AN INVESTIGATION OF THE EFFECT OF ELEMENTAL COMPOSITION AND GOLD GRAIN SIZE ON GOLD ANALYSIS AND RECOVERY AT OTJIKOTO GOLD MINE

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Abstract

Gold extraction from different type of ores is largely driven by elemental compositions and gold particle size and distribution. Another major factor that contribute to inaccurate gold analysis/ estimation is the nugget effect (presence of gold particulates in different sizes and shapes). Therefore the purpose of the study was to quantify and evaluate the effect of different elements on gold extraction/analysis, establish gold particle size distribution and determine the most suitable analytical technique that will effectively give accurate gold values in different samples from different host rocks in the Otjikoto mine gold deposit or other nugget gold ores. Sulphur (when present at the concentration of more than 2%), Iron (when present at the concentration more than 4%) and copper (when present at concentration at concentration more than 10 ppm) were found to be the major elements that cause concerns with both leaching and fire assaying. It was also determined that gold in both Otjikoto and Wolfshag occurs as nuggets and for all the samples analysed more than 50% of gold is reporting in +106 µm fractions. The three gold analysis techniques used found that Leachwell assisted cyanide leaching is the ideal method for gold analysis as it is mineralogical/elemental independent, less prone to errors and gave results which are the average of the other techniques used. Aqua regia, direct fire assay, cyanide leaching without leachwell and mixed acids techniques were found to be very sensitive to elemental compositions and gold particle sizes (nugget effect). It was discovered also that when analysing Leachwell solutions on the AAS, precipitates forms that clog the burner and the nebuliser, hence it is recommended that DIBK extraction is performed to prevent clogging.

List of publications/conference proceedings

N/A

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List of abbreviations and acronyms

Abbreviation and acronym	Definition
SFA	Screen Fire Assay
Aregia	Aqua Regia
Lwell	Leachwell
DFA	Direct Fire Assay
Macids	Mixed/multi Acids
ppm	parts per millions
PSD	particles size distributions
ACE	Accelerated Cyanide-leach Extraction
MIBK	Methyl Isobutyl Ketone
DIBK	Di-Isobutyl Ketone
AAS	Atomic Absorption Spectroscopy
CRM	Certified reference Material

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DECLARATIONS

I, Fillipus Haulofu Thomas, hereby declare that this study is my own work and is a true reflection of my research, and that this work, or any part thereof has not been submitted for a degree at any other institution.

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Chapter 1. Introduction

1.1. Background of the study

The discovery of the Otjikoto gold deposit in 1999 on the Northern zone of the Damara belt with two orebodies, the Otjikoto and Wolfshag deposits, led to the construction of the second gold mine in Namibia adding to Navachab gold mine located on the central zone of the Damara belt (1). In order to keep the mine operational and profitable for the estimated 12 years life time (1); accurate gold analysis and optimized metallurgical processing of gold ore need to be considered. Separation and recovery of gold from gangue in a commercial viable method mainly depends on mineralogical factors such as, gold particle size and association of gold with other minerals (combination of elements) (2,3, 4). Minerals are made up of either one element or more, hence a composition of elements can be referred to as a minerals. Gold bearing ores vary in composition and mineralogy and thus they are classified as free-milling ores and/or refractory ores. Knowledge of the elemental composition of an ore and understanding of the local geology of the deposit is required for sampling protocols design, sample collection, selection of suitable sample preparation and assaying protocols, selection of the appropriate quality controls, prediction of the response of the ore to various metallurgical processing of the ore and determine the nature of gold problems likely to be encountered (2, 4, 5, 6). The elements/minerals that are mostly found associated with gold and create difficulties during gold analysis and recoveries include; antimony, sulfur, tin, telluride, pyrite (FeS_2), arsenopyrite ($FeAsS$), pyrrhotite (Fe_7S_8) and much more, causes for example gold loss (2, 5). Even though there are those factors mentioned above that cause difficulties, there are different known techniques such as fire assay, leaching and acids digestions that can completely recover gold provided the composition is known (7). Hence, this study considered some common mineralogical factors affecting gold recovery and analysis and recommend the investigated routes of overcoming those factors.

1.2. Statement of the problem

Very limited (if there is any) research on the particle size of gold as well as the associated elements and minerals which can interfere with gold analysis and recovery have been conducted on the Otjikoto gold mine ore. This resulted in challenges with accurate gold estimation and significant varying gold grade between the resource estimate and the actual gold produced. The significant differences in mineralisation between the Otjikoto and Wolfshag deposits within the Otjikoto mine gold deposit (8) could cause challenges when recovering and analysing for gold. It is noted that fire assay-gold analysis technique currently used by Otjikoto site laboratory is labor intensive, time consuming, consumable demanding, mineralogy influenced and has many handling steps, where each handling step increases the chance of errors (9). Hence a better technique that can efficiently overcome these challenges is required. On the other hand, the cyanide leaching method used for gold recovery; its recovery efficiency largely depend on the elemental composition of the ore and also the gold particle sizes. The study will address the aforementioned problems by investigating the nature and occurrence of gold in the Otjikoto and Wolfshag deposits as well as elements which influence gold analysis and recovery.

1.3. Objectives of the study

(a) To determine and quantify elements (elements making up minerals) which are known to interfere with gold recovery and analysis.

(b) To establish the gold particle size distribution in the deposit.

(c) To determine the most suitable analytical technique for gold recovery/analysis for nugget gold ores.

1.4. Hypothesis of the Study

Fire assay (Pyro metallurgy) is the best technique to recover gold from ores containing minerals or elements that are known to affect gold recovery such as copper, Iron, Sulphur, arsenic, nickel and zinc just to mention few as well from ores where gold occurs as nuggets.

1.5. Significance of the study

Knowledge of the ore matrix, grain size distribution and presence of minerals or elements which appears to interfere with gold recovery and analysis will improve the accuracy of grade control and optimize gold recovery [2]. The results of this study will contribute new knowledge on the appropriate methods required to enhance gold recovery and analysis from similar gold bearing deposits in Namibia and elsewhere and form a foundation and guide to sampling, sample collection and preparation as well as selection of suitable analytical and recovery techniques. Furthermore, the findings of this study will ensure that the existing gold mines will remain in operation for a longer period and also be a guide to discoveries of other gold mines through explorations, thus contributing to reduction of unemployment and meet the high world gold demand.

1.6. Limitations

Due to continuous mining activities, samples were collected from mineralized rejects stored at the laboratory. Samples were collected/sampled by reverse circulation sampling technique (2 meters) rather than diamond drill sampling technique, contributing to sample dilution from neighboring barren materials and elemental loss due to massive dust loss. The sample weight was limited to 10 kilograms.

1.7. Delimitations

In this study only samples from albitite, marble, amphibole and fault breccia host rocks were investigated. Furthermore only three commonly used gold analysis techniques were investigated and compared.

Chapter 2. Literature Review

2.1. Outline of Geology and Mineralization of Otjikoto mine gold deposit.

Geological understanding of an ore deposit is the key to improved confidence in mining operation. Effective geological data collection can assist in development of a representative and quality sampling/drilling program as well as effective handling of samples and the use of the right assay preparation methods (10). Therefore it is important to understand the geology of the Otjikoto mine gold deposit.

The Otjikoto mine gold deposit is sub-divided into two deposits namely the Otjikoto and Wolfshag deposits. Gold in the Otjikoto and Wolfshag deposits is hosted by a sheeted vein system in the amphibole-albitite hornfels and albitite zones of units. The vein system is dominated by thin veins with varying proportions of iron rich minerals of pyrite, magnetite and pyrrhotite (8, 11, 12). Bruce et al (8) indicated that the Otjikoto deposit was a result of the metamorphism of the pelitic sediments and greywackes of the Okonguarri Formation which were metamorphosed to metagraywacke and garnet-biotite schist up to amphibolite facies grade.

Generally it is known that gold occurs in a variety of hydrothermal deposits and due to its chemical inertia and high density it can be concentrated in placer deposits, where it may form large nuggets (13, 14). Ross and David (15) indicated that gold at Otjikoto mine occurs as native gold grains on the margins of veins and between mineral grains with grains up to 650 microns and strongly associated with sulphide minerals of pyrrhotite and pyrite.

Gold mineralisation at Otjikoto is hosted by thick massive equigranular amphibole-albite granofels units with thin interbedded garnet-amphibole-albite schist units dominated by either pyrrhotite or pyrite with or without magnetite (16, 17). It is further noted by Ross and David (15) that the carbonate is generally dolomite/ferroandolomite rather than calcite. Bruce (17) pointed out that the deposit consists of magnetite-pyrrhotite-pyrite veins and magnetite-pyrrhotite-carbonate veins associated with some high temperature ganert-pyrrhotite veins. also draw attention to that based on geological studies, it is believed that the rocks were mafic greywacke and interbedded semi-pelitic sediments which were rich in iron and relatively carbonate poor before alteration and metamorphism. Figure 1 below shows typical veining in the deposit, a) magnetite-pyrrhotite-carbonate veins, b) a garnet-pyrrhotite vein and c) a microscopic image of a pyrite-magnetite veins (16, 17, 15);

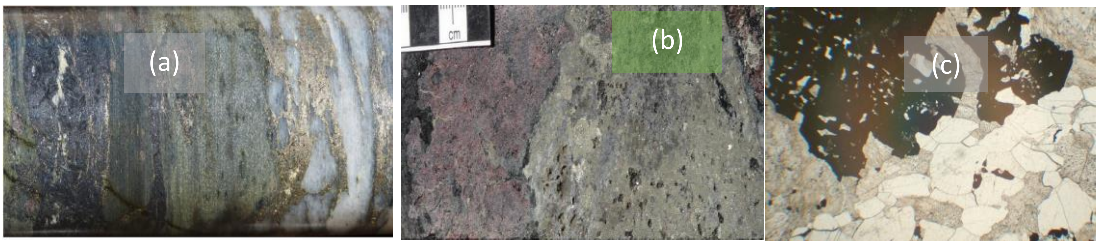


Figure 1: (a) sheeted magnetite-pyrrhotite-carbonate vein in the Otjikoto deposit, (b) High temperature garnet-pyrrhotite vein and (c) microscopic image of pyrite-magnetite concentrated in a vein (15, 16, 17).

On the other hand, gold mineralisation in the Wolfshag deposit is hosted by relatively thin massive albitite units interbedded with banded albitite and thin marble units. The albitite is dolomite rich with practically no mafic minerals (15, 17). It is believed that the first rocks were probably carbonate rich greywacke with interbedded aluminium and iron deficient marl and dolomitic limestone. The deposit consists of coarse grained pyrite-calcite veins and pyrite-calcite-magnetite brittle veins as illustrated in figure 2 below (16, 17, 15);

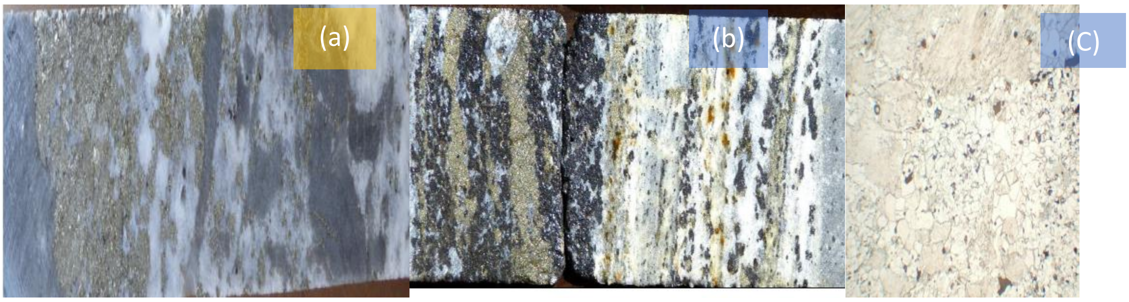


Figure 2: (a) Pyrite-calcite-magnetite brittle vein, (b and C) Normal and microscopic image of coarse grained pyrite-calcite-magnetite vein.

The significant differences in mineralisation such as host rocks, elements and minerals making up veins between Otjikoto and wolfshag deposit within the Otjikoto mine gold deposit could cause challenges when recovering and analysing for gold and factors that may contribute to the challenges need to be investigated and well understood.

2.2. The factors affecting gold analysis and recovery

From the perspective of gold analysis and recovery, gold ores can be classified as free-milling ore or refractory ore (5, 18, 19, 20). Free-milling ore is where the gold is present as native gold and can easily be liberated from its matrix, while in the refractory ore, gold is found combined with others elements such as silver or telluride or as inclusions in sulphide mineral structures and it is not easy to liberate it from the matrix. The extraction of gold from the two classes of gold ores is largely driven by mineralogical factors such as (20, 5);

a. Gold particle size.

b. Association of gold with other elements which can interfere with its recovery or analysis.

Knowledge of ore mineralogy helps to address problems encountered during gold processing or analysis, provide useful information on process and analytical methods selection, improved gold recovery, improve accuracy of gold analytical results and reagents consumption optimization (21, 22). The two factors that influence gold extraction and analysis are discussed in detail below;

2.2.1. Gold particle size

According to Dominy, Xie and platen (6), gold particle size and their distribution affects the sampling characteristics of the deposit and characteristics of the samples, and also affects how effective sampling and assaying will eventually be. They further pointed out that, the samplability of a deposit ranges from relatively simple for fine-grained disseminated gold particles, through to extremely difficult for coarse nuggety mineralisation. Presence of gold as nuggets in the ore is believed to be the major contributor to the fundamental sampling errors, grouping and segregation errors and preparation errors as the particles or distribution of gold in the parent unit does not have same probability of being selected for inclusion in the sample and sub-sample (23, 10, 24). A common tool applied to optimise sampling errors is the equation proposed by Pierre Gy known as Gy’s equation as follows (25, 26, 27, 28, 6, 29, 24, 30);

$$w = \frac{Cu^3}{S^2}$$

Whereby;

W= weight of the sample required in grams

u – Top particle size in centimetres

S^2 *= Sampling relative variance square*

C = Sampling constant for particular materials to be sampled.

The sampling constant (C) used in the equation depends on the properties of the material being sampled and it depends on four parameters namely; particle shape factor (*f*), particle size distribution factor (*g*), liberation factor (degree to which gold is entrenched in other rock forming minerals or matrix) and the mineralogical composition factor. Once sampling can be done correctly to provide representative samples regardless of the gold particle sizes and their distribution with high probability of the nugetty unevenly distributed particles included in the sample, then the samples can be confidently taken or submitted for further preparation for analysis.

During the sample preparation stage where heavy and big field samples are being reduced to small manageable analytical laboratory sub-samples, incorrect handling, mixing, grinding and sub-sampling can result in loss, contamination or alteration of the sample in such a way that it is no longer an accurate representation of the material sampled (23). When preparing samples containing unevenly distributed native metals of varying sizes, a special sample preparation is required to separate the metallic grains before assaying (25).

In order to assume the precision of the analytical method and obtain the sample size that will ensure sub-sampling uncertainty will not exceed 1 per cent, as well as to minimise the errors caused by gold particle sizes and their distribution in an ore sample, optimum sub-sample is obtained by applying Visman's theory where constant A and B are calculated (22, 31). A is the homogeneity constant and B is the segregation constant. Optimum sample weight is obtained by the following equation;

$$W_{opt} = \frac{A}{B}$$

W_{opt} is the optimum sub-sample weight.

Once a laboratory sample has been prepared, the next question is on how much material should be used in an analysis based on the analytical method being used that will minimise the effect of gold particle size in the sample. To answer that question the Ingamell theory is applied to calculate the weight required.

Ingamell's equation is as follow;

$$K_s = \frac{(100S)^2}{(X_{av})^2}\, w$$

K_s is the weight of sample in grams required to ensure that the analytical-sample's uncertainty would not exceed 1 per cent and is obtained by analysing one sample of a known weight (w) repetitively and use the obtained assays to obtain the mean (X_{av}) and the standard deviation (s) from which the required sample size will be obtained (22).

The above equations are some of the measures in place to overcome nugget effect or any other problem encountered in gold analysis that is due to the sizes of the gold particles as well as their distribution in the sample/ore. The sampling process, inclusive of sample collection, preparation and assaying is a critical component to all stages in a mining operation as it forms the basis for mineral resource and reserve estimate (32, 33, 34). Correct sampling and assaying gives accurate assays that will be used in metal accounting and will determine the gold recovery efficiency.

Gold grain size is believed to be a significant factor driving the efficiency of gold recovery processes. This is due to the fact that coarse gold may be incompletely leached, as the gold particles may get trapped upstream in the cyanidation circuit (35, 3). On the other hand when gold is very fine and associated with sulphide minerals, leaching process will yield poor gold recovery (3).

2.2.2. Association of gold with other elements/minerals which can interfere with its recovery or analysis.

Gold occurs principally as free gold in the form of flakes, scales, crystals or nuggets of native metal or found associated with other minerals such as silver, telluride or with sulphide minerals of arsenic, copper, iron, silver and other metals (4). There are also a few minerals that are listed as gold minerals, in which small amounts of gold substitute for other metals, such as aurostibite ($AuSb_2$), calaverite ($AuTe_2$) and sylvanite ($[Au, Ag]Te_2$) which are regarded as important ores of gold (4). Furthermore Sulphide ores, silica and sulphur-bearing minerals such as sphalerite (ZnS), chalcopyrite ($CuFeS_2$), cinnabar (HgS), galena (PbS), pyrite (FeS_2), reagar (AsS), arsenopyrite ($FeAsS$), ellisite (Tl_3AsS_3), and others contains varying amount of native gold occluded in them (36, 37, 20). It is noted that gold in pyrite can be several hundred ppm while in arsenopyrite it can be over 10 000 ppm (4). Similarly Woo (4) noted that oxide ores, silica (SiO_2), limonite ($FeO.OH.nH_2O$), calcite ($CaCO_3$), clay minerals and iron oxides can contain varying amount of gold. Almost all the minerals/elements found associated with gold create challenges during gold analysis and recovery (24, 25).

Apart from the challenges caused by the minerals found associated with gold, studies have also pointed out that the wide variability of concentration and heterogeneous distribution in the rock samples as well as nugget effect also present special challenges during gold analysis and recovery (34, 38, 32). Therefore the analytical methods require a large sample and highly sensitive instrumental technique for a successful analysis (26, 39). To fully appreciate and have confidence in the analytical method being used, an understanding on the properties and mode of occurrence of gold and its associated elements is required as well as the capability of the analytical method to quantitatively recover gold from the ore being analyzed (39).

2.3. Gold analysis/recovery techniques and methodologies.

The common analytical techniques for determining or recovering gold from geological samples or ores are; Fire assay, Cyanide leach and Aqua regia or mixed acids digestion (26, 40). Cyanide leaching is used both at the laboratory scale for gold analysis (Bottle roll) and at larger scale for gold recovery from thousands of tons of ore.

The common methodologies for gold recovery and analysis mentioned above are all mineralogical dependent/influenced to different extents. When developing or choosing an analytical method the selectivity should be based on the method's ability to determine the analyte without interferences from other substances present in the analyte matrix (40, 25). The minerals that affect the analytical methods and the extended to which they affect the methods will be discussed in detail.

2.3.1. Fire Assay

Fire assaying is the industry standard process for obtaining analytical gold and platinum group elements from all sample types including drill core, soil and chip samples. The technique has been used for centuries (4). It can as well be defined as a quantitative determination in which a metal or metals are pre-concentrated into a lead button and separated from impurities by fusion process (4, 41, 42, 43). The gold once recovered from impurities, is weighed to determine the amount present in the original sample. The advantages that makes fire assay an industry standard process includes(4, 40, 39);

- It provides a quick elimination of all the associated gauge minerals at relatively low cost.
- It uses a large quantity of sample per analysis, reducing sampling uncertainties.

- It is effective for collection of gold, silver, platinum and palladium in one crucible fusion.

Fire assay is based on four principles that makes it suitable for gold analysis as follow (4):

i. The high solubility of precious metals in molten metallic lead and their almost insolubility in slags of a suitable composition, hence molten lead serves as a collector for precious metals and the slag serves as a collector of the impurities.
ii. The difference in specific gravity of the molten lead and the molten slag cause easy separation of the two liquids.
iii. Lead can practically completely be removed from the precious metals by careful controlled oxidation fusion involving differential filtration.
iv. The separation of silver from gold is possible because of the solubility of silver and completely insolubility of gold in dilute nitric acid.

Fire assaying involves fluxing, fusion, oxidation fusion (cupellation), parting and dissolving and end with weighing the gold or analyzing for gold using different instruments such as AAS, ICP-OES/MS and others. The efficiency of fire assay is dependent on the minerals found associated with gold.

Fluxing and fusion is when a pulverized sample is thoroughly mixed with a flux consisting of litharge (PbO), soda ash (Na_2CO_3), borax ($Na_2B_4O_7$), silica (SiO_2), flour, niter and a small amount of silver(39, 44, 43, 45, 46, 41, 42, 47, 48, 49, 4). The amount and ingredients to add to the sample depend on the mineralogy of the sample. The mixture of the sample and fluxes (charge) is then fused in a furnace at 1050°C for about 40 to 60 minutes. During fusion litharge is reduced to elemental lead which will collect the precious metals. When oxide minerals are present, they will oxidize the metallic lead once reduced and ending up without a lead button. If Sulphur is present or sulphides, and there is no sufficient oxidizing agents to oxidize it, it will report in the lead button causing a large dark brittle lead button and also interfere by forming a mixture of metallic sulphides known as a matte (25), above the lead button. The matte always retain some of the gold and silver and when the lead button is hammered the matte is brittle, it falls off causing gold loss.

Arsenic and antimony forms metallic arsenides and antimonides known as speisses (44, 45, 50, 51) which retain some of the gold and silver. The speiss is hammered off together with slag causing gold loss (52, 42). Presence of copper in the lead button cause the lead button to be brittle (45). When chromium is present in the charge in large amount, the charge will not fuse and chromium can also occlude gold (45).

Cupellation- the lead button obtained from fusion process is hammered into a cubic shape and then placed in a pre-heated cupel made of bone ash, Portland cement or magnesia (25, 39). The cupel containing the lead button is then placed in a furnace at 950°C for about 30 to 40 minutes. Timing can be set based on the size of the lead button (1 gram per 1 minute). During cupellation lead is converted to lead oxide of which 98.5% is absorbed by the cupel and 1.5 % volatilized (4). When this process is carried out to completion, the gold, silver and most of platinum and associated metals that may be present are left on the surface of the cupel, in the form of a metallic bead. One of the causes of cupellation loss is the impurities in the lead button. These impurities and their effects on cupellation are summarized below in table 1 below (4, 41);

Impurity	Effect on cupellation
Antimony & Arsenic	Interfere only when present in a content more than 2%, they cause the formation of scoriers that immediately solidify and expand. Cause the cupel to split and gold gets lost into the cupel cracks.
Bismuth	Impact an orange-yellow stain on the cupel around the bead and cause silver and gold loss due to increased absorption loss into the cupel.
Copper	Causes slight silver and gold losses by cupel absorption, Large content of copper in the button can cause lead button to freeze or to flatten out and look coppery.
Iron, Cobalt & Manganese	Cause brown or black scoriers.
Nickel	Nickel will form scorier covering the bead completely and causing it to freeze.
Selenium & Tellurium	Wet the surface of the cupel, causing gold absorption by the cupel, Cause poor coalescence of the dore bead, resulting in many small beads in the cupel, which is regarded as primary mechanism for gold losses.
Tin	Oxidizes to tin oxide forming yellow scorier during cupellation, sufficient tin in the lead button, its scoriers can choke off the oxygen supply and stop cupellation.
Zinc	Burns and form a dense white fumes of ZnO which may condense on the cupel, covering the molten metal, chocking off oxygen supply and cause the bead to freeze.

Table 1: Effects of elements on cupellation.

Parting and dissolving – After cupellation, in the analysis of gold, the resulting prill is processed further using two available options. Option one is to dissolve the silver into dilute nitric acid and leave the gold called parting (48). The remaining gold is weighed (gravimetric method). The other option is to dissolve the silver and the gold in aqua regia and quantify it by atomic absorption spectrometry or ICP-OES (26).

2.3.2. Cyanide Leach

Gold leaching using cyanide, which is also referred to as MacArthur-forrest process or cyanidation, was introduced in 1887 by John Stewart MacArthur as a processing technology for gold and silver ores (53, 54). The method is believed to be the most effective, simple and economical process for treating a wide range of gold bearing ores. This introduction came after the discovery that gold dissolves in aqueous solution of cyanide in 1783. The equations illustrating the dissolution of gold in cyanide solution are given below (55, 53, 56, 4);

It is suggested that the equation for gold dissolution in cyanide consists of two steps.

$$2Au + 4CN^- + O_2 + 2H_2O \rightarrow 2Au(CN)_2^- + 2OH^- + H_2O_2 \ldots\ldots\ldots (i)$$

$$H_2O_2 + 2Au + 4\ CN^- \rightarrow 2Au(CN)_2^- + 2OH^- \ldots\ldots\ldots (ii)$$

From the equations, it is believed that peroxide acts as an intermediate product. The overall equation referred to as elsner's equation is the sum of the two equations as follows;

$$4Au + 8CN^- + O_2 + 2H_2O \rightarrow 4Au(CN^-)_2 + 4\ OH^- \ldots\ldots\ldots (iii)$$

The use of cyanidation in 1890 in Witwatersrand, South Africa recovered up to 96% of gold from the ore (53). Today it is believed that more than 75% of the gold produced worldwide is extracted by cyanidation process (5), where grounded materials are reacted with cyanide in oxygen rich/enriched slurry. The parameters that dictate the rate of gold dissolution/leaching as well as the rate of reagent consumption is the mineralogy of the ore being processed (57).

Several investigations were conducted to study the mineralogical factors that affect gold dissolution rate in cyanide. It was found that factors affecting gold dissolution rate includes particle size, degree of liberation of gold particles from the ore matrix, alkalinity of the solution, cyanide concentration and content of impurities (58, 59, 60). It was also discovered that oxygen is one of the most important variables in cyanidation. It reduces the overall cyanide consumption by decreasing the contact time between the cyanide and non-desirable reactants species (53). Increased oxygen concentration causes reduction in free sulphide ions in solution and passivation of reactive sulphide minerals resulting in accelerated leaching kinetics (53).

Cyanide leaching can be used at a small scale in mineral testing laboratories where large volume of samples are being tested and referred to as bulk cyanide leachable gold test (bottle roll). The method is believed to work well for samples where gold is known to be free and work poorly for samples where gold is associated with other minerals or it can be said that the method has limitations with regard to ore mineralogy. In refractory ores (where gold is associated with other minerals), the gold is contained within the crystalline structure of sulphide minerals such as pyrite, pyrrhotite, arsenopyrite and tellurides (19). Since the gold in sulphide minerals is in their lattice they cannot be ground fine enough to liberate the gold particles and for cyanide to leach gold. The cyanide solution must come into direct contact with gold in order to dissolve gold. Similarly, in other refractory ores such as silicates and carbonates the gold is encapsulated in the lattice of the minerals. Carbonaceous ores allow cyanide to dissolve gold, but quickly adsorb gold back onto the active carbon in the ore (54, 53, 61).

The basic cyanide leach technique is based on leaching large samples of about 0.5 to 5.0 kilograms in a 0.25% to 1 % sodium cyanide solution (39), but this can vary from operation to operation. The cyanide solution pH should be maintained at 9 to 11 by adding lime or sodium hydroxide to overcome the chemical loss of cyanide that may affect the extraction process (35). The extraction process consists of mixing the sample with the cyanide solution with a pulp density of 50% solids in a bottle and rolled for 24 hours, allowed to settle before a portion of the clear liquor is extracted for analysis by atomic absorption spectrometry (AAS) or other available analytical in . The factor that affect the assay quality from bottle roll technique includes liberation grind size, mineralogy, amount of free milling gold and the pulp density (44, 35, 4).

(62). The method in some operations is referred or known as ACE which stand for Accelerated Cyanide-leach Extraction. Gold cyanide leaching using Leachwell 60x is slowly becoming a common method of analysis for gold especially when working with coarse gold deposits or highly heterogeneous materials (63, 35). Leachwell 60x is a reagent grade catalyst formulated for fast cyanide leach gold assaying. It is believed that when used with 5% cyanide the dissolution rate of gold, copper and silver is increased more than sixty folds (63). The leach rate of gold under appropriate conditions is known to be 1 µm per minute, therefore with the use of Leachwell 60 µm of gold can be leached in one minute. The advantages of using Leachwell for gold cyanide leach are as follow (61, 64)(4);

- Large samples can be assayed accurately and very quickly
- Poor reproducibility when coarse gold is present is minimized and meaningful results can be consistently produced.

- Gold, copper and silver can be read from the same sample.
- Preg-robbing by organics or clay is practically eliminated.
- The procedure is simple and requires no special skills and coarse gold is dissolved quickly.
- Leachwell 60x has built in oxidizing agent so that sealed jars can be used when leaching.

In general for both laboratory scale and mining gold extraction scale the following elemental composition affects cyanidation process even though there is little literature on investigation of their effect when using Leachwell (53, 55, 19, 61, 65, 54);

- If gold particle sizes are too large they may not be totally leached during rolling time or during their residence time in the tanks and hence will report to the tails.
- Copper dissolves in cyanide causing increased use of cyanide and when copper is present above 0.3% cause formation of copper cyanide complex ($Cu(CN)_2$) which inhibit the dissolution of gold in cyanide
- Zinc bonds with the cyanide to form zinc cyanide compounds consuming cyanide meant for gold dissolution.
- Nickel causes precipitation of gold from cyanide solution.
- In gold extraction, once the gold is leached into solution, gold is separated from the slurry by carbons in pulp, When elements such as Calcium are present they get adsorbed onto the carbons decreasing the number of active sites and this reduces the carbon's ability to adsorb gold.
- When pyrrhotite is present in an ore, trouble is usually experienced both in regard to cyanide consumption and gold extraction as it reduces both free cyanide and the oxygen content of the solution (61).

- High silver content in the ore may give poor gold extraction due to tarnishing of silver.
- Arsenic and antimony reacts with the cyanide and use up all of the excess oxygen.
- Iron sulphide and arsenic sulphide ores host different proportion of non-refractory and refractory gold. Refractory gold extraction can only be achieved by employing a pre-oxidation process prior to cyanide leaching.
- Antimony, bismuth sulphide and tellurides ore contain gold minerals such as aurostibite, maldonite and gold telluride which are slow-dissolving gold minerals.

2.3.3. Acids digestion

Acids digestions or wet digestion is one of the techniques used for gold analysis. Two common used wet digestion techniques are multi (four) acids digestion and aqua regia (two acids digestion). The method is simple, fast and inexpensive. It is noted that the ratio of the sample weight to the volume of acids and an appropriate sample mesh size are important factors, predominantly in the analysis of complex matrices (20, 64). It is said that incomplete wetting of the sample or when the particles of the analyte are occluded in the grains of other minerals and the acids cannot crack those minerals open to access the analyte particles can result in a non-quantitative recovery of the metals (43, 46, 39). Acids extraction effectiveness strongly depends on the chemical solubility of individual metals, their concertation and kind of matrix.

In aqua regia digestion, the sample is subjected to attack by a mixture of concentrated hydrochloric acid and nitric acid in a ratio of 3:1. Aqua regia is useful for gold and base metals dissolution even though it is considered as the weakest of the digestions due to its inability to attack silicate minerals (47, 66, 67). Nitric acid and reaction products in combination of nitric acid with hydrochloric acid such as nitrosyl chloride are strong oxidizers forming gold ions Au^{3+} in solution following dissolution of Au-bearing materials. On the other hand hydrochloric acid form strong aqueous chlorourate ($AuCl_4^-$) complexes allowing Au dissolution and oxidation reaction to progress (40) as indicated in equations below;

$Au_{(s)} + 3NO^{3-}_{(aq)} + 6\ H^{+}_{(aq)} \rightarrow Au^{3+(}{}_{aq)} + 3NO_{2(g)} + 3H_2O_{\ (l)}$……………………………………(iv)

$Au^{3+}_{(aq)} + 4Cl^{-}_{(aq)} \rightarrow AuCl^{-}_{4(aq)}$………………………………………………………………………………(v)

The two equations above illustrate how the combination of the two acids are able to dissolve gold since neither nitric acid nor hydrochloric acid alone can dissolve and keep gold in solution.

When gold is occlude in minerals such as alumino silicates, silicates, chromites, rutile, cassiterite, garnet, ilmenite, barite and other minerals that do not get attacked by aqua regia, the recovery of gold will not be 100% (68, 25). When these minerals are present then other digestion techniques such as multi acid digestion should be engaged.

Multi acids digestions is a very effective dissolution technique for a large number of mineral species and is suitable for a wide range of elements. Multi acid digestion use a combination of nitric acid (HNO_3), hydrofluoric acid (HF), Perchloric acid ($HClO_4$) and hydrochloric acid (HCl) (39, 69). Addition of hydrofluoric acid is required for sample's complete digestion as it dissolves silicate minerals in which gold may be encapsulated in and facilitate the liberation of gold. Perchloric acid has high boiling point than hydrofluoric acid, hence its addition ensure that once HF boils off, the sample redissolves and the metal fluorides are converted to salts that are more soluble. Nitric and hydrochloric acid serve the same purpose as in aqua regia (69).

Due to the fact that wet acid digestion does not involve pre-concentration and based on the mineralogy of the sample, the total dissolved solids can be high, which will definitely clog or interfere with the analysis on the analytical instruments such as AAS or ICP-OES (38). The clogging of instruments can result in low gold values obtained. On the other hand dissolved metals like Iron (Fe) interferes with gold analysis on the AAS due to relatively strong absorption line of iron 242.4 nm near the analytical line for gold 242.8 nm as iron can be picked up as gold resulting in high gold obtained values (70, 71). To reduce the matrix interference on gold analysis by acids digestions, gold is extracted from the solution using organic solvent such as MIBK (methyl isobutyl ketone) or DIBK (di-isobutyl ketone) together with Methyltrioctylammonium chloride (aliquat 336) (4, 72, 38, 25, 32). The digested solution is shaken with the organic solvent and then the organic solvent analyzed for gold. Since gold exist in solution as $AuCl_4^-$ the oxygen with unpaired electrons in the organic solvent tends to unite with proton (H^+) in acidic solution and react with $AuCl_4^-$, hence extracting gold from the acidic solution. The extracted solution is then analyzed for gold on the atomic absorption spectrometer or on ICP.

Chapter 3. Methodology

3.1. Research Design

This is a quantitative research study which focused on identifying and quantifying different minerals and elements. The flow chart below illustrates the design of the study.

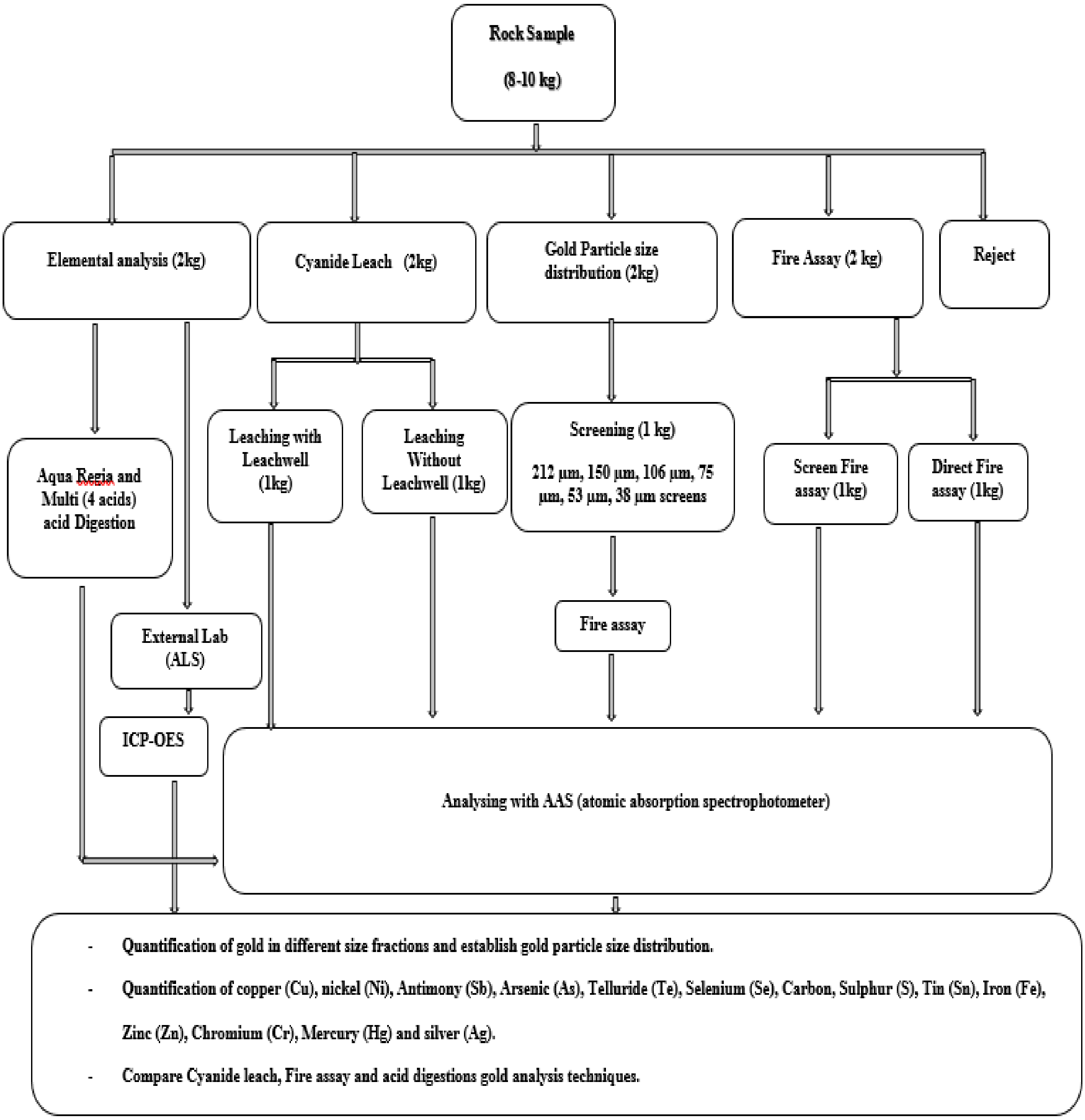


Figure 3: Project design flow chart.

3.2. Sampling/sample selection and Identification

Samples were handpicked from the laboratory grade-control rejects which were already analyzed by screen fire assay by the Otjikoto site laboratory. It is theoretically known that different minerals are colored differently due to their composition (73, 74, 75), hence sample selections was based on the color in order to cover a wide range of host rocks as well as different compositions of alteration zones. Samples ranging from whitish grey, yellow, reddish yellow, grey and dark grey in color, weighing between 8 kilograms to 12 kilograms were selected (See Appendix B). The samples selected for the study were confirmed by the geologists that they cover the full suite of the expected rock types in Otjikoto and Wolfshag.

Samples Identification: The samples were given new identification numbers as follow; B2G_XX_YY, where YY represent numbers from 1 to 30, XX- is the location the samples were sampled, where WS- indicates that the sample was taken/sampled from wolf-shag pit and OTJ indicates that the sample was taken/ sampled from Otjikoto pit.

3.3. Research Tools and instruments

Research tool/instrument	**Purpose**
Atomic Absorption Spectrophotometer (AAS)	Quantify gold in mg/L from prepared solutions.
Screen (4mm), Jaw crusher, splitter, cone blender & LM2 pulverizer.	Samples preparations and homogenization.
Drying ovens and furnaces	Samples drying, fusion, cupellation and annealing.
inductive coupled plasma optical emission spectroscopy (ICP-OES)	Quantify elements, other than gold.
Mixing Wheel	Rolling and mixing leaching bottles
Hot blocks/plates	Heating for dissolving and acid digestion.

Table 2: Research tools and instruments

3.4. Procedures

Sample preparations: The entire sample was screened through a 4mm screen and the oversize crushed to ensure that 87% of the sample is passing through a 2mm sieve/screen. After sieving and crushing, 10 % v/w of 3% v/v methanol solution was added to the sample to reduce the chances of segregation due specific gravity or particle size differences during blending. The samples were transferred to a conical Gilson/Sepor 20 litre blender (see figure 4) and blended for 30 minutes. The samples were then split into 4 x 2 kg portions and transferred to labelled plastic sample bags.

If the sample mass exceeded 8 kilograms the excess sample was returned to its original sample bag for storage. Figure 4 below shows a conical cone blender, which is one of the effective ways to fully homogenise the samples before splitting.

Figure 4: conical Gilson/Sepor 20 litre Cone blender

The cone blender consists of a motor that rotate the cones. The samples are placed and enclosed inside the cones, once the motor is turned on it moves the cones allowing the sample inside to move in an up-down motion.

3.4.1. Elemental and mineralogy analysis

The 2 kilograms portion split for elemental and mineral analysis, was further split into 2 times 1-kilogram samples, One 1-kg portion was sent to ALS laboratory Johannesburg for gold and elemental analysis and the remaining 1 kg portion was dried for 4 hours at 105 °C in the drying oven. After drying the entire 1 kg sample was pulped for 6 minutes using LM2 pulveriser, homogenised by bottle blending and packaged into brown geo-chem envelops. These samples were analysed for gold using aqua regia and 4 acids digestions technique. 10 grams of the sample for each procedure was weighed out into porcelain crucibles, mixed with 1 gram of ammonium nitrate and roasted for an hour in a bench top muffle furnace at 550°C [5]. Figure 5 below show the samples before roasting and after roasting.

Figure 5: Samples before roasting and after roasting

a. Aqua regia digestion

The roasted samples were transferred to a 400 ml beakers and 50 ml of freshly prepared aqua regia (1 part nitric acid to 3 parts hydrochloric acid) solution added. 1 gram of sodium chloride was added to each sample to stabilise the gold chloride complex during evaporation (76, 77). Each beaker was covered with a watch glass and heated for 4 hours on a hot plate, enough aqua regia was added to maintain free acid level at least 1 cm above the sample. The watch glasses were removed and the content evaporated to near dryness. 50 ml of 3 M HCl was added and solution warmed, after boiling it was removed and filtered. The residues were washed with 50 ml 3 M hydrochloric acid. To the filtrate 50 ml of DIBK-336 was added and shake for 5 minutes. Two phases formed, the aqueous layer and organic layer, the organic layer was collected for F-AAS gold determination and the aqueous layer was analysed to see if there was gold left after extraction process and then discarded.

b. Multi/Four acids digestions.

The roasted sample was transferred to a 400 ml beaker and 20 ml of concentrated hydrochloric acid plus 10 ml of concentrated hydrofluoric acid added. The content was swirled to ensure complete mixing. The content was heated gently until the volume was about 10 ml. Then 25 ml of decomposing acid (4 parts of nitric acid to 1 part of perchloric acid) was added, mixed and the content evaporated to almost dryness. 50 ml of concentrated hydrochloric acid was added and the content boiled. After boiling the content was allowed to cool and transferred to a 100ml volumetric flask and topped up to volume. The solution was further transferred to a 200 ml volumetric flask, 50 ml of DIBK-336 added, shaken for 5 minutes and the organic and aqueous layers analysed for gold using AAS.

c. ALS Multi-elements analysis procedure

A prepared sample (0.25 g) is digested with perchloric, nitric and hydrofluoric acids. The residue is leached with dilute hydrochloric acid and diluted to volume.

The final solution is then analysed by inductively coupled plasma-atomic emission spectrometry and inductively coupled plasma-mass spectrometry. Results are corrected for spectral inter-element interferences.

3.4.2. Gold particle size distribution (PSD)

A 2 kilogram sample portion for gold particle size distribution analysis was further split into 2x 1 kg samples and dried at 105° C for 4 hours. 1 portion (1kg) was kept as a backup sample and another portion used for particle size distribution analysis. The portion for PSD analysis was allowed to cool to room temperature and pulped using LM2 pulveriser for three minutes. The pulp was weighed and taken for wet sieving using different size sieves mounted together on an agitator.

Wet sieving method was used to evaluate the gold particle size distribution by determining the gold content in the materials retained on each sieve. The sieves used were 212 µm. 150 µm, 106 µm, 75 µm, 53 µm and 38 µm screen/sieve. An agitator was used to aid the gravitational pull and controlled water pressure used to aid particles to go through the sieves without clogging it in a way to avoid sample loss during wet sieving. The retained materials were dried and the weight recorded. The materials retained on each sieve were assayed and gold content obtained using the AAS. Percentage contribution of each fraction was determined by multiplying the fraction's weight contribution to the total used weight by the obtained gold content from that fraction.

3.4.3. Fire assaying

The 2 kilogram sample for fire assay was further split into 2x 1 kg samples. The two samples were dried at 105°C for four hours. After drying the samples were allowed to cool to room temperature and their exact weights after drying were captured. One 1 kg split was used for direct fire assay and another split was used for screen fire assay.

Direct fire

The samples for direct fire assay were pulped for three minutes with addition of methanol to prevent the sample from sticking on the disk and bowl. LM2 pulveriser was used to pulverise the samples to about 90 % particles less than 106 microns. After pulverizing, the samples were bottle-blended and transferred on a Kraft paper where the portion for analysis was scooped from by scooping from all sides of the heap into the geo-chem envelope.

Screen Fire

The samples for screen fire assay were pulped using LM2 pulveriser for three minutes and methanol added to reduce possible sticking of the sample on the disk and bowl. After pulping, the sample was transferred to a screen fitted with a 106 micron nylon screen and screened for 2 minutes using LM2 pulveriser. The plus 106 fractions were transferred to a geo-chem envelop together with the nylon screen to ensure no gold particles that may be stuck on the screen are lost. The minus 106 micron fractions were bottle blended, transferred to kraft paper and scooped into the geo-chem envelops by scooping from both sides of the heap to get a representative sample.

After the sample preparation steps outlined above for each technique, the subsequent steps are common to both techniques as follow;

Fluxing- The plus 106 fractions for screen fire assay technique were weighed and transferred to a crucible fitted with a clear plastic bag and then fluxes added based on the sulphur, carbon, magnetite content and sample weight. The bottom part of the envelop was cut and the piece transferred to the crucible with the sample together with the nylon screen. The minus 106 fraction and the pulps for the direct fire were homogenised again and different aliquots 10 grams, 30 grams and 50 grams weight into different crucibles and fluxes added as per calculated flux ration based on their mineralogy.

Fusion- The samples were fused for 1 hour at 1050 °C and the molten slag poured into an iron moulds. The metallic lead button separate from the slag due to difference in density. The slag was hammered of the slag and the lead button hammered into a nice metallic cube. the metallic cube-lead buttons were cuppelled using #6 cuppels for lead buttons weighing less than 50 grams and #8 cuppels for lead button weighing more than 50 grams.

Parting and dissolving- The gold-silver prill/bead remaining after cupellation for the plus 106 fractions was flattened and transferred to a parting cup, silver was dissolved in dilute nitric acid (1:5) and the remaining gold was dried, annealed and weighed using the micro-balance (gravimetric analysis).

The silver-gold prills from the pulps and minus 106 fractions were transferred into test tubes and dissolved into dilute nitric acid (1:1) and concentrated hydrochloric acid. The resulting solution was analysed using AAS machine.

Instrument- New gold standards were prepared and calibration was performed prior to reading the samples, certified reference materials were analysed to validate the results.

ALS Screen fire assay method summary

1000 g of the final prepared pulp is passed through a 100 micron (Tyler 150 mesh) stainless steel screen to separate the oversize fractions. Any +100 micron material remaining on the screen is retained and analysed in its entirety by fire assay with gravimetric finish and reported as the Au (+) fraction result. The –100 micron fraction is homogenized and two sub-samples are analysed by fire assay with AAS finish (Au-AA25 and Au-AA25D). The average of the two AAS results is taken and reported as the Au (-) fraction result. All three values are used in calculating the combined gold content of the plus and minus fractions.

In the fire assay procedure, the sample is fused with a mixture of lead oxide, sodium carbonate, borax, silica and other reagents as required in order to produce a lead button. The lead button, containing the precious metals is cupelled to remove the lead and the resulting precious metal bead is parted in dilute nitric acid, annealed and weighed to determine gold content.

The gold values for both the +100 and -100 micron fractions are reported together with the weight of each fraction as well as the calculated total gold content of the sample.

3.4.5. Cyanide leaching

The 2 kilogram sample portion for Leaching was dried at 105° C for 4 hours. The entire sample was pulped for four minutes and homogenised in the plastic bags by inverting the closed bag several times. After the homogenisation, the pulp was split into two ±1 kg sub-samples/portions. The weighed sub-samples from the pulp were transfered into 2-litres wide mouth bottles labelled as bottle A and B. 20 grams of Leachwell (catalyst) was added to bottle A and none to bottle B. 1 litre of 5 % cyanide solution was added to each bottle to make up 50% solids slurry. The bottles were placed into a mixing wheel blender and leached for 12 hours. After 12 hours the samples were filtered and two samples taken (solution and solids). The tails were taken for fire assay and the solutions were read on AAS direct.

3.5. Data analysis

In order to interpret data in an unbiased manner, maximise reliability of the data and assure that the tests were used in ways that are valid and scientifically defensible, statistical analysis are applied to the data. Statistical analyses provide a measure of proof that the analytical values are true. In this study Statistical parameters such as mean, standard deviation, F-test and large deviation for determining outliers are applied to validate and compare methods in order to determine the method that gives more accurate and precise data.

Mean – Is used to measure the centre of sample or population value from a set of data and applied using the following equation;

$$X_{Av} = \frac{\Sigma x}{n}$$

X_{av} = is the mean (average)

Σ – Epsilon (operation of adding)

x– Individual datum

n- Is the number of data points.

Standard Deviation – Is used to measure the spread of data from the mean, whereby the smaller the standard deviation of a set of data, the closer the measurements to the mean which is referred to as high precision. It is applied using the equation below;

$$S = \sqrt{\frac{\Sigma(X - X_{av})^2}{(N - 1)}}$$

$S = Standard\ deviation$

$X = individual\ datum$

$X_{av} = mean\ value\ for\ the\ data\ set$

$(N - 1) = degree\ of\ freedom$

Determining outliers - an outlier in this study is regarded as either extremely large or small value relative to the rest of the data. The outliers are determined by visual inspection or by determining “large deviation” whereby if a datum differ from the mean by a predefined multiple of the standard deviation then it is an outlier. The formula below is used;

$$M = \frac{|X_q - X_{av}|}{S}$$

M – Is the predefined multiple (usually 4), X_q – Is the datum in question, X_{av} – Is the means and S- is the standard deviation.

F-test for the equality of two variances – is used to compare the variance of the analytical methods for analysing gold and to see if there is a significant difference between the tested methods and the screen fire assay method in use. The formula used is;

$$F = \frac{{S_1}^2}{{S_2}^2}$$

whare S_1 *and* S_2 *are standard deviations of method* 1 *and method* 2 *respectively*.

The obtained F *value will be compared to the tabulated value of* F *at* 29 *degree of freed*

(n−1 =30-1 =29)

This will be followed by t-test using the following formula;

$$t_{cal} = \frac{[Mean1 - Mean2]}{S_p}\sqrt{\frac{n1n2}{n1+n2}}$$

Mean1 and mean2 are population mean for data set 1 and data set 2, S_p is the pooled standard deviation of the sets and n1 and n2 are number of data set 1 and 2.

Pooled standard deviation is calculated as follow:

$$S_{pooled} = \sqrt{\frac{(n1-1)S1^2+(n2-1)S2^2}{n1+n2-2}}$$

S1 and S2 are standard deviations of data set 1 and 2 while n1 and n2 are number of data set 1 and 2.

Chapter 4. Results and Discussions

In this chapter, the results obtained using different analytical techniques and instruments as outlined in chapter 3 (Methodology) are presented. Tables and graphical representations are used to illustrate the result and the results are presented in an orderly sequence as per the objectives of the study, the results are discussed in details in section 4.2.

4.1. Results

4.1.1. Problematic elements identification, quantification and evaluation of their effects.

a. Elemental effect on cyanide leaching

The table below illustrates the content of the quantified elements and the calculated % gold recovered;

Sample ID	**% Au recovery**	**Cu ppm**	**Fe %**	**S %**	**C %**	**As ppm**	**Ni ppm**	**Sb ppm**	**Zn ppm**	**% of Au. Reportingin+106µ**
B2G_WS_01	92.0	3	5	1	5	12	37	1	5	64.50
B2G_WS_02	84.1	1	5	2	3	4	36	0	7	83.85
B2G_WS_03	99.4	1	6	1	6	5	31	0	4	72.97
B2G_WS_04	92.7	1	8	4	4	4	35	1	5	82.64
B2G_WS_05	90.2	3	5	2	4	5	42	1	3	81.21
B2G_WS_06	72.7	3	9	7	7	7	45	0	5	74.28
B2G_WS_07	95.8	1	3	1	3	3	33	1	3	64.77
B2G_WS_08	86.7	1	3	2	9	3	11	0	3	92.83
B2G_WS_09	95.0	1	4	2	3	4	55	0	2	70.89

B2G_WS_10	91.8	3	7	5	3	5	35	0	5	70.94
B2G_WS_11	75.5	3	12	11	5	14	38	0	4	59.56
B2G_WS_12	77.5	2	9	7	5	5	34	0	4	73.91
B2G_WS_13	86.1	4	7	4	3	4	33	0	6	62.38
B2G_OTJ_14	74.6	149	12	3	3	1	39	0	9	62.08
B2G_OTJ_15	63.5	91	10	4	2	2	44	0	17	73.85
B2G_OTJ_16	78.3	38	7	1	1	1	50	0	25	62.34
B2G_OTJ_17	84.8	371	20	10	2	1	61	0	7	52.78
B2G_OTJ_18	79.0	376	19	9	3	1	59	0	13	54.02
B2G_OTJ_19	62.8	394	22	9	2	1	51	0	13	59.01
B2G_OTJ_20	71.1	109	10	4	2	1	43	0	19	72.73
B2G_WS_21	83.4	18	11	0	5	37	44	1	26	70.12
B2G_WS_22	67.5	18	8	0	5	18	39	1	35	50.88
B2G_WS_23	93.3	30	7	0	5	13	46	1	44	54.57
B2G_WS_24	81.1	18	10	0	5	35	42	1	26	71.65
B2G_WS_25	87.6	12	6	3	4	11	31	0	8	57.35
B2G_WS_26	94.2	4	11	2	9	3	57	0	8	65.25
B2G_WS_27	92.6	7	3	0	3	2	31	0	8	54.91
B2G_WS_28	74.6	2	4	6	4	6	12	0	5	77.04
B2G_WS_29	81.5	2	7	3	5	4	30	0	7	72.22
B2G_WS_30	95.4	1	7	2	5	3	31	0	8	70.74
Recovery Mean	83.5									

Table 3: Effect of elements on gold cyanide leaching.

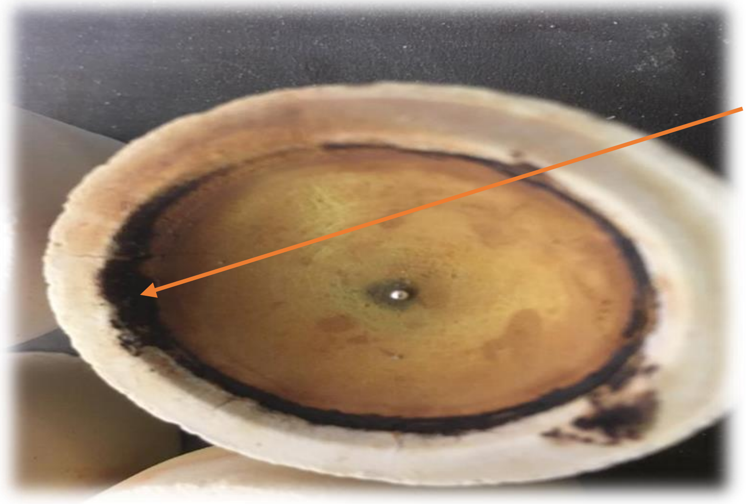
The effects of the elements on leaching are revealed by the percentage gold recovery by leaching, in the column titled % Au recovery. The lower the percentage recovery the more the effects of the elements that appear in high concentration.

a. Elemental/mineralogical effect on Fire assaying

Figure 6 below show the lead button for sample B2G_OTJ_17 after trying hammering it into a cube. This sample contains 10.3696 % S which is equivalent to 103 696.0 ppm sulphur and 371 ppm of copper.

Figure 6: Brittle button (the button breaks into scattered pieces once hammered).

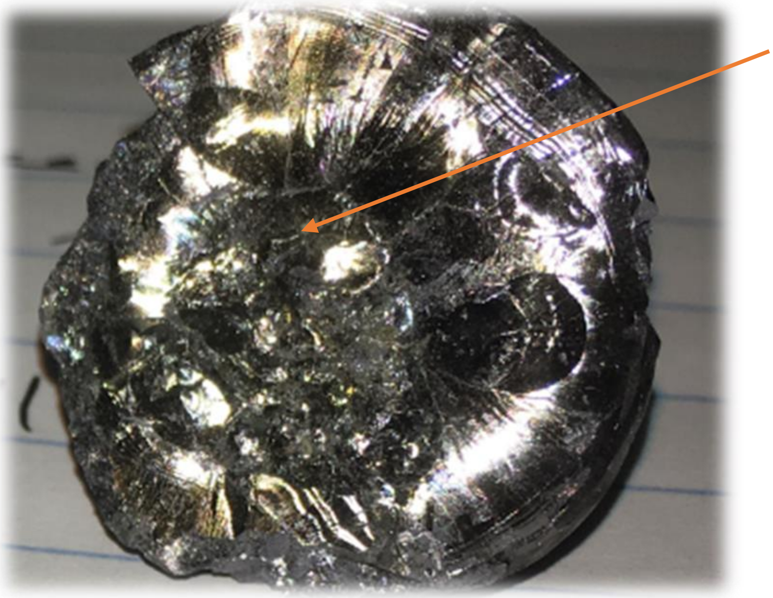
Figure 7 shows the black and brown scoriers that formed on the side of the cupel for sample B2G_WS_11. The sample contains 17.248 % Iron (Fe) which is equivalent to 172 480.0 ppm Fe.



Scoriers forming on the side of the cupel can trap preciou metals preventing them fro settling in the bead.

Figure 7: Black and brown scoriers and stains formed on the sides of the cupel.

When elements like copper, nickel, Iron and other base metals are smelted a molten metal sulphide called a matte forms and can dissolve gold and silver. The matte is brittle when solidified. Figure 8 below shows a matte that form on top of the lead button of sample B2G_OTJ_19 which contains 8.9834 % Sulphur (89 834 ppm S), 22 % Iron (220 000.0 ppm Fe) and 394 ppm copper.



A metals sulphide matte that appears like slag stuck on the lead button. The matte dissolve gold and silver and it is very brittle, hence during hammering it falls off causing gold loss.

Figure 8: Matte formed between the lead button and the slag.

Table 4 below summarises the finding on the 8 samples investigated and it compares the lead button qualities, cupellation efficiency and the variation of gold content Au1 and Au2 done on duplicated samples to the elemental content of sulphur, Carbon, Iron, copper and zinc.

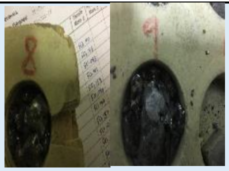
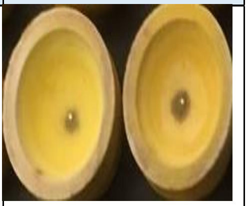
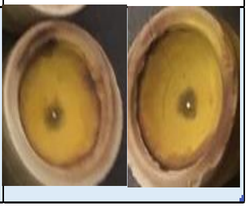
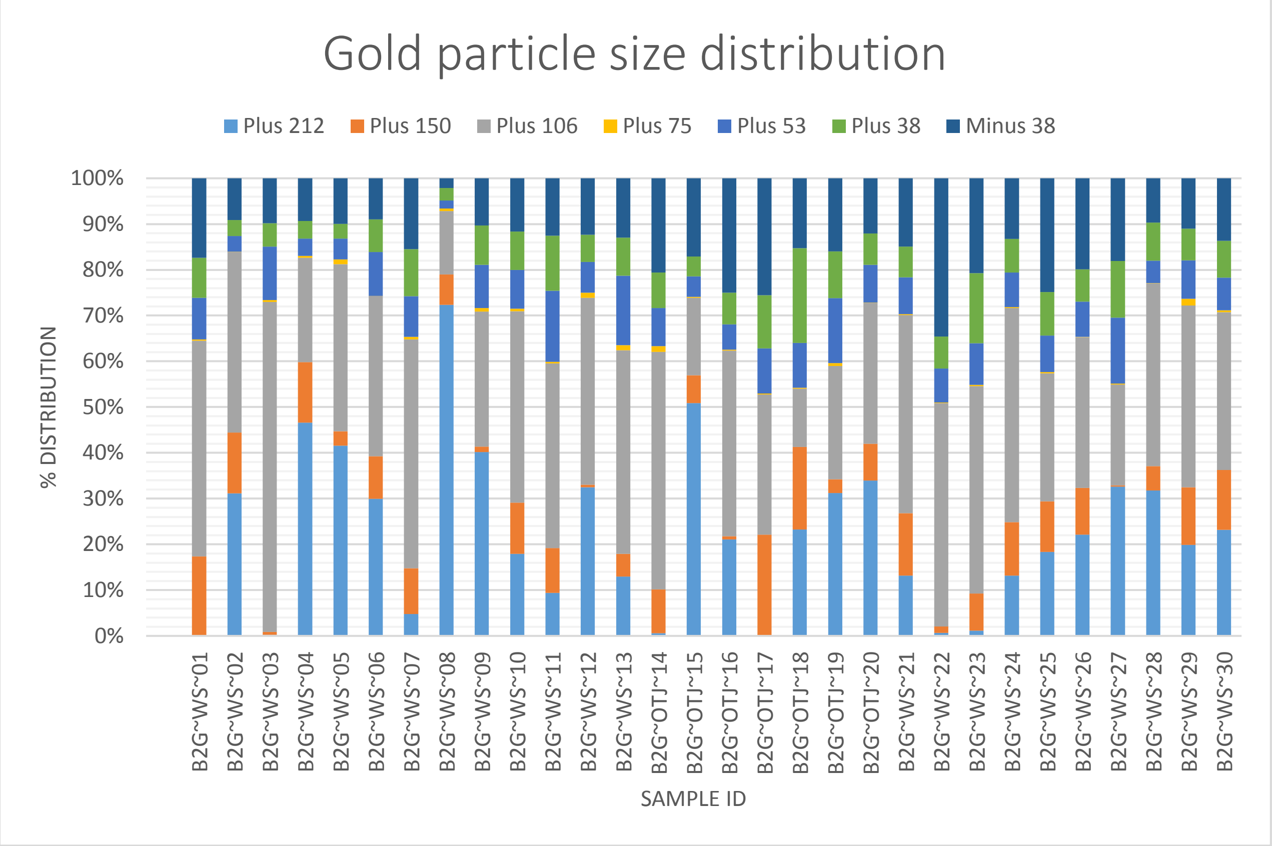
Sample ID	S %	C %	Fe (%)	Cu (ppm)	Zn (ppm)	Au 1	Au 2	Lead buttons	Cupel condition After cupellation
B2G_WS_08	1.6616	9.3358	2.83	0.9	3	0.441	0.242		
B2G_OTJ_19	8.9834	2.3485	22	394	13	27.85	19.691		
B2G_WS_26	1.6612	9.102	10.55	3.5	8	0.777	0.639		
B2G_WS_11	11.248	5.1126	12.4	2.6	4	17.145	12.902		
B2G_WS_21	0.1593	5.0595	10.6	18.3	26	18.219	10.424		
B2G_OTJ_17	10.3696	1.7532	19.55	371	7	0.83	0.005		
B2G_OTJ_14	3.4943	3.0113	11.95	149	9	0.645	1.122		
B2G_OTJ_18	8.6079	3.2037	19	376	13	0.159	0.441		

Table 4: Brittle buttons and scoriers on the cupels due to presence iron, sulphur and copper

4.1.2. Gold particle size distribution in samples

Graph 1 below show the content of gold in each size fraction of each sample;



Graph 1: Gold particle sizes distribution in samples (% contribution of different size fractions to the total gold in each sample).

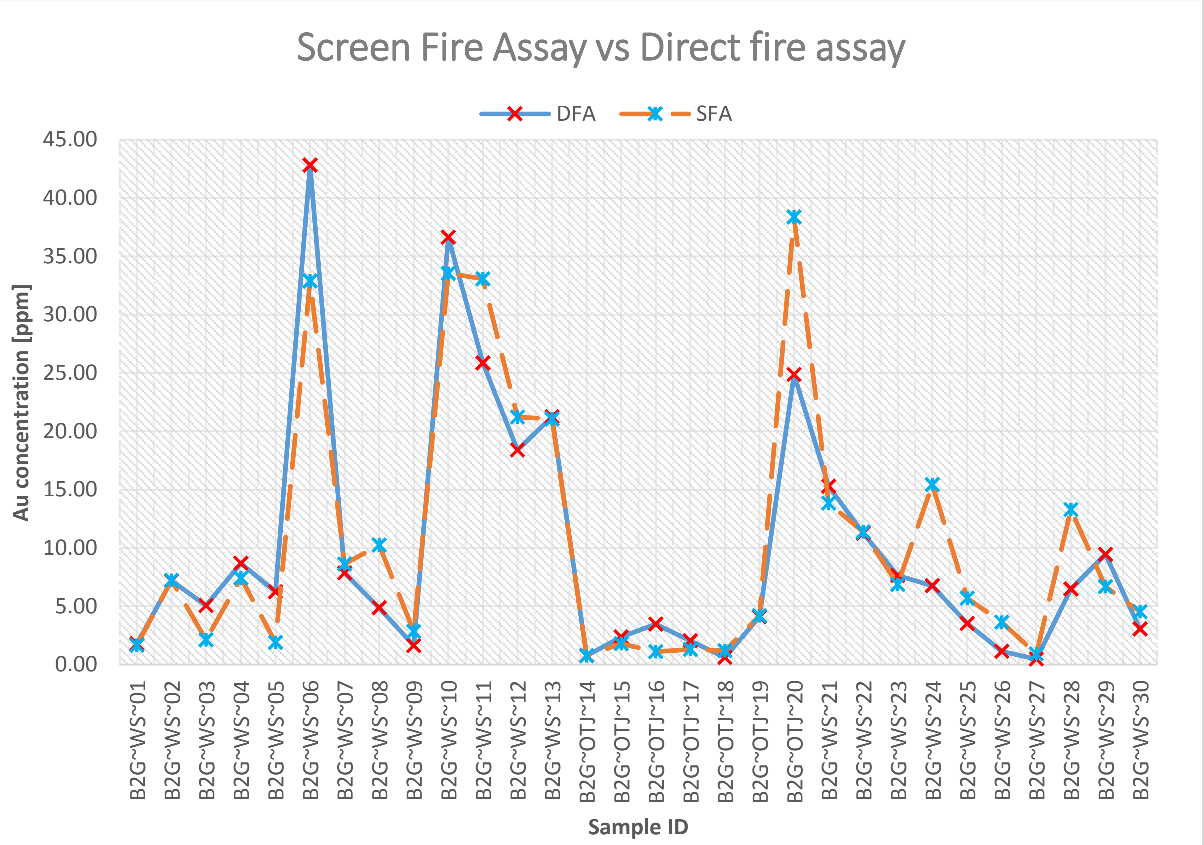
From the graph, the total gold content is regarded as 100% and the contribution of gold content from each size fraction is represented by the color of each size fraction given in the legend on graph.

4.1.3. Analytical techniques for gold analysis

The results of the techniques are displayed below;

a. Fire Assay results

The results obtained from direct fire assay are represented by the line labelled DFA and for Screen fire assay are represented by SFA line as presented in graph 2 below;



Graph 2: gold assays comparison of direct fire assay and screen fire assay.

From the data on graph 1 (gold assays comparison of direct fire assay and screen fire assay) the following statistical parameters were found;

Population mean for direct fire assay = 9.72

Population mean for Screen fire assay = 10.49

Population Standard deviation for direct fire assay = 10.58

Population Standard deviation for Screen fire assay = 10.94

Outliers= None

F_{cal} *value = 1.069*

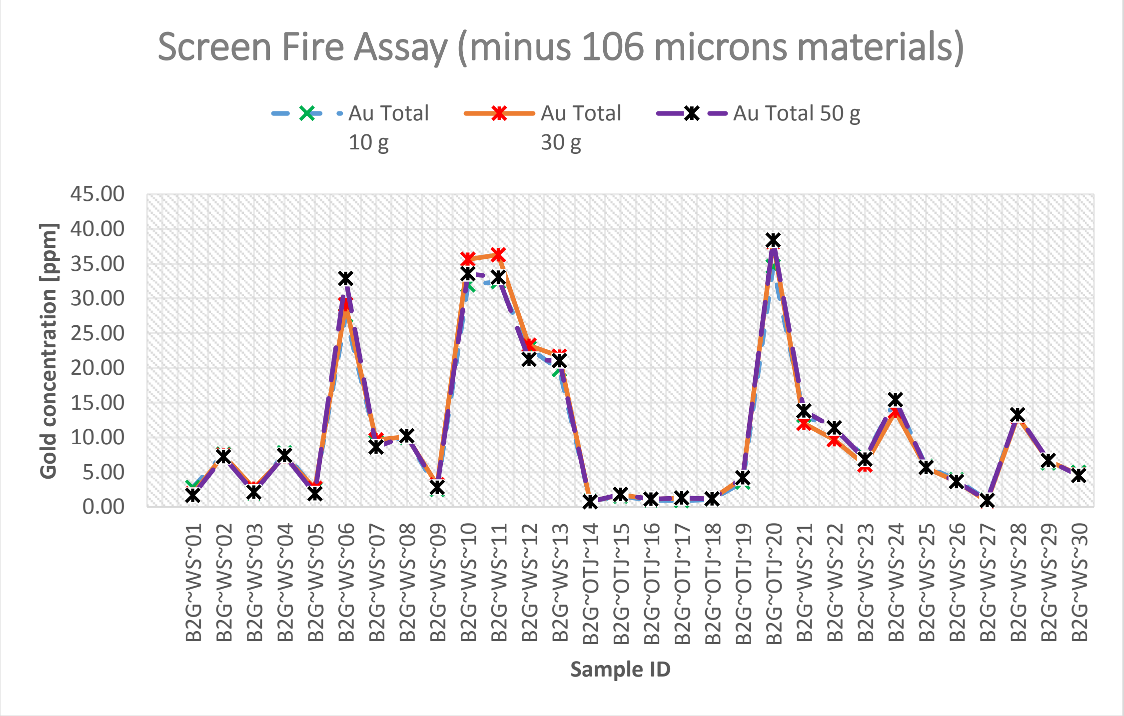
t_{cal} *value =1.08*

The tabulated F and t values when working 95% confidence level and with 29 degree of freedom (df=n-1) are;

t= 1.699

F= 1.901

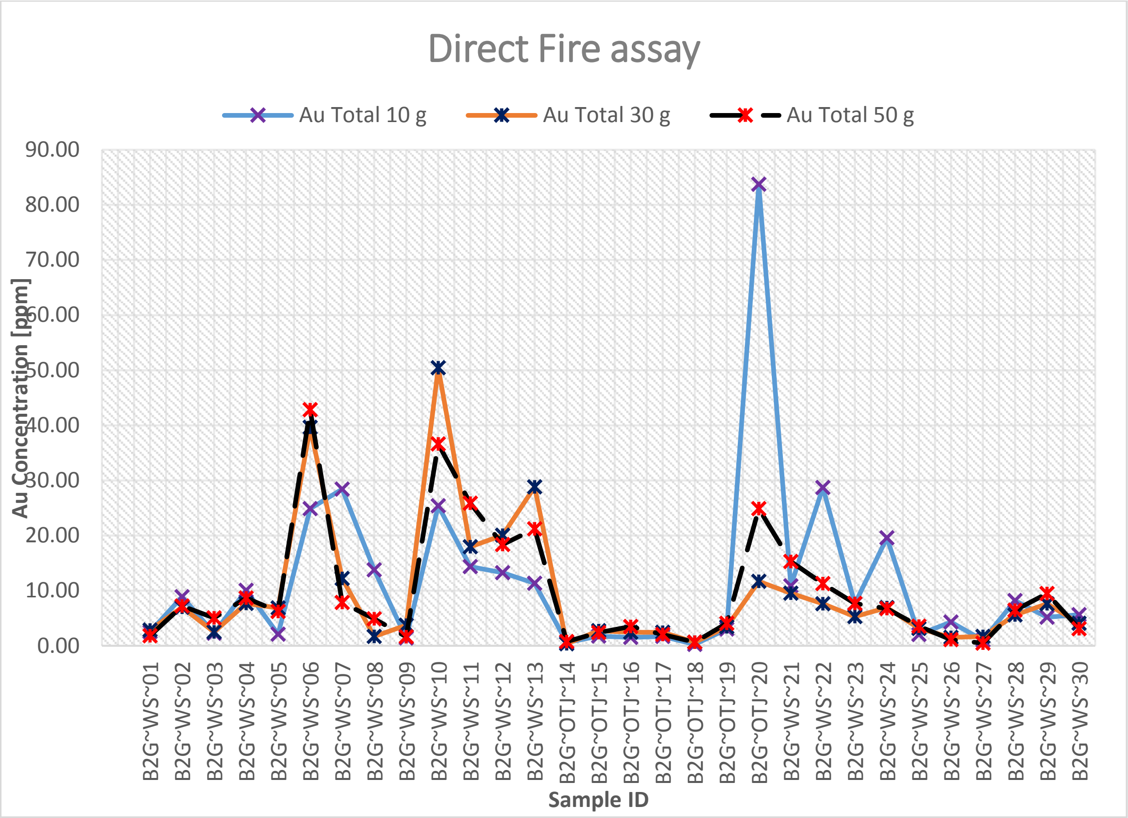
The obtained results from different size aliquot with coarse gold screened out are represented by graph 3 below;



Graph 3: Investigation results on the effects of the coarse gold on assays using different size aliquots on screened materials for screen fire assay technique.

The population means/average obtained using the three aliquots are; 10 grams = 10.16, 30 grams = 10.50 and 50 grams = 10.49 ppm.

The obtained results from different size aliquot for materials with coarse gold not screened out are represented by graph 4 below;



Graph 4: Investigation results on the effects of the coarse gold on assays using different size aliquots on direct fire assay technique.

The population means/average for direct fire assay using the three aliquots are; 10 grams = 11.47, for 30 grams = 9.27 and for 50 grams gave 9.72 ppm.

The reason for the differences in average is discussed into details in section 4.2.

b. Cyanide leaching results

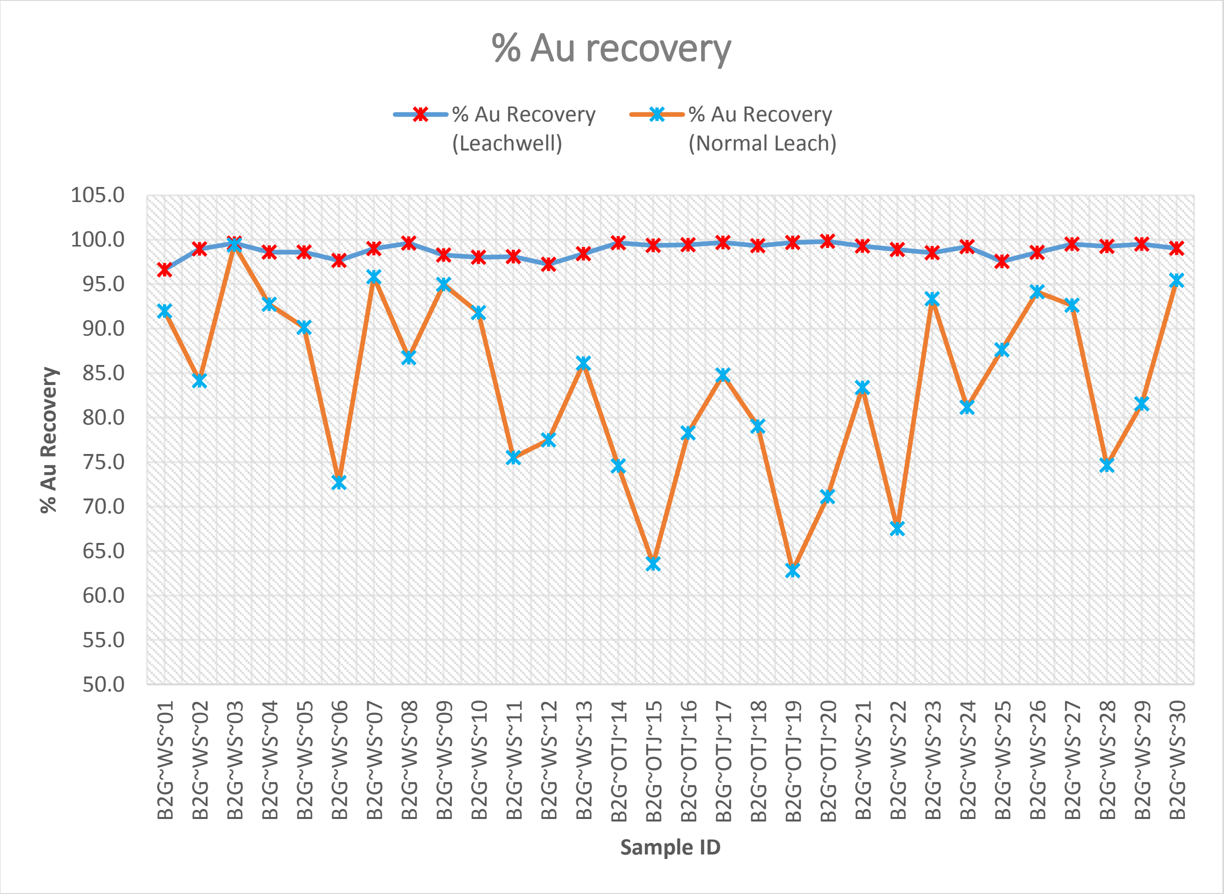
The table below display the results obtained from gold cyanidation with leachwell and without leachwell as well as from the tails;

SAMPLE ID	**Leaching**			
	Leaching with Leachwell		**Leaching Without Leachwell**	
	Au (ppm) Solution	**Au (ppm) Tails**	**Au (ppm) Solution**	**Au (ppm) Tails**
B2G~WS~01	1.498	0.052	2.150	0.187
B2G~WS~02	8.076	0.084	8.757	1.653
B2G~WS~03	3.511	0.014	2.039	0.012
B2G~WS~04	5.664	0.080	5.540	0.434
B2G~WS~05	2.780	0.039	1.887	0.206
B2G~WS~06	28.273	0.678	20.699	7.776
B2G~WS~07	6.666	0.066	6.760	0.294
B2G~WS~08	16.984	0.063	15.530	2.378
B2G~WS~09	2.802	0.049	2.187	0.116
B2G~WS~10	27.776	0.559	26.148	2.339
B2G~WS~11	19.810	0.383	15.036	4.881
B2G~WS~12	17.549	0.501	13.100	3.808
B2G~WS~13	17.322	0.277	17.239	2.777
B2G~OTJ~14	14.118	0.048	6.438	2.197
B2G~OTJ~15	4.484	0.029	3.164	1.815
B2G~OTJ~16	2.685	0.015	1.208	0.335
B2G~OTJ~17	2.2361	0.007	1.944	0.349
B2G~OTJ~18	1.038	0.007	0.937	0.25
B2G~OTJ~19	3.744	0.012	3.010	1.78
B2G~OTJ~20	15.110	0.028	9.677	3.93
B2G~WS~21	13.078	0.093	12.413	2.47
B2G~WS~22	12.703	0.143	9.636	4.64

B2G~WS~23	6.3588	0.094	6.069	0.43
B2G~WS~24	12.039	0.094	10.567	2.46
B2G~WS~25	3.281	0.082	2.716	0.38
B2G~WS~26	2.943	0.043	1.934	0.12
B2G~WS~27	1.372	0.007	1.144	0.09
B2G~WS~28	12.768	0.095	9.020	3.06
B2G~WS~29	7.347	0.037	5.703	1.29
B2G~WS~30	3.3231	0.032	3.868	0.19

Table 5: Comparison of gold cyanide leaching using Leachwell and without Leachwell.

Percentage gold recovery was calculated and the percentage gold recovery is shown in the graph below;



Graph 5: % gold recovery by cyanide leaching using Leachwell verses normal cyanide leaching.

The gold leached using Leachwell as a catalyst are represented by the blue line with red data points while the gold leached by normal cyanide leach are represented by the orange line with blue data points on the graph.

From the data obtained when samples are analyzed using cyanide leaching and its modification Leachwell accelerated cyanide leaching the following statistical parameters are found;

Population mean for Normal cyanide leaching assays = 7.55

Population mean for Leachwell accelerated cyanide leaching assays = 9.24

Population Standard deviation for Normal cyanide leaching assays = 6.338

Population Standard deviation for Leachwell accelerated cyanide leaching assays = 7.550

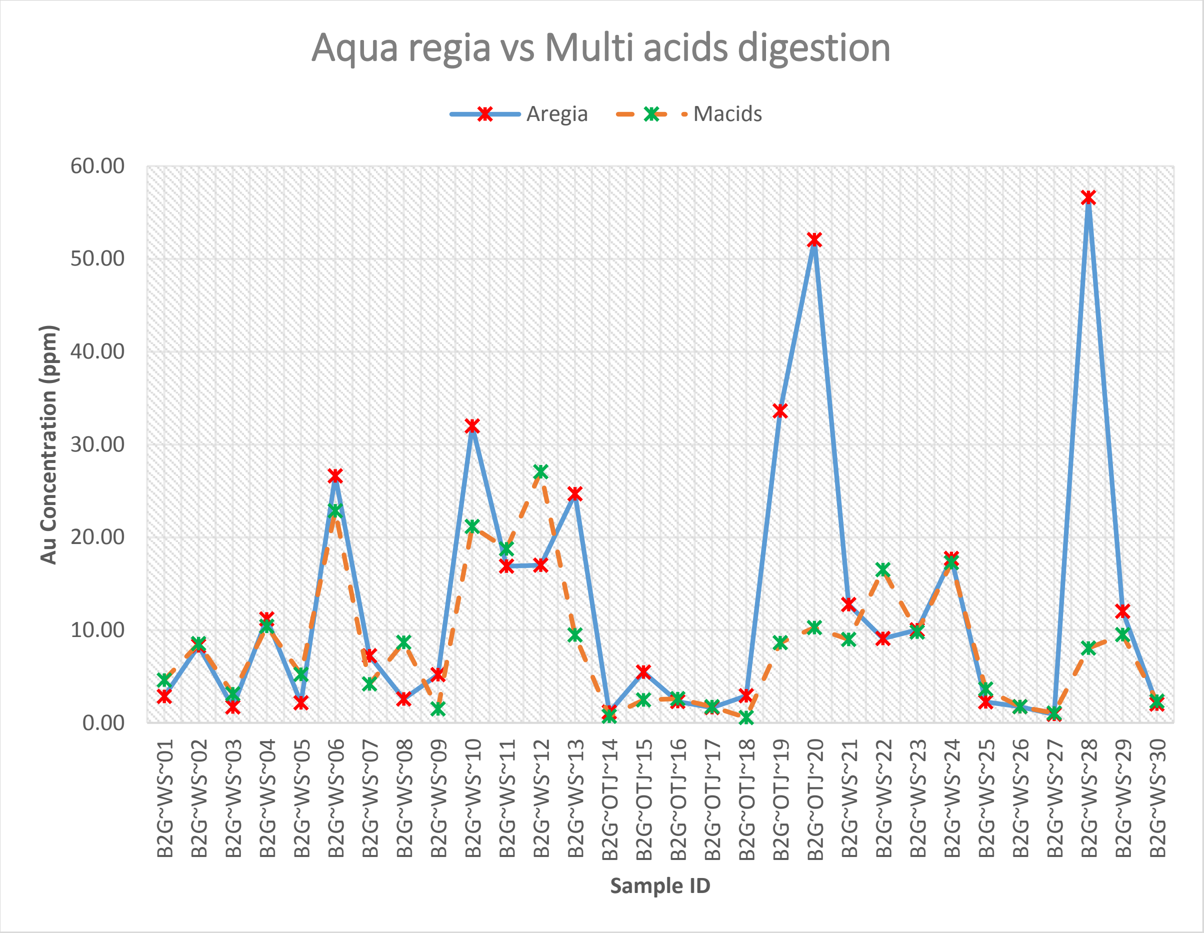
Outliers= None

F_{cal} *value = 1.419*

t_{cal} *value =3.65.*

c. Acid digestion results

Acid digestions well known for gold analysis aqua regia and its modification multi acid digestion’s results are presented by the graph below;



Graph 6: Comparison of gold assays from aqua regia digestion and multi acids digestion.

On the graph above, the gold content in each sample analyzed by aqua regia is represented by the blue line with red data points and for multi acids is represented by the orange dotted line with green data points.

From the data represented by the graph, the following statistical parameters are obtained;

Population mean for aqua regia assays = 12.75

Population mean for multi acids assays = 8.38

Population Standard deviation for aqua regia assays = 14.422

Population Standard deviation for multi acids assays = 7.052

Outliers= by visual inspection three data for sample $B_2G\_OTJ\_19$, $B_2G\_OTJ\_20$ *and* $B_2G\_WS\_28$

F_{cal} *value = 4.183*

t_{cal} *value =6.79*

d. Combined techniques results

The three common used techniques are compared to their modified versions, all the techniques are then compared on one table to see each techniques' performance relatively to others (See appendix E).

On the table below DFA represent direct fire assay, SFA represent screen fire assay, LWELL represent leaching using Leachwell, Leach is the normal leach without Leachwell, Aregia represent aqua regia digestion and Macids represent multi or mixed acids digestion.

Comparing the six techniques, we use the population mean and see which technique' mean is deviating from the entire data set mean as illustrated in the table below;

Technique	**Mean**
DFA	9.72
SFA	10.49
LWELL	9.25
Leach	7.55
Aregia	12.75
Macids	8.38

Mean 9.69

Table 6: Comparison of the mean for different gold analytical techniques

If the two outliers on obtained from aqua regia for sample B2G_WS_29 and B2G_OTJ_19 are taken out from the data set the average/ mean for all the methodology means becomes **9.27 ppm** gold.

4.2. Discussions

4.2.1. Effects of the elemental composition

a. Elemental effects on Cyanide Leaching.

From table 3 the effect of the elements are clearly revealed, it can be said with confidence based on the results that the percentage gold recovery is inversely proportional to the elements contained in the sample. Notable differences that can be used to justify this argument is given by sample B2G_WS_03 which has the highest percentage gold recovery of 99.4 % and looking into the elemental composition of the above mentioned sample, it contains 72.97% of coarse gold, 1 ppm copper, 6% iron and 1% Sulphur which is an indication that the sample is low in copper and iron sulphide minerals, which by literature (50) are the main cause of poor gold dissolution by cyanide. The remaining 0.6% that was not dissolved can be said that it is due to the presence of more coarse gold which make up 72.97% of the total gold in the sample. Comparably, sample B2G_OTJ_19 with the lowest percentage gold recovery with only 62.8% recovery, contains 59.01% of coarse gold, 394 ppm copper (highest sample in copper content), 22% Iron (that is 220 000 ppm Iron), 9% Sulphur (that is around 90 000 ppm Sulphur), 51 ppm Nickel (second highest in nickel content after B2G_OTJ_18 with 79% gold recovery), the poor recovery can be said that is due to the high content of the problematic elements. Looking from the table the trend that can easily be picked up from the table is that samples with a combination of high copper, Iron and Sulphur had the lowest recovery.

The effect of carbon (Total combustible carbon) on leaching can be observed using sample B2G_WS_08 and B2G_WS_26 both with 9% carbon, but different gold percentage recoveries. Sample B2G_WS_08 has total gold percentage recovery of 86.7% while from B2G_WS_26, 94.4% of gold was recovered. From this observation it can be said that the carbon contained in the two samples is not a preg-robber as literature outlined (54, 53, 61) which stated that carbon adsorb gold back onto the carbons from solution. Therefore other elemental and grain size factors on the two samples that could have cause the difference are; sample B2G_WS_08 contains 92.8% of coarse gold of which 70% (see graph 1) of the gold was recovered from +212 µm which is an indication that the gold on this sample are very coarse and according to the literature (53, 55, 61) if gold particles are too large they may not be totally leached. On the other hand B2G_WS_26 only contained 65.3% of coarse gold of which only about 21% was recovered from the +212 µm. Therefore it can be established that the difference in the two sample recoveries are due to gold particle size differences and their distribution in the sample.

In this study it was observed that the samples containing high copper or containing high Iron and Sulphur or containing coarse gold, the gold recovery by leaching is poor. It was unfortunate that the other elements were all below 100 ppm in all the samples and their effects are mostly visible when they are in high concentrations.

b. Elemental composition and gold grain size effects on Fire Assaying.

As it is outlined in literature review that there are elements that causes difficulties when analyzing samples for gold using fire assay (4, 41, 44, 45, 50, 52), this was verified by the results. The elements found to have the most effect are those that are present in large amount in the Otjikoto and Wolfshag ore. The elements found to cause much of trouble with fire assay are Sulpur (when present at the concentration of more than 2%), Copper (when present at the concentration more than 10 ppm) as well as Iron (when present at the concentration more than 4%). The evidence from the study that back the above information are an example of sample B2G_OTJ_17 (Amphibole) as shown in figure 6, Due to presence of 10.3696% sulfur and 371 ppm copper the lead button was very brittle which lead to gold loss, the gold loss was verified from the gold value from the duplicated sample, where 2x 50 grams samples were assayed the same and total gold content compared, Au 1 was 0.83 ppm Au for original and duplicate gave Au2 of 0.005 ppm Au. With these big discrepancies the deposit can easily be underestimated as the average will be lowered due to gold loss.

Sample B2G_OTJ_19 (Albitite) has Sulphur content of 8.9834%, 22% Iron and 394 copper and due to these high content of Sulphur and base metals a matte was formed as shown in figure 8. The matte has a potential of dissolving and keeping some of the gold and silver. Au 1 and Au 2 gave 27.85 ppm Au and 19.091 ppm Au, where there is also a big discrepancy, this can be said is due to formation of different matte size as well as brittleness of the button. Another factor that can cause this big differences can be high Iron content which lead to the formation of scoriers during cupellation as indicated in the images in table 4. When the scoriers are forming they can trap gold and silver preventing them to settle in the Dore bead with the rest of the gold and silver, hence resulting in inaccurate gold assays/values. Another example of scorier formation is shown in figure 7 for sample B2G_WS_11 from Biotite Banded Amphibole rock.

Carbon seems not to have any effect on fire assaying process this is indicated by samples such as B2G_WS_08 (marble) and B2G_WS_26 with 9% (90 000 ppm) total combustible carbon (TCC), but the buttons and assays do not seem to be affected. The Au [1] and Au [2] for B2G_WS_26 are 0.777 ppm and 0.689 ppm nicely close to one another. The buttons for oxides or Fault Breccia samples were small, which implies that there was too much of oxidizing agents in the crucible, oxidizing lead once it is reduced and returning it into the slag. This can result in insufficient lead in the crucible to collect all the precious metals as also stated in the literature (50).

4.2.2. Gold particle size distribution.

The established gold particle size distribution as displayed by graph 1, indicates that most of the gold in Otjikoto mine gold deposit occurs as coarse gold as indicated in the document titled Otjikoto ore mineralization and by Ross and David (15). This study has therefore verified that the gold in the Otjikoto and Wolfshag based on the representative samples analyzed occurs as nugget as seen on the graph that in all samples 50% of the gold is reporting in +106 microns fractions.

4.2.3. Gold analysis techniques

b. Fire assay

The population mean or average for direct fire assay is 9.72 ppm Au and for screen fire assay is 10.49 ppm Au, this implies better recovery with the screen fire assay compared to direct fire assay. This does not mean what it looks like, the recovery should be the same, but the cause of the difference is the gold nuggets whereby with direct fire assay it works with the probability of scooping a nugget when taking the aliquot for analysis, while with screen fire assay all the nuggets are excluded from the assays. In addition, the calculated F-value and t-value are both less than the tabulated values (See appendix C and D), this suggests that there is no significant difference between the two methods, meaning that both methods are capable of recovering gold. The differences can be due to other factors such as nuggets, handling error and so forth. Graph two (2) also clearly show the correlation between the two techniques.

To determine the consistency as well as the effect of nuggets on the fire assay techniques, different aliquot (10g, 30g & 50g) were assayed and the results are presented in graph 3 and graph 4. The aliquots for screen fire assay all gave the average in the same range which is an indication that when the materials are screened for coarse gold, then the aliquot used for minus fraction does not make any difference in the results. On the other hand for direct fire assay there is a difference in average of the assays obtained from different aliquots hence with direct fire assay it seems that the bigger the aliquot used the better the assays/results.

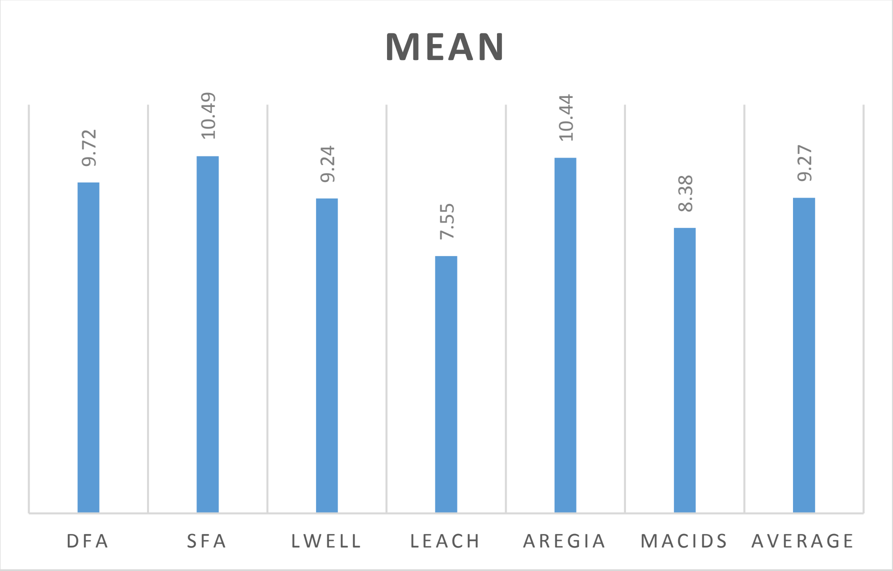
c. Cyanide Leaching

Table 4 and the percentage gold recovery graph 5 clearly demonstrate poor gold recovery with the normal cyanide leaching and a very good recovery with Leachwell assisted cyanide leaching. The tabulated t- value for the two methods at 29 degree of freedom and 95% confidence level is more than the calculated t-value and this implies that there is a significant difference between gold leaching with cyanide alone and gold leaching with Leachwell assisted cyanide. It is evident that the big differences were caused by the elements present in the samples. Leaching with Leachwell assisted cyanide show very good and consistent gold recovery regardless of the elements present, hence it can be said with confidence that cyanide leaching using Leachwell is mineralogical/elemental independent or not influenced by the elements present. It is unfortunate that limited work has been published on leachwell as stated under literature review, hence making it difficult to compare to previous studies.

d. Aqua Regia/Acids Digestions

In some literature it was said that aqua regia or acids digestions was an ideal technique due to its simplicity, time and low cost could replace fire assay (26), but this study found this techniques as an unreliable technique when it comes to analyzing samples with coarse gold. It is likely that the studies that suggest acids digestion to be a replacement for fire assay could have been done on gold deposits where the gold is disseminated and fine as well. The technique uses small sample size with high chances of either picking or missing a nugget particle and this will result in either extremely high or low values, overestimating or underestimating the resource. The clear evidence from the results is the presence of three outliers, sample B2G_OTJ_19, B2G_OTJ_20 and B2G_WS_28. Going back to graph one, there is 59% of coarse gold in sample B2G_OTJ_19, 75 % of coarse gold in sample B2G_OTJ_20 and 78% of coarse gold in sample B2G_WS_28. The coarse gold particles present in the samples that came out as outlier is a clear indication that a particle or particles of gold were picked and included in the aliquot used. Both the calculated F value, t-value are more than the tabulated value showing significant difference between the two acid digestion techniques.

e. Combination and comparison of the techniques used



Graph 7: Comparison of different techniques mean with outliers removed.

Looking and judging from the chart the population mean for different techniques are not that far apart from one another. The mean for aqua regia only came in range with others after the three outliers were removed/rejected using visual inspection. With outliers removed the average of all the techniques combined is 9.27 ppm Au which is closer to Leachwell assisted cyanide leaching with an average of 9.24 ppm Au. The poorest of all the averages is normal cyanide leaching without Leachwell. As discussed earlier direct fire assay, aqua regia digestion and mixed acids digestion cannot be used when analyzing samples with coarse gold as they give erratic results. On the other hand leaching without Leachwell is affected so badly by different elements. Therefore the competition between the best techniques remains only between screen fire assay and Leachwell assisted cyanide leaching.

Although it is well known that the most suitable method for dealing with samples containing nugget gold is to process a large mass of sample by the Screen Fire Assay (SFA) procedure, (present method being used by Otjikoto mine laboratory.) This procedure is labor intensive, time consuming, consumable demanding and by its nature requires many handling steps. Each handling step increases the chance for errors. In contrast the Leachwell procedure may allow the processing of a sample with an equally large or larger sample mass while significantly reducing consumable consumption, handling steps as well as labor and maintenance costs. A further advantage of the Leachwell procedure being investigated is that it represents the total sample mass taken for analysis whereas the SFA method quantifies all the +106µ fraction but only 10% or less of the -106µ fraction. This led to the rejection of the null hypothesis which states that Fire assay (Pyro metallurgy) is the best technique to recover gold from ores containing minerals or elements that are known to affect gold recovery such as copper, Iron, Sulphur, arsenic, nickel and zinc just to mention few as well from ores where gold occurs as nuggets.

Chapter 5. Conclusions and Recommendations

In conclusion the aim/objectives of the study were met. The elements that are known to interfere with gold recovery and analysis or elements that make up minerals that are known and has been studied and verified that causes difficult during gold analysis and recovery were identified, quantified and their effects assessed. The study has demonstrated that presence of Iron (<4 % or 40 000 ppm), sulfur (<2%), Copper (<10 ppm) and Zinc (<5 ppm) in high concentration pose a problem with almost all the gold analytical procedures as it was determined from the results obtained. The high sulfur and iron obtained in the study confirms Bruce et al (8) and Ross and David (15) that the minerals associated with gold in the Otjikoto and Wolfshag deposit are pyrite and pyrrhotite which are made up of iron and sulfur and magnetite and carbonate containing carbon and iron which were the major elements found. It can be assumed that these samples are likely present in the form of the aforementioned minerals as these elements are not found native in nature.

Furthermore the study has also successfully established the gold particle size distribution in the samples and verified that the gold in Otjikoto mine gold deposit in both Otjikoto and Wolfshag pits/zones occurs as free coarse gold, with all the sample containing 50 % of its total gold content reporting in plus 106 µm fractions. The effect of coarse gold or “Nugget effect” was illustrated by the erratic results of the direct fire assay, aqua regia as well as mixed acid digestion.

Lastly during determining the most suitable analytical technique for gold recovery/analysis for nugget gold ore, erratic results were obtained from direct fire assay, aqua regia as well as mixed acid digestion, where only a small portion (10 grams to 50 grams) of the sample is used in analysis and based on this problem the three techniques do not qualify to reliably analyze samples when coarse gold is present and/or when the gold is associated with elements that cause difficulties in gold assaying and recovery. Leaching without Leachwell is mineralogical dependent/influenced and cannot be relied on. Screen fire assay and Leachwell assisted cyanide leaching have proven to be the most suitable techniques when working with nugget gold and/or in presence of problematic elements.

Although the Leachwell recovery may only be 98% of the total gold content on average, as confirmed by the assay of the tailings portion, the Leachwell results are statistically acceptable when comparing to SFA. Based on the samples used in this study, it also appears that the Leachwell procedure is immune to different elemental compositions. Screen fire assay is labour intensive, consumable demanding and prone to errors with about 25 handling steps compared to Leachwell with only about 5 handling steps. Leachwell has short turn-around time and reduced cost as well, which may make it a viable option to replace the SFA. Leachwell procedure could provide more efficient, cheaper and more reliable analysis for the Otjikoto and Wolfshag samples as well as elsewhere in the world. Due to the fact that it also uses larger sample quantity, this makes cyanide leaching using Leachwell the ideal gold analytical technique and led to the rejection of the null hypothesis.

Recommendations

- Further work must be done comparing the Leachwell and SFA procedure but this preliminary work provides a strong argument for the adoption of the Cyanide leaching using Leachwell technique for the analysis of samples containing coarse gold and/or for gold associated with challenging elements/minerals.
- Detailed cost study must be done to estimate the consumable and maintenance costs.

- Detailed investigation on the cause and preventive measure on the clogging of the burner and nebuliser when reading Leachwell solutions on the AAS direct or alternatively gold extraction from the Leachwell solutions using DIBK and aliquat-336 should be investigated into details.

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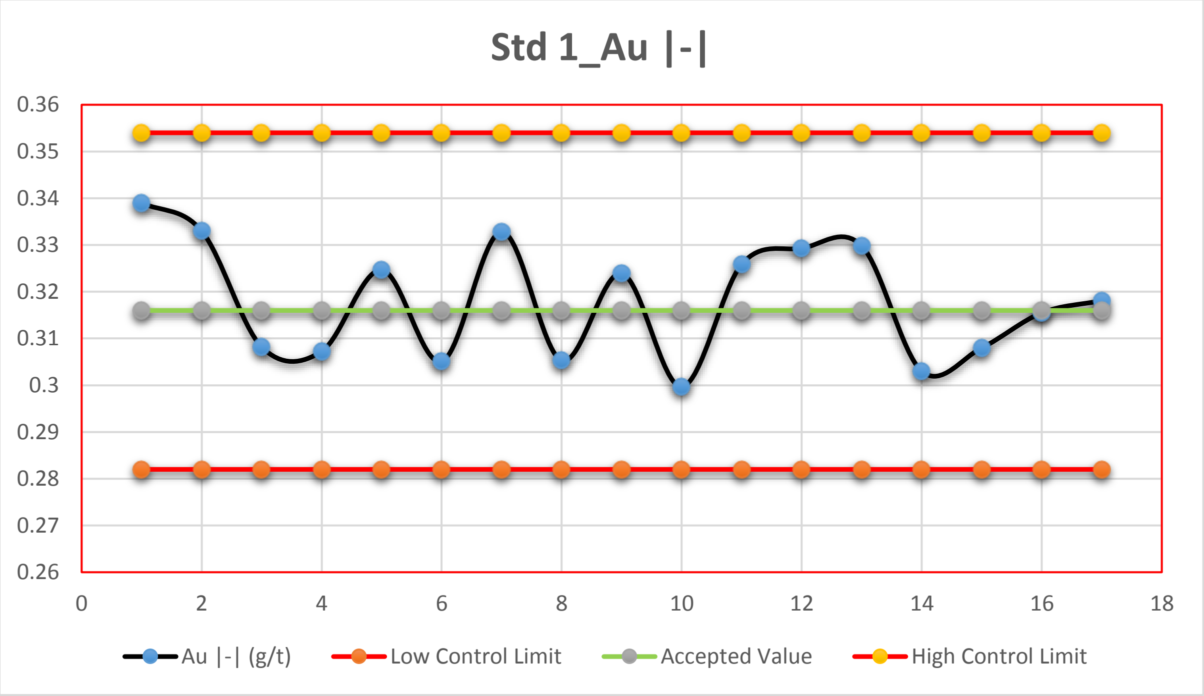
7. Appendix

Appendix A: Quality controls and quality assurance

To assure and control the quality of the assays/Data obtained in the study, certified reference materials (CRM) were used to control accuracy and precision of the assays. Three standards with different gold content in different ranges were used, namely standard 1, standard 2 and standard 3. The assays of the above mentioned standards are graphically presented below;

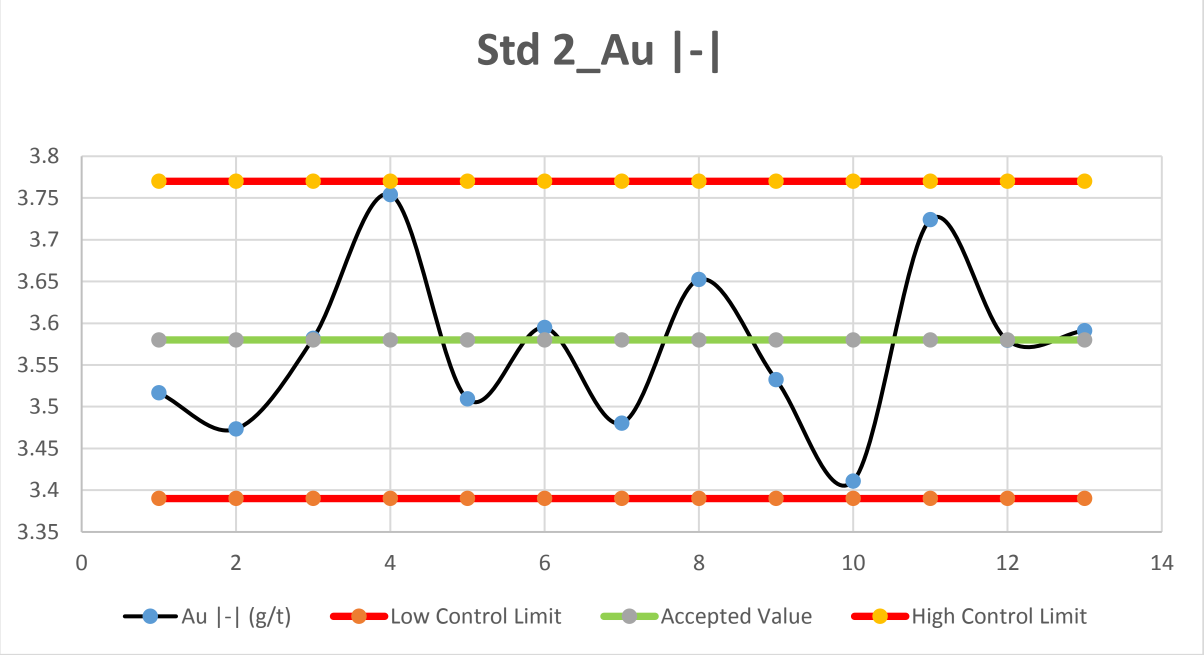
a. Standard 1 used (direct and screen fire assay).

Standard 1 has a certified gold value of 0.316 g/Mt, with acceptable range from 0.282 to 0.350 g/Mt Au, used to control samples with low Au value as from 0.6 g/Mt to zero. Standard 1 was only used to control assay quality of minus 106 microns fractions and pulps. 17 samples of standard 1 certified reference materials were used to control research assays and presented below.



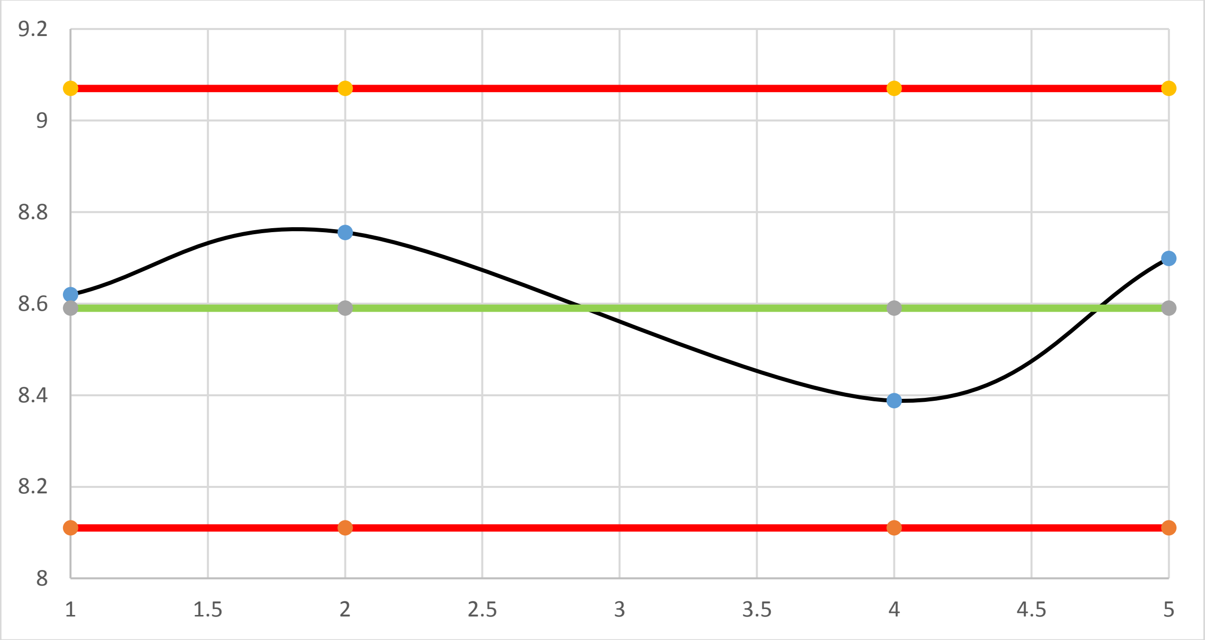
b. Standard 2 used (direct and screen fire assay).

Standard 2 has a certified gold content of 3.58 g/Mt, with acceptable range from 3.39 to 3.77 g/Mt Au. Standard 2 was used to control samples with moderate to high Au content as from 0.7 g/Mt upward and was only used to control assay quality of minus 106 microns fractions and pulps. 13 samples of standard 2 were used as shown below.



c. Standard 3 used (screen fire assay)

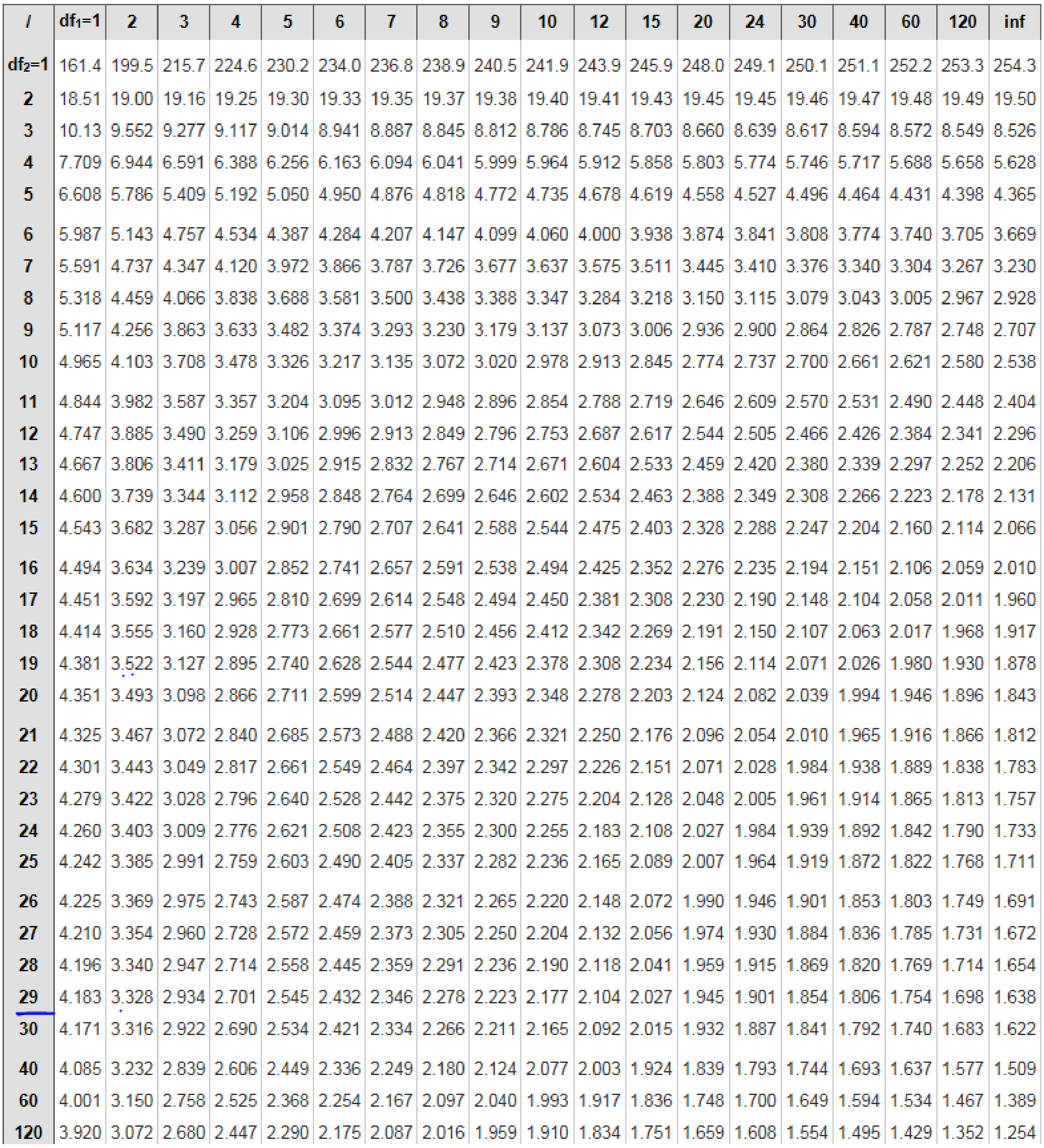
Standard 3 was used to control and assure quality of gravimetric assays obtained from plus 105 micron fractions. The gravimetric certified gold value in standard 3 is 8.59 g/Mt, with range from 8.11 to 9.07 g/Mt Au.



Appendix B: Samples Information

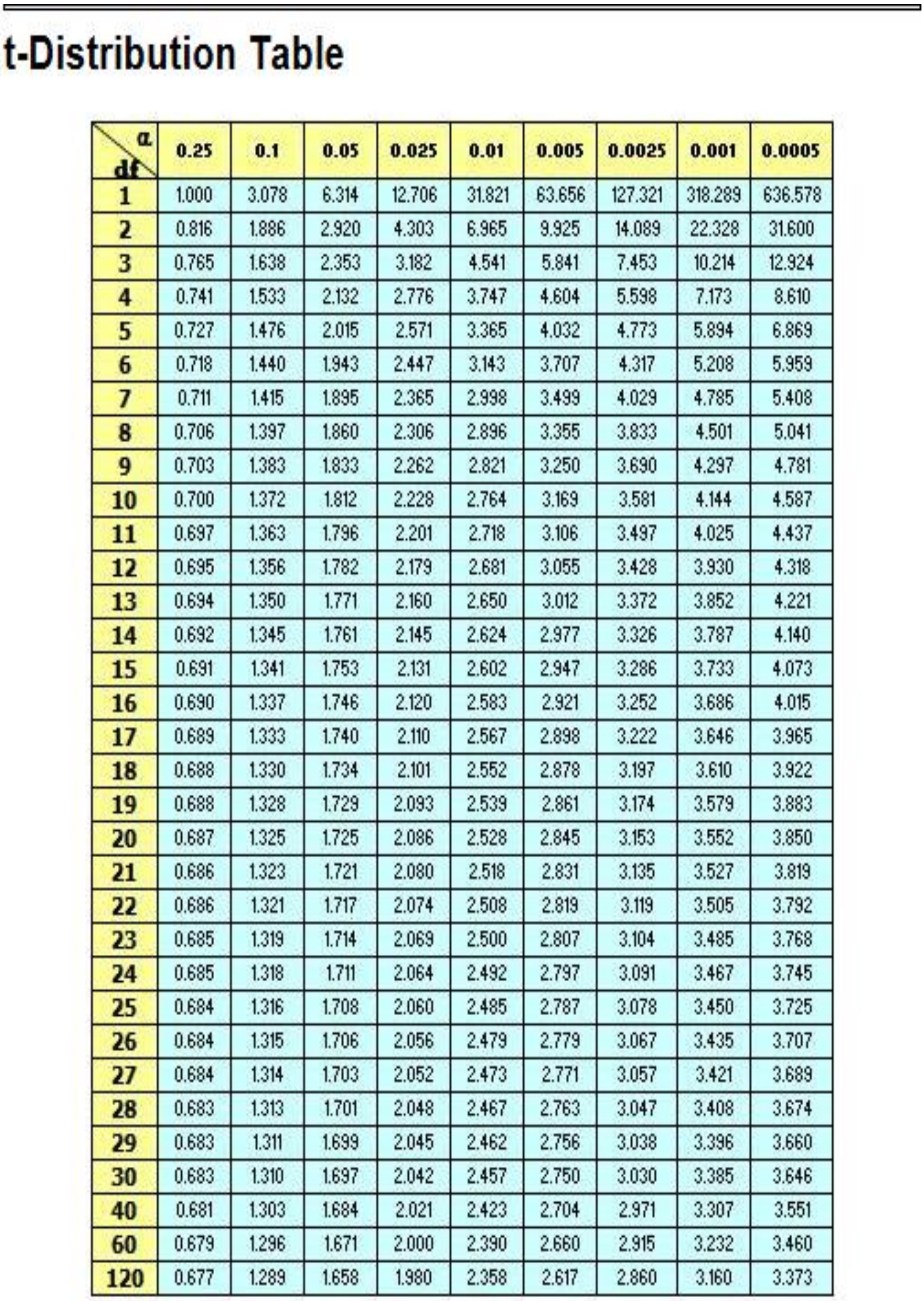
Sample ID	Sample Colour	Gross Sample weight	HOLE NUMBER	coordinates.Easting	coordinates.Northing	coordinates.Elevation	Rock type	Date sampled	Sample length(m)	Samp
B2_WS_01	Yellow	8.4645	W425_006_042	7790075.156	721114.576	1435	Albitite	18 August 2017	2	Cl
B2_WS_02	Yellowish Red	8.5415	W455_008_143	7790031.769	721050.16	1465	Marble	21 March 2017	2	Cl
B2_WS_03	Redish Yellow	8.7745	W455_008_139	7790055.406	721046.001	1465	Albitite	21 March 2017	2	Cl
B2_WS_04	Purplish Grey sample	8.464	W455_008_143	7790031.769	721050.16	1465	Albitite	21 March 2017	2	Cl
B2_WS_05	Grey	10.914	W455_007_249	7790110.998	721084.957	1465	Albitite	05 April 2017	2	Cl
B2_WS_06	Redish grey	12.295	W435_008_071	7790015.735	721019.449	1445	Albitite	12 July 2017	2	Cl
B2_WS_07	Whitish Grey	10.935	W425_007_189	7789969.854	721035.663	1435	Albitite	17 August 2017	2	Cl
B2_WS_08	Greyish white	10.937	W425_008_271	7789932.078	720977.355	1434.739	MARBLE	09 August 2017	2	Cl
B2_WS_09	Grey white (Hard to pulp)	9.5115	W455_008_140	7790049.497	721047.041	1465	Albitite	21 March 2017	2	Cl
B2_WS_10	Dark grey	8.6255	W435_008_066	7790014.346	721011.571	1445	Albitite	12 July 2017	2	Cl
B2_WS_11	Grey	12.055	A455_032_794	7788688.367	719904.276	1465	Biotite Banded Amphibole	12 May 2017	2	Cl
B2_WS_12	Grey	10.1815	W435_008_066	7790014.346	721011.571	1445	Albitite	12 July 2017	2	Cl
B2_WS_13	grey	9.3525	W435_008_066	7790014.346	721011.571	1445	Albitite	12 July 2017	2	Cl
B2G_OTJ_14	Dark grey	11.268	A445_031_083	7788691.059	720012.416	1455.316	Albitite	01 November 2017	2	Cl
B2_WS_15	Grey	8.6135	A455_032_825	7788705.465	719922.155	1465	Amphibole	11 May 2017	2	Cl
B2G_OTJ_16	Grey	8.348	A455_032_794	7788688.367	719904.276	1465	Biotite Banded Amphibole	12 May 2017	2	Cl
B2G_OTJ_17	Dark grey	9.0425	A445_031_082	7788701.279	720018.796	1455.19	Amphibole	01 October 2017	2	Cl
B2G_OTJ_18	Dark grey	8.889	A445_031_086	7788700.691	719997.336	1455.562	Albitite	01 December 2017	2	Cl
B2G_OTJ_19	Dark grey	10.301	A445_031_081	7788704.428	720013.531	1455.183	Albitite	01 September 2017	2	Cl
B2G_OTJ_20	Dark grey	8.4655	A455_032_822	7788695.861	719937.38	1465	Albitite	11 May 2017	2	Cl
B2G_WS_21	Red	9.1575	W455_008_145	7790019.951	721052.24	1465	Fault Breccia	20 March 2017	2	Cl
B2G_WS_22	Red	9.1575	W455_008_144	7790025.86	721051.2	1465	Fault Breccia	20 March 2017	2	Cl
B2G_WS_23	Red	9.311	W455_008_146	7790014.042	721053.28	1465	Fault Breccia	20 March 2017	2	Cl
B2G_WS_24	Red	11.063	W455_008_145	7790019.951	721052.24	1465	Fault Breccia	20 March 2017	2	Cl
B2G_WS_25	Redish-Yellowish grey	8.25	W425_007_169	7790025.812	721042.043	1435	Albitite	21 August 2017	2	Cl
B2G_WS_26	Redish Yellow	8.323	W455_009_059	7790001.564	720982.369	1465	Fault Breccia	27 March 2017	2	Cl
B2G_WS_27	Whitish Grey	8.1185	W455_007_288	7790083.617	721101.655	1465.119	Albitite	24 March 2017	2	Cl
B2G_WS_28	Purplish Grey	8.7325	W425_008_234	7789955.692	721005.586	1434.602	Albitite	10 August 2017	2	Cl
B2G_WS_29	grey	8.129	W455_008_141	7790043.588	721048.081	1465	Albitite	21 March 2017	2	Cl
B2G_WS_30	grey	8.9465	W425_008_225	7789956.876	721013.651	1434.807	Albitite	08 August 2017	2	Cl

Appendix C: F Table



/	$df_1=1$	2	3	4	5	6	7	8	9	10	12	15	20	24	30	40	60	120	inf
$df_2=1$	161.4	199.5	215.7	224.6	230.2	234.0	236.8	238.9	240.5	241.9	243.9	245.9	248.0	249.1	250.1	251.1	252.2	253.3	254.3
2	18.51	19.00	19.16	19.25	19.30	19.33	19.35	19.37	19.38	19.40	19.41	19.43	19.45	19.45	19.46	19.47	19.48	19.49	19.50
3	10.13	9.552	9.277	9.117	9.014	8.941	8.887	8.845	8.812	8.786	8.745	8.703	8.660	8.639	8.617	8.594	8.572	8.549	8.526
4	7.709	6.944	6.591	6.388	6.256	6.163	6.094	6.041	5.999	5.964	5.912	5.858	5.803	5.774	5.746	5.717	5.688	5.658	5.628
5	6.608	5.786	5.409	5.192	5.050	4.950	4.876	4.818	4.772	4.735	4.678	4.619	4.558	4.527	4.496	4.464	4.431	4.398	4.365
6	5.987	5.143	4.757	4.534	4.387	4.284	4.207	4.147	4.099	4.060	4.000	3.938	3.874	3.841	3.808	3.774	3.740	3.705	3.669
7	5.591	4.737	4.347	4.120	3.972	3.866	3.787	3.726	3.677	3.637	3.575	3.511	3.445	3.410	3.376	3.340	3.304	3.267	3.230
8	5.318	4.459	4.066	3.838	3.688	3.581	3.500	3.438	3.388	3.347	3.284	3.218	3.150	3.115	3.079	3.043	3.005	2.967	2.928
9	5.117	4.256	3.863	3.633	3.482	3.374	3.293	3.230	3.179	3.137	3.073	3.006	2.936	2.900	2.864	2.826	2.787	2.748	2.707
10	4.965	4.103	3.708	3.478	3.326	3.217	3.135	3.072	3.020	2.978	2.913	2.845	2.774	2.737	2.700	2.661	2.621	2.580	2.538
11	4.844	3.982	3.587	3.357	3.204	3.095	3.012	2.948	2.896	2.854	2.788	2.719	2.646	2.609	2.570	2.531	2.490	2.448	2.404
12	4.747	3.885	3.490	3.259	3.106	2.996	2.913	2.849	2.796	2.753	2.687	2.617	2.544	2.505	2.466	2.426	2.384	2.341	2.296
13	4.667	3.806	3.411	3.179	3.025	2.915	2.832	2.767	2.714	2.671	2.604	2.533	2.459	2.420	2.380	2.339	2.297	2.252	2.206
14	4.600	3.739	3.344	3.112	2.958	2.848	2.764	2.699	2.646	2.602	2.534	2.463	2.388	2.349	2.308	2.266	2.223	2.178	2.131
15	4.543	3.682	3.287	3.056	2.901	2.790	2.707	2.641	2.588	2.544	2.475	2.403	2.328	2.288	2.247	2.204	2.160	2.114	2.066
16	4.494	3.634	3.239	3.007	2.852	2.741	2.657	2.591	2.538	2.494	2.425	2.352	2.276	2.235	2.194	2.151	2.106	2.059	2.010
17	4.451	3.592	3.197	2.965	2.810	2.699	2.614	2.548	2.494	2.450	2.381	2.308	2.230	2.190	2.148	2.104	2.058	2.011	1.960
18	4.414	3.555	3.160	2.928	2.773	2.661	2.577	2.510	2.456	2.412	2.342	2.269	2.191	2.150	2.107	2.063	2.017	1.968	1.917
19	4.381	3.522	3.127	2.895	2.740	2.628	2.544	2.477	2.423	2.378	2.308	2.234	2.156	2.114	2.071	2.026	1.980	1.930	1.878
20	4.351	3.493	3.098	2.866	2.711	2.599	2.514	2.447	2.393	2.348	2.278	2.203	2.124	2.082	2.039	1.994	1.946	1.896	1.843
21	4.325	3.467	3.072	2.840	2.685	2.573	2.488	2.420	2.366	2.321	2.250	2.176	2.096	2.054	2.010	1.965	1.916	1.866	1.812
22	4.301	3.443	3.049	2.817	2.661	2.549	2.464	2.397	2.342	2.297	2.226	2.151	2.071	2.028	1.984	1.938	1.889	1.838	1.783
23	4.279	3.422	3.028	2.796	2.640	2.528	2.442	2.375	2.320	2.275	2.204	2.128	2.048	2.005	1.961	1.914	1.865	1.813	1.757
24	4.260	3.403	3.009	2.776	2.621	2.508	2.423	2.355	2.300	2.255	2.183	2.108	2.027	1.984	1.939	1.892	1.842	1.790	1.733
25	4.242	3.385	2.991	2.759	2.603	2.490	2.405	2.337	2.282	2.236	2.165	2.089	2.007	1.964	1.919	1.872	1.822	1.768	1.711
26	4.225	3.369	2.975	2.743	2.587	2.474	2.388	2.321	2.265	2.220	2.148	2.072	1.990	1.946	1.901	1.853	1.803	1.749	1.691
27	4.210	3.354	2.960	2.728	2.572	2.459	2.373	2.305	2.250	2.204	2.132	2.056	1.974	1.930	1.884	1.836	1.785	1.731	1.672
28	4.196	3.340	2.947	2.714	2.558	2.445	2.359	2.291	2.236	2.190	2.118	2.041	1.959	1.915	1.869	1.820	1.769	1.714	1.654
29	4.183	3.328	2.934	2.701	2.545	2.432	2.346	2.278	2.223	2.177	2.104	2.027	1.945	1.901	1.854	1.806	1.754	1.698	1.638
30	4.171	3.316	2.922	2.690	2.534	2.421	2.334	2.266	2.211	2.165	2.092	2.015	1.932	1.887	1.841	1.792	1.740	1.683	1.622
40	4.085	3.232	2.839	2.606	2.449	2.336	2.249	2.180	2.124	2.077	2.003	1.924	1.839	1.793	1.744	1.693	1.637	1.577	1.509
60	4.001	3.150	2.758	2.525	2.368	2.254	2.167	2.097	2.040	1.993	1.917	1.836	1.748	1.700	1.649	1.594	1.534	1.467	1.389
120	3.920	3.072	2.680	2.447	2.290	2.175	2.087	2.016	1.959	1.910	1.834	1.751	1.659	1.608	1.554	1.495	1.429	1.352	1.254

Appendix D: t- distribution table



t-Distribution Table

α df	0.25	0.1	0.05	0.025	0.01	0.005	0.0025	0.001	0.0005
1	1.000	3.078	6.314	12.706	31.821	63.656	127.321	318.289	636.578
2	0.816	1.886	2.920	4.303	6.965	9.925	14.089	22.328	31.600
3	0.765	1.638	2.353	3.182	4.541	5.841	7.453	10.214	12.924
4	0.741	1.533	2.132	2.776	3.747	4.604	5.598	7.173	8.610
5	0.727	1.476	2.015	2.571	3.365	4.032	4.773	5.894	6.869
6	0.718	1.440	1.943	2.447	3.143	3.707	4.317	5.208	5.959
7	0.711	1.415	1.895	2.365	2.998	3.499	4.029	4.785	5.408
8	0.706	1.397	1.860	2.306	2.896	3.355	3.833	4.501	5.041
9	0.703	1.383	1.833	2.262	2.821	3.250	3.690	4.297	4.781
10	0.700	1.372	1.812	2.228	2.764	3.169	3.581	4.144	4.587
11	0.697	1.363	1.796	2.201	2.718	3.106	3.497	4.025	4.437
12	0.695	1.356	1.782	2.179	2.681	3.055	3.428	3.930	4.318
13	0.694	1.350	1.771	2.160	2.650	3.012	3.372	3.852	4.221
14	0.692	1.345	1.761	2.145	2.624	2.977	3.326	3.787	4.140
15	0.691	1.341	1.753	2.131	2.602	2.947	3.286	3.733	4.073
16	0.690	1.337	1.746	2.120	2.583	2.921	3.252	3.686	4.015
17	0.689	1.333	1.740	2.110	2.567	2.898	3.222	3.646	3.965
18	0.688	1.330	1.734	2.101	2.552	2.878	3.197	3.610	3.922
19	0.688	1.328	1.729	2.093	2.539	2.861	3.174	3.579	3.883
20	0.687	1.325	1.725	2.086	2.528	2.845	3.153	3.552	3.850
21	0.686	1.323	1.721	2.080	2.518	2.831	3.135	3.527	3.819
22	0.686	1.321	1.717	2.074	2.508	2.819	3.119	3.505	3.792
23	0.685	1.319	1.714	2.069	2.500	2.807	3.104	3.485	3.768
24	0.685	1.318	1.711	2.064	2.492	2.797	3.091	3.467	3.745
25	0.684	1.316	1.708	2.060	2.485	2.787	3.078	3.450	3.725
26	0.684	1.315	1.706	2.056	2.479	2.779	3.067	3.435	3.707
27	0.684	1.314	1.703	2.052	2.473	2.771	3.057	3.421	3.689
28	0.683	1.313	1.701	2.048	2.467	2.763	3.047	3.408	3.674
29	0.683	1.311	1.699	2.045	2.462	2.756	3.038	3.396	3.660
30	0.683	1.310	1.697	2.042	2.457	2.750	3.030	3.385	3.646
40	0.681	1.303	1.684	2.021	2.423	2.704	2.971	3.307	3.551
60	0.679	1.296	1.671	2.000	2.390	2.660	2.915	3.232	3.460
120	0.677	1.289	1.658	1.980	2.358	2.617	2.860	3.160	3.373

Appendix E: Comparing the gold assays obtained using six different analytical techniques

Sample ID	DFA	SFA	Lwell	Leach	Aregia	Macids	Average
B2G~WS~01	1.81	1.67	1.50	2.15	2.84	4.59	2.42
B2G~WS~02	7.20	7.22	8.08	8.76	8.29	8.54	8.01
B2G~WS~03	5.06	2.11	3.51	2.04	1.70	3.13	2.92
B2G~WS~04	8.67	7.43	5.66	5.54	11.20	10.38	8.15
B2G~WS~05	6.24	1.91	2.78	1.89	2.15	5.23	3.37
B2G~WS~06	42.81	32.89	28.27	20.70	26.60	22.83	29.02
B2G~WS~07	7.86	8.62	6.67	6.76	7.21	4.21	6.89
B2G~WS~08	4.86	10.24	16.98	15.53	2.58	8.66	9.81
B2G~WS~09	1.63	2.82	2.80	2.19	5.19	1.51	2.69
B2G~WS~10	36.64	33.55	27.78	26.15	32.01	21.17	29.55
B2G~WS~11	25.85	33.06	19.81	15.04	16.88	18.75	21.57
B2G~WS~12	18.39	21.23	17.55	13.10	16.97	27.07	19.05
B2G~WS~13	21.24	21.05	17.32	17.24	24.67	9.45	18.50
B2G~OTJ~14	0.76	0.75	14.12	6.44	1.16	0.72	3.99
B2G~OTJ~15	2.37	1.79	4.48	3.16	5.47	2.48	3.29
B2G~OTJ~16	3.48	1.10	2.69	1.21	2.31	2.60	2.23
B2G~OTJ~17	2.04	1.29	2.24	1.94	1.65	1.73	1.81
B2G~OTJ~18	0.62	1.18	1.04	0.94	2.90	0.55	1.21
B2G~OTJ~19	4.08	4.19	3.74	3.01	33.61	8.64	9.54
B2G~OTJ~20	24.86	38.38	15.11	9.68	52.07	10.27	25.06
B2G~WS~21	15.29	13.84	13.08	12.41	12.73	9.00	12.72
B2G~WS~22	11.26	11.38	12.70	9.64	9.10	16.49	11.76
B2G~WS~23	7.65	6.87	6.36	6.07	10.03	9.78	7.79
B2G~WS~24	6.77	15.43	12.04	10.57	17.70	17.25	13.29
B2G~WS~25	3.53	5.69	3.28	2.72	2.26	3.65	3.52
B2G~WS~26	1.14	3.64	2.94	1.93	1.75	1.75	2.19

B2G~WS~27	0.46	0.91	1.37	1.14	0.90	1.05	0.97
B2G~WS~28	6.47	13.29	12.77	9.02	56.61	8.05	17.70
B2G~WS~29	9.45	6.67	7.35	5.70	12.01	9.51	8.45
B2G~WS~30	3.06	4.53	3.32	3.87	2.01	2.33	3.19
Mean	9.72	10.49	9.24	7.55	12.75	8.38	9.69