅	(wt.%)	0.125	0.113	0.100
SO ₃	(wt.%)	10.094	4.717	7.872
K ₂ O	(wt.%)	1.242	0.998	1.101
CaO	(wt.%)	0.117	0.114	0.112
TiO ₂	(wt.%)	0.906	1.173	0.798
V ₂ O ₅	(wt.%)	0.028	0.059	0.023
Cr ₂ O ₃	(wt.%)	0.153	0.129	0.142
MnO	(wt.%)	0.064	0.032	0.050
Fe ₂ O ₃	(wt.%)	9.768	5.203	7.481
Sum Major Elements		99.246	97.828	98.738
Cl	ppm	84.430	86.971	85.588
Co	ppm	362.861	302.563	327.979
Ni	ppm	895.606	1108.140	831.075
Cu	ppm	LLD	LLD	LLD
Zn	ppm	171.411	492.842	225.705
As	ppm	406.615	470.596	413.786
Rb	ppm	0.000	0.000	0.000
Sr	ppm	134.247	124.335	118.449
Y	ppm	120.236	425.148	145.935
Ag	ppm	3.922	8.338	6.438
Cd	ppm	10.578	7.308	7.565
Sn	ppm	0.000	64.519	39.348
Sb	ppm	10.811	18.357	15.459
Ba	ppm	398.200	1568.972	1229.490
Ce	ppm	438.803	452.588	438.295
Hf	ppm	18.917	20.738	18.619
Ta	ppm	12.299	200.072	46.336
W	ppm	1.996	3.061	2.125
Pb	ppm	3637.980	2814.579	3738.676
Bi	ppm	1080.193	1267.962	1135.688
Th	ppm	333.999	695.765	410.592
U	ppm	2675.274	6718.085	3505.897
Zr		1270.324	1382.959	1233.815
Sum Trace Elements		12068.702	18233.898	13976.860
Sum Trace Elements		1.207	1.823	1.398

Appendix G: Detailed Mineral Maps



Crystalkop Reef



Appendix G: Grain Size Distribution

Vaal Reef grain size distribution results

VR1	Sieve Size	Retained wt. %	Cum. Retained wt. %	Cum. Passing wt. %
	19	0,00	0,00	100,00
	16	65,63	65,63	34,38
	13,5	0,00	65,63	34,38
	11,4	34,38	100,00	0,00
	9,6	0,00	100,00	0,00
VR5	Sieve Size	Retained wt. %	Cum. Retained wt. %	Cum. Passing wt. %
	8,1	0,00	0,00	100,00
	6,8	73,84	73,84	26,16
	5,7	26,16	100,00	0,00
	4,8	0,00	100,00	0,00
	2	0,00	100,00	
	1,75	0,00	100,00	
VR9	Sieve Size	Retained wt. %	Cum. Retained wt. %	Cum. Passing wt. %
	16	0,00	0,00	100,00
	13,5	34,27	34,27	65,73
	11,4	0,00	34,27	65,73
	9,6	18,25	52,53	47,47
	8,1	9,59	62,11	37,89
	6,8	14,05	76,17	23,83
	5,7	4,73	80,89	19,11
	4,8	13,85	94,75	5,25
	4,1	0,00	94,75	5,25
	3,4	2,23	96,98	3,02
	2,9	1,58	98,56	1,44
	2,4	0,92	99,47	0,53
	2	0,00	99,47	0,53
	1,75	0,53	100,00	0,00
	1,45	0,00	100,00	0,00

Cystalkop Reef grain size distribution results

CR1	Sieve Size	Retained wt. %	Cum. Retained wt. %	Cum. Passing wt. %
	90	0	0	100
	75	51,6	51,6	48,4
	63	0	51,6	48,4
	53	0	51,6	48,4
	45	0	51,6	48,4
	38	0	51,6	48,4
	32	0	51,6	48,4
	27	0	51,6	48,4
	22	4,43	56,03	43,97
	19	6,17	62,2	37,8
	16	2,46	64,66	35,34
	13,5	3,03	67,69	32,31
	11,4	3,52	71,21	28,79
	9,6	6,01	77,22	22,78
	8,1	6,99	84,22	15,78
	6,8	2,46	86,68	13,32
	5,7	8,78	95,46	4,54
	4,8	1,36	96,82	3,18
	4,1	0,64	97,46	2,54
	3,4	0,69	98,15	1,85
	2,9	0,56	98,71	1,29
	2,4	0,58	99,29	0,71
	2	0,4	99,69	0,31
	1,75	0,31	100	0
	1,45	0	100	0
	1,2	0	100	0
	1	0	100	0
	0,87	0	100	0
	0,73	0	100	0
CR2	Sieve Size	Retained wt. %	Cum. Retained wt. %	Cum. Passing wt. %
	27	0,00	0,00	100,00
	22	22,55	22,55	77,45
	19	16,54	39,09	60,91
	16	0,00	39,09	60,91
	13,5	17,12	56,20	43,80
	11,4	6,13	62,33	37,67
	9,6	0,00	62,33	37,67
	8,1	12,07	74,40	25,60
	6,8	4,28	78,68	21,32
	5,7	9,98	88,66	11,34
	4,8	5,87	94,53	5,47
	4,1	0,89	95,41	4,59

Annexure

	3,4	2,39	97,80	2,20
	2,9	0,77	98,57	1,43
	2,4	0,62	99,19	0,81
	2	0,19	99,38	0,62
	1,75	0,62	100,00	0,00
	1,45	0,00	100,00	0,00
CR7	Sieve Size	Retained wt. %	Cum. Retained wt. %	Cum. Passing wt. %
	27	0,00	0,00	100,00
	22	45,91	45,91	54,09
	19	0,00	45,91	54,09
	16	0,00	45,91	54,09
	13,5	0,00	45,91	54,09
	11,4	0,00	45,91	54,09
	9,6	10,13	56,03	43,97
	8,1	0,00	56,03	43,97
	6,8	10,21	66,24	33,76
	5,7	13,09	79,33	20,67
	4,8	7,25	86,58	13,42
	4,1	1,77	88,35	11,65
	3,4	5,40	93,75	6,25
	2,9	0,84	94,60	5,40
	2,4	1,11	95,71	4,29
	2	2,78	98,49	1,51
	1,75	1,51	100,00	0,00
	1,45	0,00	100,00	0,00

Annexure A: AngloGold Ashanti Great Noligwa Plant Processing Standards.

Milling: Mill to 80 % passing 75 micron and 3 % retained on 150-micron screen (Appendix F).

Uranium Leach Test	➤ Sample was re-pulped to a density of 1.500. ➤ Slurry heated = 50 degrees Celsius ➤ Add 9.9 kg/t Sulfuric Acid while heating the slurry. ➤ Contact for 1 hour 40 minutes. ➤ Maintain the temperature for the duration of the test. ➤ Add 6.0 kg/t Manganese Dioxide ➤ Contact for 1 hour 40 minutes. ➤ Add 8.1 kg/t Sulfuric Acid ➤ Contact for 1 000 minutes. ➤ Filter the solids and liquids
Gold Leach	➤ Gold leach on uranium tailings ➤ Adjust slurry density to 1.450. ➤ Add oxygen to achieve a DO level of 20 mg/l. ➤ Neutralise residue from uranium leach with CaO in three stages as follows: • Increase pH to 6.5 and agitate for 60 minutes. • Increase pH to 7.5 and agitate for another 60 minutes. • Increase pH to 10.5 and agitate for another 60 minutes. ➤ Add 360 g/t sodium cyanide ➤ Contact for 24 hours at room temperature.

Annexure

	➤ Measure DO level and spike where necessary until the required 20 mg/l of oxygen. ➤ Maintain dissolved oxygen levels. ➤ Continue measuring DO and spiking where necessary.
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Annexure B: Sample preparation for mineralogical study

Sample preparation for mineralogical study	
Cutting and trimming	➤ Large rock samples were cut into smaller blocks of approximately 5cm using a diamond cutting saw. Water and cutting oil were applied as lubricants on the diamond saw during cutting. ➤ After cutting, the samples were trimmed using a Geoform-105 cutting machine to further reduce the size to approximately 2cm to fit into the mounting cups. The samples were then dried overnight using a Vacutec drying oven at 50°C. ➤ For thin sections, samples were cut into small rectangular blocks of approximately 30x40 mm using a Geoform 150 cutting machine.
Mounting and bonding	➤ The bonding agent was prepared by mixing the Akasel Epoxy resin and Akasel hardener using a 2.5:0.3 ratio (2.5 g of resin and 0.3 g of hardener). Mounting cups were cleaned and dried to avoid contamination. Sixteen mounting cups were used for the 16 samples, which were numbered according to the sample number. ➤ Polished samples were then put into the mounting cups first before pouring the bonding agent (mixture of resin and hardener) into the cup. ➤ The samples were left to dry 24 hours before polishing. ➤ The bonding agent used for mounting ore blocks was also used to mount rectangular blocks on frosted glass slides for thin sections. ➤ The glass slides were frosted using the Geoform-150 machine grinding compartment.

Grinding and polishing	➤ The Struers LaboPol-5 polishing machine was used for grinding and polishing the ore blocks. Grinding was done to remove the surface irregularities of the blocks, reduce the thickness and prepare a smooth surface. ➤ Four grit disks (150, 220, 1000 and 1200) were used to grind the ore blocks from the 150, which is more course, to 1200, which is finer. ➤ After grinding, polishing of the blocks followed, and this was done to improve the transparency and optical quality of the samples to enhance mineral identification and to smoothen surface irregularities. ➤ The Struers LaboPol-5 polishing machine was used to polish the surfaces of the ore blocks using a 3-micron polishing disk. ➤ Thin section samples were firstly cut before grinding to reduce the thickness from 40 mm to 10 mm using a Geoform-150 cutting machine. Samples were then manually grinded to 50 µm. ➤ Grinded samples were placed on a LaboPol-5 polishing machine and polished on a 3-micron polishing disk for approximately 3 hours until the required thickness of 30 microns. ➤ Both polished ore blocks and polished thin section samples were analysed under a microscope using a Leica ICC50 W petrographic light microscope.
Crushing	➤ The reef samples were crushed using a jaw crusher to reduce the size to a smaller manageable size that would fit into the mill rings for pulverising.
Splitting	➤ After crushing individual samples, splitting was done using a riffle splitter in order to produce smaller representative samples which were later mixed to make three composite samples.

	➤ Bulk samples were poured into the riffle splitter tray, which then allowed the material to flow down the chutes of the splitter. ➤ Subsamples were then collected at the bottom of each chute for further milling into smaller sizes.
Milling	➤ Samples were milled to powder using a ring milling machine at the North-West University in the Department of Geology and Soil Science milling lab. ➤ Each crushed sample was milled for approximately until 80% passed a 75 µm sieve as per the mine plant standard. ➤ The sample size fractions were virtually tested using a Malvern and the tests were run in triplicates to validate the results. ➤ To prevent cross contamination between samples, mill rings were thoroughly washed with water and thoroughly cleaned with acetone.

Annexure C: MLA-SEM Conditions

Brightness	76
Contrast	34
Working Distance	13mm
kV	25
Magnification	600
Spot Size	5.958
Time	3 hours per block
Pixel Size	0.6µm

Annexure D: Hazard identification and risk assessment (HIRA)

Probability / Likelihood categories:

Probability	Rating	Incident has occurred / What is the Chance of Incident will or may occur
ALMOST	5	Incident has occurred; has a 90%> chance of reoccurring. Incident may or will occur; has a 90%> chance of occurring.
LIKELY	4	Incident has occurred; has a 50% - <90% chance of reoccurring. Incident may or will occur; has a 50% - <90% chance of occurring.
MODERATE	3	Incident has occurred; has a 20% - <50% chance of reoccurring. Incident may or will occur; has a 20% - <50% chance of occurring.
UNLIKELY	2	Incident has occurred; has 1% - <20% chance of reoccurring. Incident may or will occur; has 1% - <20% chance of occurring.
RARE	1	An incident has never occurred, has a <1% chance of occurring

Consequence categories:

Consequence	Rating	Injury / Damage		
		Productivity	Injury	Asset damage
Catastrophic	5	More than month	Fatality	>R 5 mil
Major	4	More than week lost	Disabling injury	<R 5 mil
Moderate	3	3 – 8 hr lost	More that first aid	R 100 000 – R1 mil
Minor	2	2 hr lost	First aid	R 50 000 – R 100 000
Insignificant	1	1 hr lost	No injury	R 1000 – R 50 000

Actions to be taken when:

RED – HIGH RISK

(Take immediate action to reduce, mitigate & control the risk)

Implement safe work procedure(s) and task observations

ORANGE – MEDIUM RISK

(Take action to reduce, mitigate & control the risk)

Implement safe work procedure(s) and task observations

GREEN – LOW

(Tolerate the risk with current controls and supervision)

Annexure E: HAZARD IDENTIFICATION & RISK ASSESSMENT

Item Nr	Task / Process / Activity	Risk(s) – Potential incident or loss	Risk Calculation (Pure Risk)			Current Controls	Safety Plan			Risk Calculation (Residual risk)		
			C	P	RV		Additional Controls	Responsible person	Target Date	C	P	RV
1. Bottle Role Procedure												
1.1	Obtain the dried solids from the Uranium leach and ensure that it is milled to the following specifications (80% passing 75micron and 3% retained on 150micron screen)	If sample is not milled within the prescribed specifications, it will not conform plant conditions and incorrect results will be obtained. Inhalation of dust particles affecting the respiratory system.	3	2	6	Sample is checked using Particle size analyser to confirm the value before continuing with analysis. Students are trained in the use of material. Correct PPE is supplied and used (Dust mask)				2	1	2
1.2	Weigh the solids and record the mass of sample on a top pan balance and record the weight.	Incorrect mass could be weighed resulting in incorrect results. Inhalation of dust particles.	2	2	4	Balances are inspected and checked with calibrated mass pieces before use.				1	1	1

Annexure

	Transfer to glass reagent bottle (500ml).	Sample spillages resulting in incorrect mass transferred. Breakage of glass can result in cuts.				Spillages are cleaned up immediately. Students are trained in following correct procedures. Dust masks are used when working with samples. Correct rubber gloves are used when working with glass.						
1.3	The sample should be prepared to a density of 1.45 to simulate plant conditions – add the correct amount of water using the following formula: $D = m/v$ $1.45 = \text{kg of solids}/v$	Spillages resulting in incorrect quantity added to solid. Spillages can result in slips. Breakage if glass resulting in cuts.	1	2	2	Students are trained to follow procedures. Spillages are cleaned up immediately. Correct PPE (gloves are used to prevent injuries)				1	1	1

Annexure

	V = $\frac{xg}{1.45}$ = xml of distilled water.											
1.4	Mix the water and solid to obtain a pulp.	Spillages resulting in incorrect quantity added to solid. Spillages can result in slips. Breakage if glass resulting in cuts.	1	2	2	Students are trained to follow procedures. Spillages are cleaned up immediately. Correct PPE (gloves are used to prevent injuries)				1	1	1
1.5	Use a densitometer to check if the density is in the correct range.	Glass Breakage resulting in cuts. Temperature sensitivity – sudden temperatures can cause glass to crack resulting in accuracy of measurements.	1	2	2	Students are trained in the use of equipment and safety procedures are followed. Correct PPE (gloves) are used when working with glass.				1	1	1

Annexure

1.6	Measure the pH of the pulp and increase to pH 6.5 and agitate for 1hour using CaO powder. Increase pH to 7.5 by a further addition of CaO and stir for 1hour. Add CaO to pH of 10.5 and stir for 1hour.	Electrode contamination – resulting in inaccurate measurements. Calibration drift Electrode damage due to rough handling Temperature affects. Chemical burns Corrosive effects Inhalation from dust particles. If pH is not adjusted to >10 the reaction between Cyanide and low pH sample can result in HCN gas to be released.	5	1	5	Students are trained in the use of pH meters. pH meters are calibrated before use. pH meter is regularly maintained. Correct PPE (Gloves) are used to ensure student is protected from chemical burns. Standard procedure is followed.				2	1	2
1.7	Add 360g/t NaCN (converted to 100gram sample). 360gNaCN for 1ton, therefore for 360g in 1000kg = xNaCN in 100g = 0.1kg x 360/1000	Toxicity – Sodium cyanide can be absorbed through the skin, inhalation of powder, irritation to skin and eyes. Cyanide poisoning depletes oxygen in the body – rapid breathing,	5	1	5	Students are trained in using Sodium Cyanide. Correct protocols are followed, Cyanide is weighed and signed out and				2	1	2

Annexure

	= 0.036g / 100g.	seizures, loss of consciousness. Chemical reactivity – when reacting with acids it can produce dangerous gas (HCN), which is deadly. Unauthorized usage and removal from store can result in people removing without knowledge.				return and locked away (Logbook in place). Sample is treated with CaO to ensure pH >10 before Cyanide addition. All work is conducted under extraction unit. Students are not allowed to work alone when working with Cyanide. Correct PPE is (Gloves, goggles, mask, and lab coat). Safety shower is available and operational.						
1.8	Place lid on bottle containing hole to	If no oxygen enters the reaction the dissolution of	5	1	5	Students are trained using				2	1	2

Annexure

	allow oxygen into the sample and place on roller bed and agitate for 4hrs, 8hrs, 12hrs and 24hrs. A new sample for each time needs to be used.	gold is not effective or insufficient. Sample spillages resulting in contamination of the area with cyanide. Inhalation of fumes if in contact with other chemicals like acids.				the safe work practice. The correct procedures are followed. Correct PPE is used and provided (Gloves blue latex, lab coat, goggles).						
1.9	Remove the bottle and slurry and filter to obtain the filtrate and solid.	Sample spills when filtering resulting in splashing and contact with skin. Inhalation of fumes affecting the respiratory system.	2	2	4	Students are trained as per safe work practice. Filtration is conducted under extraction unit. Correct PPE is used when handling slurry (Gloves latex, safety glasses and lab coat)				2	1	2
1.10	Transfer the solution to a sample container for analysis.	Dirty glassware resulting in contamination and incorrect results.	2	2	4	Students are trained in the correct Safe work practice.				2	1	2

Annexure

		Glass breakage resulting in cuts.				Glassware is inspected and cleaned before use. Correct PPE (gloves latex) is used to assist in the prevention of cuts						
1.11	Re-pulp the solid three times with distilled water and dry the solids with the filter paper at 105°C overnight.	If sample is not re-pulp completely soluble metals will stay behind and incorrect result will be obtained. Burns from heat generated from drying oven.	2	2	4	Correct Safe work practice is followed, and students are trained. Correct tools are used to handle hot objects. Heat resistant leather gloves are available to prevent student from injuries.				2	1	2
1.12	The solution sample is analysed for gold on the ICP and residual	Only a note										

Annexure

	cyanide will be titrated with silver nitrate. The solid will be digested using aqua-regia (Nitric and Hydrochloric acid) to determine gold. The remainder of the solids will be submitted for XRF analysis and a portion the mine laboratory for comparative gold analysis using Fire Assay.											
2. Titration of residual Cyanide using silver nitrate solution												
2.1	Prepare a solution of 0.01mol dm^{-3} Silver Nitrate	Eye and respiratory irritation – coughing, shortness of breath and irritation to nose and throat. Corrosive – can cause damage to skin and burns as well as discolouration. Photo sensitive -light sensitive and can decompose resulting in incorrect value.	2	2	4	Students are trained as per safe work practice how to handle chemicals. Correct tools like spatula are used to remove chemicals.				2	1	2

Annexure

						Silver Nitrate is stored in dark bottles to prevent it from any external light. Correct PPE (latex gloves are used when handling chemicals).						
2.2	Prepare Rhodanine indicator in acetone. (0.2g Indicator in 100ml acetone)	Rhodanine - Low irritation to skin. Acetone – flammable. Inhalation of fumes can cause irritation to the respiratory system. Irritation to skin and eyes.	2	2	4	Chemicals are used in small quantities so limited exposure. Acetone is stored away from heat and only small quantities used at a time. Correct PPE (Rubber gloves and not latex is used when handling acetone).				2	1	2

Annexure

2.3	Transfer the Silver Nitrate solution to a clean burette.	Chemical spillages resulting in slips and reaction with other chemicals. Glass breakage resulting in cuts. Contamination due to dirty glassware resulting in incorrect results.	2	2	4	All spillages are cleaned up immediately. All glassware is cleaned before use. Correct PPE is used when handling glassware like rubber or latex gloves.				2	1	2
2.4	Add 50ml of the solution sample to an Erlenmeyer flask and add few drops Rhodanine indicator	Chemical spillages resulting in slips and reaction with other chemicals. Glass breakage resulting in cuts. Contamination due to dirty glassware resulting in incorrect results.	2	2	4	All spillages are cleaned up immediately. All glassware is cleaned before use. Correct PPE is used when handling glassware like rubber or latex gloves.				2	1	2
2.5	Titrate with Silver nitrate solution to end point (Orange to salmon pink).	Incorrect reading if the student does not read the meniscus correctly can result in incorrect reading.	1	2	2	Students are trained in following correct procedures				1	1	1

Annexure

2.6	Calculate the residual cyanide in the sample.	Only note										
3.Microwave digestion for solid sample												
3.1	Ensure that all the equipment is clean before use.	Contamination resulting in incorrect results. Cuts from broken crystal vessels. Loss of equipment resulting in expenses due to cost of crystal vessels.	2	2	4	All vessels are cleaned as per procedure with Nitric and Hydrochloric acids. Students are trained in handling the equipment. Correct PPE is used (Latex gloves when handling equipment).				1	2	1
3.2	Weigh 5 – 10 grams of solid material into the crystal microwave container and record the mass.	Sample spillage Breakage of glass resulting in cuts. Incorrect mass if balance is not calibrated correctly.	2	2	4	Spillages are cleaned up immediately and correct procedure is followed. Balances are checked with calibrated mass pieces before use.				2	1	2

Annexure

						Correct PPE is used when using Glassware (Rubber or latex gloves).						
3.3	Add the Nitric and Hydrochloric acids to the container and allow the reaction to seize completely.	Acids are corrosive and can cause severe burns and tissue damage. The reaction of acids with solids can be violent and generate fumes that are toxic when inhaled.	3	2	6	When acids are handled students are trained in the risks and hazards, The correct PPE is used when handling acids (Latex and rubber gloves). When acids are used it is done under the extraction unit.				2	1	2
3.4	Place the lid on the container and transfer to the Microwave sample holder	Spillages if containers are dropped resulting in burns. Glass breakage can result in cuts.	3	2	6	Spillages are cleaned up immediately, Correct PPE (Gloves, glasses, safety shoes and lab coat is used).				2	1	2

Annexure

3.5	Choose the correct program on the microwave and start the process.	Incorrect program can result in incomplete dissolution and low results.	3	1	3	Student is trained on the correct procedure.				2	1	2
3.6	Once the process is completed, remove from the microwave, and transfer the contents to a sample holder.	Burns from handling hot objects. Inhalation of fumes when lids are removed and resulting in irritation to the throat.	3	2	6	Correct PPE is used (Leather gloves when handling hot objects). When the containers are opened it is conducted under the extraction unit.				2	1	2
3.7	Filter the contents and submit for ICP analysis.	If samples are not filtered it can block the nebulizer resulting in low results.	2	1	2	Standard procedure is followed and students are trained in the correct procedure.				1	1	1

