



# **Flotation of non-sulphide PGM ores - Optimization of flotation reagent suite and conditions**

by

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the degree of Master of Science in Engineering

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## **DECLARATION**

I declare that this dissertation is my own unaided work besides where stated otherwise. It is being submitted to the degree of Master of Science in Engineering to the University of the Witwatersrand, Johannesburg. It has not been submitted before for any other degree or examination in any other University.

A handwritten signature in black ink, appearing to read 'S. S.' or similar, positioned above a horizontal dotted line.

Katlego Sekgarametso

08 November 2018

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## **PUBLICATIONS**

Sefako, R., **Sekgarametso, K.** & Sibanda, V., 2017. Potential processing routes for recovery of platinum group metals from Southern African oxidized PGM ores: A review. *Journal of Sustainable Metallurgy*, 3(4), pp. 797-807.

## **ABSTRACT**

The aim of this study is to improve the flotation of non-sulphide PGM ores from the Mimosa Mine in the Great Dyke of Zimbabwe by evaluating a variety of collector reagents that have not been tested on such material before and applying a full factorial experimental design to investigate the effects of the main primary collector, co-collector and depressant on PGM recovery and grade.

The mineralogical studies by XRD revealed that the non-sulphide PGM ore had substantial amounts of gangue material, comprising of 45% quartz, 21% chabazite and 33% magnetite. The ICP-OES analysis showed that this particular non-sulphide PGM ore is a low-grade ore with an average 4E head assays of 2.37ppm.

In the preliminary flotation stage, three reagent suites made up of (i) a collector, (ii) a co-collector and (iii) a depressant i.e. (SIBX, DTP, M98B); (SIBX, C7133, M98B) and (SIBX, AM810, M98B) respectively were tested. It was observed that (SIBX, AM810, M98B) reagent suite gave the best performance with respect to both recovery and grade of the PGM concentrate from the ore. Attempts were made to optimize the dosage levels of the 3 reagents. The optimization studies revealed that 78.5% Pt and 69.3% Pd can be recovered at grades of 17.90g/t Pt and 9.44g/t Pd respectively. This represents a significant upgrade for the roughing stage from the 1.42g/t Pt and 0.85g/t Pd in the feed. These results were obtained at optimized dosages of 86g/t SIBX and 80g/t AM810, with depressant M98B at 50g/t.

The observations from the experiments indicated that recovery of PGEs was on the upward trend as the dosage of hydroxamate was increasing hence the effect of the hydroxamate co-collector was further tested at higher dosages while fixing SIBX at 100g/t. The experiments were carried out using 50g/t, 60g/t, 70g/t and 80g/t hydroxamate (AM810) with the depressant M98B at 50g/t. It was observed that the Pt recovery only increased slightly with increasing hydroxamate (AM810) dosage.

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## Nomenclature

PGM	Platinum Group Metals
BC	Bushveld Complex
MSZ	Main Sulphide Zone
UG	Upper Ground
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
SIBX	Sodium Isobutyl Xanthate
IPETC	O-isopropyl-N-ethyl thionocarbamate
TTC	Trithiocarbonates
CMC	Carboxymethyl cellulose
PPM	Pilanesburg Platinum mine
DTP	Dithiophosphate
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
NFG	Naturally Floating Gangue
DOE	Design of Experiments
$k_f$	fast floating constant rate
$k_s$	slow floating constant rate
$P_f$	fast floating mass fraction
$P_s$	slow floating mass fraction

## **CHAPTER 1 : INTRODUCTION**

### **1.1. Background**

Platinum Group Metals (PGMs) are used in a range of applications such as the manufacture of catalysts for chemical process facilities, catalytic converters for exhaust control in transport vehicles, hydrogen fuel cells, electronic components in the medical sector and in jewellery manufacture. It's due to their unique properties such as high conductivity, high corrosion resistance that they have numerous applications and are in high demand (Glaister & Mudd, 2010; Jones, 2005). The six PGMs; platinum, palladium, rhodium, ruthenium, iridium and osmium usually occur together with gold and base metals such as nickel, cobalt and copper. Platinum, rhodium and palladium are the most important of the PGMs due to their abundance and economic significance whilst the other three PGMs are produced as by-products (Jones, 2005).

The Southern African region is home to the largest deposits of PGMs in the world, with the Bushveld Igneous Complex (BIC) in South Africa holding 70% of the world's platinum and 40% of the world's palladium (Kraemer, et al., 2017). The Main Sulphide Zone (MSZ) of the Great Dyke of Zimbabwe is believed to host the second largest deposits (Vermaak, 2005). The Bushveld Complex comprise of three reefs; Merensky reef, Upper Group 2 (UG2) reef and the Platreef. The Merensky reef has been the most exploited and continues to be due to its relatively higher PGM grades. The UG2 reef has similar PGM content as the Merensky, is rich in chromite but has lower levels of nickel and copper while the Platreef has lower PGM values and it is enriched in palladium and base metals (Jones, 2005).

The most economical reserves of PGMs in the Great Dyke of Zimbabwe are found in the sulphide ores of the MSZ, which are currently mined at the Ngezi, Unki and Mimosa mines. The sulphide-rich MSZ and BIC are treated by conventional metallurgical processes such as comminution, flotation, smelting and matte production and lastly chemical refining (Oberthur, et al., 2013). The PGM pristine ores (sulphide-rich PGM ores) contains between 3 to 10 ppm of PGMs and are treated by comminution to liberate the sulphide mineral grains associated with PGM minerals. This is followed by the separation of liberated PGM and sulphide mineral grains from gangue minerals by froth flotation ( Crundwell, et al., 2011).

The froth flotation concentrate typically contains about 70 to 100 ppm PGMs consisting of base metal sulphide minerals such as chalcopyrite, pentlantite and pyrrhotite and over 85% of Pt recovered. It is the close association of base metal sulphide minerals with PGM minerals in pristine ores that has been the foundation of the success in the froth flotation of pristine PGM ores, due to the easy floatability of sulphide minerals (Oberthur, et al., 2013; Kraemer, et al., 2017).

The fast depletion of pristine PGM ores and rising mining costs caused by mining increasingly happening at deeper levels, has triggered interest in the processing of weathered non-sulphide PGM ores found near the surface or from open pit mines. These attempts have proved futile as recoveries lower than 50% have been consistently achieved in the flotation stage, as it was the case at the Old Wedza mine (1926-1928) and Hartley mine (1997-1999) (Oberthur, et al., 2013; Vermaak, 2005). Attempts have also been made in the Bushveld Complex UG2 Reef at Smokey Hills mine, Impala Platinum mine in the western Bushveld and at the Pilanesburg Platinum Mine (PPM) to process the non-sulphide PGM ores. All efforts in the Bushveld Complex also resulted in low recoveries using conventional metallurgical processes (Becker, et al., 2014; Oberthur, et al., 2013).

Numerous researchers suggest that the non-sulphide PGM ores were originally the pristine sulphidic ores that were altered by exposure to atmospheric conditions and underground waters leading to oxidation (Becker, et al., 2014; Kraemer, et al., 2017). Oberthur et al. (2013) states that in oxidised MSZ PGM ores, sperrylite and cooperite/braggite grains, which make up about 25% of the original Pt content of the ore, largely remain stable as relict PGM minerals, whereas the (Pt,Pd)-bismuthotellurides are disintegrated. They further assert that base metal sulphides are destroyed during weathering, partly releasing their base metal and PGM contents, and are replaced by iron oxides or hydroxides (Oberthur, et al., 2013). Legrand et al (2005) also found that hydrophilic iron (III) oxyhydroxide (FeOOH) is formed due to oxidation.

It is understood that the oxidation of base metal sulphide minerals such as pentlandite and pyrrhotite occur when iron moves from the bulk to the gas-solid interface where it is oxidised (Legrand, et al., 2005; Buckley & Woods, 1984; Mielczarski, et al., 1996; Vaughan, et al., 1997).

Becker et al (2014) also attributes the poor flotation response of non-sulphide ores to high contents of naturally floating gangue such as talc, chlorite and aluminosilicates, a lack of base metal sulphide association with PGMs and the Pd remobilization from the weathered zone to the lower zone which reduces the flotation efficiency by changing Pt:Pd ratio, an important metric in the flotation of PGM ores (Becker, et al., 2014; Bulatovic, 2003; Kraemer, et al., 2017)

Currently, an estimated resource of about 250 to 400 Mt of non-sulphide PGM ores measuring approximately 1.5ppm 4E in grade is reported to be available in the Great Dyke of Zimbabwe either left in situ, stockpiled, or discarded as waste. It is also estimated that these ores are as much as 337 Mt (2.4ppm 4E) in the Bushveld Complex (Oberthur, et al., 2013). These large resources of non-sulphide PGM ores have an enormous economic potential that justifies further research into improving flotation recoveries or developing alternative routes of beneficiating them.

The correct selection and dosing of collectors remains a huge challenge in the flotation of non-sulphide PGM ores. This research, therefore, focused on trying to improve the flotation of non-sulphide PGM ores from the Mimosa Mine in the Great Dyke of Zimbabwe by applying a full factorial experimental design to investigate the effects of the main primary collector, co-collector and depressant on PGM recovery and grade.

In the current study, batch flotation tests were conducted on a non-sulphide PGM ore using primary collector SIBX, co-collectors hydroxamate AM810, Flomin C7133 and Flomin C5460. According to the hydroxamate supplier, Axis House Pty(Ltd), AM810 is an improved formulation of the previously promising AM28, it is a C8-C10 alkyl hydroxamate used to collect heavily oxidized or tarnished minerals. AM810 alkyl hydroxamate has froth characteristics which require that frother dosage is controlled carefully. The co-collectors Flomin C5460 and Flomin C7133 are new reagents which have not been tested on non-sulphide ores before; Flomin C5460 is a blend of sodium isobutyl dithiophosphate and IPETC and Flomin C7133 is a blend of xanthogen formate and IPETC. IPETC is a dialkyl

thionocarbamate, comprising of a branched isopropyl group and a short-chained ethyl hydrocarbon group, and is known to have frothing properties (Maree, et al., 2017) .It is envisaged that the improved reagents and conditions will increase the recovery of PGE and associated base metals from what has been achieved by previous workers.



## 1.2. Problem statement and research questions

The oxidised PGM ores present a challenge when processed using conventional methods and reagent regimes, as poor flotation recoveries (typically less than 50%) are experienced. Different workers have attributed the poor flotation response in oxidized PGM ores to several factors such as high contents of naturally floating gangue such as talc, chlorite and aluminosilicates. A lack of base metal sulphide association with PGMs and the Pd remobilization which reduces the flotation efficiency by changing Pt:Pd ratio also contributes to poor flotation recoveries. (Becker, et al., 2014; Bulatovic, 2003; Kraemer, et al., 2017).

This study seeks to improve the rougher flotation recovery of non-sulphide PGM ores by testing a variety of flotation reagents and combinations of reagents that have not been tested before by any other workers in order to find a balance between an acceptable grade and recovery of the rougher concentrate and then optimizing the reagent dosages.

The research questions that will be addressed in this investigation are as follows:

- i. What is the mineralogical composition of the non-sulphide PGM ore?
- ii. What are the main and interactive effects of the selected flotation reagent suites on the flotation recovery and grade of PGM concentrates?
- iii. What are the optimum conditions when floating non-sulphide PGM ores?

### **1.3. Research objectives**

The aim of this research is to contribute towards development of a process that can improve the recovery of PGM from an oxidised PGM ore by testing different reagents suites. The specific objectives are:

- To perform a mineralogical and chemical analysis on the non-sulphide/oxidised ore to better understand the nature of this complex PGM non-sulphide ore.
- To perform rougher flotation experiments to investigate the effect of the selected flotation reagents on the recovery and grade of the PGMs in terms of their dosages using design of experiments (DOE) approach.
- To determine the optimum flotation conditions for the upgrading of this ore type.

### **1.4. Dissertation Layout**

The dissertation is divided into five chapters. Chapter 1 gives a background of the problem and outlines the objectives of this research. Chapter 2 reviews the main principles of the froth flotation process and discusses previous work done on the processing of non-sulphide or complex PGM ores using froth flotation and hydrometallurgical processing routes. Chapter 3 describes the methods used to prepare the samples, the analytical techniques used to characterize the ore material and the experimental design used to carry out the experiments. Chapter 4 presents the results and discussion of the effect of the three reagent suites tested on the rougher flotation of a non-sulphide PGM ore, this includes the statistical analysis, optimization studies and the kinetic studies. The conclusions and recommendations based on this research are presented in Chapter 5.

## CHAPTER 2 : LITERATURE REVIEW

The main purpose of this literature review is to discuss the main principles of the froth flotation process and discuss previous work done on the processing of non-sulphide or complex PGM ores using froth flotation and hydrometallurgical processing routes.

### 2.1. Principles of froth flotation

Froth flotation is a separation process that relies on the difference in surface properties of the valuable minerals and gangue minerals, with the former expected to be hydrophobic and the latter hydrophilic. This process is used to treat complex or low-grade ores where the average particle size for liberation is too small for efficient gravity separation or where the density difference between minerals is very small (Gupta & Yan, 2006). The feed material to the flotation stage is a slurry prepared by grinding the mined ore in the presence of water in order to liberate the valuable minerals by reducing the ore to an appropriate size distribution.

The difference in surface properties of the valuable and gangue minerals is achieved by the addition of various flotation reagents in a conditioning tank or laboratory cell. This is accompanied by sparging in air and agitating the slurry to promote formation of air bubbles and collision of particles with the bubbles as illustrated in Figure 2-1.

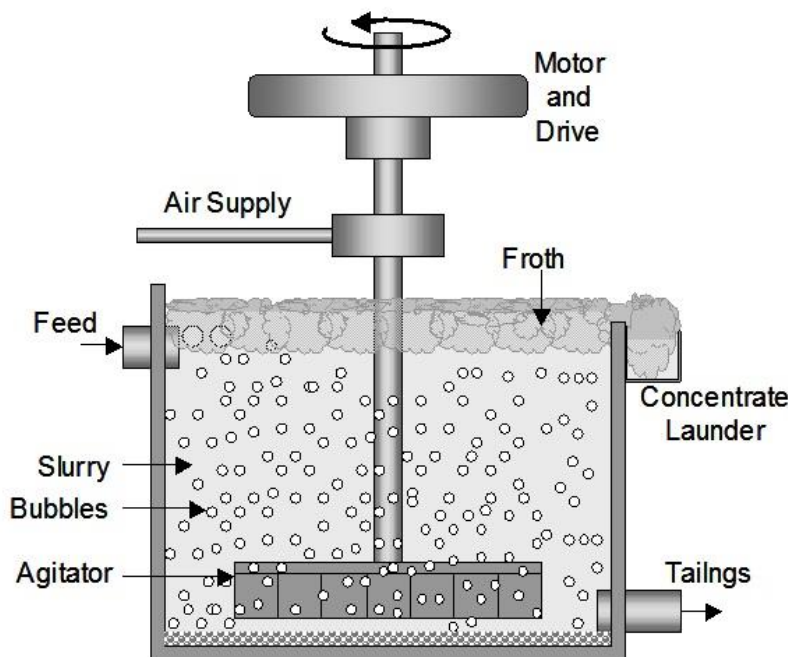


Figure 2-1: Schematic representation of froth flotation

The desired minerals attach to the bubbles and rises to the froth phase where they are recovered whilst the gangue material remain in the pulp. The flotation mechanism to recover minerals from ore pulp can be explained as follows:

- a) Selective attachment of minerals to air bubbles (true flotation)
- b) Entrainment of particles in the water which passes through the froth
- c) Physical entrapment of particles between particles in the froth attached to air bubbles

### 2.1.1. True flotation

True flotation occurs when particles collide with the surface of rising air bubbles in the aerated, agitated pulp and the mineralized bubbles are lifted to the froth phase where they are scraped off and recovered. True flotation is selective as only hydrophobic particles are recovered in the froth phase. The flotation phenomenon can be explained by a three-phase system (i.e. water-solid-air). Equilibrium in the system is reached when the solid-air, solid-liquid and liquid-air interfacial forces are in contact, as shown in Figure 2-2.

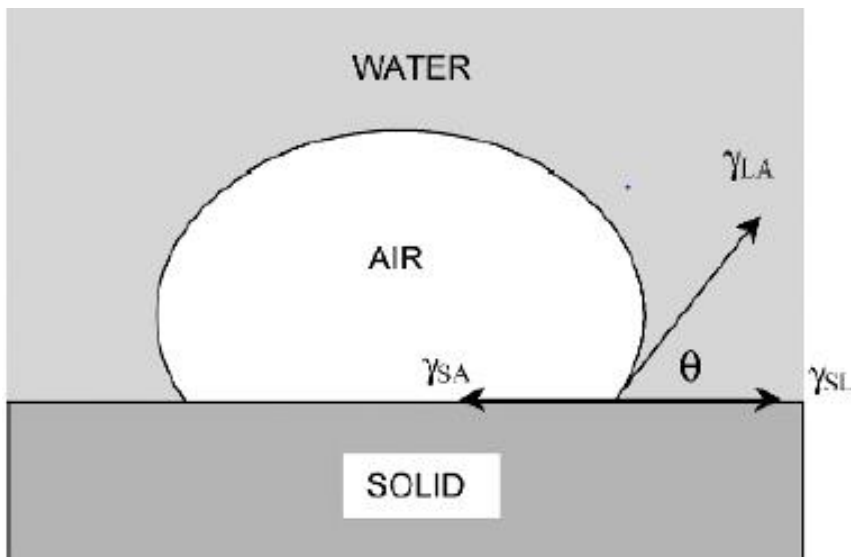


Figure 2-2: Three phase contact between solid, liquid and air (Gupta & Yan, 2006)

At equilibrium, the balance of the solid, liquid and air tensile forces is given by Young's equation:

$$\gamma_{SA} = \gamma_{SL} + \gamma_{LA}\cos\theta \quad (2.1)$$

where  $\gamma_{SA}$ ,  $\gamma_{SL}$  and  $\gamma_{LA}$  are the surface energies between solid and air, solid and liquid and liquid and air respectively, and  $\theta$  is the contact angle between the three-phase contact and the solid surface.

A small contact angle indicates a hydrophilic surface while a greater angle represents a hydrophobic surface which will result in the surface coming into contact with air instead of water due to a lower free energy and hence will readily *stick* to an air interface if one is available.

In a three-phase contact as shown in Figure 2-2, when the air bubble detaches from the solid surface then the solid/air interface is replaced by a solid/water and an air/water interface. The change in free energy due to this replacement of a unit area of the solid/air interface by a solid/liquid interface is given as:

$$\Delta G = \gamma_{SL} + \gamma_{LA} - \gamma_{SA} \quad (2.2)$$

Combining with Eq. (2.1) gives

$$\Delta G = \gamma_{LA}(1 - \cos\theta)$$

Where  $\Delta G = \text{the free energy}$

There is a direct proportionality between the contact angle and the work of adhesion between the particle and the bubble, resulting in the system being resilient to disruptive forces

### 2.1.2. Entrainment

Entrainment occurs when both hydrophobic and hydrophilic particles suspended in the water occupying the spaces between bubbles are carried upwards in to the froth phase and leave with particles recovered by true flotation. The quality of the flotation product is diluted by the presence of the entrained hydrophilic (gangue) particles. It has been observed that significant ultrafine hydrophilic mineral particles are recovered into the flotation product by entrainment (Wang, et al., 2015; George, et al., 2004; Warren, 1985). Different researchers have found that some of the most influential factors that affect entrainment of particle minerals during flotation are as follows:

#### 1. Water recovery

It has been previously shown that there is a direct relationship between the water recovered to the concentrate and the solids recovered by entrainment, for both ultrafine and coarse particles. An increase in the water recovery leads to more solids recovered by entrainment, resulting in a reduced concentrate grade as more gangue is recovered, as shown in Figure 2-3.

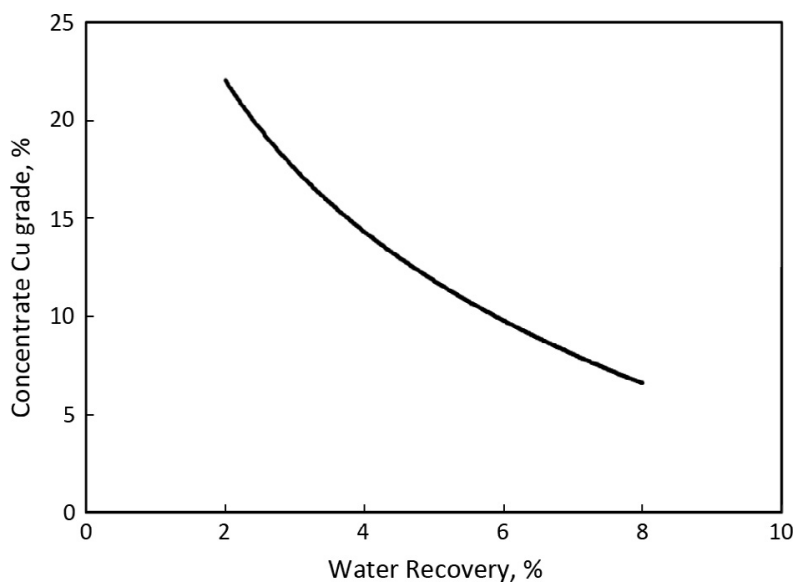


Figure 2-3: Relationship between water recovery and concentrate grade (Yianatos & Contreras, 2010)

## 2. Particle size

Fine particles have been found to be more entrained compared to coarse particles. This is explained by the fact that fine particles are easily suspended in the water whereas coarse particles drain more easily. Figure 2-4 shows that fine hydrophilic silica particles leads to more entrainment.

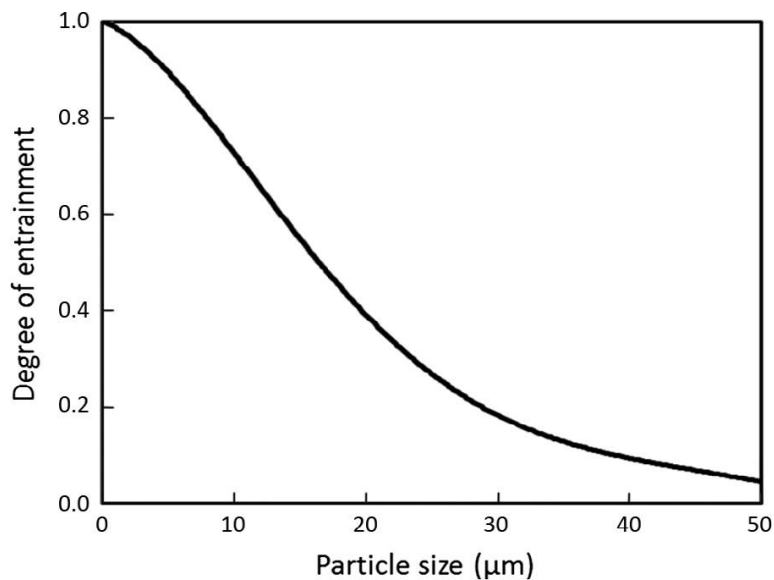


Figure 2-4: Effect of particles size on the degree of entrainment (Bisshop, 1974)

## 3. Froth height

The froth phase improves the selectivity by reducing the recovery of entrained gangue material to the concentrate stream, while selectively recovering and retaining the valuable mineral resulting in increased concentrate grade. The general consensus is that the recovery of mineral particles is lower when the froth height is deeper, this is because the froth residence time is increased which leads to the effective drainage of hydrophilic particles and some hydrophobic particles.

## 2.2. Factors influencing flotation

The flotation process is controlled by several inter-related factors as indicated in Figure 2-5. Relationship of these factors need to be considered in froth flotation operations as they have a synergetic effect (Klimpel, 1995) on the flotation performance. This thesis will largely focus on the optimisation of reagent suites hence only the chemical components will be discussed in this review.

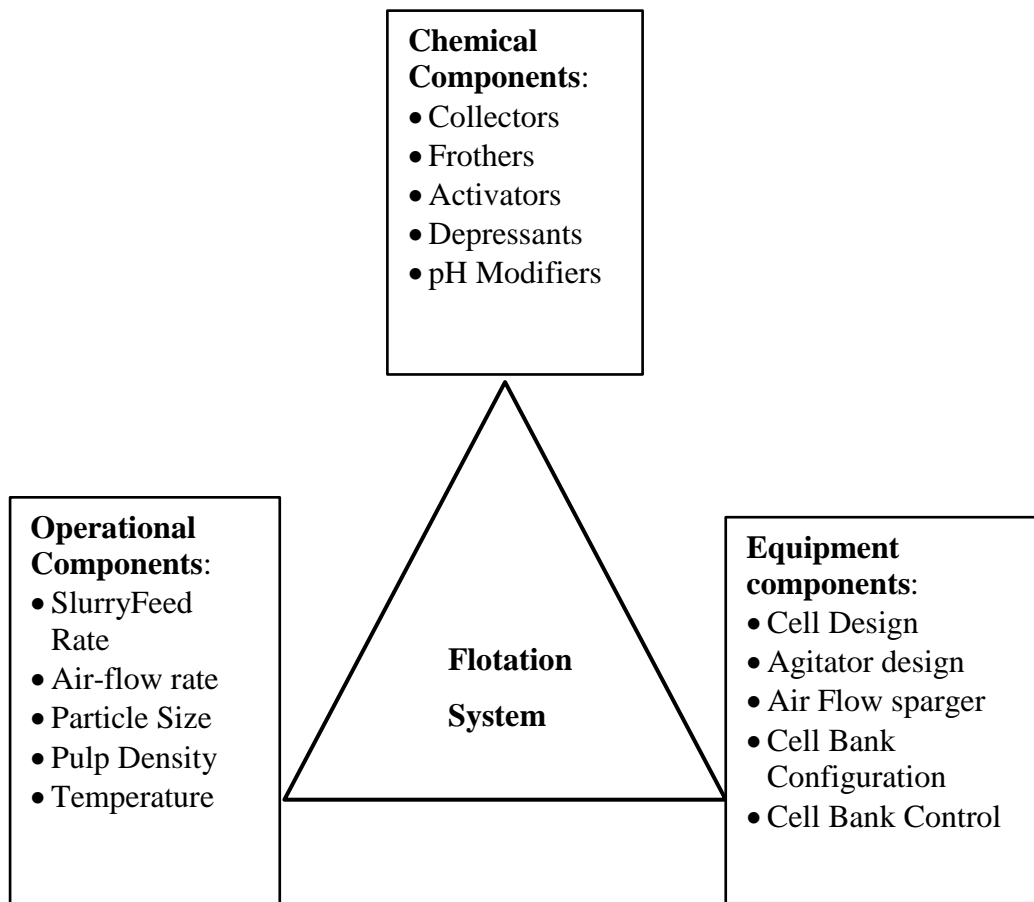


Figure 2-5: Schematic diagram of factors influencing flotation. Adapted (Klimpel, 1995)



### 2.2.1. Collectors

Collectors are organic chemical compounds used to make the mineral surfaces of interest hydrophobic, this happens by adsorption of molecules and ions onto the mineral surface. Collectors are generally heteropolar organics with a charged polar group and an uncharged non-polar group as shown in Figure 2-6. Collectors adsorb on mineral surfaces with their polar group ends and the non-polar group part is oriented towards the water phase rendering the mineral surface water-repellent. The non-polar group is typically a hydrocarbon chain which does not react with water and the polar group can react with water. The chain length and structure of a collector affects the solubility and adsorption of a collector. In the processing of sulphide minerals, collectors with shorter chain length of two to five carbon atoms are favoured as they are more soluble in water than longer chained collectors, and branched chains have higher solubility than straight chains (Wills & Napier-Munn, 2006).

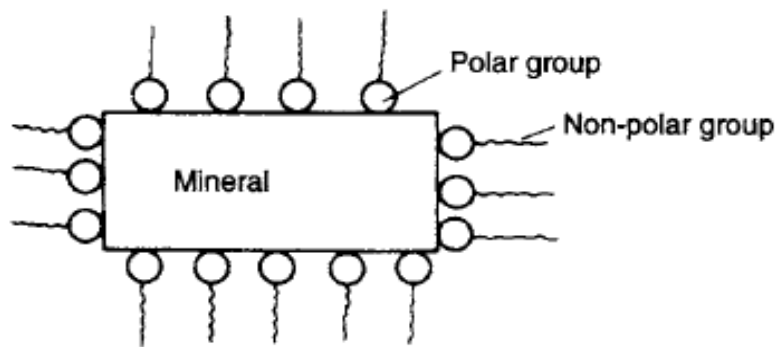


Figure 2-6: Collector adsorption on mineral surface ( (Wills & Napier-Munn, 2006)

The collectors are divided into two groups i.e. ionizing compounds and non-ionizing compounds. The ionizing collectors dissociate into ions in water and the non-ionizing compounds are known to be insoluble in water and make the mineral water-repellent by covering its surface with a thin film. The ionizing collectors are further categorized as anionic and cationic collectors as shown in Figure 2-7.

The anionic collectors have a negatively charged anion part that attaches to the positively charged mineral surface. The anionic collectors are subdivided into oxyhydril collectors and sulphydryl collectors. Oxyhydril collectors are based on organic and sulfo-acid anions as their polar groups and sulphydryl collectors have bivalent sulphur as their polar group.

The sulphhydryl collectors such as xanthates and dithiophosphates are used for the flotation of sulphide minerals due to their high selectivity for sulphide surfaces as they chemically bond to the surface. Xanthates such as amyl are also used for the flotation of oxidized minerals such as malachite, cerussite and anglesite, however higher concentrations are required (Bulatovic, 2007; Wills & Napier-Munn, 2006).

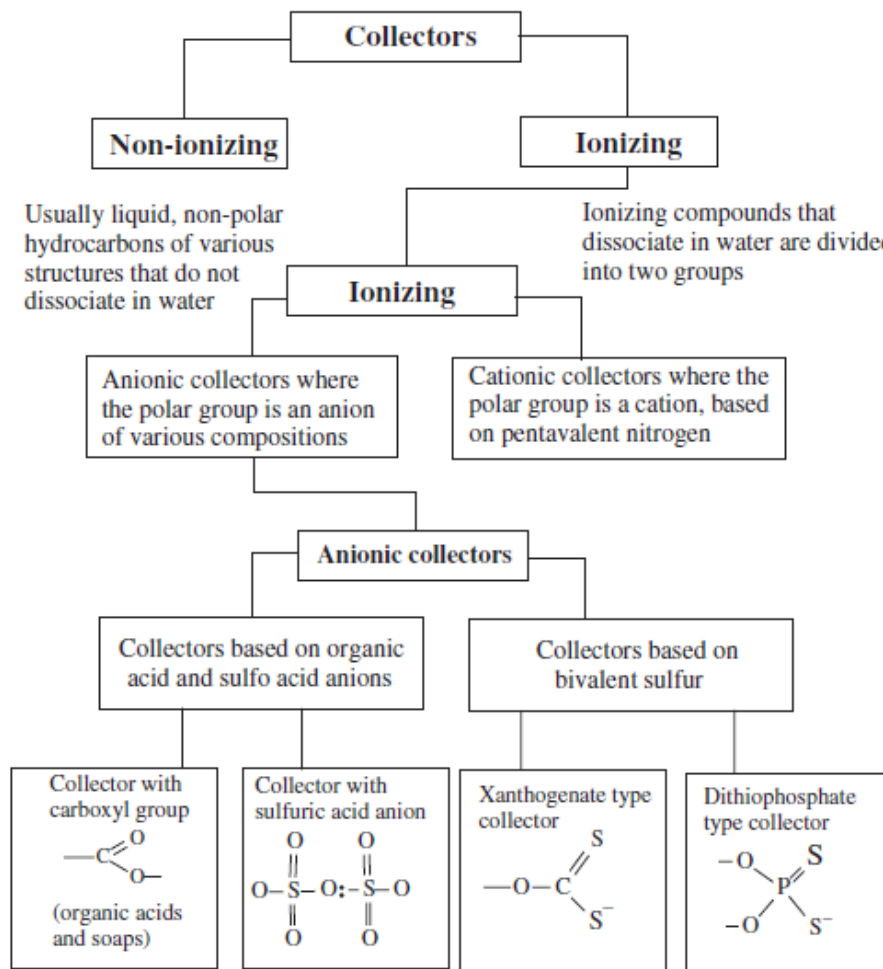


Figure 2-7: Classification of collectors (Bulatovic, 2007)

The traditional collectors used in PGM flotation are xanthates; normally SIBX which is often mixed with secondary-collectors such as dithiophosphate (DTP) or trithiocarbonates (TTC) as they have been shown to improve collector effectiveness and sometimes reduce collector consumption (Wiese, et al., 2005; Vos, et al., 2007). A pairing of SIBX with Flomin C5460 (blend of sodium isobutyl dithiophosphate and IPETC) and Flomin C5460 (blend of xanthogen formate and IPETC) respectively were investigated in this study.

IPETC is a dialkyl thionocarbamate, comprising of a branched isopropyl group and a short-chained ethyl hydrocarbon group, and is known to have frothing properties (Maree, et al., 2017). The structure of IPETC is shown in Figure 2-8. IPETC was initially used for sulphide mineral flotation and earlier studies have showed that it has higher selectivity against pyrite compared to xanthates and dithiophosphates (Buckley, et al., 2004).

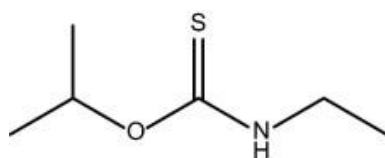


Figure 2-8: Structure of O-isopropyl-N-ethyl thionocarbamate

Xanthogen formates have historically been known to be strong collectors of sulphide minerals, attaching to base metal sulphides by chelation (Ackerman, et al., 2000). Xanthogen formates are made by reacting a xanthate with an alkyl chloroformate.

Oxyhydril collectors are mainly used for the flotation of oxidic minerals like silicates, carbonates and oxides. These collectors are less selective than sulphhydril collectors as they attach to the surface by electrostatic attraction hence they can also adsorb to gangue minerals. Examples of oxyhydrils include the hydroxamate collector used in this study.

Hydroxamates as secondary collectors have been tested on a range of oxide ores and it has been found that AM28 alkyl hydroxamate in conjunction with xanthate can successfully recover oxide minerals in mixed copper sulphide/oxide blends (Lee, et al., 2009). Becker, et al (2014) found that AM28 alkyl hydroxamate slightly improves the recovery of PGE from oxidised PGM ores, though it is significantly sensitive to flotation residence time and dosages (Ramonotsi, 2011).

Hydroxamates are chelating reagents also classified as oxyhydril collectors and are used as oxide collectors because of their affinity to selectively complex with metal oxides, however they are known to have very slow adsorption kinetics which mainly depends on mineral solubility. Hydroxamate-type collectors are synthesised from alkyl hydroxamic acid. The three basic structures from which hydroxamates are synthesized are shown in Figure 2-9

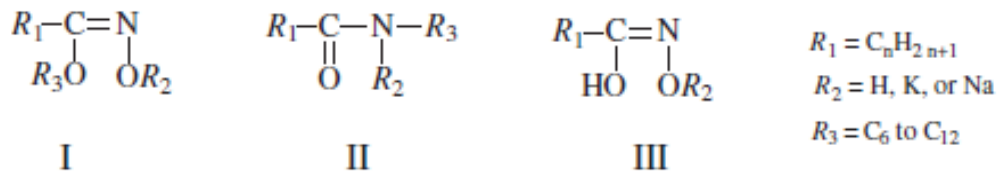


Figure 2-9: Basic structures of alkyl hydroxamic acids

where  $R_1$  is usually an organic ligand,  $R_2$  and  $R_3$  may be organic or inorganic. The structure shown in III is the most used in the synthesis of hydroxamates. The typical hydroxamate structure is shown below:

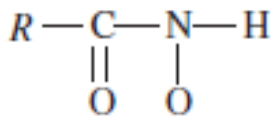


Figure 2-10: Typical hydroxamate structure

Improved hydroxamates selection is observed when the target mineral is the most soluble and the chelate formed between the cation in the lattice and hydroxamate is the most stable (Assis, et al., 1996). It has also been found that when the carbon chain of the hydroxamate is higher than  $C_9$ , then flotation performance is reduced. A mixture of hydroxamates with a carbon-chain length between  $C_7$  and  $C_9$  are the most successful (Bulatovic, 2007). In this study, AM810 alkyl hydroxamate was used as a secondary collector. According to the supplier Axis House Pty (Ltd) AM810 is an improved formulation of the previously promising AM28, it is a  $C_8$ - $C_{10}$  alkyl hydroxamate used to collect heavily oxidized or tarnished minerals. AM810 alkyl hydroxamate has frothing characteristics which require that frother dosage is controlled carefully.

The cationic collectors are organic compounds where the cation renders the mineral surface hydrophobic. These collectors perform best in slightly acid solutions. They are used for the flotation of oxides, carbonates, silicates and alkali earth metals. Both anionic and cationic collectors adsorb on oxide and silicates minerals electrostatically, with anionic collectors adsorbed by chemisorption on some oxides resulting in the formation of hydroxyl complexes of the metal ions comprising the mineral. Cationic collectors adsorb effectively on oxide minerals when the potential of the mineral surface is negative and anionic collectors when the mineral surface is positive. Insoluble oxides and silicates require enough conditioning time to allow for surface conditions to stabilize before and after collector addition (Kelly & Spottiswood, 1982).

The mixing of different collectors has been observed to have beneficial effects on the flotation performance due to their synergistic effect. In a study by Glembotskii (1958), it was found that when a weak collector is used in conjunction with a strong collector, there was a 2-5% recovery increase compared to when only a strong collector is used. Lotter & Bradshaw, (2010) summarized the benefits of mixed collectors as follows:

- i. Improved flotation rate
- ii. A low dosage requirement
- iii. Better recovery of coarse particles
- iv. An optimum mixture ratio results in better flotation performance

This study will explore the effect of using collector mixtures on the flotation of a non-sulphide PGM ore. It is envisaged that the different adsorption mechanisms of the different collectors will improve the PGM recovery and grade.

### **2.2.2. Frothers**

Frothers are water-soluble organic reagents that absorb at the air-water interface. They are heteropolar molecules with a polar group to provide water solubility, and a non-polar hydrocarbon group. Frothers are used to stabilise bubble formation in the pulp phase so that hydrophobic minerals can be captured and attached to the bubbles. It is important that the froth is stable enough to avert froth breakage and to ensure valuable particles do not return to the pulp before the froth is removed.

A frother is not supposed to adsorb on mineral particles as this will mean its behaving as a collector. This behaviour would reduce the selectivity of the collector (Rao, 2004; Kelly & Spottiswood, 1982).

### **2.2.3. Depressants**

Depressants alter the mineral surface to prevent or hinder the effect of the collector so as to increase the selectivity of flotation. The typical depressants used in PGM flotation to decrease the flotation of naturally floating gangue (NFG) are carboxymethyl cellulose (CMC) and modified guar gum (Wiese, et al., 2005). According to Becker, et al ( 2014), the Pilanesburg Platinum Mine (PPM) oxidised ores showed significant amounts of NFG which required high dosages of CMC i.e. 750g/t to control the NFG and prevent it from diluting the concentrate. This shows that a depressant is very important in improving the flotation performance hence its effect was also considered in this study. The depressant M98B supplied by Chemquest Pty (Ltd) was used in this study.

### 2.3. Flotation kinetics modelling and parameter estimation

Assuming that flotation is a first -order rate process and that a mineral i consists of j subclasses.

$$C_i = \sum_j^n C_{ij} \quad (2.3)$$

Where  $C_i$  represents the concentration (mol/l) of the floatable mineral in the float cell and  $C_{ij}$  represents the fraction of that mineral in the jth class

For a first order batch process, equation (1) can be expressed as:

$$\frac{dC_{ij}}{dt} = -k_{ij}C_{ij} \quad (2.4)$$

Integrating both sides of equation (2)

$$C_{ij}(t) = C_{ij}(0)\exp(-k_{ij}t)$$

$$C_i(t) = \sum_j C_{ij}(0)\exp(-k_{ij}t)$$

$$C_i(t) = C_i(0) \sum_j P_{ij}(0)\exp(-k_{ij}t)$$

Where  $k_{ij}$  is the flotation rate constant for that fraction,

$P_{ij}(0)$  is the fraction of mineral i which occurs as subclass j in the feed.

If  $R_i(t)$  is the fraction of the mineral i recovered in the concentrate at time t then,

$1 - R_i(t)$  is the fraction of the mineral i in the tailings at time t,

At constant volume,  $1 - R_i(t) = \frac{C_i(t)}{C_i(0)}$  hence the flotation kinetics can be expressed as follows:

$$1 - R_i(t) = \frac{C_i(t)}{C_i(0)} = \sum_j P_{ij}(0)\exp(-k_{ij}t) \quad (2.5)$$

A number of first order batch flotation mathematical models that have been developed to describe the flotation kinetics are presented in Table 2.1.

Table 2.1: First-order batch flotation models

Model	Assumptions	Equation
Classical model	Monodisperse feed with an average rate constant	$R = R_{\infty}(1 - e^{-kt})$
Klimpel model	Rate constant distribution is uniform	$R = R_{\infty}(1 - \frac{1}{kt}(1 - e^{-kt}))$
Kelsall (Unmodified) Model	Two fractions i.e. fast-floating and slow-floating fractions both comprising of rate constants. Ultimate recovery is assumed to be 100%	$R = (1 - \varphi)(1 - e^{-k_f t})(1 - e^{-k_s t})$
Modified Kelsall Model	Includes the effect of ultimate recovery	$R = R_{\infty}(1 - \varphi)(1 - e^{-k_f t}) + \varphi(1 - e^{-k_s t})$
Three fraction model	Three fractions i.e. fast-floating, medium-floating and slow-floating fractions both comprising of rate constants. Ultimate recovery is assumed to be 100%	$R = R_{\infty}[\phi_f(1 - e^{-k_f t}) + \phi_m(1 - e^{-k_m t}) + \phi_s(1 - e^{-k_s t})]$

In this study, it is assumed that the minerals/metals have three subclasses, i.e. fast floating ( $P_f$ ), slow floating ( $P_s$ ) and non-floating components ( $P_n$ ). The flotation parameters are determined graphically using equation (2.5), hence equation (2.5) becomes:

$$1 - R_i(t) = P_f e^{-k_f t} + P_s e^{-k_s t} + P_n \quad (2.6)$$

Where  $k_f$  is the fast floating rate constant

$k_s$  is the slow floating rate constant

$P_f$  is the fast floating mass fraction

$P_s$  is the slow floating mass fraction



$P_n$  is the non-floating mass fraction and is the approximate value of  $1 - R_i(t)$  as time approaches infinity

Assuming that  $k_f \gg k_s$  and that at large  $t$  values  $P_f e^{-k_f t}$  is negligible, equation (4) becomes:

$$1 - R_i(t) = P_s e^{-k_s t} + P_n \quad (2.7)$$

Taking natural logarithms on both sides, equation (5) becomes:

$$\ln\{1 - R_i(t) - P_n\} = \ln P_s - k_s t \quad (2.8)$$

Equation (6) is in a straight-line form, where  $\ln P_s$  is the intercept and  $k_s$  is gradient. The values of  $\ln P_s$  and  $k_s$  determined from the straight-line can be substituted back into equation (4) to determine values of  $P_f$  and  $k_f$  after taking natural logarithms on equation (5), which becomes:

$$\ln\{1 - R_i(t) - P_s e^{-k_s t} - P_n\} = \ln P_f - k_f t \quad (2.9)$$

Plotting  $\ln\{1 - R_i(t) - P_s e^{-k_s t} - P_n\}$  versus time  $t$  gives a straight-line with intercept  $\ln P_f$  and gradient  $k_f$ .

## 2.4. Source of the oxidised/non-Sulphide PGM Ore under study – The Main Sulphide Zone (MSZ), Great Dyke, Zimbabwe

The Great Dyke is a  $2575.4 \pm 0.7$  million years old ultramafic layered intrusion that drifts over 550 km NNE with a maximum width of about 11 km, and cuts Archean granites and greenstone belts of the Zimbabwe Craton as shown in Figure 2-11. Stratigraphically, the rock sequence of the Great Dyke is divided into a lower ultramafic sequence and an upper ultramafic sequence with the former composed of dunites, harzburgites, olivine bronzitites, pyroxenites, and thin layers of chromitites and the latter made up of plagioclase-rich norites, gabbronorites, and olivine gabbros (Locmelis, et al., 2010; Oberthur, et al., 2013).

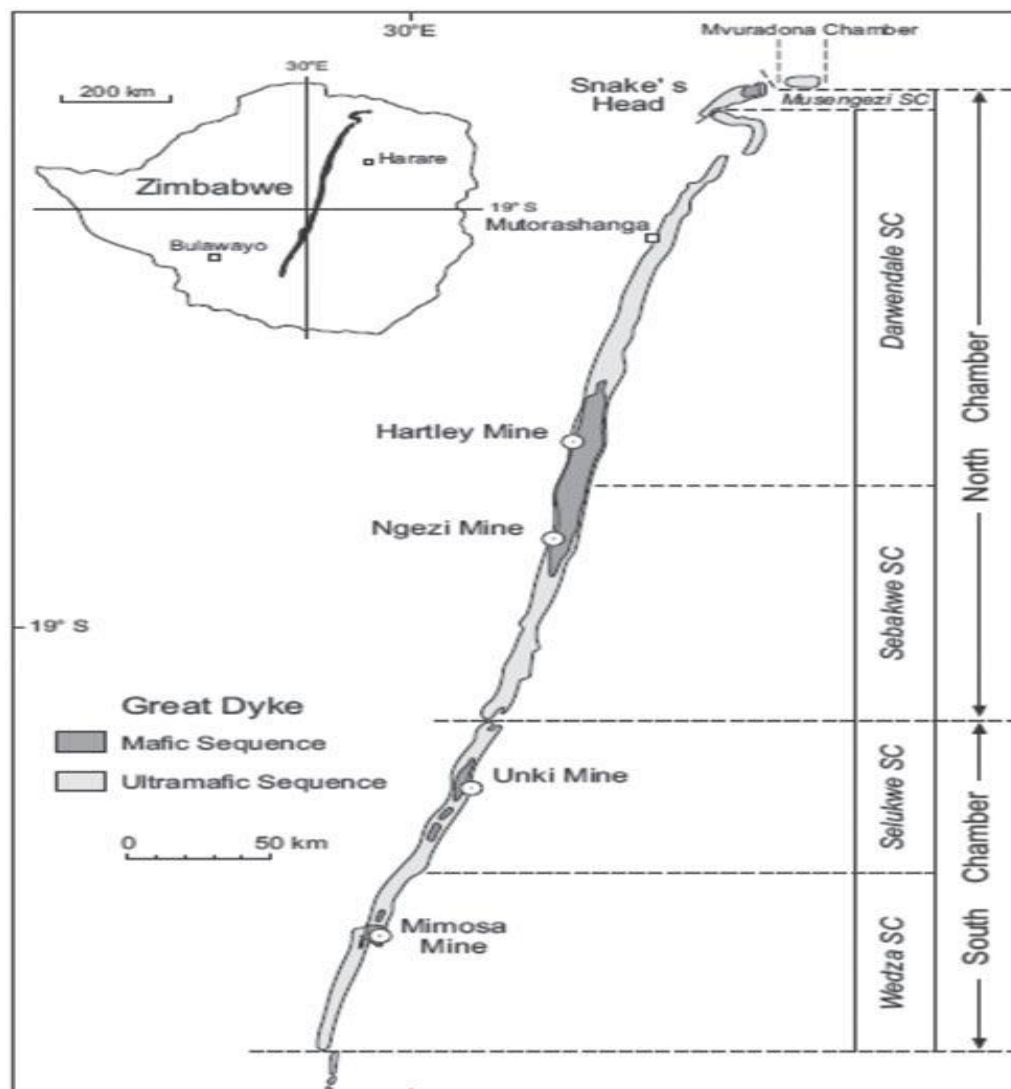


Figure 2-11: Geological map of the Great Dyke, after (Prendergast & Wilson, 1989)

The MSZ is subdivided into a lower PGE-rich subzone and an upper Cu- and Ni-rich subzone and comprises of bronzitites with average sulphide contents of about 2 vol.% (locally up to 7 vol.%). The most common sulphides are pyrrhotite, pentlandite, chalcopyrite, and pyrite that mainly form polyphase aggregates. The main PGM minerals in the MSZ are sperrylite [PtAs<sub>2</sub>], (Pt–Pd)-bismuthotellurides, cooperite, and braggite [(Pt,Pd,Ni)S], and Rh-Ir-Pt sulfarsenides. PGE, particularly Pd, can also be found in solid solution in the crystal lattices of the sulphides, mostly in pentlandite (Locmelis, et al., 2010).

#### **2.4.1. Mineralogy of the oxidised ores of the MSZ**

The mineralogy of pristine ores is transformed when they are exposed to weathering conditions i.e. atmospheric conditions and underground waters interact with primary minerals (pyroxene, feldspar, sulphides) transforming them to secondary minerals like Fe/Mn hydroxides, smectite and clay minerals (Evans, 2002; Evans & Spratt, 2000). This behaviour is consistent with the observations made on the weathering of BIC pristine ores (Junge, et al., 2015; Kraemer, et al., 2017).

It has been shown that PGMs in pristine ores are bimodally distributed into discrete PGM minerals and are associated with sulphide minerals such as pentlandite and pyrite. The investigations by Oberthur et al. (2013) on the oxidised PGM ores at the Hartley mine showed that PGMs are polymodally distributed into several phases as follows;

##### **i. Relict primary PGM**

The concentrates extracted from several discrete PGM grains showed that the carriers of PGM in primary PGM minerals are sperrylite (57.2%), cooperite/braggite (28.3%) and Pt-Fe alloy (3.1%). The proportion of sperrylite to cooperite/braggite is nearly identical to that of the pristine MSZ, showing that these minerals are relicts of the pristine MSZ ores. Sperrylite mostly displays idiomorphic crystal shapes with grain sizes up to about 100 µm whereas cooperite/braggite grains are present as splinters of irregular shape or elongated grains with clean surfaces and grain sizes between 1 µm and 100 µm (Oberthur, et al., 2013).

## **ii. Relict primary sulphides**

The base metal sulphides in the oxidized MSZ are primarily pentlandite, pyrrhotite, and chalcopyrite in about equal amounts, whereas pyrite, galena, bornite, and arsenopyrite are found in minor to trace amounts. Pentlandite and pyrrhotite are the only sulphides that contain PGE concentrations above the detection limit of the electron microprobe (~40–50 ppm Pt and Pd) (Locmelis, et al., 2010).

## **iii. (Pt,Pd)-oxides and/or hydroxides**

It has been confirmed by several authors that Pt and Pd contents are found in secondary PGE oxides or hydroxides in the oxidised MSZ ores (Evans & Spratt, 2000; Oberthur, et al., 2013). The PGE oxides/hydroxides are formed because of oxidation and they replace the primary PGMs. The PGE oxides/hydroxides are unnoticeable in reflected light as they are dark grey or brownish in colour and have low reflectivity, often comparable to Fe or Mn oxide/hydroxide phases whilst some have internal textures such as concentric rings, alternating bands, and botryoidal textures (Oberthur, et al., 2013).

## **iv. PGE bearing secondary phases**

The secondary phases that host PGE are iron oxides/hydroxides (up to 3,600 ppm Pt and 3,100 ppm Pd), manganese oxides/hydroxides (up to 1.6% Pt and 1,150 ppm Pd), and secondary phyllosilicates (up to a few 100 ppm Pt and Pd). The amounts of the various PGE-bearing phases differ significantly from mine to mine and between samples. Locmelis et al, (2010) asserts that this could be due to both the variation in primary ore mineralogy and depth within the weathering profile.

Locmelis, et al. (2010) reported that there is a loss of palladium during the weathering process due to its mobility resulting in a higher Pt/Pd ratio in oxidised ores as indicated in Figure 2-12.

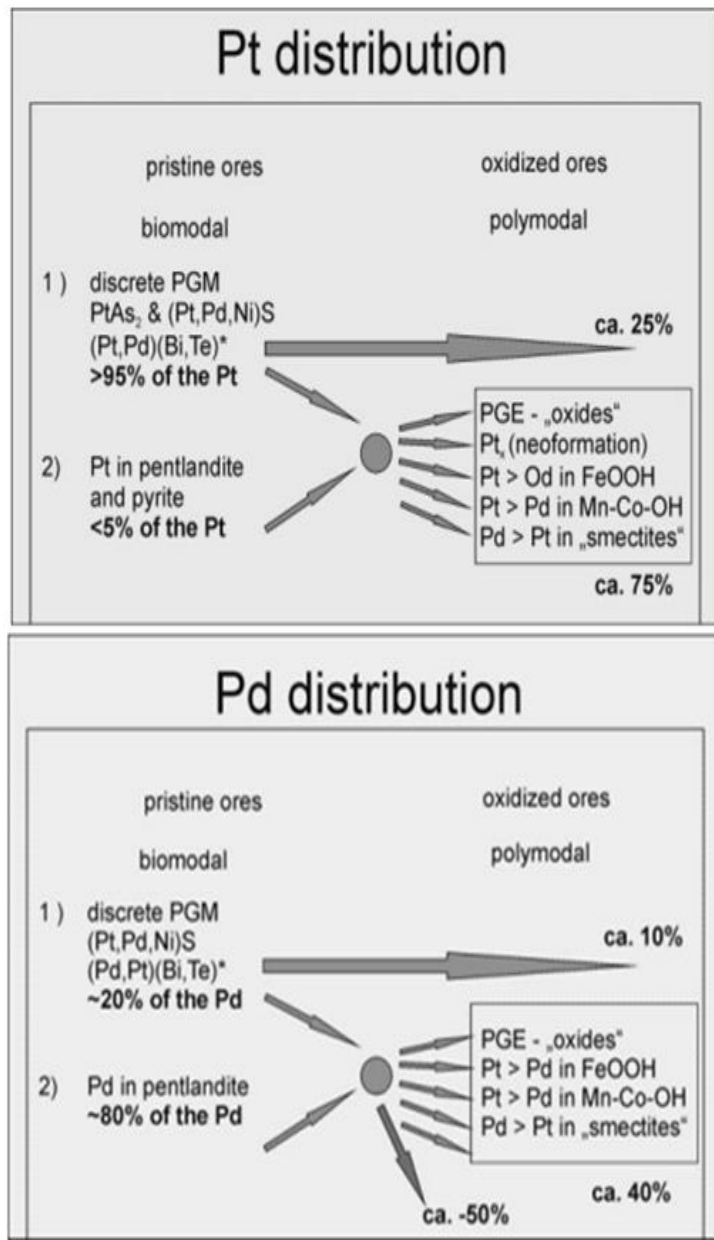


Figure 2-12: Phase changes and distributions of Pt and Pd from pristine ore to oxidized ore (Adapted from (Locmelis, et al., 2010))

## **2.5. Processing of oxidized/non-sulphide/complex PGM ores**

The depletion of pristine PGM ores and rising mining costs as the mines become deeper has triggered interest in the processing of weathered non-sulphide PGM ores found near the surface or mined from open pit mines. Attempts to recover these PGM ores using conventional means have proved futile as achieved recoveries have been consistently lower than 50% in the rougher flotation stage as reported in the work conducted at the Old Wedza mine (1926-1928) and Hartley mine (1997-1999) (Oberthur, et al., 2013 ; Vermaak, 2005).

Oberthur et al (2013) suggests that only bulk leaching methods could be viable for the treatment of the oxidised ores due to their complexity, while Kraemaer et al (2017) proposes in situ leaching or atmospheric leaching at ambient or slightly elevated temperatures as probably the most economically promising options for treating oxidized ores in the event that adaptations in flotation process continues to be unsuccessful.

There are considerable published and unpublished studies done to improve the recovery of oxidised PGM ores by several workers using both the flotation and leaching methods (Becker, et al., 2014; Evans & Spratt, 2000; Kraemar, et al., 2015).

### **2.5.1 Strategies to improve flotation of complex/non-sulphide PGM ores**

Numerous strategies have been proposed to improve the flotation of oxidised/non-sulphide PGM ores. Some of these strategies include; ore pre-treatment using leaching, sulphidisation, ultrasonic pre-treatment and the use of different improved reagent schemes.

It has been postulated that oxidised sulphide minerals are surrounded by an oxide/hydroxide layer which inhibits the easy recovery of PGMs by flotation using normal sulphide collectors. What has also been considered to hinder the floatability of oxidized PGM ores is the varying degrees of oxidation and mineralogy within the same mine ore and the lack of the usual association of PGM minerals with base metal sulphides (Kraemer, et al., 2017; Becker, et al., 2014). This suggests that beneficiation solutions will be more specific than general owing to the different ore deposits and mineralogy of individual deposits.

Ramonotsi (2011) attempted to remove the perceived oxidation layer by pre-treating the oxidised PGM ores of the Pilanesburg Platinum Mine. The pre-treatment stage comprised of leaching the oxidised PGM ores with sulphuric acid in an attempt to remove the oxidation

layers and expose the sulphide surface. The results from this study showed that after the pre-treatment by acid leaching and followed by flotation, flotation recoveries increased by over 20%. However, the drawback with this strategy is that a neutralizing stage and corrosion resistant new infrastructure will be required due to the corrosive nature of the acid pre-treatment, meaning new investment will be needed (Ramonotsi, 2011).

A study by Mphela (2010) on the influence of cleaning oxidised pyrrhotite with gaseous carbon dioxide using electrochemical and micro-flotation measurements showed an improved pyrrhotite flotation response. In this approach carbon dioxide (CO<sub>2</sub>) gas is absorbed in water on the mineral surfaces to form carbonic acid which ionizes to digest hydroxyl groups followed by the deprotonation of the absorbed bicarbonate, the reaction scheme is as follows



In this study, 30 minutes of carbon dioxide pre-treatment improved the PGMs recovery from 40.6% to 50.4 %. The electrochemical measurements showed that the oxidised surfaces can be depassivated with CO<sub>2</sub> treatment and ToF-SIMS measurements demonstrated a reduction in the oxide layer thickness after CO<sub>2</sub> treatment (Mphela, 2010).

The sulphidisation process has also been proposed, this is an aqueous based method, which converts non- sulphide surface mineral to sulphide surface mineral by use of soluble sulphide salts such as sodium hydrosulphide and sodium sulphide. Sodium sulphide hydrolyses and dissociates in the pulp releasing hydroxide ions (OH<sup>-</sup>), hydrosulfide ions (HS<sup>-</sup>) and sulphide ions S<sup>2-</sup> into solution summarized by reactions 5 to 8.



The sulfidisation mechanism involves adsorption of produced hydrosulfide ions onto the oxidised mineral surface followed by formation of a sulfide surface through anionic exchange (Castro, et al., 1974). This process has been used extensively in the flotation of base metal oxidised ores such as copper and lead oxides (John, et al., 1991; Newell & Bradshaw, 2007). Newell et al. (2007) successfully restored the floatability of thermally and chemically oxidised sulphide minerals of the Nkomati massive sulphide ore and pentlandite by using the control potential sulphidisation (CPS) method. The sulphidisation of oxidised pentlandite was initially challenging in these studies as oxidised pentlandite could not be readily sulphidized. This difficulty was overcome by the addition of base-metal ions (copper and iron) during the sulphidisation to form copper and iron sulphides on the oxidised pentlandite surfaces. It was found that copper ions were more significant in the sulphidisation or activation of oxidised pentlandite than iron. In industrial application, the main drawback of sulphidisation is that the optimum conditions are highly dependent on reagent conditioning time, mixing and other properties which lead to poor reproducibility in a plant situations (Lee, et al., 2009).

Previous attempts in pre-treating the oxidized ore from the MSZ at the Great Dyke in Zimbabwe by gravity concentration followed by froth flotation also yielded recoveries less than 50% (Prendergast, 1990).

The correct selection of collectors remains a huge challenge in the flotation of oxidised PGM ores. However, alkyl hydroxamates have previously shown good potential of recovering floatable oxide copper minerals (Lee, et al., 1998). The application of N-octyl hydroxamate collector AM28 in successfully recovering the oxide minerals malachite and azurite from a copper sulphide/oxide ore blend without affecting the sulphide recovery validated the prowess of hydroxamates to recover oxide minerals. Taking this into account, Becker et al. (2014) evaluated alkyl hydroxamate (AM28) as a co-collector to treat oxidised PGM ores of the Pilanesburg Platinum Mine (PPM) with xanthate as the primary collector. The PPM ore was characterised by high contents of alteration minerals, Fe oxide/hydroxides and very small amounts of base metal sulphides. It was found that the use of hydroxamate AM 28 as a co-collector with CPS and without CPS resulted in an improved PGE (Pt, Pd, Rh and Au) recovery of about 11%, the recovery improved to 39% from 27% at a depressant (CMC) dosage of 750 g/t. However, after careful analysis it was observed that this improvement in the PGE recoveries was mainly due to the increased froth stability and water recovery than the selective recovery of PGMs (Becker, et al., 2014).



In the work conducted by Bulatovic (2003) in evaluating different reagent suites on various PGM ores, it was found that the floatability of PGM ores mainly depends on the mineralogy of the ore. It was found that the floatability of a high chromium oxidised PGM ore from the Panton Sill deposit in Australia can be improved by using a modified xanthate (PM230) with a phosphate ester in conjunction with dithiophosphate. The reagent suite achieved recoveries of 79.3% Pt and 79.2% Pd respectively compared to 68% Pt and 67% Pd recoveries when conventional xanthate and dithiophosphate were used. The study also revealed that careful selection of depressant type and dosing is important in preventing depression of sulphides (Bulatovic, 2003).

### **2.5.2. Hydrometallurgical processing routes – Leaching of complex/non-sulphide PGM ores**

Oberthur et al (2013) suggested that only bulk leaching methods could be viable for the treatment of oxidised PGM ores due to their complexity, while Kraemaer et al (2017) proposes in situ leaching or atmospheric leaching at ambient or slightly elevated temperatures as probably the most economically promising options for beneficiating the oxidized PGM ores. The following section summarises the work done in processing non-sulphide/complex PGM ores using leaching methods.

According to a study by Becker and Wotruba (2008), bulk leaching of oxidised PGM ore from the Hartley Mine in Zimbabwe using hydrochloric acid at room temperature and pH 3 for 72 hours yielded maximum recoveries of 18.9% Pt and 18.5 Pd. Bulk leaching with sulphuric acid also did not have much success as only 11.1% Pt and 13.8% Pd were recovered.

However, leaching experiments conducted on a magnetite ore containing platinum, palladium and gold using concentrated chloride solutions and ozone gas injection as oxidizing agent resulted in >90% Pt, 70 % Pd and 50% Au extractions. These extractions were obtained after 3 hours of leaching under atmospheric pressure at ambient temperatures and a pH 4. The workers observed that the magnetite mineral matrix was not attacked during the leaching process (Torres & Lapidus, 2016). The mineralogy of the magnetite ore used in this study might be different from that of southern African oxidized PGM ores, however both minerals consist of oxide matrix hence this leaching method or an adaptation of it is worth exploring (Sefako, et al., 2017).

The recovery of PGMs from oxidized ores through leaching was also explored using a two-stage method consisting of a hydrochloric acid pre-treatment followed by siderophore leaching. The hydrochloric acid pre-treatment is used to liberate PGM minerals and reduce quantities of cations such as Fe which may compete with Pt and Pd in the subsequent stage. The experimental results were promising as up to 80% Pt extraction was achieved, however due to the possibility of excessive costs associated with this method and selectivity challenges in the leaching process the method might not be feasible on an industrial scale and as such heap or in-situ leaching using siderophore might be the option (Kraemar, et al., 2015).

Mwase et al. (2012) proposed a conceptual flowsheet for heap leaching of PGMs from a low-grade ore concentrate. The proposed process involved the bioleaching of low-grade flotation concentrate with a mixture of thermophiles and mesophiles in order to extract the base metals and then leach the residue with cyanide to extract Pt, Pd and Rh. The base metals were extracted first to reduce the potentially high consumption of the cyanide lixiviant as cyanide readily complexes with the base metals as well. The PGM flotation concentrates used in the experiments were oxidized in nature and occurring in the form of sulphides, telluride, selenide, arsenide and ferroalloy. From the bioleaching system, it was found that extractions of 52% Cu, 95% Ni and 85% Co could be achieved after 30 days, and extractions of 20% Pt, 87% Pd and 46% Ru were achieved after 21 days of cyanidation (Mwase, et al., 2012).

Subsequently, another study involving heap and tank leaching of PGM flotation concentrates using ammoniacal solutions was proposed as an alternative to recovering base metals before PGM recovery. In this study, a solution containing 4M  $\text{NH}_4\text{OH}$ , 4M  $(\text{NH}_4)_2\text{CO}_3$ , buffered in the pH range 9–10, was used as base case lixiviant in column and batch stirred tank reactor tests. It was found that at optimal conditions almost 100% of Cu and Ni respectively were successfully extracted in the batch stirred tank reactor and in the columns 95% Cu and 60% Ni were extracted after 42 days. It was also observed that both methods would require high concentrations of ammonia, with the ammonia lost in heap leaching due to evaporation losses but in tank leaching the ammonia could be recycled (Muzawazi & Petersen, 2015; Sefako, et al., 2017).

The earlier work by Bruckard, et al.(1992) at Coronation Hill showed that platinum, palladium and gold can be extracted by cyanidation at elevated temperatures. The ore treated in this case was highly oxidized with a sulphur assay as low as 0.1%. It was found that after amalgamation to extract gold, the concentrate could be leached with cyanide under alkaline conditions to extract 90% and 80% of Pd and Pt respectively at 100-125°C and pH 9.0-10.0 for 6 hours (Bruckard, et al., 1992). The success of this cyanidation is explained by the fact that the ore in this case had large amounts of metallic Pt and Pd which is not the case in the oxidized PGM ores in southern Africa (Evans, 2002).

BHP Minerals patented a process to treat oxidised ores involving roasting of the oxidized PGM ore followed by acidic bromine leaching of the oxidized PGM ore. The dissolution was attained with a sulphuric acid solution at a pH that enables formation of PGM complexes with bromine ions, with bromide acting as an oxidizing agent. In the experiments carried out

using the oxidized Hartley mine ore it was found that 85% platinum, 65% rhodium and 95% could be recovered (Duyvesteyn, et al., 1992; Duyvesteyn, et al., 1994).

## 2.6. Summary

The different research efforts discussed in this paper have been grouped into two broad groups of flotation methods and leaching methods and they are summarized in Table 2.2 and Table 2.3. From the flotation grouping, there is room for more research in improving the reagent suite selection in the flotation of oxidized/non-sulphide PGM ores as evidenced by the promising flotation performance of novel flotation reagents suites such as the modified xanthate (PM230) combined with the DTP as a co-collector and the use of hydroxamates. Based on this observation, this study will test a range of collector combinations in an effort to improve the recovery of PGMs.

Table 2.2: Challenges and opportunities in flotation of oxidized PGM ores (Sefako, et al., 2017)

Recovery method	Method and process conditions	Recovery (%)	Salient aspects
Ore-pretreatment and flotation	(i) Leach with 0.5M H <sub>2</sub> SO <sub>4</sub> for 20 minutes, Flotation with SIBX and Sendep depressant	70 % (up from 50%)	<ul style="list-style-type: none"> <li>Moderate recoveries</li> <li>High equipment cost - due to corrosiveness of H<sub>2</sub>SO<sub>4</sub></li> </ul>
	(ii) Continuous bubbling of CO <sub>2</sub> for 30-45 minutes. Flotation using SIBX, CuSO <sub>4</sub> , NaOH at pH 9	50.4% (up from 40.6%)	<ul style="list-style-type: none"> <li>Low recoveries</li> <li>Moderate equipment cost – less corrosive</li> </ul>
Sulfidisation and flotation	(i) Addition of base metal ions (Cu and Fe) at potential of - 650 mV for 10 minutes, decantation and flotation with SIBX, CuSO <sub>4</sub> , NaOH at pH 9. (ii) Sulfidisation as above followed by flotation with SIBX and hydroxamate at pH 9, at depressant (CMC) dosage of 750 g/t	80% (up from insignificant recovery)  Increased overall recovery of four elements (Pt, Pd, Rh and Au) from 27 to 39%	<ul style="list-style-type: none"> <li>High recoveries</li> <li>Poor reproducibility</li> <li>Low recoveries</li> <li>Improved recovery mostly due to increased frothiness rather than the hydroxamate selectivity</li> </ul>

Novel flotation reagent suites	Flotation with modified Xanthate (PM230) and dithiophosphate of a rich-chromium, highly oxidized ore	79.3% Pt and 79.2% Pd (up from approximately 50%)	<ul style="list-style-type: none"> <li>• High recoveries</li> <li>• Promising reagents</li> </ul>
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Table 2.3: Challenges and opportunities in the leaching of oxidized PGM ores (Sefako, et al., 2017)

Leaching method	Method and process conditions	Extraction (%)	Salient aspects
Ammonia pre-leaching + Cyanide leaching	(i) Ammonia leaching and Cyanidation for 40 days with 0.5M cyanide	40% and 73% total extraction Pt and Pd respectively	<ul style="list-style-type: none"> <li>• Low to moderate extraction</li> <li>• High reagent costs</li> </ul>
	(ii) Heap and tank ammonia leaching + PGM recovery leaching	Approximately 100% Cu and Ni extraction in tank leaching 95% Cu and 60% Ni extraction in heap leaching	<ul style="list-style-type: none"> <li>• Successful base metal recovery, development of PGM recovery process still required, possibly cyanidation</li> </ul>
	(ii) Gravity separation for Au and Cyanidation at 125 <sup>o</sup> C for 6 hours followed by CIP	80 % Pt and 90-95% palladium	<ul style="list-style-type: none"> <li>• High extraction</li> <li>• High reagent costs</li> </ul>
Acid leaching	(i) Leaching with HCl at 25 <sup>o</sup> C for 72hours at pH 3	18.9 % Pt and 18.5% Pd extraction	<ul style="list-style-type: none"> <li>• Low extractions</li> <li>• High reagent costs</li> <li>• High equipment cost due to corrosiveness of HCL</li> </ul>
	(ii) Leaching with H <sub>2</sub> SO <sub>4</sub> at 25 <sup>o</sup> C for 72hours at pH 3	11.1% Pt and 13.8% Pd	<ul style="list-style-type: none"> <li>• Low extractions</li> <li>• Low reagent costs</li> <li>• High equipment cost due to corrosiveness of H<sub>2</sub>SO<sub>4</sub></li> </ul>
	(iii) Leaching with 0.5M HCl for 24 hours, followed by Buffered siderophore (Desferrioxamine	Up to Pt 78% extraction	

	B) leaching for 70-120 hours at ambient temperatures		<ul style="list-style-type: none"> <li>• High extractions</li> <li>• High reagent costs</li> <li>• High equipment cost due to corrosiveness of HCL</li> <li>• Highly sensitive to ore type</li> </ul>
Bromide leaching	Roasting of the PGM ore then Bromine leaching	95% for gold, 85% for platinum and over 65% for rhodium	<ul style="list-style-type: none"> <li>• High extractions</li> <li>• High equipment and operating costs due to a roasting stage</li> </ul>

## **CHAPTER 3 : METHODOLOGY**

This chapter describes the methods used to prepare the samples, the analytical techniques used to characterize the ore material and experimental design used to carry out the experiments.

### **3.1. Ore sample preparation and characterization**

An oxidised PGM ore sample from the Mimosa Mine in the Great Dyke, Zimbabwe was used in this study. The crushed oxidised PGM ore sample was supplied at 70% passing 1 mm. The ore was then blended, riffled and split using a rotary splitter into representative samples of 1 kg. The crushed ore sub-samples were then ground in a ring mill with the aim of achieving a grind size of 60% passing 75 $\mu$ m, typically used in PGM concentrator operations.

The standard riffle and rotary splitter method was used to split approximately 300 g representative samples from the bulk. A ring mill was used to pulverize the samples to ~90% -75  $\mu$ m in preparation for head analyses. PGM analyses (in duplicate) were done by fire assaying, and lead collection with ICP-EOS Finish. X-ray diffraction (XRD) analysis was also carried out to establish the bulk mineralogical composition of the ore by identifying the major crystalline phases. The bulk mineralogical composition from XRD technique needed to be validated by elemental analysis because amorphous minerals were not detectable by XRD since it can only identify crystalline particles. Hence additional pulverized samples were analysed using X-Ray Fluorescence (XRF) and EDX techniques to establish the elemental composition of the ore.

### **3.2. Process water preparation**

All tests were conducted using synthetic water which was prepared by adding ionic salts to distilled water to simulate typical process water at PGM concentrator plants (Wiese, et al., 2005).

### **3.3. Flotation Reagents**

The flotation reagents investigated in this study were collector SIBX, co-collectors Flomin C7133, Flomin C5460 and AM810. The depressant M98B and the frother Flomin F200, a polyethylene glycol monobutyl ether mixture were supplied by Chemquest (Pty).

The co-collectors Flomin C7133 and Flomin C7133 were supplied by Chemquest (Pty) Ltd and were touted as potential collectors for non-sulphide PGM ores. The supplier described Flomin C7133 as a blend of xanthogen formate and IPETC, and Flomin C5460 as blend of sodium isobutyl dithiophosphate and IPETC. IPETC is a dialkyl thionocarbamate, comprising of a branched isopropyl group and a short-chained ethyl hydrocarbon group, and is known to have frothing properties (Maree, et al., 2017). The suppliers did not provide any recommendations with regards to reagent preparation or dosing, hence a factorial design was used to find the conditions.

According to the supplier Axis House Pty (Ltd), AM810 is an improved formulation of the previously promising AM28; it is a C8-C10 alkyl hydroxamate used to collect heavily oxidized or tarnished minerals. AM810 alkyl hydroxamate has frothing characteristics which require that frother dosage is controlled carefully.

The reagent SIBX was received in powder form at 90% strength. M98B and the activator  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  were also received in powder form and the secondary collectors were received as liquid. Stock solutions of SIBX and the co-collectors were prepared to 1% wt. by mixing 1 g of the powder with de-ionised water in a 100ml volumetric flask as it is standard in some PGM concentrator operations.



### 3.4. Flotation Experiments

The non-sulphide Mimosa mine PGM ore was received crushed at 70% passing 1 mm. The crushed samples were ground in a ring mill to achieve a grind size of 60% passing 75 $\mu$ m and packaged into representative 1160g sample bags. A Denver flotation machine with a 3.0 L cell was used in this work. The flotation experimental set-up that was used is shown in Figure 3-1.



Figure 3-1: Flotation experimental set-up

The ground samples and 2700ml prepared synthetic water were mixed in a flotation cell to form slurry with 30 % solids by weight. The machine was turned on and the slurry was agitated with a variable speed impeller at 1200 rpm to keep the solids homogeneously suspended in the slurry before addition of reagents in the flotation cell. The activator, copper sulphate, was dosed at 50g/t and conditioned for 5 minutes and the frother at 50g/t and conditioned for 1 minute. The rest of the reagents for each suite were added to the flotation cell and conditioned as shown in Table 3.1.

**Table 3.1: Flotation reagent suites and experimental conditions**

Reagent Suite	Reagents	Reagent dosages(g/t)		Conditioning time
		Low	High	
1	SIBX	100	150	2
	Flomin C5460	30	50	2
	Depressant M98B	50	100	3
2	SIBX	100	150	2
	Flomin C7133	30	50	2
	Depressant M98B	50	100	3
3	SIBX	100	150	2
	AM 810	30	50	2
	Depressant M98B	50	100	3

All flotation tests were carried out at the natural pH of the slurry which was measured at an average of 9. All tests were conducted using synthetic plant water prepared by adding ionic salts to deionized water to simulate typical process water in concentrator plants as described by Wiese et al. (2005). The rougher flotation tests were initiated after 13 minutes conditioning time, by opening the gas valve at a flowrate of 7ml/min. The concentrates were collected into trays by scraping off the froth after every 15 seconds using scraper blades for 20 minutes' flotation time.

### 3.5. Experimental design

Flotation experiments were carried out using a full factorial design of experiments. The aim of carrying out this statistical analysis was to identify and evaluate the significance of the main factors and their interactions, and develop a regression model. This was done by constructing a half-normal probability plot of the effect estimates. The non-significant factors are normally distributed and will lie on a straight line and significant factors will be further away from the straight line.

The Design-Expert® 11 software was used to plot the half-normal probability and develop a regression model. The factors investigated were the collector, secondary collector and depressant, and the response studied was the platinum and palladium recovery. The variables were studied at two levels, i.e. low level coded as (-) and high level as (+), hence eight experimental tests were conducted for each reagent suite as illustrated in Table 3.2.

**Table 3.2: Full factorial design matrix for flotation of a non-sulphide PGM ore**

Test no.	Level		
	A	B	C
1	-	-	-
2	+	-	-
3	-	+	-
4	+	+	-
5	-	-	+
6	+	-	+
7	-	+	+
8	+	+	+

For Reagent suite 1: A - SIBX, B - Flomin C5460, C – M98B

Reagent suite 2: A - SIBX, B - Flomin C7133, C – M98B

Reagent suite 3: A - SIBX, B – AM810, C – M98B

## **CHAPTER 4 : RESULTS AND DISCUSSION**

The results and discussion of the effect of the three reagent suites tested on the rougher flotation of a non-sulphide PGM ore are presented in this chapter. The three reagent suites tested are made up of (i) a collector, (ii) a co-collector and (iii) a depressant i.e. (Suite 1 - SIBX, FLOMIN C5460, M98B), (Suite 2 - SIBX, FLOMIN C7133, M98B) and (Suite 3 - SIBX, AM810, M98B).

The flotation performance was analysed by measuring solids, water, 3E (Pt, Pd, Au) recoveries and grades. Water recovery is calculated as a fraction of the total water in the flotation cell that is recovered to the concentrate. The platinum performance was presented independent of the 3E performance in order to show its significance as it was observed that platinum has a much higher content than that of palladium and gold in the ROM ore, with gold in very small quantities.

Section 4.1 show the ore preparation results, Section 4.2 shows the characterisation results, Section 4.3 show the effect of the 3 reagent suites on the non-sulphide PGM ore flotation performance, Section 4.4 show the statistical analysis performed on the platinum recovery, Section 4.5 shows the optimization results based on the initial flotation experiments and Section 4.6 shows the kinetics and flotation performance data of the best conditions obtained from the optimization results.

#### 4.1 Particle size distributions

A non-sulphide PGM ore sample from the Mimosa Mine in the Great Dyke, Zimbabwe was used in this study. The crushed oxidised PGM ore sample was supplied at 70% passing 1 mm as shown in the PSD in Figure 4-1

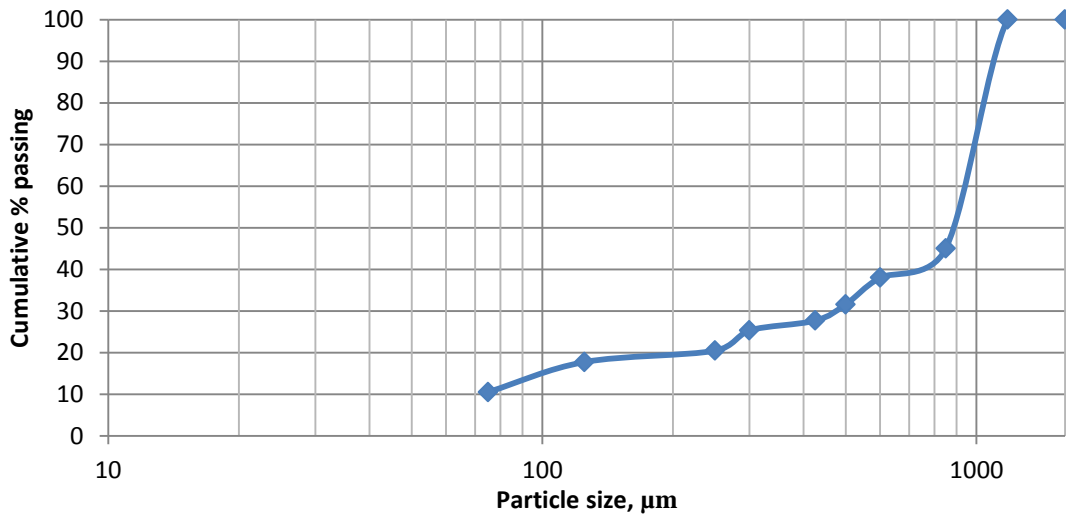


Figure 4-1: Particle size distribution of “as received” oxidized ore from Mimosa mine

The crushed ore sub-samples were then milled in a ring mill with the aim of achieving a grind size of 60% passing 75 μm. A grind size of 63% passing 75 μm was achieved. The ground ore was packaged into 1kg sample bags. The particle size distribution of the ground ore is shown in Figure 4-2.

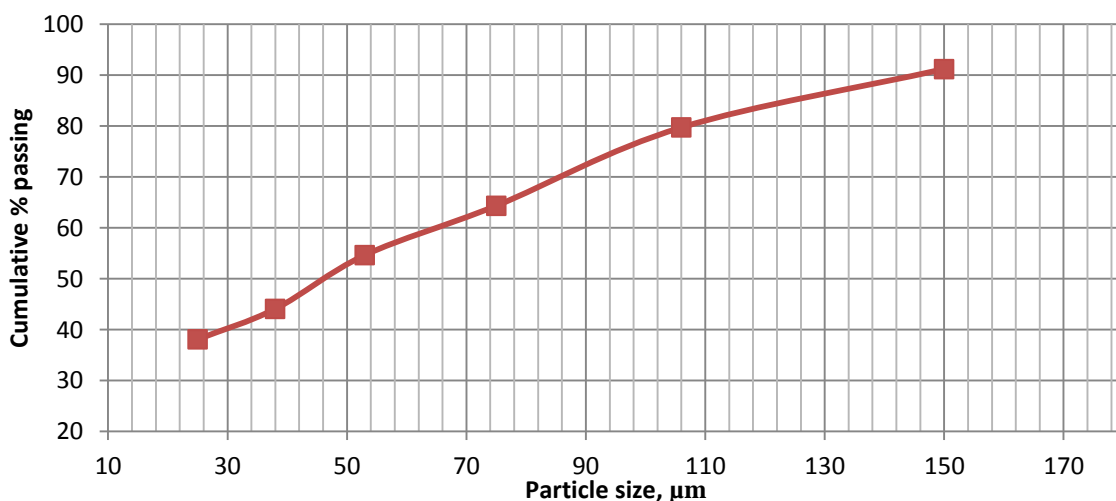


Figure 4-2: Particle size distribution of the milled oxidized ore from the Mimosa mine

## 4.2. Characterisation of the oxidised/non-sulphide PGM ore sample- Mineralogical and elemental analysis

A representative powder sample for ore characterization was prepared from the milled ore. The powder sample was characterized using XRD for mineral identification and quantification of minerals present in the ore. The diffraction pattern in Figure 4-3 shows minerals such as quartz, chabazite, enstatite, chromite and magnetite were identified but no base metal sulphide or PGM minerals could be identified which could be due to low concentrations of PGMs. However previous studies on the Great Dyke oxidised PGM ores have revealed that the main PGM carrying minerals found in this ore are sperrylite and cooperite. The mineral chabazite identified in the ore under study has previously not been reported in non-sulphide/oxidized PGM ores.

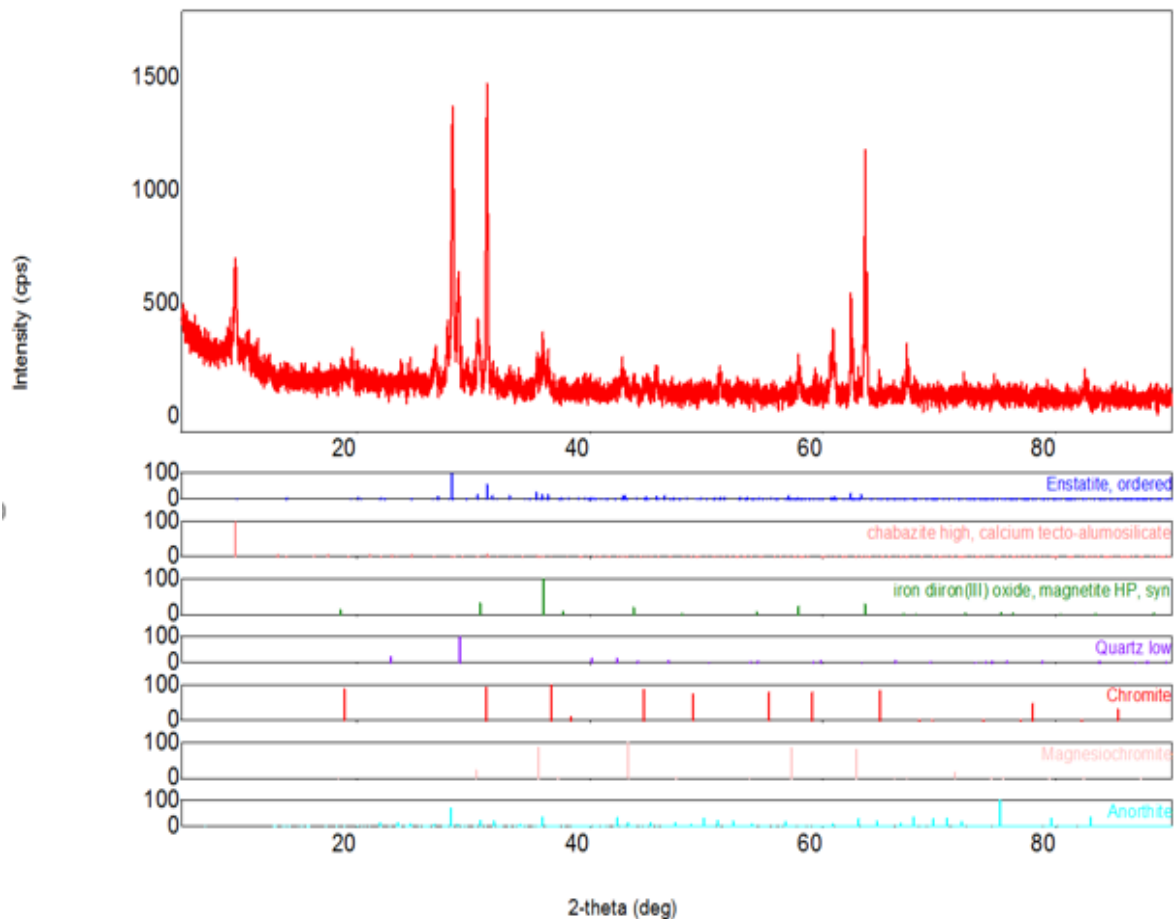


Figure 4-3: X-ray diffraction pattern of the non-sulphide Mimosa mine PGM ore

The results of the XRD mineral quantification are shown in Table 4.1 . It can be seen that the ore is dominated by gangue material such as quartz, chabazite and a high content of magnetite.

**Table 4.1: XRD mineral quantification of Mimosa mine non-sulphide PGM ore**

Mineral phase	Formula	Content (%)
Chabazite	$\text{Ca}_{1.85}(\text{Al}_{3.7} \text{Si}_{8.3} \text{O}_{24})$	21.82
Quartz	$\text{SiO}_2$	45.10
Magnetite	$\text{Fe}_3\text{O}_4$	33.07
Others		0.01
Total		100

An XRF and ICP-OES analysis were also carried out to determine the elemental composition of the feed. The ore was analysed for 4E at Mintek using the ICP-OES characterization technique and it was found that the ore is low-grade as observed in Table 4.2. The Pd content is much lower than Pt, indicating Pd remobilization from the weathered zone to the lower zone which reduces the flotation efficiency by changing Pt:Pd ratio. The XRF technique could not identify any PGMs but it identified base metals such copper, nickel and cobalt as shown in Table 4.2. The XRF technique also identified high contents of iron and silicon indicating agreement with the XRD results. Detailed XRF results are in Appendix A.

**Table 4.2: Elemental composition of Mimosa mine non-sulphide PGM ore**

ICP-OES		XRF	
Element	Content (g/t)	Element	Content (wt. %)
Pt	1.42	Cu	0.31
Pd	0.85	Ni	0.58
Au	0.09	Fe	25.9
Rh	<0.02	S	0.64
		Cr	7.97
		Co	0.051
		Si	32.00

### 4.3. Flotation test work results and discussion

#### 4.3.1. The effect of reagent suite 1 on 3E and platinum flotation performance

Figure 4-4 show that 3E recoveries are generally higher at low depressant dosage (Tests 1 to 4) and lower at high depressant dosage (Tests 5 to 8) whereas 3E grade is generally low at low depressant dosages and high at high depressant dosages. This is due to increased depression of gangue minerals caused by high depressant dosages, resulting in a cleaner concentrate. The highest 3E recovery of 55.14% was obtained from the 3<sup>rd</sup> test where both the SIBX and depressant M98B were at low dosages and the co-collector Flomin C5460 was high. The corresponding 3E grade of this test was 31.58g/t. The platinum recovery at these conditions is 66.12% as shown in Figure 4-5 and the palladium recovery is 40.06% as presented in Figure 4-6. The significant difference in Pt and Pd recovery could be due to the different flotation behaviour of minerals associated with each metal or the low Pd content in the ore due to the remobilization from the weathered zone to the lower zone which reduces the flotation efficiency by changing the Pt:Pd ratio. The recoveries of both palladium and gold reduce the overall 3E recovery.

The best 3E grade of 43.02g/t was obtained from the 7<sup>th</sup> test where both the co-collector Flomin C5460 and the depressant M98B were at high dosages. However this resulted in a relatively low 3E recovery of 45.55%. Figure 4-7 show that Test 7 had the lowest solids recovery of 2.49%, which suggest that there was more selectivity at these conditions. This could be due to the co-collector Flomin C5460 selectively recovering the PGM minerals or the depression of the gangue material by the high depressant dosage.

An increase in the dosages of both SIBX and the co-collector Flomin C5460 in the 4<sup>th</sup> test resulted in the second best 3E recovery of 54.61% at a 3E grade of 10.86g/t from 55.12% at 31.58g/t when the SIBX was low and Flomin C5460 was high. This suggests that SIBX at high dosages reduces the selectivity of Flomin C5460. The low grade in Test 4, which was the lowest in all tested conditions, corresponds with the highest solids and water recoveries of 11.82% and 18.63% respectively as shown in Figure 4-7. This is consistent with the findings by (Yianatos & Contreras, 2010) who carried a study and found that as the water recovery increased, the copper grade decreased. The high mass recovery and low grade in Test 4 could be due to the frothing properties of the IPETC in the Flomin C5460 co-collector. This leads to entrainment of gangue material in the froth, increasing the gangue material recovered



relative to the PGM minerals which dilutes the concentrate grade. This poor selectivity is due to the more stable froth observed at these conditions. This observation is also consistent with the findings by Maree et al. (2017) on using IPETC as a collector on a sulphide ore, though when paired with a xanthate PAX, they obtained better recoveries.

The co-collector Flomin C5460 seems to have a positive and significant effect on the 3E recovery which is more than that achieved when the other two reagents were increased or decreased individually or in combination.

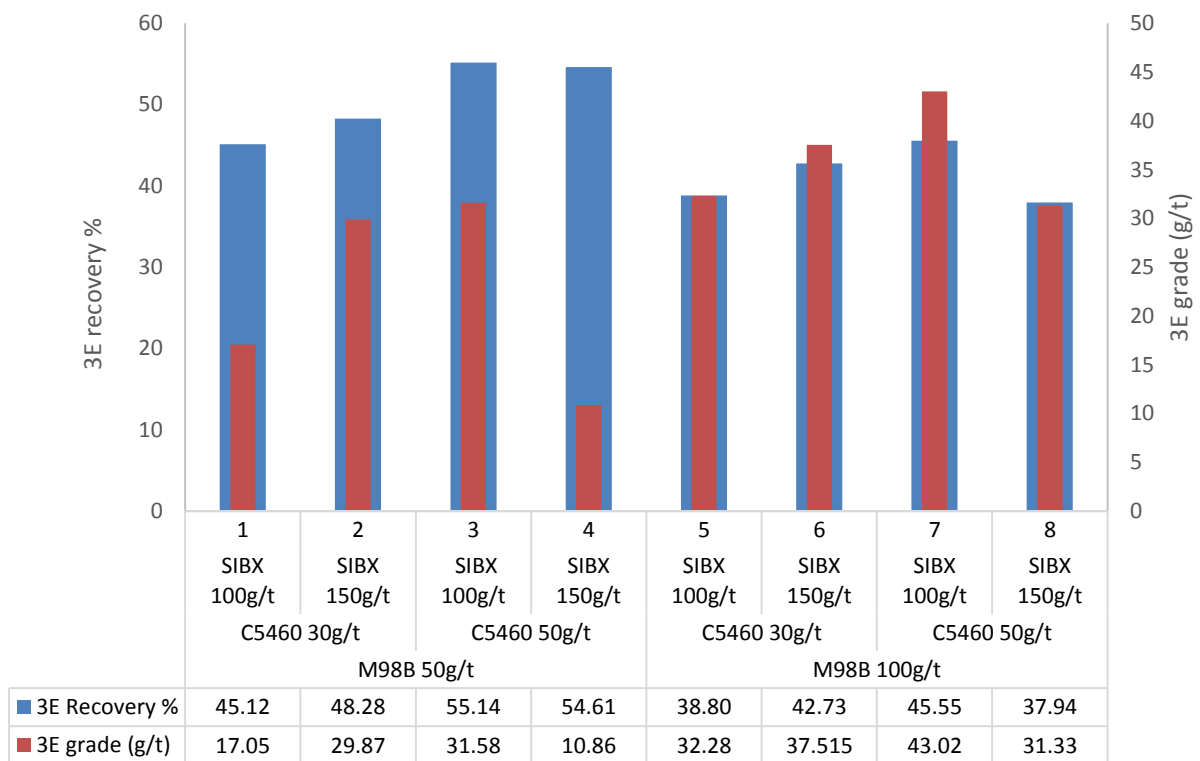


Figure 4-4: Effect of reagent suite 1 on the 3E recovery and grade

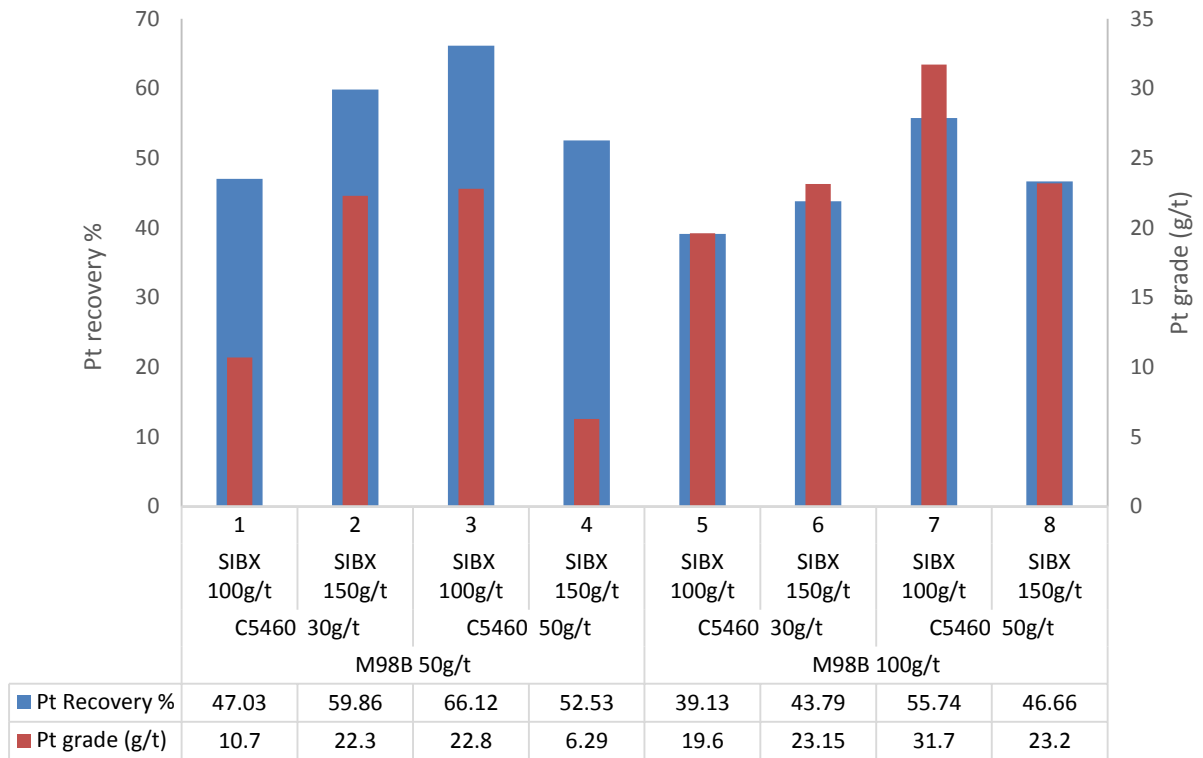


Figure 4-5: Effect of reagent suite 1 on the Pt recovery and grade

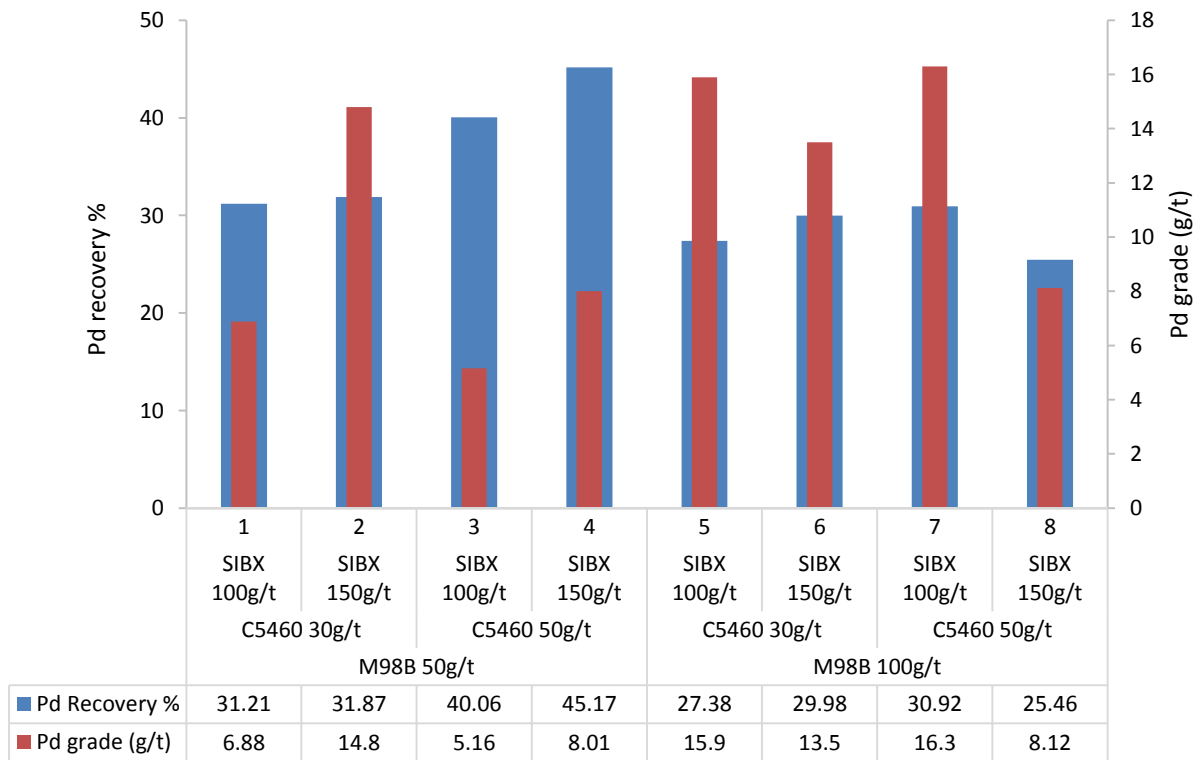


Figure 4-6: Effect of reagent suite 1 on the Pd recovery and grade

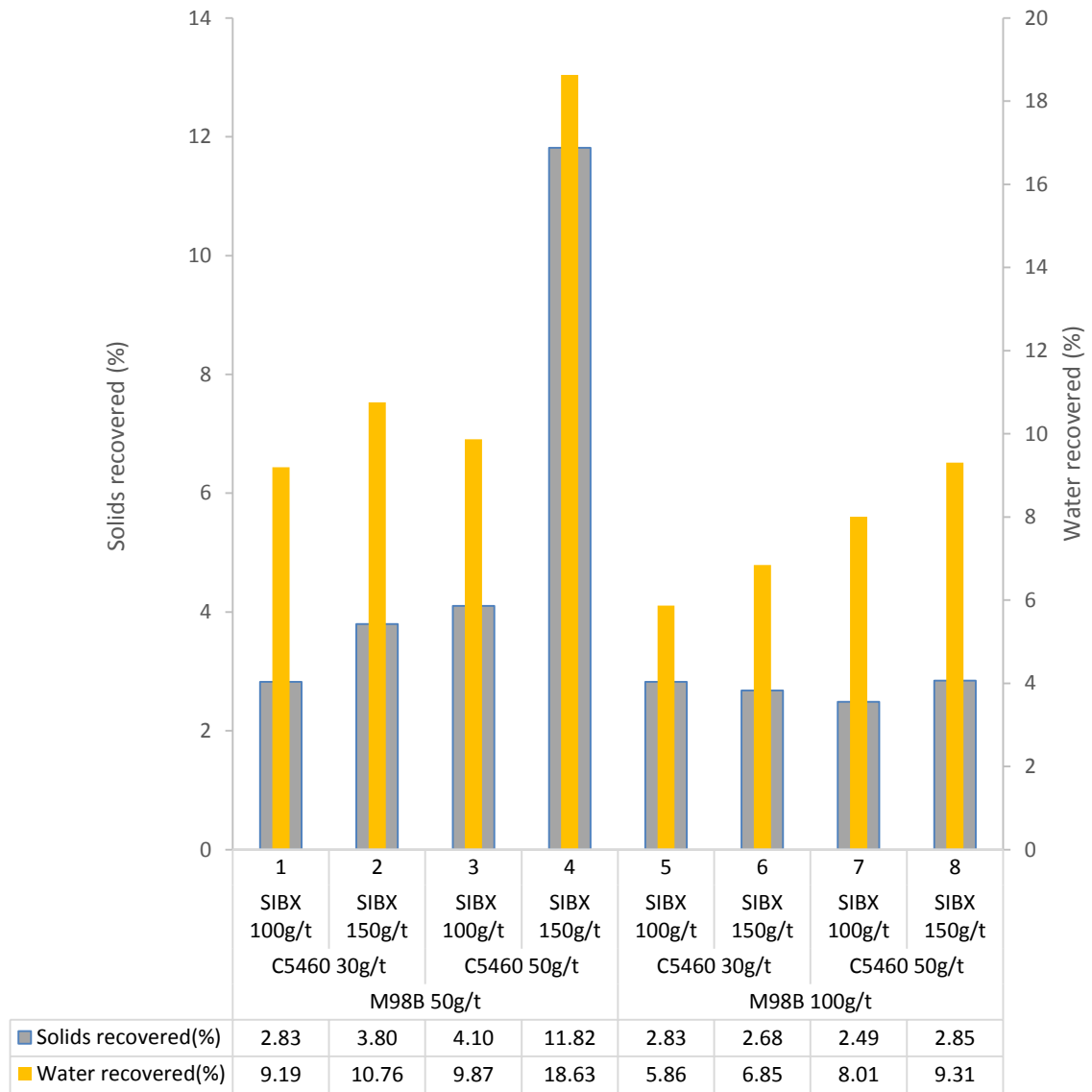


Figure 4-7: Effect of reagent suite 1 on the mass recovery

### 4.3.2. The effect of reagent suite 2 on 3E and platinum flotation performance

Figure 4-8 shows that 3E recoveries are generally higher for Tests 1-4 where the depressant M98B was at a low dosage of 50g/t. However there is no clear trend observed with the behaviour of grade. In Figure 4-8, the best 3E recovery of 51.80% at a grade of 45.46 g/t was obtained from the 2<sup>nd</sup> test where SIBX was at a high dosage of 150 g/t and both the co-collector, Flomin C7133 and the depressant M98B were at low dosages of 30 g/t and 100 g/t respectively. The solids recovery of 2.68% obtained at these conditions is the lowest in all tested conditions as shown in Figure 4-10, which suggest that the high 3E grade at these conditions is due to better selectivity. The platinum recovery at the same conditions is 60.93% as shown in Figure 4-9.

However, when the SIBX is reduced to 100g/t as in the 1<sup>st</sup> test, the 3E recovery does not change as much whereas the grade is halved, indicating that SIBX at higher dosages improves the selectivity when paired with Flomin C7133. This suggests that higher SIBX dosages and lower xanthogen formate will be preferable in optimising this reagent suite.

Increasing the dosage of only the co-collector Flomin C7133 or a combination of the reagents had a negative effect on the recovery of 3E, compared to when all the reagents had low dosages. A similar trend was observed for both the recovery and grade of 3E and platinum. SIBX seems to have the greatest effect in improving the 3E recovery and grade in this reagent suite albeit slightly from what it is when all reagents are at their low dosage levels. The effect of the different reagents on the solids and water recoveries is not clear as only minimal changes are observed as seen in Figure 4-10.

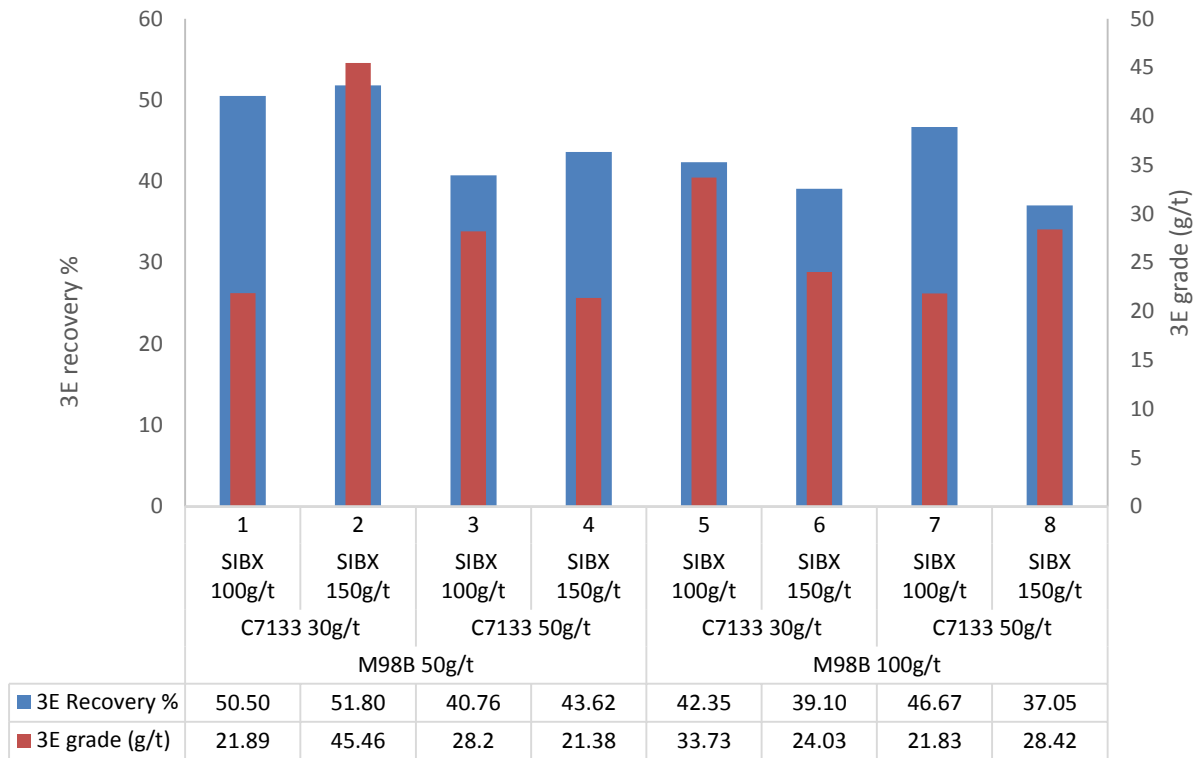


Figure 4-8: Effect of reagent suite 2 on the 3E recovery and grade

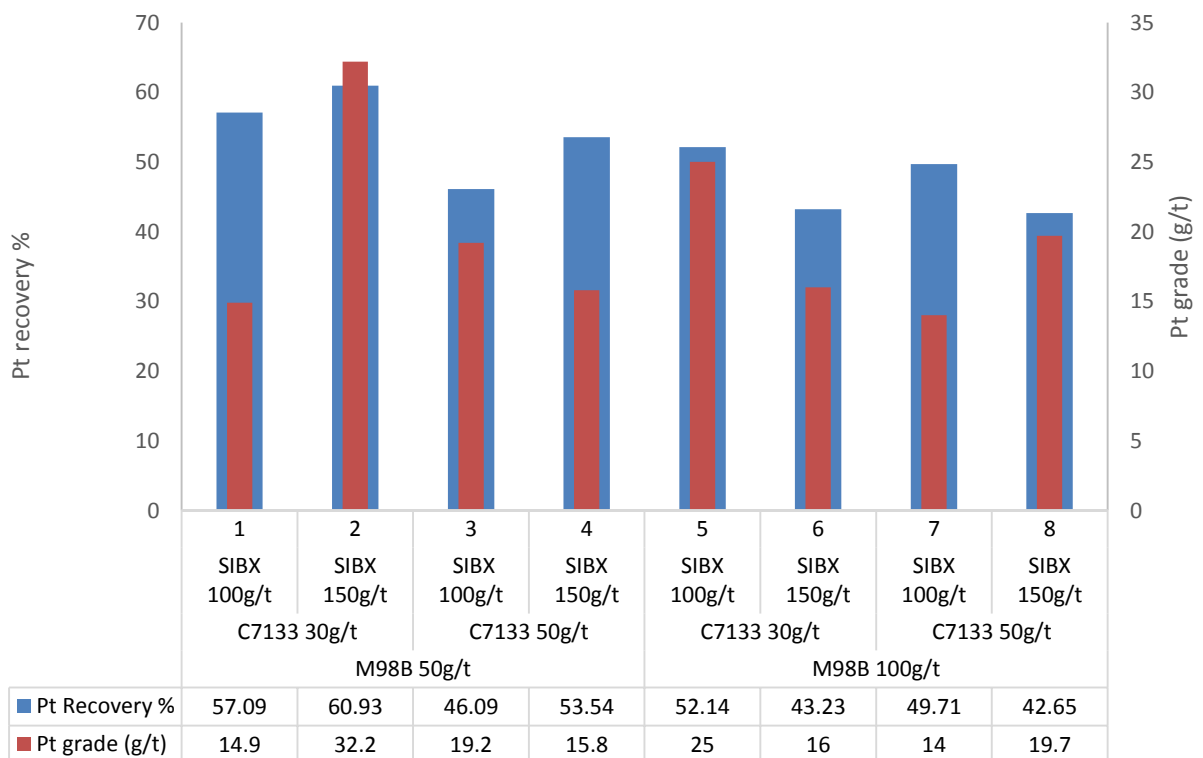


Figure 4-9: Effect of reagent suite 2 on the Pt recovery and grade

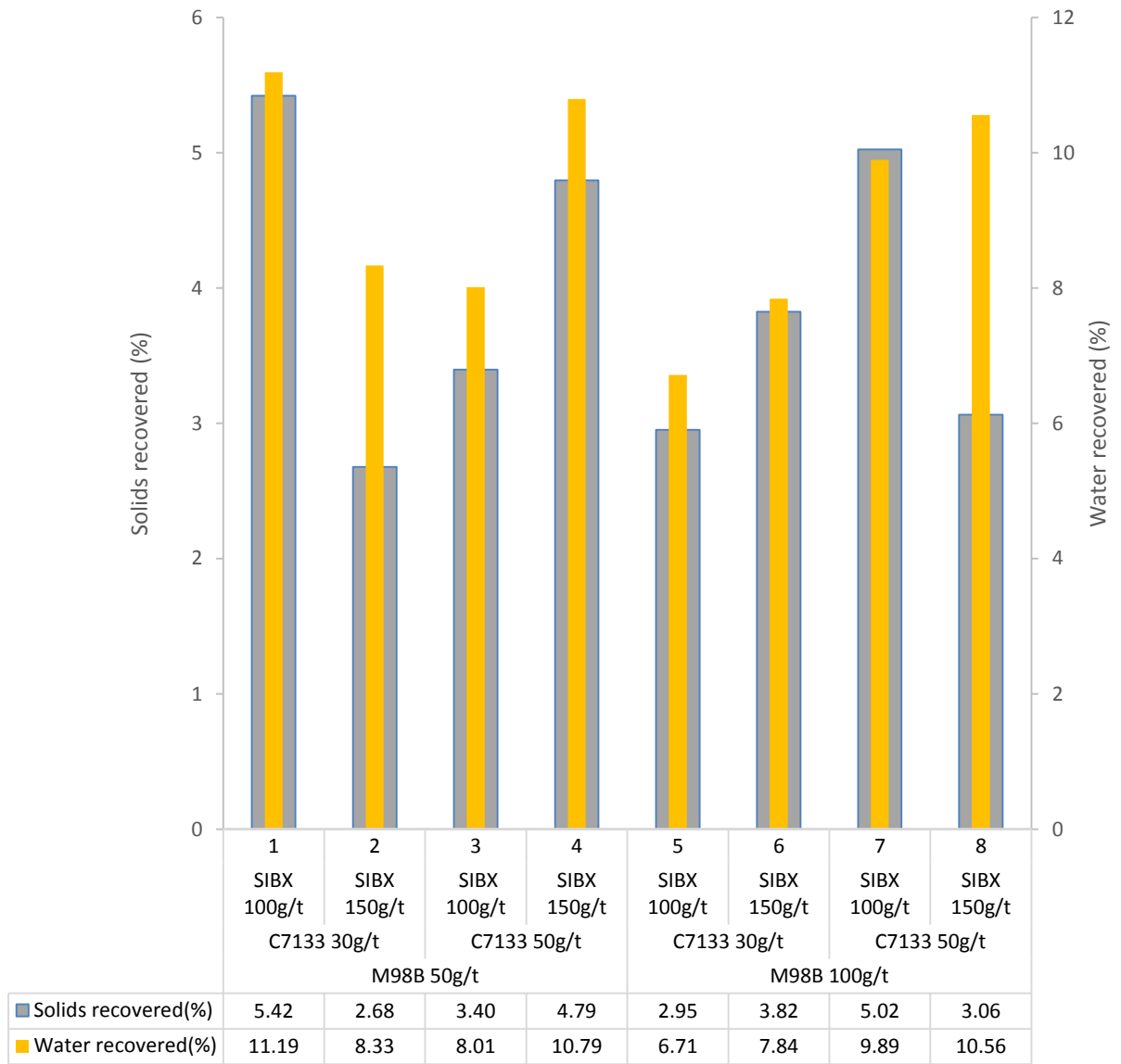


Figure 4-10: Effect of reagent suite 2 on the mass recovery

### 4.3.3. The effect of reagent suite 3 on 3E and platinum flotation performance

It can be observed in Figure 4-11 that 3E grades are generally higher at high depressant dosage (Tests 5 to 8) and lower at low depressant dosage (Tests 1 to 4), which suggests that the gangue was well depressed at high depressant dosage and the only gangue recovered was by entrainment as explained by Becker et al. (2014). The high grade obtained when using the hydroxamate AM810 indicates improved attachment of PGM bearing minerals to the bubbles when this reagent is used. It can also be observed that at high depressant dosage, the 3E recovery does not change significantly when other reagents are varied except when all the reagents are at high dosages as in Test 8.

The Pd recoveries and grades shown in Figure 4-13 follow a similar trend to Pt though much lower compared to Pt, as was the case in the previous reagents suites. The significant difference in Pt and Pd recovery could be due to different flotation behaviour of minerals associated with each metal. It can also be a result of the difference in the ROM ore due to the Pd remobilization from the weathered zone to the lower zone which reduces the flotation efficiency as observed in the low content of Pd in the ROM ore.

The highest recoveries for Pt (73.95%), Pd (48.41%) shown in Figure 4-12 and 3E were obtained from the 3<sup>rd</sup> test where the hydroxamate was at a high dosage and both the SIBX and depressant were at low dosages. This represents the highest Pt and Pd recoveries achieved, in comparison with what was achieved using other reagent suite investigated in this study.. The 3E recovery and grade at these conditions is 63.08% and grade 18.7g/t, indicating a substantial difference from the 4E recovery of 39% obtained by Becker et al. (2014) on the flotation of an oxidized PGM ore. Improved hydroxamates selection is observed when the target mineral is the most soluble and the chelate formed between the cation in the lattice and hydroxamate is the most stable (Assis, et al., 1996). Since the oxidised PGM ore contained high concentrations of iron as shown in the characterisation test work, it was deduced that recovery of iron oxide minerals by the hydroxamate collector resulted in improved PGM recoveries.

It has also been found that when the carbon chain of the hydroxamate is higher than C9, then flotation performance is reduced. Bulatovic (2007) found that hydroxamates with a carbon-chain length between C7 and C9 are the most successful. The application of N-octyl hydroxamate collector AM28 in successfully recovering the oxide minerals malachite and azurite from a copper sulphide/oxide ore blend without affecting the sulphide recovery

supports this claim (Lee, et al., 2009). The C8-C10 alkyl AM810 hydroxamate used in this study was able to improve 3E recoveries to 63.08% at a grade 18.7g/t, indicating a substantial difference from the 4E recovery of 39% obtained by Becker et al. (2014) on the flotation of an oxidized PGM ore.

However, grades for both Pt and Pd were low in Test 3, which could be due to entrainment and the recovery of gangue caused by the frothing properties of the hydroxamate. The large solids and water recovery in Test 3 illustrated in Figure 4-14 also suggests that more gangue material was recovered, reducing the concentrate grade. This is consistent with the findings by Yianatos & Contreras (2014) who found that water recovery is inversely proportional to grade. The best 3E grade (68.01g/t) was obtained from the 5<sup>th</sup> test where both the hydroxamate and SIBX were at low dosages and the depressant at a high dosage.

The recoveries obtained in Test 3 after using reagent suite 3 are much improved than the recoveries reported in previous studies in literature hence it was decided that reagent suite 3 based on a hydroxamate collector is selected and more conditions tested to optimize its application.



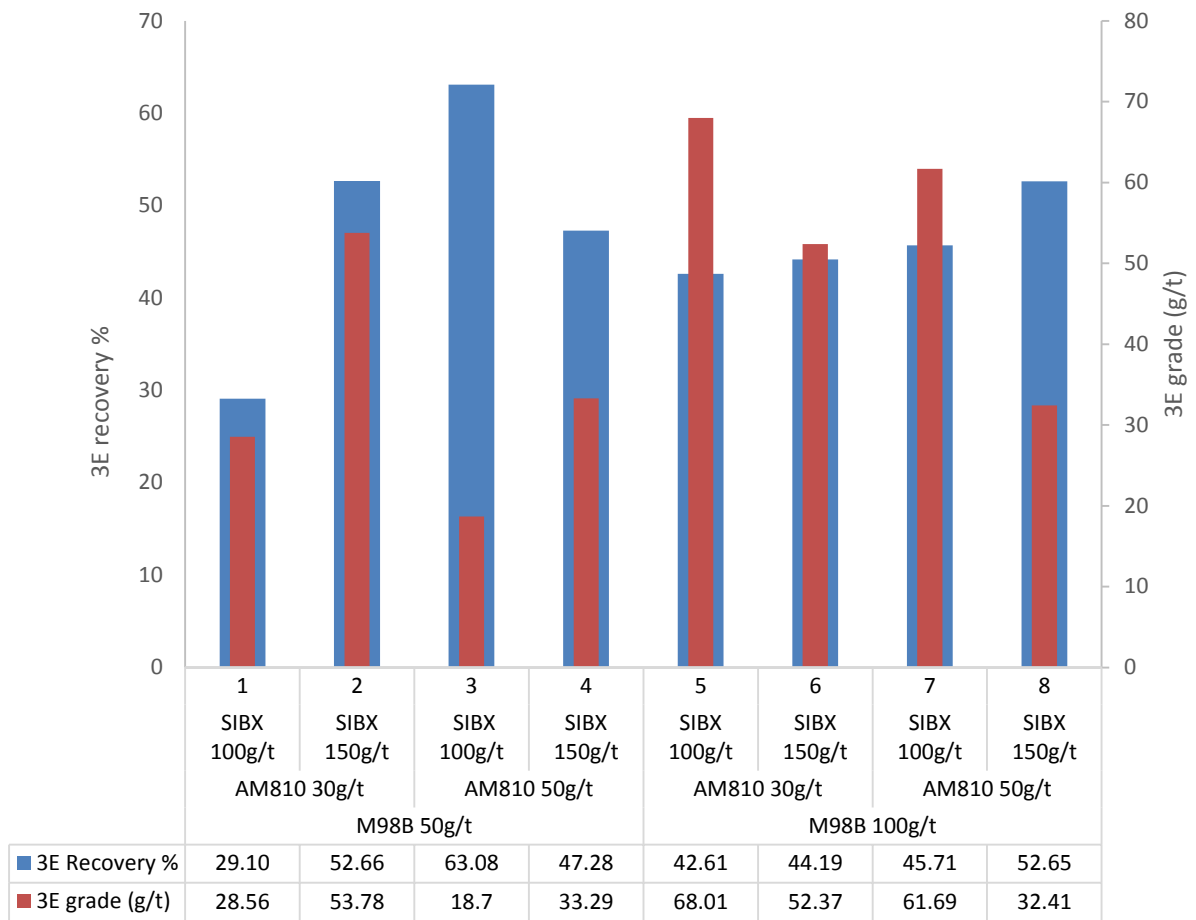


Figure 4-11: Effect of reagent suite 3 on the 3E recovery and grade

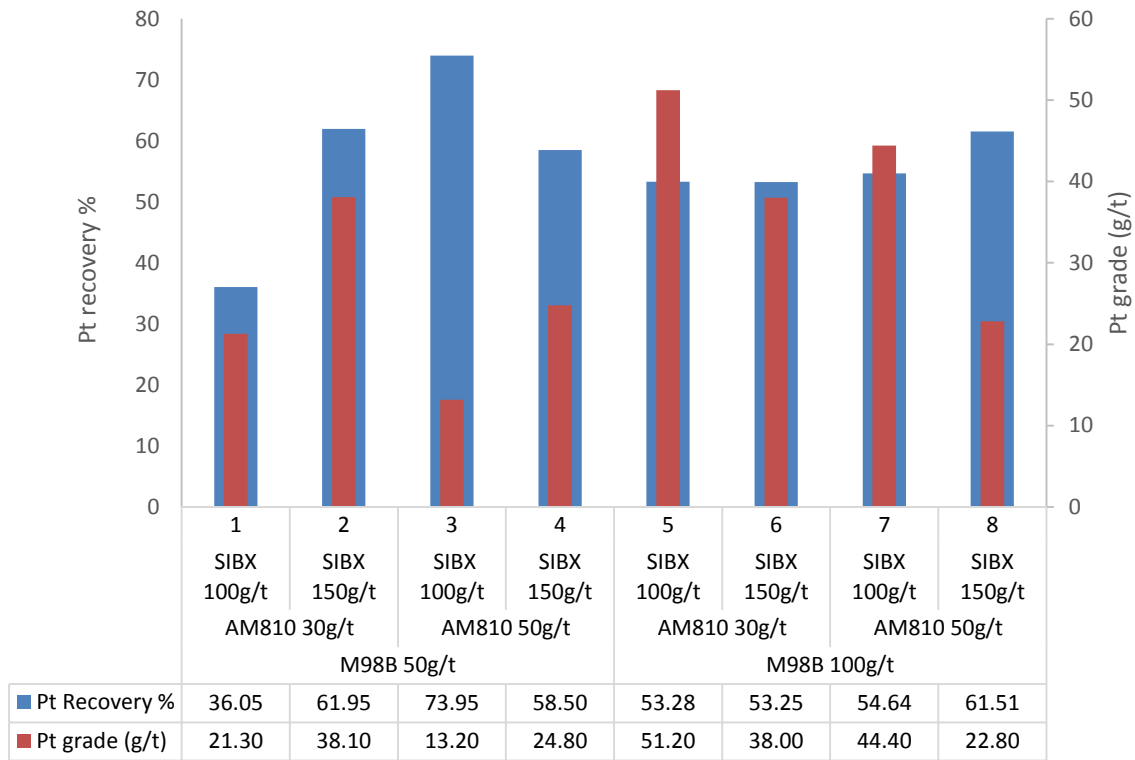


Figure 4-12: Effect of reagent suite 3 on the Pt recovery and grade

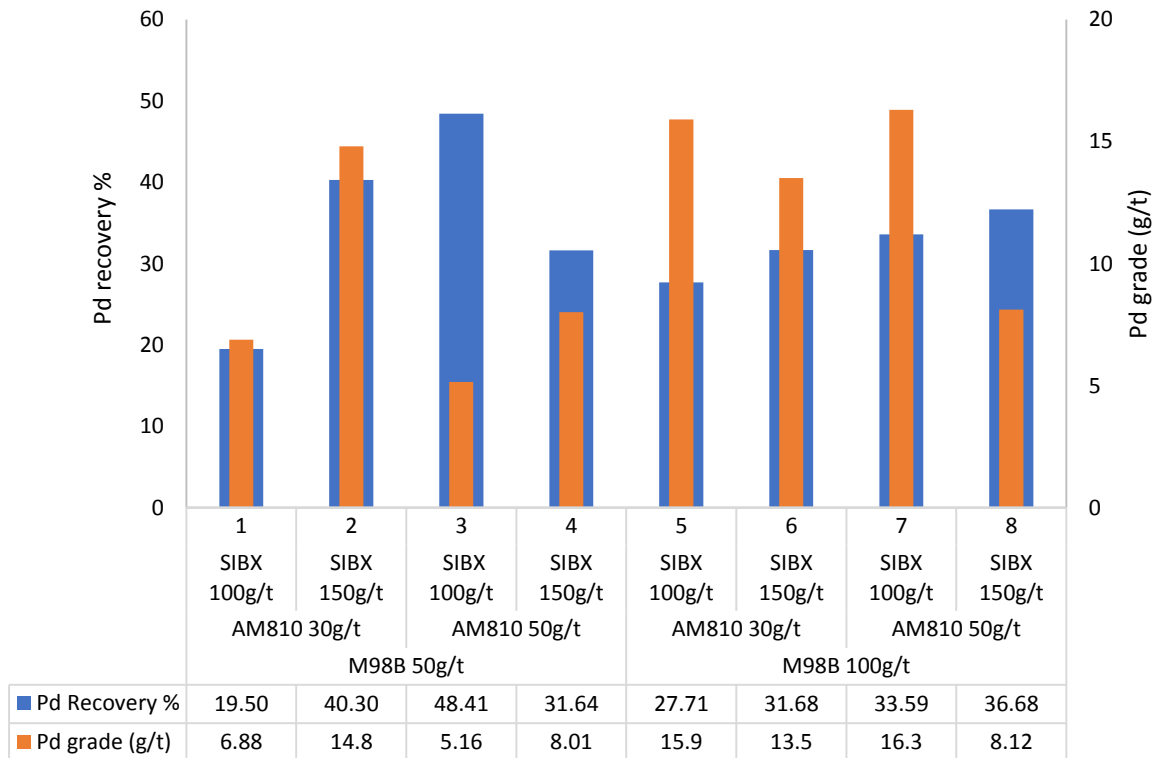


Figure 4-13: Effect of reagent suite 3 on the Pd recovery and grade

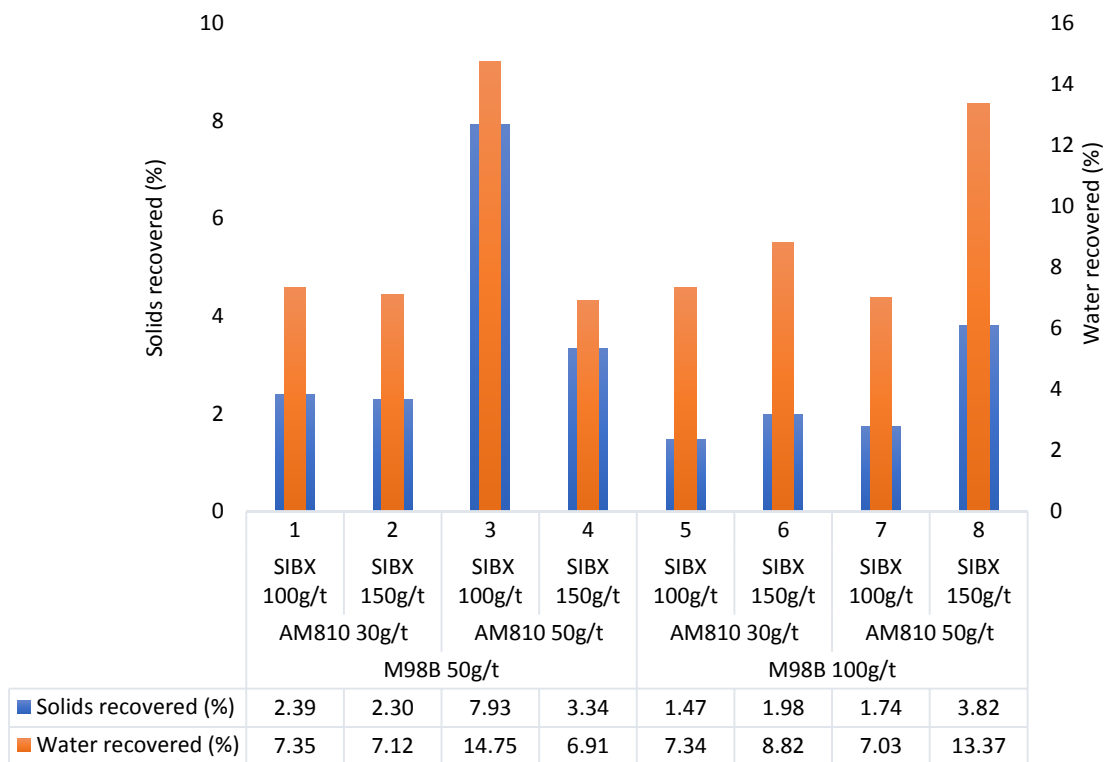


Figure 4-14: Effect of reagent suite 3 on mass recovery

#### 4.4. Statistical analysis

Reagent suite 1 and 3 flotation results were chosen for statistical analysis as they showed significantly more promising results and the response of Pt was selected as it was observed to have the greatest effect on the 3E recovery . The matrix of the variables and response studied for both reagent suites 1 and 3 respectively are shown in Table 4.3. The experimental data in Table 4.3 was used to plot the main and interaction effects, and these are shown in Figure 4-15 and Figure 4-16.

Figure 4-15 shows that SIBX and M98B have a negative effect on the Pt recovery, while Flomin C5460 has a positive effect on the Pt recovery. The interaction between SIBX and Flomin C5460 shows that higher Pt recoveries will be obtained at high Flomin C5460 and low SIBX dosages respectively. Figure 4-16 shows that AM810 has a positive effect on the Pt recovery and M98B has a negative effect on the Pt recovery, while SIBX-AM810 shows that higher Pt recoveries will be obtained at low SIBX and high AM810 dosages.

**Table 4.3: Full factorial design results for flotation of a non-sulphide PGM ore**

Test no.	Level			Pt recovery %	
	A	B	C	Reagent suite 1	Reagent suite 3
1	-	-	-	47.03	36.05
2	+	-	-	59.86	61.95
3	-	+	-	66.12	73.95
4	+	+	-	52.53	58.50
5	-	-	+	39.13	53.28
6	+	-	+	43.79	53.25
7	-	+	+	55.74	54.64
8	+	+	+	46.66	61.51

For reagent suite 1: A – SIBX, B - Flomin C5460, C- M98B

reagent suite 3: A – SIBX, B -AM810, C - M98B

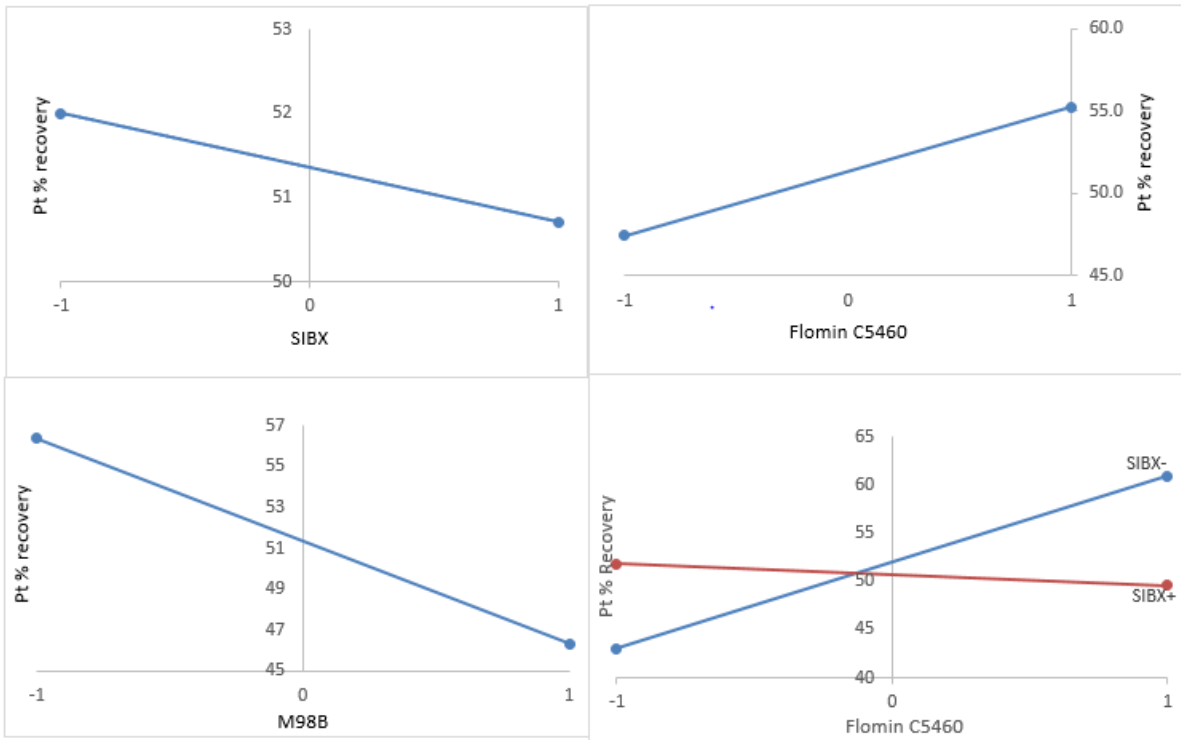


Figure 4-15: Effect of SIBX, Flomin C5460, M98B and SIBX-FLOMIN C5460 interaction on Pt recovery

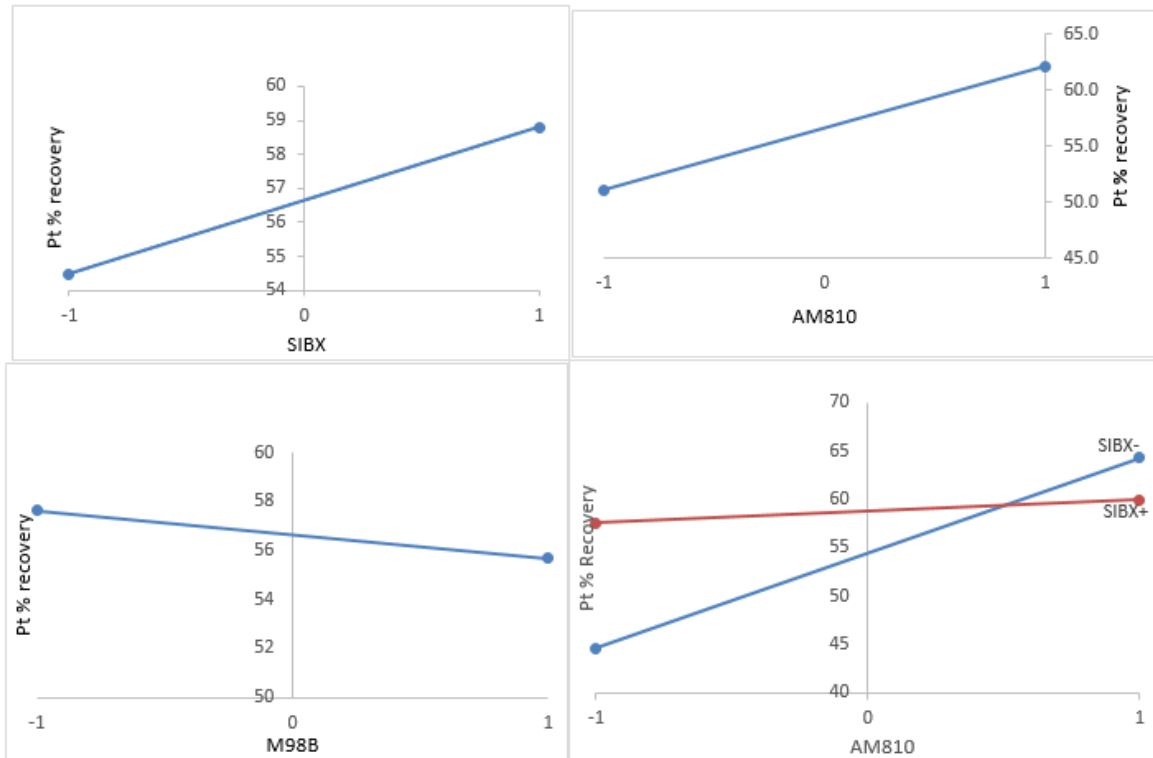


Figure 4-16: Effect of SIBX, AM810, M98B and SIBX-AM810 interaction on Pt recovery

In analysing factorial designs with only few effects estimates such as the one carried out in this study, it is often recommended that a half-normal probability plot be used instead of a normal probability plot as it is easier to interpret. The absolute values of the effect estimates are plotted against their cumulative normal probabilities. The straight line on the half-normal plot always passes through the origin and should also pass close to the fiftieth percentile data value (Myers, et al., 2009).

The analysis of the half-normal probability plot of effects presented in Figure 4-17 show that the co-collector Flomin C5460, depressant M98B and the interaction between SIBX and Flomin C5460 were more statistically significant as they are further away from the straight line. When floating with reagent suite 3 it was observed that there were significant interactions between the factors, with AM810 being the most statistically significant main factor, SIBX-AM810 was also found to be significant as shown in Figure 4-18.

The regression models obtained from the Design Expert software based on the experimental data for both reagent suites are expressed below as:

$$Pt_{recovery,suite\ 1} = 51.36 + 3.91B - 5.025C - 5.02AB \quad (10)$$

$$Pt_{recovery,suite\ 3} = 56.64 + 2.16A + 5.51B - 4.31AB - 3.11BC + 6.03ABC \quad (11)$$

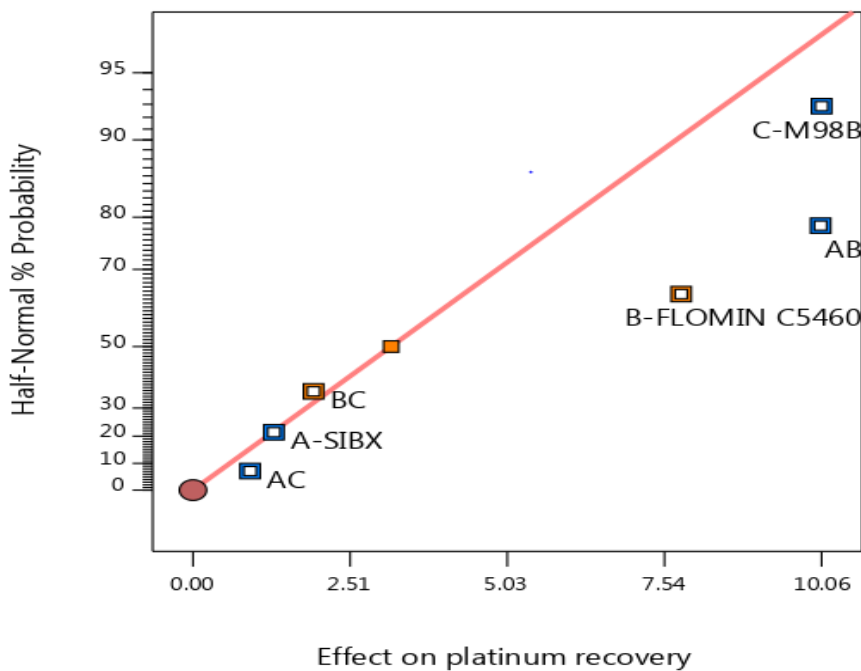


Figure 4-17: Half-normal plot of effects of main factors and factor interactions for reagents suite 1

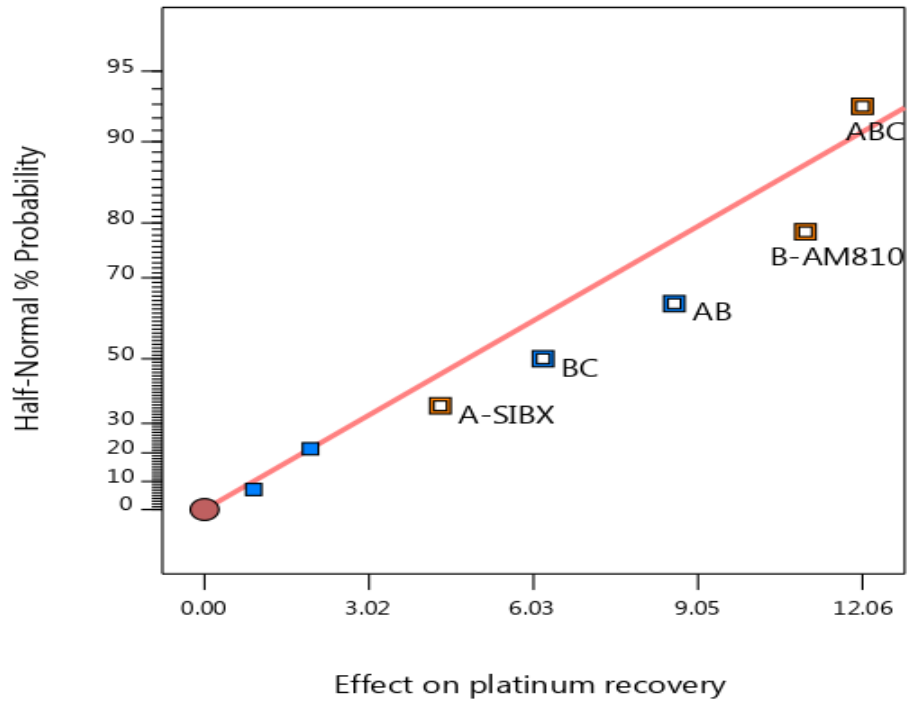


Figure 4-18: Half-normal plot of effects of main factors and factor interactions for reagents suite 3

#### 4.5. Optimization studies

Following the initial reagent scouting phase, an optimization campaign based on the analysis of results was proposed to determine optimum reagent conditions that can maximise 3E and Pt recovery. The flotation tests were conducted as described in Section 3.5. Based on the flotation performance results obtained in Test 3 using reagent suite 3, it was decided to investigate the effect of increasing the hydroxamate AM810 dosage incrementally while fixing SIBX at 100g/t. The experiments were carried out using 60g/t, 70g/t, 80g/t and 90g/t hydroxamate (AM810) with the depressant M98B at 50g/t. The relationship between the AM810 dosage and 3E recovery and grade is shown in Figure 4-19 and the relationship between 3E recovery, % solids recovery and AM810 dosage is shown in Figure 4-20.

A study by Fuerstenau et al. (1970) revealed that better recoveries and selectivity were achieved at longer conditioning times and higher reagent dosage. In this optimization study, it was found that 3E and Pt recoveries slightly increased with increasing hydroxamate (AM810) dosage. However, the grades decreased contrary to the claim by Fuerstenau et al. (1970) as shown in Figure 4-19.

From Figure 4-20, it can be observed that as the AM810 dosage increases, the solids recovery also increases. These observations could be due to the increasing froth as a result of increasing the hydroxamate, leading to increased recovery of both the PGMs and entrained gangue material.

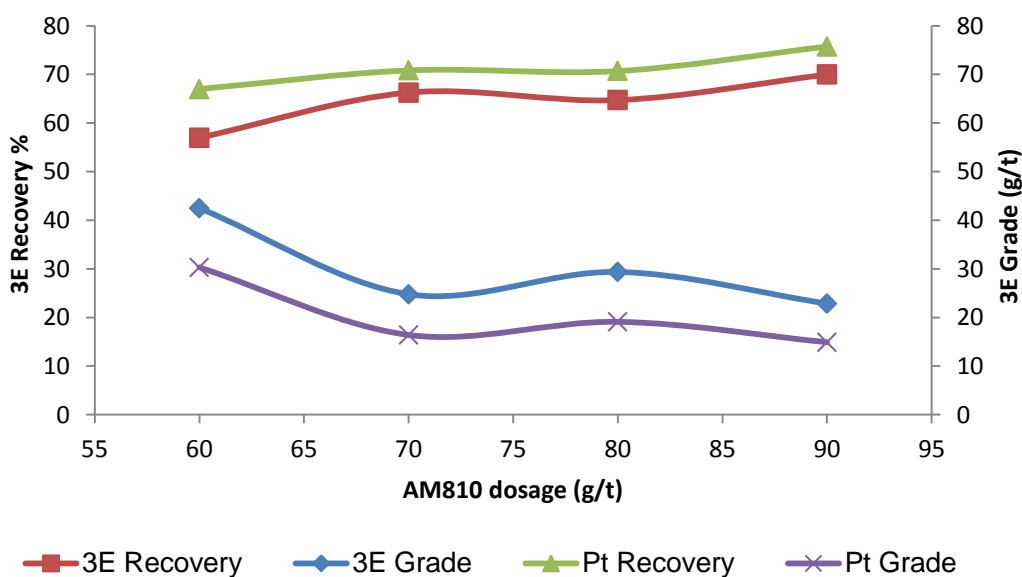


Figure 4-19: Relationship between AM810 dosage and 3E recovery and grade



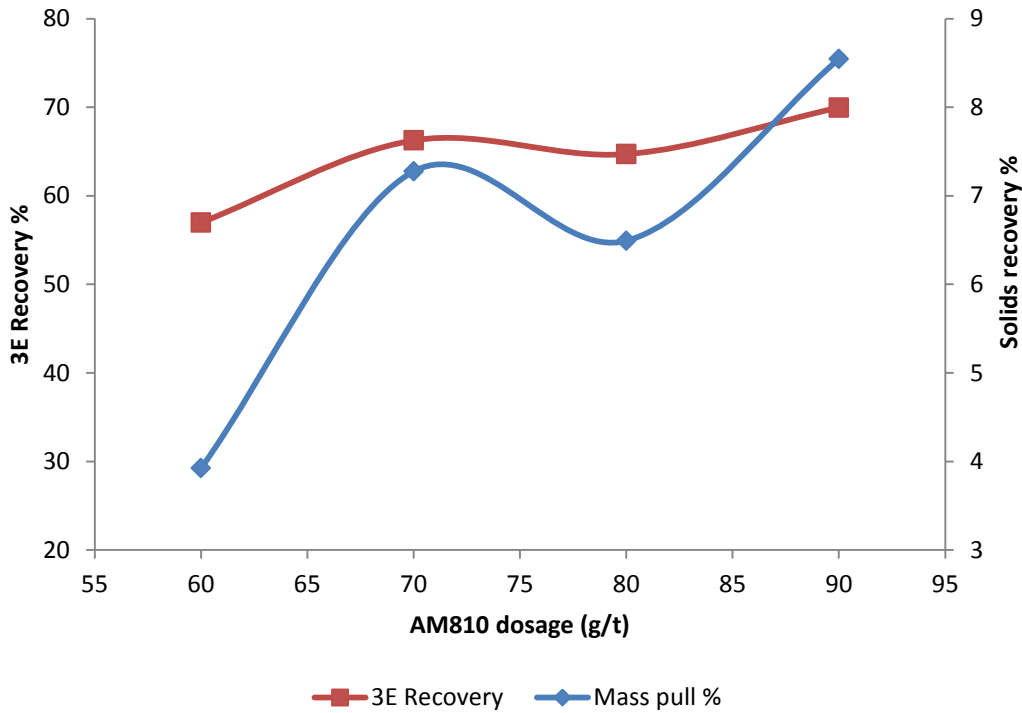


Figure 4-20: Relationship between AM810 dosage and solids recovery

Based on the statistical analysis results, a proposal was made to determine optimum reagent conditions that can maximise platinum recovery. The positive signs of the main effects SIBX(A) and AM810(B) in the Pt recovery equation suggest that platinum recovery may be increased by high dosages of SIBX (A) and AM810 (B).

$$Pt_{recovery, suite 3} = 56.64 + 2.16A + 5.51B - 4.31AB - 3.11BC + 6.03ABC$$

However, the interactions between SIBX and AM810 had to be taken into consideration when maximizing the response.

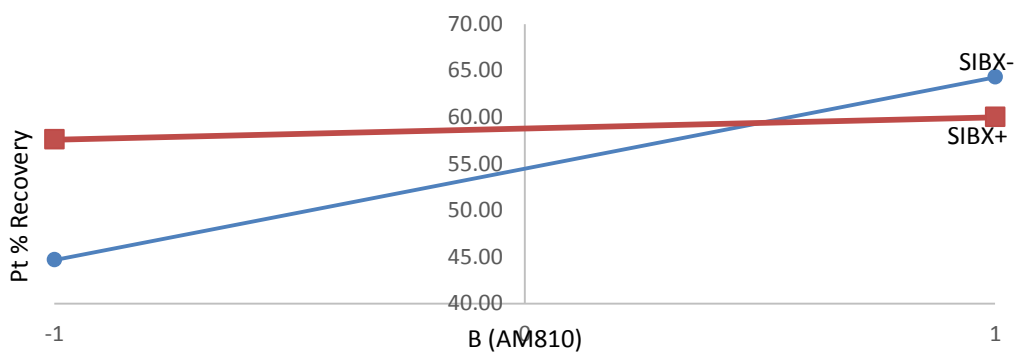


Figure 4-21: SIBX and AM810 interaction plot

The AB interaction shown in Figure 4-21 suggests that higher platinum recoveries were achieved when the hydroxamate was high (positive) and SIBX was low (negative). This implies that to maximize recovery, higher dosages of the hydroxamate and lower dosages of SIBX should be used. This observation was used to find the optimum region by using the steepest ascent method.

The first step in using this method was choosing a step size in one of the process variables, which is significant. In this case variable B (hydroxamate) was chosen and assigned a factor of 1.0, corresponding to 10g/t.

$$\Delta B = +1.0 \rightarrow 10 \text{ g/t}$$

The step size of A (SIBX) is calculated using the coefficients in the regression equation and multiplying the product by the actual dosage average as follows;

$$\Delta A = \frac{2.16}{5.51} = 0.39$$

$$\Delta A = 0.39 \times \frac{150-100}{2} = 9.75 \text{ g/t}$$

Because the dosage of A (SIBX) should be low in order to maximise the recovery, the step size takes a negative value in order to decrease the dosage incrementally,  $\Delta A = -9.75 \text{ g/t}$  so the path of maximum recovery can be followed. Table 4.4 shows the path of steepest ascent. Experimental runs from (Base + $\Delta$  to Base +4 $\Delta$ ) were conducted to search for maximum platinum recovery. The dosage of the depressant M98B was kept at a constant low of 50 g/t in order to maximise the PGM recovery during rougher flotation as it was also found to be statistically insignificant.

**Table 4.4: Path of steepest ascent**

Point	Coded variables		Natural variables	
	SIBX	AM810	SIBX (g/t)	AM810 (g/t)
Base (Starting point)	0	0	125	40
Increment $\Delta$	-0.39	1.0	-9.75	10
Base + $\Delta$	-0.39	1.0	115.25	50
Base +2 $\Delta$	-0.78	2.0	105.5	60
Base +3 $\Delta$	-1.17	3.0	95.75	70
Base +4 $\Delta$	-1.56	4.0	86	80

The optimization results obtained using the steepest ascent method are shown in Figure 4-22 and

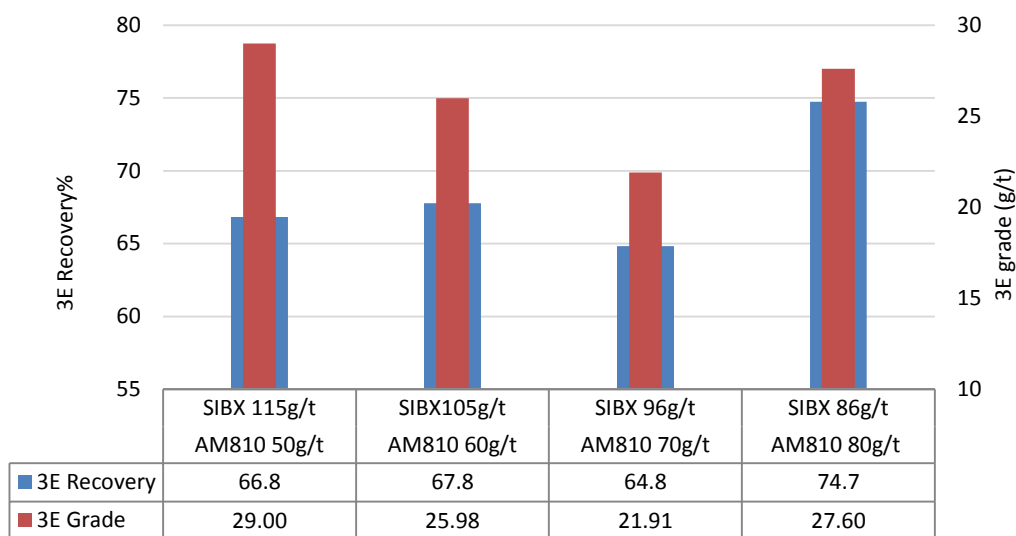


Figure 4-23. It can be observed from the results that as the SIBX dosage decreases and hydroxamate dosage is increased the recovery of both platinum and 3E generally increases. The optimization studies revealed that maximum recoveries of 78.5% Pt and 74.7% 3E can be achieved at grades of 17.90g/t Pt and 27.60g/t 3E at the roughing stage. This is a significant upgrade from the 1.42g/t Pt and 0.85g/t Pd in the feed material. These results were obtained at optimized parameters of 86g/t SIBX and 80g/t AM810, with depressant M98B at 50g/t.

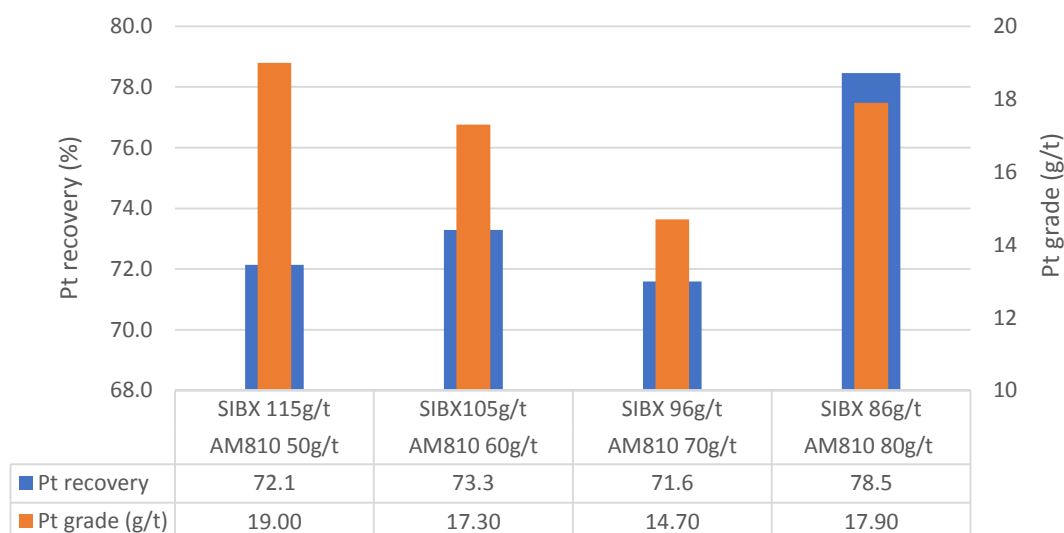


Figure 4-22: Effect of AM810 and SIBX on Pt recovery and grade using the steepest ascent method

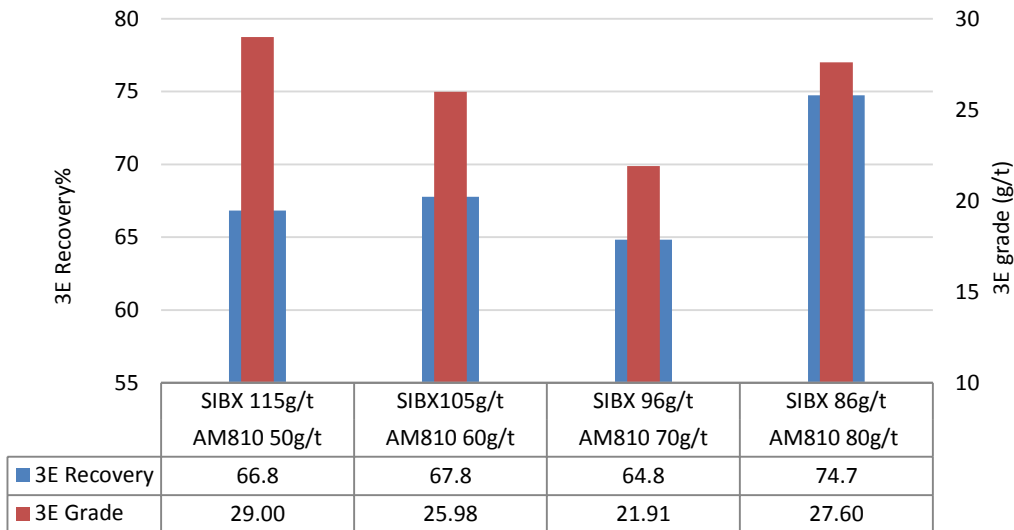


Figure 4-23: Effect of AM810 and SIBX on 3E recovery and grade using the steepest ascent method

When comparing the methods used to attempt to maximize PGM recovery i.e. steepest ascent method and incrementally increasing the hydroxamate dosage while other reagents are kept constant, it was found that the first method resulted in a higher 3E recovery. This suggest that the AM810 co-collector functions better in collecting oxidised PGM bearing minerals when the AM810 dosage is high and the SIBX dosage is low.

#### 4.6. Kinetic studies

Flotation kinetics rate tests were conducted on the optimum conditions obtained in Section 4.4. The optimum conditions are shown in Table 4.5. Flotation tests were conducted as described in Section 3.5, however in the flotation kinetics rate tests, concentrates were collected into different trays after 1, 3, 7, 15 and 20 minutes and then dried, weighed and sent for analysis. The flotation test was repeated twice for repeatability purposes.

Table 4.5: Kinetic rate tests conditions

Reagents in order of addition	Reagent dosage(g/t)	Conditioning time (min)
Activator	50	5
SIBX	86	2
AM810	80	2
Depressant M98B	50	3
Frother	50	1

Figure 4-24 show the plot of recovery of 3E, Pt and Pd vs time. Generally, the flotation kinetics appears to be slow as the experimental flotation rate curves show a small gradient in the first few minutes of flotation and do not seem to approach a plateau after 20 minutes flotation time. According to literature hydroxamates are associated with slow adsorption kinetics (Assis, et al., 1996), which is consistent with the observations made in these tests. This suggests that longer flotation times would be required to increase recovery, consistent with the study by Fuerstenau et al. (1970) who found that better recoveries and selectivity were achieved at longer conditioning times. However, this happens at the expense of grade which becomes increasingly poor due to increased recovery of gangue material.

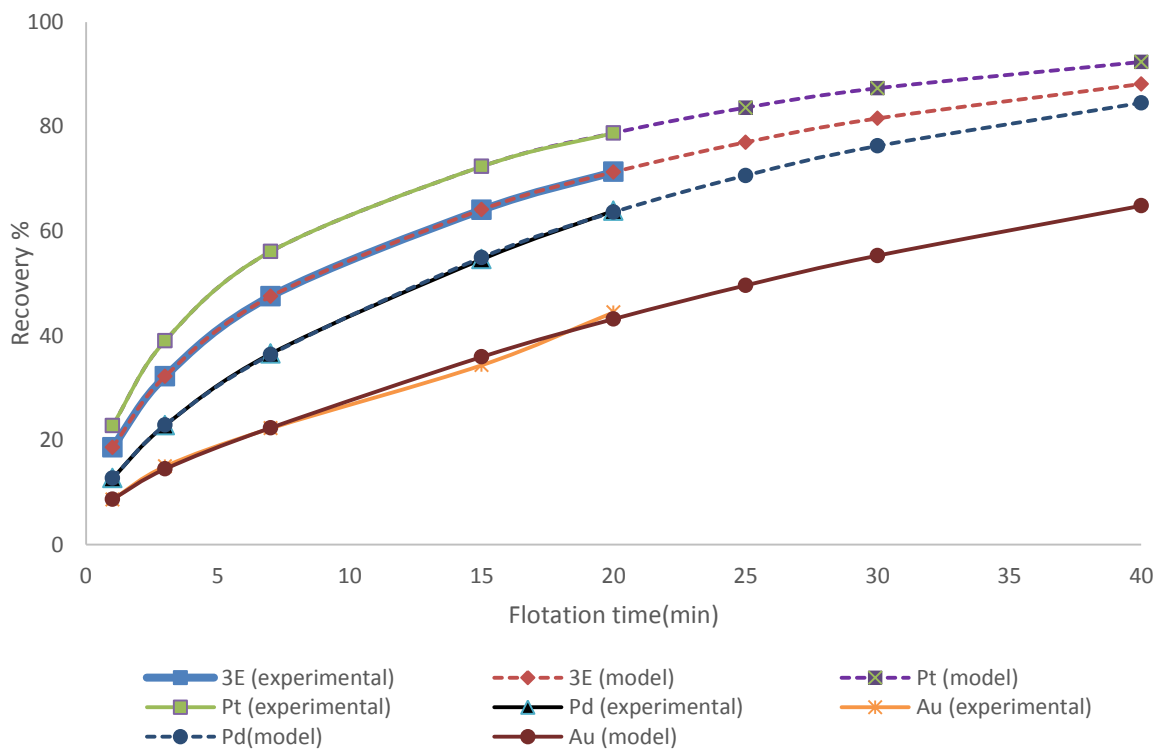
From the recovery-time curve in Figure 4-24, it can be seen that the PGMs were recovered relatively faster in the first 7 minutes and then slower onwards, this can be explained by the fact that the PGMs were being depleted in the flotation cell. The 3E recovery achieved after 20 minutes was 71.32%, hence it was decided to investigate the recovery at longer flotation times.

Further investigations were done to predict the PGM recovery at longer flotation times. This was achieved by using the flotation kinetic model developed in Section 2.3 to determine the flotation kinetic parameters i.e. the fast floating constant rate ( $k_f$ ), slow floating constant rate

( $k_s$ ), fast floating mass fraction ( $P_f$ ), slow floating mass fraction ( $P_s$ ) and the non-floating mass fraction ( $P_n$ ), using the minimization of sum of squares (SOS) to minimise the residual error and then using the fitted model to predict the recovery. The flotation kinetic parameters for 3E, Pt and Pd shown in Table 4.6 were used to predict the recovery of the respective components at longer flotation times. The fast -floating fraction of Pt is much greater than that of Pd and Au which suggest that the fraction associated with Pt is much better liberated. The predicted 3E and Pt flotation rate curves shown in Figure 4-24 were modelled using the established parameters.

**Table 4.6: Flotation kinetic parameters**

	Pf	Kf	Ps	Ks	Pn
3E	0.218	0.385	0.696	0.044	0
Pt	0.291	0.38	0.602	0.0529	0.0011
Pd	0.0906	0.525	0.855	0.0428	0
Au	0.0996	1.798	0.919	0.0240	0



**Figure 4-24: Experimental and model recovery-time curves**

According to Hay (2005), the minimum rougher residence time required to recover the fast-floating fraction of PGMs in a plant processing sulphide PGM ores is approximately 25 minutes, hence a similar time was used for rougher flotation in this testwork. The predicted flotation rate curves in Figure 4-24 show that there is a good agreement between the experimental and predicted recovery values. Based on the predicted recovery and data, it was decided that the optimum flotation time to recover PGMs is 30 minutes as the curve starts to plateau out.

#### 4.7. Summary

The mineralogical and elemental analysis performed on the oxidic/non-sulphide PGM ore under study revealed that the ore is a low grade non-sulphide PGM ore. The XRD characterization technique identified minerals such as quartz (45.10%), chabazite (21.82%), magnetite (33.07%) and minor contents (<0.01%) of enstatite and chromite indicating the ore is rich in silicate and iron minerals. However, PGMs and base metal sulphides were not detected by XRD due to their low concentrations. The XRF characterization technique further revealed that the ore is rich in base metals such nickel, copper, chromium and iron, and has a low content of sulphur, which is consistent with findings of the XRD analysis. The ICP-OES characterization technique used to identify PGE in the ore found that the ore is a low-grade PGM ore ( $3E = 2.36\text{ppm}$ ) with a high Pt/Pd ratio owing to the weathering process which results in the loss of palladium. The non-sulphide PGM ore under study has similar characteristics to the oxidized PGM ores previously investigated in the Bushveld Complex and the Great Dyke, where poor flotation recoveries (typically less than 50%) were experienced.

The effect of the selected flotation reagents on the recovery and grade of the PGMs in terms of their dosages and interactive effects based on the design of experiments (DOE) approach was investigated. The three reagent suites tested are made up of (i) a collector, (ii) a co-collector and (iii) a depressant i.e. (Suite 1 - SIBX, FLOMIN C5460, M98B), (Suite 2 - SIBX, FLOMIN C7133, M98B) and (Suite 3 - SIBX, AM810, M98B).

The best 3E recovery (55.41%) using reagent suite 1 was obtained when a high dosage of co-collector FLOMIN C5460, low SIBX dosage and a low dosage of the depressant M98B were used. The statistical analysis corroborated that the co-collector has a positive effect on the PGM recovery and higher PGM recoveries can be obtained at high FLOMIN C5460 and low SIBX dosages respectively. The best 3E recovery (51.8%) using reagent suite 2 was obtained when low dosages of both co-collector FLOMIN C7133 and the depressant M98B, and high SIBX dosages were used. Reagent suite 2 performed poorly in comparison to the other reagent suites tested. The best 3E recovery (63.08%) using reagent suite 3 was obtained when a high dosage of co-collector AM830, low SIBX dosage and a low dosage of the depressant M98B were used. The statistical analysis corroborated that the co-collector AM810 has a positive effect on the PGM recovery and that higher recoveries can be obtained at high AM810 and low SIBX dosages respectively



It was observed that reagent suite 3 gave the best performance as it was the most effective in recovering PGM from the ore. Attempts were made to optimize the dosage levels of the reagent suite using two methods i.e. incrementally increasing the hydroxamate dosage while other reagents were kept constant and using the steepest ascent method. The observations from the experiments described above indicated that recovery of PGEs was on the upward trend as the dosage of hydroxamate was increasing hence the effect of the hydroxamate co-collector was further tested at higher dosages while fixing SIBX at 100g/t and depressant M98B at 50g/t. The experiments were carried out using 50g/t, 60g/t, 70g/t and 80g/t hydroxamate (AM810). It was observed that the 3E recovery only increased slightly with increasing hydroxamate(AM810) dosage reaching a maximum of 70.0% at a grade of 22.85g/t, with a platinum recovery and grade of 75.7% and 14.9g/t respectively. These results were obtained at 100g/t SIBX and 90g/t AM810.

The steepest ascent optimization studies revealed that 74.7% 3E at a grade of 22.60g/t (78.5% Pt at a grade of 17.90g/t) can be achieved which is a significant upgrade for roughing stage from the 1.42g/t Pt and 0.85g/t Pd in the feed. These results were obtained at optimized dosages of 86g/t SIBX and 80g/t AM810, with depressant M98B at 50g/t.

## CHAPTER 5 : CONCLUSIONS AND RECOMMENDATIONS

### 5.1. Conclusions

The aim of this study was to improve the rougher flotation recovery of non-sulphide PGM ores by testing a variety of flotation reagents and combinations of reagents in order to find a balance between an acceptable grade and recovery of the rougher concentrate and then optimizing the reagent dosages.

The research questions investigated and the findings are presented below:

- i. What is the mineralogical composition of the non-sulphide PGM ore?

The ore under study was found to be a low grade PGM ore with a significant amount of base metals such as copper, nickel and iron. The characterization techniques used in this study could not identify any PGM minerals, which could have been due to their low concentrations. The ore was found to contain silicate minerals such as silica and chabazite, and magnetite in substantial amounts.

- ii. What are the main and interactive effects of the selected flotation reagent suites on the flotation recovery and grade of PGM concentrates?

Three flotation reagent suites were tested, comprising of (i) a collector, (ii) a co-collector and (iii) a depressant i.e. (Suite 1 - SIBX, FLOMIN C5460, M98B), (Suite 2 - SIBX, FLOMIN C7133, M98B) and (Suite 3 - SIBX, AM810, M98B) respectively. Flotation with the first and second reagent suite resulted in moderate recoveries ranging between 50-55% 3E, with the recovery positively influenced by the co-collector FLOMIN C5460 in the first reagent suite and a combination of SIBX and FLOMIN C7133 had a poor effect on the PGM recovery and grade with second reagent suite use. Reagent suite 3 gave the best performance as it was the most effective in recovering PGM from the ore resulting in a 3E recovery of 63.08%, 73.95% Pt and Pd 48.41% recovery. The improved recovery was because of the co-collector AM810, which seemed to provide a more stable froth due to its froth properties resulting in increased solids and water recovery. The significant difference in Pt and Pd recovery could be due to different flotation behaviour of minerals associated with each metal or the difference in the ROM ore due to the Pd remobilization from the weathered zone to the lower zone which reduces the flotation efficiency as seen by the low content of Pd in the ROM ore.

iii. What are the optimum conditions when floating non-sulphide PGM ores?

Reagent suite 3 comprising of the primary collector SIBX, co-collector AM28 and depressant M98B was optimized as it resulted in improved PGM recovery. The optimization studies revealed that 74.7% 3E at a grade of 22.60g/t (78.5% Pt at a grade of 17.90g/t) can be achieved which is a significant upgrade for roughing stage from the 1.42g/t Pt and 0.85g/t Pd in the feed. These results were obtained at optimized dosages of 86g/t SIBX and 80g/t AM810, with depressant M98B at 50g/t.

## **5.2. Recommendations**

- The non-sulphide ore under study should be characterized with more advanced techniques such as QEMSCAN in order to identify and quantify PGM minerals present in this ore as this will assist in understanding the flotation behaviour of the ore.
- There should be attempts to clean rougher concentrate obtained at optimum conditions or the concentrate can be directly leached in order to recover the PGMs and base metals in the ore.
- The optimum conditions obtained in this study should be further investigated by varying the dosage of the depressant M98B, to determine if the grade can be improved without reducing the recovery.

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## APPENDIX

### Detailed XRF results

2017-3-27 10:23

SQX Calculation Result							
Sample : PGMT				Date analyzed :		2017- 3-23 13:05	
Application : EZS002MNV		Model : Bulk		Balance :			
				Matching library:			
				File :		EZS2712	
No.	Component	Result	Unit	Det.limit	El.line	Intensity	w/o normal
1	Na	0.543	mass%	0.02942	Na-KA	0.6027	0.3272
2	Mg	18.1	mass%	0.02305	Mg-KA	62.5293	10.8908
3	Al	5.25	mass%	0.00974	Al-KA	45.6443	3.1611
4	Si	32.0	mass%	0.01456	Si-KA	264.9398	19.2937
5	P	0.0254	mass%	0.00266	P -KA	0.3613	0.0153
6	S	0.640	mass%	0.00349	S -KA	7.9365	0.3859
7	Cl	0.0365	mass%	0.01225	Cl-KA	0.1015	0.0220
8	K	0.414	mass%	0.00498	K -KA	6.9024	0.2497
9	Ca	7.12	mass%	0.00831	Ca-KA	107.7247	4.2902
10	Ti	0.504	mass%	0.01562	Ti-KA	2.2638	0.3039
11	V	0.106	mass%	0.01329	V -KA	0.7807	0.0641
12	Cr	7.97	mass%	0.08523	Cr-KB1	16.7485	4.8039
13	Mn	0.424	mass%	0.01592	Mn-KA	6.4239	0.2554
14	Fe	25.9	mass%	0.01665	Fe-KA	445.4589	15.5930
15	Co	0.0505	mass%	0.01339	Co-KA	1.1574	0.0305
16	Ni	0.582	mass%	0.00884	Ni-KA	11.2537	0.3508
17	Cu	0.313	mass%	0.00790	Cu-KA	7.7089	0.1886
18	Zn	0.0458	mass%	0.00704	Zn-KA	1.4886	0.0276
19	Sr	0.0203	mass%	0.00461	Sr-KA	2.1665	0.0122

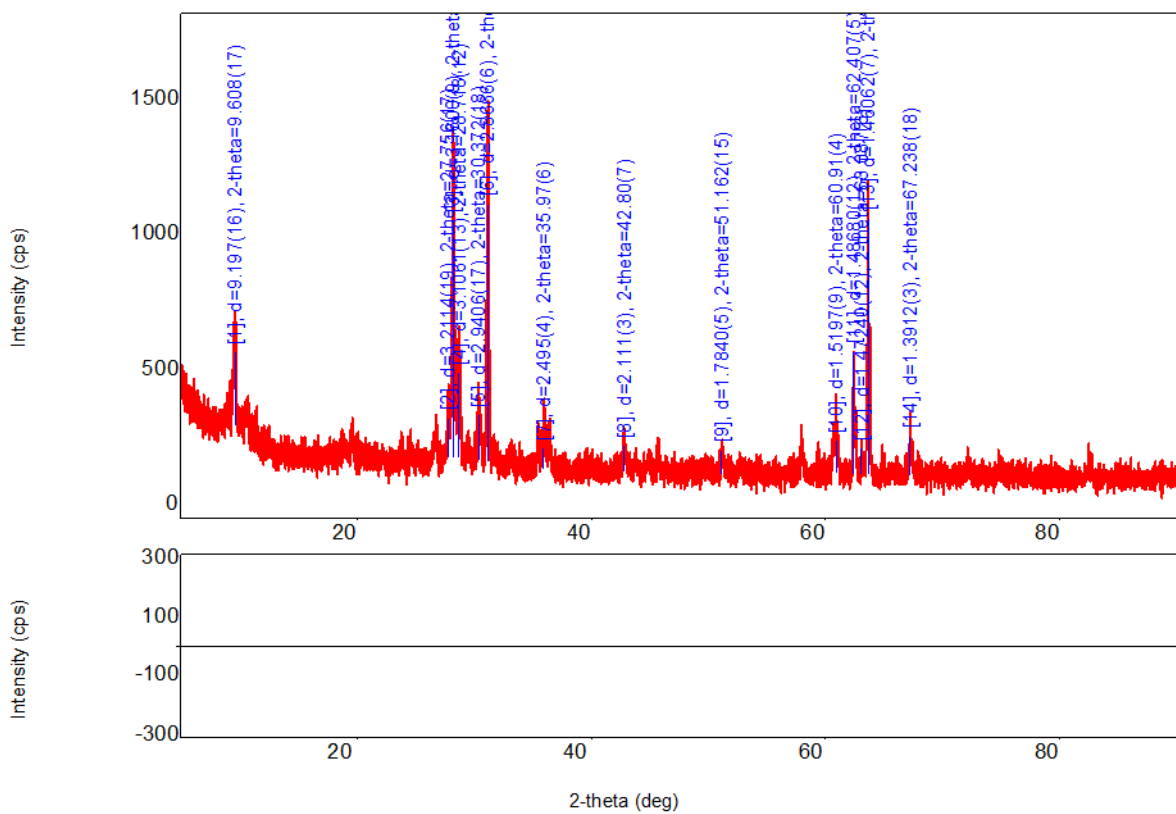
# Detailed XRD Results

## Analysis Results

### General Information

Analysis date	2017/09/26 08:34:30 AM	Measurement date	2017/03/24 07:22:08
Sample name	PGM T	Operator	User
File name	PGM T.raw		
Comment	Full Chart		

### Measurement profile

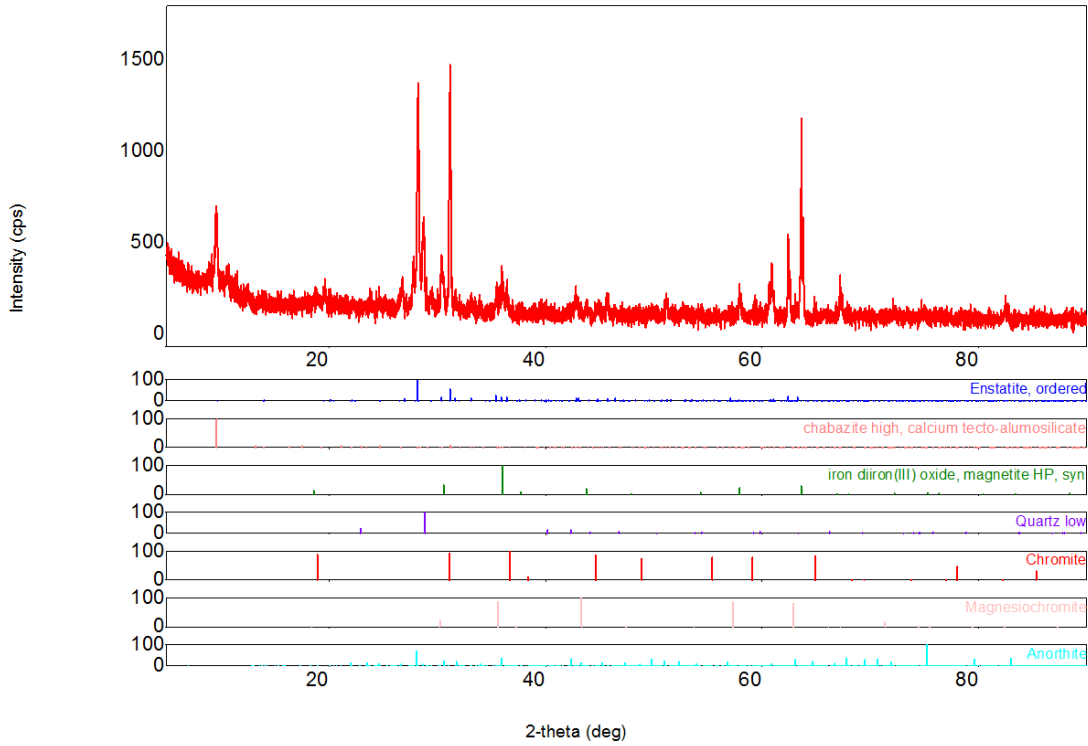


### Qualitative analysis results

Phase name	Formula	Figure of merit	Phase reg. detail	DB card number
Enstatite, ordered	Mg Si O3	0.780	ICDD (PDF 2010)	00-022-0714
chabazite high, calcium tecton diiron(II) oxide, magnetite HP,	Ca1.85 (Al3.7 Si8.3 O24) Fe3 O4	0.961 0.791	ICDD (PDF 2010) ICDD (PDF 2010)	01-085-1140 01-071-6339
Quartz low	Si O2	1.859	ICDD (PDF 2010)	01-070-2537
Chromite	Cr2 O3 · Fe O	1.949	ICDD (PDF 2010)	00-002-1083
Magnesiochromite	Mg O · Cr2 O3	2.934	ICDD (PDF 2010)	00-002-1228
Anorthite	Ca Al2 ( Si O4 )2	3.158	ICDD (PDF 2010)	00-002-0523

Phase name	Formula	Space group	Phase reg. detail	DB card number
Enstatite, ordered	Mg Si O3	61 : Pbca	ICDD (PDF 2010)	00-022-0714
chabazite high, calcium tecton diiron(II) oxide, magnetite HP,	Ca1.85 (Al3.7 Si8.3 O24) Fe3 O4	166 : R-3m,hexagonal 227 : F d-3m,choice-2	ICDD (PDF 2010) ICDD (PDF 2010)	01-085-1140 01-071-6339
Quartz low	Si O2	154 : P3221	ICDD (PDF 2010)	01-070-2537
Chromite	Cr2 O3 · Fe O	227 : F d-3m,choice-2	ICDD (PDF 2010)	00-002-1083
Magnesiochromite	Mg O · Cr2 O3	227 : F d-3m,choice-2	ICDD (PDF 2010)	00-002-1228
Anorthite	Ca Al2 ( Si O4 )2	2 : C-1	ICDD (PDF 2010)	00-002-0523



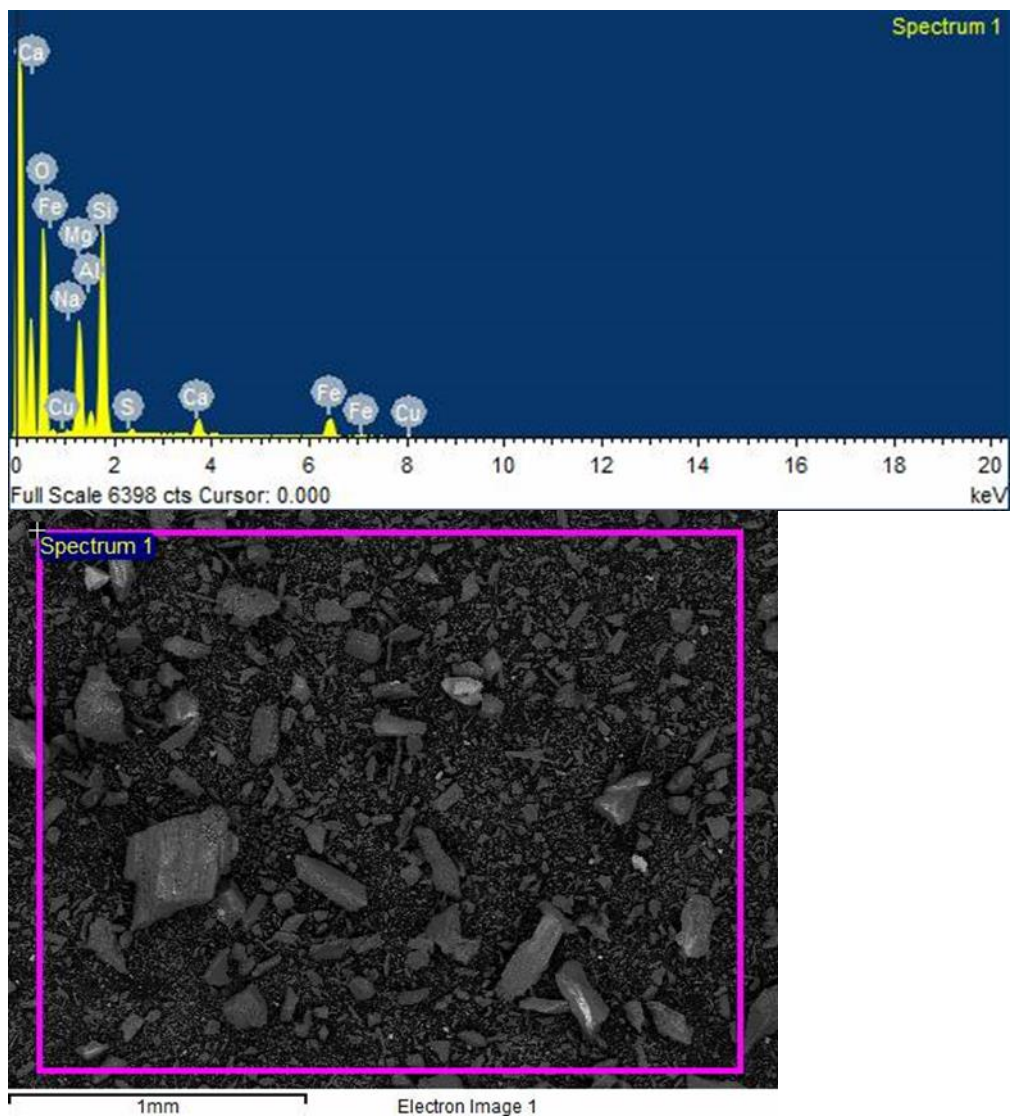
#### Quantitative analysis results (RIR)

Phase name	Content(%)
Enstatite, ordered	0.000000e+000
chabazite high, calcium tecto-alumosilicate	21.823546
iron diiron(III) oxide, magnetite HP, syn	33.070953
Quartz low	45.105501
Chromite	0.000000e+000
Magnesiochromite	0.000000e+000
Anorthite	0.000000e+000
Unknown	0.000000e+000
iron diiron(III) 00-002-1228@oxide, magnetite HP, syn	
Magnesiochromite	
iron diiron(III) 00-002-1083@oxide, magnetite HP, syn	
Chromite	
Magnesiochromite	
Anorthite	

## Detailed ICP-OES results

Assay values						
Sample	Description	Pt (ppm)	Pd (ppm)	Rh (ppm)	Au(ppm)	3E(ppm)
1	Sample 1	1.45	0.85	<0.02	0.09	2.39
2	Sample 2	1.38	0.84	<0.02	0.09	2.31
<b>Average</b>		<b>1.42</b>	<b>0.85</b>	<b>-</b>	<b>0.09</b>	<b>2.35</b>

## SEM Results



## Raw preliminary flotation results

Reagent suite	Exp no	Product	Mass (g)	Mass (%)	Grade				Distribution			
					Pt (g/t)	Pd (g/t)	Au (g/t)	3E (g/t)	Pt (%)	Pd (%)	Au (%)	3E (%)
1	1	Rconc	32.77	2.83	19.60	8.19	4.49	32.28	39.1	27.4	140.9	38.8
	2	Rconc	72.14	6.22	10.70	4.24	2.11	17.05	47.0	31.2	145.8	45.1
	3	RConc	31.05	2.68	23.15	9.47	4.90	37.52	43.8	30.0	145.7	42.7
	4	RConc	47.60	4.10	22.80	8.25	0.53	31.58	66.1	40.1	24.2	55.1
	5	Rconc	28.86	2.49	31.70	10.50	0.82	43.02	55.7	30.9	22.7	45.5
	6	Rconc	33.01	2.85	23.20	7.56	0.57	31.33	46.7	25.5	18.0	37.9
	7	RConc	44.06	3.80	22.30	7.09	0.48	29.87	59.9	31.9	20.3	48.3
	8	RConc	137.07	11.82	6.29	3.23	1.34	10.86	52.5	45.2	175.9	54.6
2	1	Rconc	55.62	4.79	15.80	5.16	0.42	21.38	53.5	29.3	22.4	43.6
	2	Rconc	35.54	3.06	19.70	7.73	0.99	28.42	42.7	28.0	33.7	37.1
	3	RConc	34.23	2.95	25.00	7.79	0.94	33.73	52.1	27.2	30.8	42.4
	4	RConc	44.35	3.82	16.00	6.21	1.82	24.03	43.2	28.1	77.3	39.1
	5	Rconc	58.28	5.02	14.00	5.75	2.08	21.83	49.7	34.2	116.1	46.7
	6	Rconc	62.89	5.42	14.90	5.78	1.21	21.89	57.1	37.1	72.9	50.5
	7	RConc	39.40	3.40	19.20	7.49	1.51	28.20	46.1	30.1	57.0	40.8
	8	RConc	31.06	2.68	32.20	11.90	1.36	45.46	60.9	37.7	40.5	51.8
3	1	Rconc	20.20	1.74	44.40	16.30	0.99	61.69	54.6	33.6	19.2	45.7
	2	Rconc	91.96	7.93	13.20	5.16	0.34	18.70	74.0	48.4	29.9	63.1
	3	RConc	44.28	3.82	22.80	8.12	1.49	32.41	61.5	36.7	63.2	52.6
	4	RConc	38.72	3.34	24.80	8.01	0.48	33.29	58.5	31.6	17.8	47.3
	5	Rconc	17.08	1.47	51.20	15.90	0.91	68.01	53.3	27.7	14.9	42.6
	6	Rconc	27.78	2.39	21.30	6.88	0.38	28.56	36.0	19.5	10.1	29.1
	7	RConc	26.69	2.30	38.10	14.80	0.88	53.78	62.0	40.3	22.5	52.7
	8	RConc	23.00	1.98	38.00	13.50	0.87	52.37	53.2	31.7	19.2	44.2
		Feed	1160									

**Reagent suite 1 conditions and results**

<b>Wits: Non-sulphide Mimosa Mine ore pr+K2:O29ocessing -FLOTATION TEST SHEET</b>				
<b>PROJECT:</b>		<b>Non-sulphide Mimosa Mine ore flotation</b>		
<b>Procedure:</b>		Rougher rate tests		
<b>Operating conditions</b>				
<b>Grind:</b>	60% - 75µm			
<b>Feed:</b>	1.16kg in a 3 L flotation cell			
<b>Make up water</b>	Distilled water modified by addition of ionic salts			
<b>pH:</b>	9			
<b>Reagent Conditions</b>				
<b>Reagent Scheme 1</b>				
<b>Purpose:</b> PGM recovery/grade investigation using SIBX and FLOMIN C5460 collector suite				
<b>Test number</b>	<b>Dosage (g/t)</b>	<b>conditioning time (min)</b>	<b>3E Recovery (%)</b>	<b>3E grade (g/t)</b>
1	100	2	38.8	32.3
	30	2		
	100	3		
2	100	2	45.1	17.1
	30	2		
	50	3		
3	150	2	42.7	37.5
	30	2		
	100	3		
4	100	2	55.1	31.6
	50	2		
	50	3		
5	100	2	45.5	43.0
	50	2		
	100	3		
6	150	2	37.9	31.3
	50	2		
	100	3		
7	150	2	48.3	29.87
	30	2		
	50	3		
8	150	2	54.6	10.86
	50	2		
	50	3		
	50	5	Activator and Frother dosages were constant for all tests	
	50	1		



## Reagent suite 2 conditions and results

Wits: Non-sulphide Mimosa Mine ore processing -FLOTATION TEST SHEET					
<b>PROJECT:</b>		Non-sulphide Mimosa Mine ore flotation			
<b>Procedure:</b>		Rougher rate tests			
<b>Operating conditions</b>					
<b>Grind:</b>	60% - 75µm				
<b>Feed:</b>	1.16kg in a 3 L flotation cell				
<b>Make up water</b>	Distilled water modified by addition of ionic salts				
<b>pH:</b>	9				
<b>Reagent Conditions</b>					
<b>Reagent Scheme 2</b>					
<b>Purpose:</b> PGM recovery/grade investigation using SIBX and FLOMIN C7133 collector suite					
Test number	Reagent name	Dosage (g/t)	conditioning time (min)	3E Recovery (%)	3E grade (g/t)
1	SIBX	150	2	43.6	21.4
	C7133	50	2		
	M98B	50	3		
2	SIBX	150	2	37.1	28.4
	C7133	50	2		
	M98B	100	3		
3	SIBX	100	2	42.4	33.7
	C7133	30	2		
	M98B	100	3		
4	SIBX	150	2	39.1	24.03
	C7133	30	2		
	M98B	100	3		
5	SIBX	100	2	46.7	21.83
	C7133	50	2		
	M98B	100	3		
6	SIBX	100	2	50.5	21.9
	C7133	30	2		
	M98B	50	3		
7	SIBX	100	2	40.8	28.2
	C7133	50	2		
	M98B	50	3		
8	SIBX	150	2	51.8	45.5
	C7133	30	2		
	M98B	50	3		
	CuSO <sub>4</sub>	50	5	Activator and Frother dosages were constant for all tests	
	F200	50	1		

### Reagent suite 3 conditions and results

Wits: Non-sulphide Mimosa Mine ore processing -FLOTATION TEST SHEET					
<b>PROJECT:</b> Non-sulphide Mimosa Mine ore flotation					
<b>Procedure:</b> Rougher rate tests					
<b>Operating conditions</b>					
<b>Grind:</b> 60% - 75µm					
<b>Feed:</b> 1.16kg in a 3 L flotation cell					
<b>Make up water</b> Distilled water modified by addition of ionic salts					
<b>pH:</b> 9					
<b>Reagent Conditions</b>					
<b>Reagent Scheme 1</b>					
<b>Purpose:</b> PGM recovery/grade investigation using SIBX and AM810 collector suite					
Test number	Reagent name	Dosage (g/t)	conditioning time (min)	3E Recovery (%)	3E grade (g/t)
1	SIBX	100	2	45.7	61.7
	Hydroxamate (AM810)	150	2		
	M98B	100	3		
2	SIBX	100	2	63.1	18.7
	Hydroxamate (AM810)	150	2		
	M98B	50	3		
3	SIBX	150	2	52.6	32.41
	Hydroxamate (AM810)	150	2		
	M98B	100	3		
4	SIBX	150	2	47.3	33.29
	Hydroxamate (AM810)	150	2		
	M98B	50	3		
5	SIBX	100	2	42.6	68.01
	Hydroxamate (AM810)	100	2		
	M98B	100	3		
6	SIBX	100	2	29.1	28.56
	Hydroxamate (AM810)	100	2		
	M98B	50	3		
7	SIBX	150	2	52.7	53.78
	Hydroxamate (AM810)	100	2		
	M98B	50	3		
8	SIBX	150	2	44.2	52.37
	Hydroxamate (AM810)	100	2		
	M98B	100	3		
	CuSO <sub>4</sub>	50	5	Activator and Frother dosages were constant for all tests	
	F200	50	1		

## Detailed optimization results

### Optimisation 1 (SIBX 100g/t, AM810 60g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	41.7	3.9	3.9	30.30	11.70	0.48	42.48	67.0	41.3	49.5	57.0
Ro Tails	1021.0	96.1	100	0.61	0.68	0.02	1.31	33.0	58.8	50.5	43.0
Head calc	1062.4	100		1.77	1.11	0.04	2.92	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

### Optimisation 2 (SIBX 100g/t, AM810 70g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	77.3	7.3	7.3	16.40	8.11	0.27	24.78	70.8	59.1	51.4	66.3
Ro Tails	985.0	92.7	100	0.53	0.44	0.02	0.99	29.2	40.9	48.6	33.7
Head calc	1062.3	100		1.68	1.00	0.04	2.72	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

### Optimisation 3 (SIBX 100g/t, AM810 80g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	69.0	6.5	6.5	19.10	9.92	0.34	29.36	70.7	56.1	51.8	64.7
Ro Tails	993.5	93.5	100	0.55	0.54	0.02	1.11	29.3	44.0	48.2	35.3
Head calc	1062.5	100		1.75	1.15	0.04	2.95	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

### Optimisation 4 (SIBX 100g/t, AM810 90g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	90.78	8.5	8.5	14.90	7.73	0.22	22.85	75.7	61.6	50.7	70.0
Ro Tails	970.90	91.4	100	0.45	0.45	0.02	0.92	24.2	38.3	49.3	30.0
Head calc	1061.7	100		1.68	1.07	0.04	2.79	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

### Optimisation 5 (SIBX 115g/t, AM810 60g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	70.3	6.6	6.6	19.00	9.69	0.31	29.00	72.1	58.9	52.3	66.8
Ro Tails	992.0	93.4	100	0.52	0.48	0.02	1.02	27.9	41.1	47.6	33.2
Head calc	1062.3	100		1.74	1.09	0.04	2.87	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

### Optimisation 6 (SIBX 105g/t, AM810 60g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	78.1	7.4	7.4	17.30	8.40	0.28	25.98	73.3	59.2	52.6	67.8
Ro Tails	984.0	92.6	100	0.50	0.46	0.02	0.98	26.7	40.8	47.3	32.2
Head calc	1062.1	100		1.74	1.04	0.04	2.82	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

### Optimisation 7 (SIBX 96g/t, AM810 70g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	85.6	8.1	8.1	14.70	6.98	0.23	21.91	71.6	54.5	50.2	64.8
Ro Tails	975.5	91.8	100	0.51	0.51	0.02	1.04	28.3	45.4	49.7	35.1
Head calc	1061.1	100		1.66	1.03	0.04	2.72	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

### Optimisation 8 (SIBX 86g/t, AM810 80g/t)

Product	Mass distribution			Fraction Assays				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt (g/t)	Pd (g/t)	Au (g/t)	3E	Pt	Pd	Au	3E
Ro Conc	82.8	7.8	7.8	17.90	9.44	0.26	27.60	78.5	69.3	52.3	74.7
Ro Tails	975.8	91.8	100	0.41	0.35	0.02	0.78	21.2	30.3	47.4	24.9
Head calc	1058.6	100		1.78	1.06	0.04	2.88	100	100	100	100
Head Meas	1160			1.47	1.05	0.05	2.56				

## Detailed kinetic results

Product	Mass			Fraction Assays (g/t)				Distribution (%)			
	(g)	indiv (%)	Cum %	Pt	Pd	Au	3E	Pt	Pd	Au	3E
RoConc 1 min	7.88	0.68	0.68	47.60	15.9	1.14	64.64	22.77	12.71	8.60	18.61
RoConc 3 min	12.88	1.11	1.79	20.80	7.76	0.32	28.88	16.26	10.13	6.36	13.58
RoConc 7 min	24.01	2.07	3.86	11.70	5.62	0.17	17.49	17.06	13.69	7.29	15.34
RoConc 15 min	39.32	3.39	7.25	6.82	4.53	0.15	11.50	16.28	18.07	12.08	16.52
RoConc 20 min	21.46	1.85	9.10	4.91	4.27	0.1	9.28	6.40	9.29	10.11	7.27
Head Measured	1160			1.42	0.85	0.09	2.36				

Product	Mass			Cum Assays (g/t)				Cum Distribution (%)			
	(g)	indiv (%)	Cum %	Pt	Pd	Au	3E	Pt	Pd	Au	3E
RoConc 1 min	7.88	0.68	0.68	47.60	15.90	1.14	64.64	22.77	12.71	8.60	18.606
RoConc 3 min	12.88	1.11	1.79	30.97	10.85	0.63	42.46	39.03	22.84	14.97	32.19
RoConc 7 min	24.01	2.07	3.86	20.64	8.04	0.38	29.07	56.09	36.53	22.26	47.53
RoConc 15 min	39.32	3.39	7.25	14.18	6.40	0.27	20.85	72.37	54.59	34.34	64.05
RoConc 31 min	21.46	1.85	9.10	12.29	5.97	0.24	18.50	78.76	63.89	44.45	71.32
Head Meas	1160										

## Statistical analysis of variance

ANOVA for reagent suite 1

Source of Variation	Sum of Squares	D.O.F	Mean Square	F <sub>0</sub>	P-Value
B	121.91	1	121.91	14.97	6.14E-03
C	202.16	1	202.16	24.83	1.60E-03
AB	201.67	1	201.67	24.77	1.61E-03
Error	32.57	4	8.14		
Total	558.31	7	79.76		

ANOVA for reagent suite 3

Source of Variation	Sum of Squares	D.O.F	Mean Square	F <sub>0</sub>	P-Value
A	37.36	1	37.36	8.11	2.47E-02
B	242.86	1	242.86	52.75	1.68E-04
AB	148.42	1	148.42	32.23	7.53E-04
BC	77.07	1	77.07	16.74	4.62E-03
ABC	291.02	1	291.02	63.21	9.49E-05
Error	9.21	2	4.60		
Total	805.93	7	115.13		