

The Relative Bias Errors of Gravimetric Fire-Assaying Practice for Platinum-Group Elements in Bushveld Merensky Ore at Rustenburg

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SUMMARY

The evaluation of PGE in Rustenburg Merensky ore and mineral process products has been studied with potential biases as a specific issue. It was found that the lead-collection fire-assaying method, which is widely used in South Africa for this purpose, is not robust to the complex form of PGE mineral host occurrence in this ore type. Mill feed and tailings both show a potential for underestimation of grade, whilst concentrate shows a potential for overstatement of grade. These biases are minimised by an approach of optimal comminution of the sample prior to fire-assaying. Ball-milling of the mill feed and tailings to 70% mass passing 75 micrometers (μm) maximises the PGE grade estimated, whereas pulverisation of the concentrate sample in analytical silica prior to fire-assaying reduces the grade estimated. It is believed that these biases are caused by two different mechanisms. The mill feed and tailings effect is believed to arise from incomplete liberation of the PGE mineral hosts in those samples, leading to incomplete recovery of the PGE to the lead collection button. The concentrate effect is believed to be caused by the heterogeneous nature of the material, viz. carrying PGE mineral hosts across six orders of magnitude in grade and across a variety of mineral densities and grain sizes. Optimal treatments for these biases are proposed in this paper. Using data accepted at the 95% level, the potential biases found are: mill feed: - 4.1%; tailings: - 9.3%, and concentrate: + 2.29%. It is shown that these biases have significant influences on estimates of recoveries in mineral processes. Unless minimised using methods outlined in this paper, these biases can seriously confound the effect of process variables. In a typical mass balance across a milling and flotation process, these errors combine to form an overall bias of some 5.2% in PGE metal recovery, or if calculated off a reconciled head grade, would typically show an unaccounted gain of some 5.2%. Using raw data with outliers present, these relative biases are: mill feed: -1.7%; concentrate: -1.3% and tailings, 12.9%, culminating in an overall bias of 2.5%. In either case, an overall positive bias is shown which would over-estimate PGE recovery across a concentrator.

Introduction

Numerous references exist for the fire-assaying of gold-bearing samples in the South African mining industry, some of which are noted here [Eissler, 1896; Adamson, 1972, Lenehan and de L. Murray-Smith, 1986]. Most of these describe a pre-concentration fusion step followed by cupellation and gravimetric determination of the gold prill after appropriate parting for silver. The adaptation of this practice to the gravimetric determination of the PGE in evaluation of the Merensky and UG2 ore types of the Bushveld Complex, South Africa, found favour because it was more rapid than the lengthy classical wet analytical technique [Lenehan and de L. Murray-Smith, 1986]. This fire-assay technique for the PGE-bearing ore types and metallurgical process products differs from the equivalent method for determination of gold in Witwatersrand gold-bearing ore types for several reasons. These include the alteration of the flux to suit the more

basic nature of the Merensky host rock, and resetting the fusion times and temperatures of the two cupelling stages.

Typical Fire-Assaying Practice for Gravimetric Determinations on Samples of Merensky Reef

Fire assaying is commonly used in the determination of trace concentrations of precious metals in ores and mineral process products. This is because it pre-concentrates the precious metals from the sample by fusion with a flux. This produces a lead button, which collects the precious metals present, leaving a slag, which is separated from the lead button. The flux used typically contains litharge, sodium carbonate and borax. A reducing agent, usually maize (corn) meal, is used to react with the litharge and produce lead metal and carbon dioxide. Occasionally potassium nitrate is added to the flux to oxidise any sulphide minerals present in the sample. This is to counteract the naturally reducing nature of the base metal sulphides.

Lead Fusion Stage

The purpose of this stage is to quantitatively pre-concentrate the precious metals into a lead button. The weighed sample is mixed with the flux and the mixture placed in a crucible. Batches of weighed sample and flux are prepared and placed in a series of crucibles. This batch of crucibles and their contents is heated in a pre-heated furnace, which is operated under controlled conditions. The rate of heating and final temperature during the fusion are critical controls. The initial furnace temperature is 1100⁰ C. As the batch of crucibles is loaded into the furnace, the temperature drops to approximately 850⁰ C, and rises again to 1100⁰ C over a 45-minute period. On completion of the fusion the crucibles are unloaded singly from the furnace, and the crucible contents poured into an inverse conical mould. The fusion products settle and solidify as they cool. After cooling the mould is inverted and the lead button removed from the slag. The efficient recovery of the PGE present in the sample to the lead button, which is critical to the reliability of this method, is dependent on appropriate comminution of the sample prior to fusion, thorough mixing of the sample and flux, and temperature control during the fusion. The mass of metallic lead is also an important factor, which needs to be controlled during the adjustment of flour or maize (corn) meal and nitre (potassium nitrate). Volatile losses have also been discussed, which have been treated or limited by the use of lids on the fusion pots [Munro, 1993]. The typical flux compositions for the fusion of samples of Merensky ore and mineral process products are summarised in Table 1 [Williams, 1994].

Table 1 - Typical Lead Fusion Flux Composition

Flux Constituent % By Mass	Ore and Tailings Samples	Flotation Concentrate Samples
Sodium Carbonate	35.1	31.5
Borax	26.3	23.6
Silica	8.8	15.7
Litharge	26.3	23.6
Maize (Corn) Meal	3.5	3.2
Potassium Nitrate	0.0	2.4

Low-Temperature Cupellation Stage

The purpose of this stage is to quantitatively remove the lead from the precious metals. The lead buttons are placed individually on preheated cupels, in a cupellation furnace at 900-1100 degrees centigrade. The cupels are made from calcined magnesite. These cupels absorb the molten lead and leave behind a prill of precious metals. Some of the lead is volatilised during this process. The product of this stage is called a 'low-temperature prill', or 'LT prill'.

High-Temperature Cupellation Stage

The purpose of this stage is to quantitatively burn off the volatile precious metals Ru, Os, and Ir, since partial losses of these occur in the LT cupellation stage. This leaves a 'high-temperature prill', or 'HT prill', after HT cupellation at 1300-1350 degrees. This also incurs secondary losses of the four remaining precious metals, Pt, Pd, Au and Rh.

Gravimetric Determination

The HT prills produced from the above processes weigh between 1 mg for flotation concentrate samples, and 0.1 mg for low-grade tailings samples. The handling of these prills is therefore a delicate matter, as breakage of the prill during handling negates all of the foregoing effort to quantitatively produce a prill for weighing on a microbalance.

Assay Replicates

Quadruplicate determinations are the standard practice in production metal-accounting laboratory operations. The arithmetic mean of these replicates is calculated after inspection of the replicate values for precision; this introduces a necessary quality control process that, unless appropriately formulated with knowledge of the complex mineralogy and skew distributions, will bias the estimation of mean grade. This matter is outside the scope of this paper, and will be separately reported. The experimental work reported here, however, used multiple replicate determinations per test condition in order to overcome this difficulty.

Correction Factors

In routine fire-assaying of production metal accounting samples which have a processing and analytical history, empirical correction factors are developed which correct for the cumulative minor loss of Pt, Pd, Au and Rh in the LT and HT cupellation stages. The factor is only applied to the HT prill mass. LT prills are an intermediate stage of the process and are not weighed. These factors are in the order of 3% in ore samples, 7% for tailings samples, and 1% for concentrate samples. Nickel collection is used to calibrate these factors opposite multiple replicate fire-assay determinations of the sample samples. The procedure of nickel collection is outside the scope of this paper, except to note that this method produces a more complete determination of the precious metals [Robert et. al., 1975, Munro, 1994]. In this paper the units of PGE quoted shall be '4 element uncorrected', i.e. the sum of Pt, Pd, Au and Rh, without correction factors applied.

Description of the Main Mineralogical Features of the Merensky Ore Type

The Bushveld Complex is an extensive geological system in the Northwest and Northern provinces of South Africa. Bushveld Complex rocks are also seen westwards into Botswana. In the Rustenburg Section of Rustenburg Platinum Mines, it was noted that the Merensky Reef Unit varies from 9 to 15 m in width. This unit starts with a pyroxenitic layer with associated thin chromitite layers, overlain by a differentiated suite of norite, anorthositic norite, spotted

anorthosite, and mottled, or poikilitic, anorthosite. The Merensky Reef forms the lowermost part of the basal pyroxenite/chromitite assemblage of the Merensky Reef Unit [Viljoen and Hieber, 1982].

Specifically the Merensky Reef commences with a 1cm thin chromitite layer often resting on anorthositic norite; the chromitite layer is well defined and is overlain by a pegmatoidal feldspathic pyroxenite. This pegmatoidal layer is generally some 25 cm thick, and comprises subhedral- to euhedral- orthopyroxene crystals with less abundant plagioclase feldspar. A second 1 cm chromitite layer occurs at the top of this coarse-grained unit, and demarcates the top of the geological entity called the Merensky Reef of Rustenburg Platinum Mines.

The regional features of this system show that variations in the Merensky Reef are emulated in the lower UG2 system in the same vicinity [Kinloch, 1982]. This variation is characterised by the relative abundance of the discrete platinum group minerals (PGM). In particular, isoferroplatinum (Pt_3Fe) and the platinum-palladium sulphides exchange as the dominant discrete PGM form at a given site, depending on the geological history of a particular area of the Bushveld. In areas where secondary activity such as a volcanic intrusion occurred, isoferroplatinum is dominant over the platinum-palladium sulphides. In Rustenburg Section of Rustenburg Platinum Mines, the platinum-palladium sulphides are dominant over isoferroplatinum.

In terms of the general base metal sulphide mineralogy, the Merensky ore type hosts chalcopyrite, pentlandite, and pyrrhotite with minor pyrite. The pentlandite and pyrrhotite are enriched by PGE in solid solution to varying degrees. Also, blebs of discrete PGM occur as intergrowths with pentlandite and pyrrhotite. For Rustenburg, the relative abundance of the PGM is described in Table 2, and the enrichment of the base metal sulphides, in Table 3.

**Table 2 - Relative Abundance of the PGM at Rustenburg
(After Kinloch, 1982)**

Platinum Group Mineral	Volume Distribution as % of all PGM
iso-Ferroplatinum Pt_3Fe	1.7
Electrum (AuAg)	3.3
Sperrylite (PtAs_2)	6.0
Laurite (RuS_2)	5.2
Pt-Pd Tellurides	2.6
Pt-Pd Sulphides	80.9

**Table 3 - Tenor of PGE in Base Metal Sulphides at Rustenburg
(After Kinloch, 1982)**

Base Metal Sulphide	Pt ppm	Pd ppm	Rh ppm
Pentlandite	11	140	23
Pyrrhotite	11	0	3
Pyrite	15	34	9

Other work, which focussed on the metallurgical implications of the modes of occurrence of PGE amongst the entire PGE host mineral set, found that silicates and oxides also accounted for a small amount of PGE in the Merensky [Peyerl, 1983]. Although the purpose of that

investigation was to account for the flotation behaviour of the various PGE hosts, it also became clear that some 5-10% of all PGE assayed in this ore was present as solid solution in silicates and oxides. The degree of enrichment of this silicate/oxide phase was dependent on the extent of serpentinisation in the particular area of the Bushveld.

When the relative grain sizes are considered, the minerals hosting PGE in Merensky become vulnerable to certain evaluation problems. The average grain size of discrete PGM in the Rustenburg Merensky is 26 μm , with a size range from 1 to 840 μm . The fire-assaying of ore and mineral process samples of this orebody therefore faces a complex task in addressing diverse mineral hosts across several orders of magnitude in PGE grade.

Technical Exercises on Replicate Sampling and Assaying

Method

Representative samples of Merensky mill feed, final tailings and final concentrate were taken from the Frank Concentrator of Rustenburg Platinum Mines Limited. The rules for representative sampling were observed [Lotter, 1995b]. The lead-collection fire-assay method described in this paper was used throughout the analysis of the samples. This included both LT and HT cupellation. Gravimetric determination was performed on the LT prills.

Mill Feed

The mill feed sample was dried in a thermostatically-controlled oven at 106° C, and then crushed to a limiting size of 3mm. The crushed product was blended in a spinning riffler using a specialised technique called 'odds-and-evens blending' [Lotter, 1995a]. The spinning riffler is known as a superior technology for this purpose [Allen, 1990], and is used without exception where thorough blending is required. The blended mill feed sample was then subsampled using the spinning riffler to produce replicate 2.5 kg test lots of ore. The proportionality of the spinning riffler allowed 25-kg charges, which were batch-weighed, to produce 2.5 kg subsample units. Test lots of 2.5 kg sample were chosen randomly from this suite and milled in a laboratory scale ball mill to produce milled charges at grinds of 40, 50, 60 and 70% mass passing 75 μ . The process was wet, *i.e.* operated at a pulp density similar to that of a primary ball mill in the production process. This is typically 80% solids w/v. Each milled charge was quantitatively removed from the mill and filtered then dried at 106° C. The milled charges were then separately reblended and subsampled using the spinning riffler in preparation for fire-assaying.

This arrangement is shown in Figure 1.

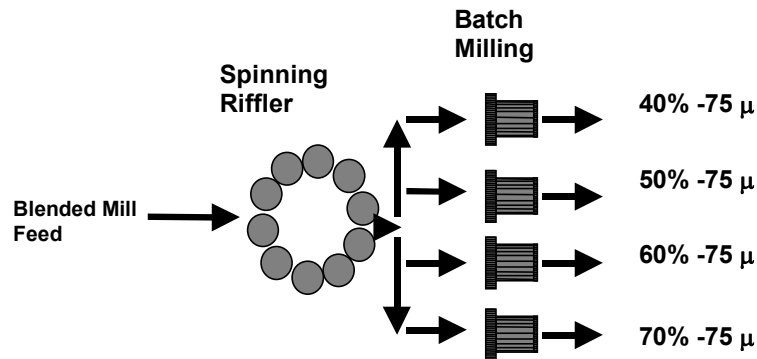


Figure 1 – Subsampling and Batch Milling Arrangement for Mill Feed

Final Concentrate

Similar arrangements to the mill feed were made, except that the sample was not crushed and milled. Drying was performed at a lower temperature (80° C), and dried final concentrate sample blended using the odds-and-evens method. Smaller subsamples (50 g) were produced from the spinning riffler.

Three groups of subsamples were extracted from this set.

1. Group A was selected and set aside 'as is'. This group is referred to as 'blended'.
2. Group B was prepared by batch-pulverising 50 g charges with matched masses of analytical silica in a ring pulveriser. The pulveriser operation time was 2 minutes, controlled using a timer. This group is referred to as 'silica ground'.
3. This pulveriser was tested for mass balance criteria prior to the testwork and was found to lose on average 0.2% of the sample mass. The product from this process was called 'pulverite'. The reason for producing group B was to test for the effect of homogenisation on the fire-assay result, since the concentrate as produced by the mineral process carries PGE in distinctly different mineral hosts that have natural PGE grades across 5 orders of magnitude. It is believed that the pulverisation-in-silica process transforms the discrete PGM (such as ferroplatinum or platinum-palladium sulphides) into an artificial solid solution in the analytical silica, thus reducing the grade of the ferroplatinum from some 40% PGE by some three orders of magnitude to an order of several hundred grams per tonne.
4. Group C was treated by laboratory scale superpanning to semi-quantitatively remove the discrete PGM present by gravity separation prior to assaying. A mass balance was determined, and both gravity concentrate and tailings were assayed. This group is referred to as 'superpanned'.

These arrangements are shown in Figure 2.

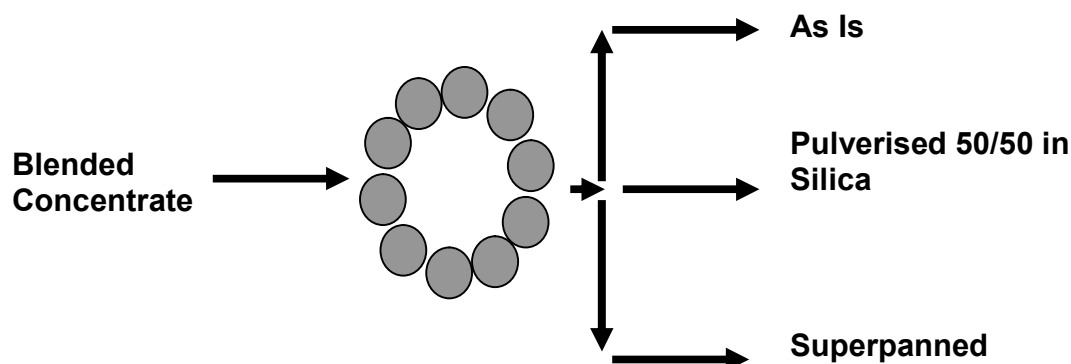


Figure 2 – Subsampling and Sample Treatments for Final Concentrate

Final Tailings

Similar arrangements to those for mill feed were made, except that there was no crushing. The tailings sample, having been produced from a production grinding process, had a particle size distribution equivalent to 43% mass passing 75 μm . Ball-milling of 2.5 kg subsamples of tailings produced ground charges of 50, 60 and 70% mass passing 75 μm . These milled charges were filtered and dried as for mill feed.

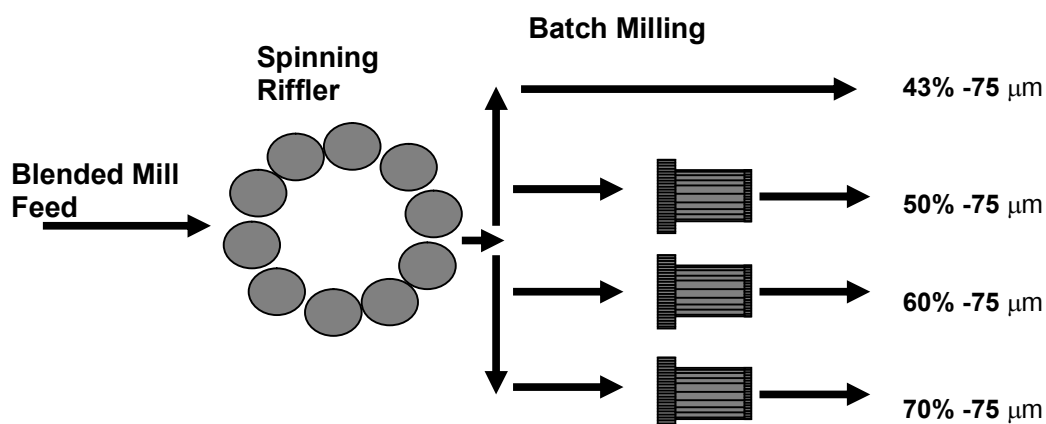


Figure 3 – Subsampling and Batch Milling of Tailings

Fire-Assaying

Lead-collection fire-assaying was used in all cases to estimate the PGE grade of the processed samples. For each category of sample, viz. mill feed, final concentrate and final tailings, the fusion mass of sample, or analyte mass, was adjusted within the test group so that prills of comparable mass would be obtained. For example, the final concentrate samples used 5 g for

Group A and 10 g for Group B, so producing similar prill masses. Mill feed analyte mass was 50 g, and final tailings, 125 g. Within each sample type, replicate fusions were arranged so that samples from each grinding condition were present in the 30-fusion tray. In this way, variation in furnace conditions could not be blamed for any difference in the results. Natural standards were included in the fusion tray layout to cross-check the unknown fusions. The degree of inspection was in the order of 4 standard fusions per tray.

RESULTS

Mill Feed

The overall results of the replicate fire-assaying of the mill feed samples by grind group are summarised in Table 5 and Figure 4. The screened data, *i.e.* those accepted within the sample mean $\pm$ 2 sample standard deviations, are also shown.

Table 4 - Analysis of Mill Feed PGE Grade by Grind Group

Parameter	Test Condition : Grind, % Sample Mass Passing 75 Microns							
	40 Raw	40 Screened	50 Raw	50 Screened	60 Raw	60 Screened	70 Raw	70 Screened
Mean Grade g/t PGE	1.000	0.988	0.998	1.003	1.026	1.010	1.017	1.029
RSD %	20.0	13.3	15.2	10.9	26.0	8.8	15.6	10.9
n	143	137	14.3	136	107	105	108	103

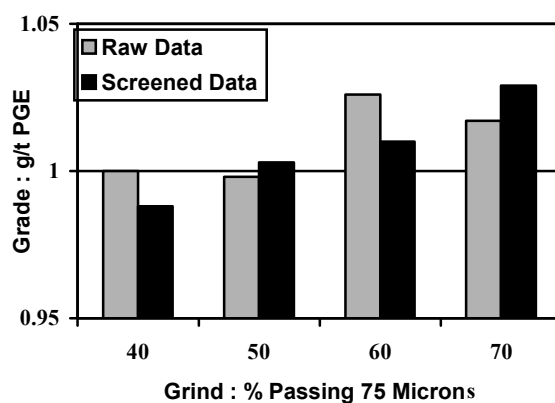


Figure 4 – Effect of Mill Feed Sample Grind on Mean Grade of PGE Estimated

Final Concentrate

The overall results of the replicate fire-assaying of the final concentrate samples by grind group are summarised in Table 5 and Figure 5.

Table 5 - Analysis of Final Concentrate PGE Grade by Sample Preparation Method

Raw Data Parameter	Test Condition : Sample Preparation Method					
	Group A As-Is		Group B Pulverised in Silica		Group C Prior Separation of Discrete PGM	
	Raw Data	Screened	Raw Data	Screened	Raw Data	Screened
Mean Grade g/t PGE	1.000	1.004	0.981	0.980	0.973	0.978
RSD %	3.06	2.28	2.66	2.45	2.14	1.45
n	60	58	56	54	12	11

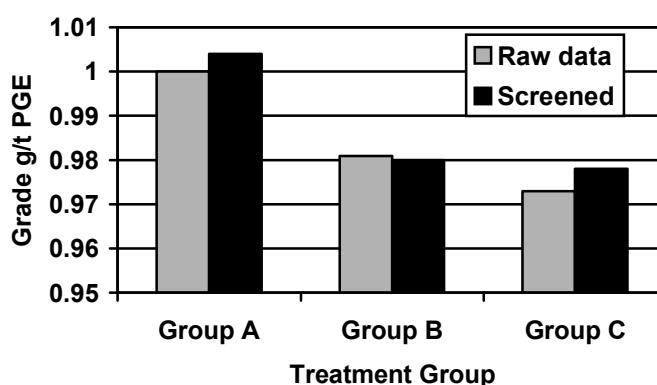


Figure 5— Effect of Sample Preparation Method of Concentrate on Mean Grade of PGE Estimated

Final Tailings

The overall results of the replicate fire-assaying of the mill feed samples by grind group are summarised in Table 6 and in Figure 3. The screened data, i.e. those accepted within the sample mean $\pm$ 2 sample standard deviations, are also shown.

Table 7 - Analysis of Tailings PGE Grade by Grind Group Raw Data

Parameter	Test Condition : Grind, % Sample Mass passing 75 Microns							
	40 Raw	40 Screened	50 Raw	50 Screened	60 Raw	60 Screened	70 Raw	70 Screened
Mean Grade g/t PGE	1.000	1.036	1.051	1.069	1.106	1.147	1.129	1.149
RSD %	20.4	14.2	14.7	9.3	20.0	12.9	18.8	11.4
n	168	158	135	127	103	97	135	126

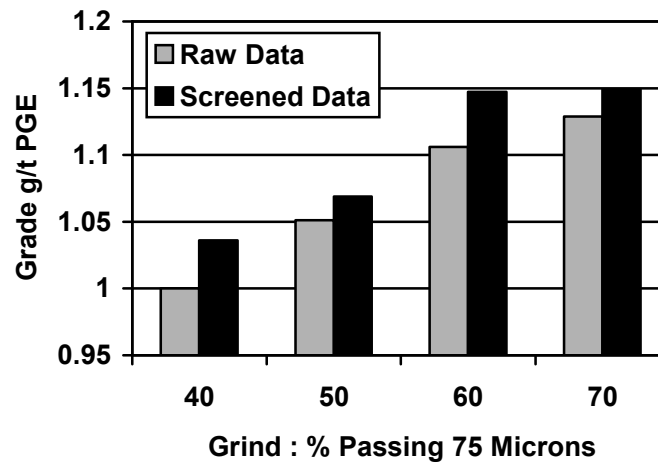


Figure 3 – Effect of Tailings Sample Grind on Mean Grade of PGE Estimated

Tests of Significance

In order to determine the significance of any observed differences in grade of PGE estimated by sample treatment group, the Analysis of Variance (ANOVA) was used [Box et al., 1978]. This process compares the variance arising within treatment type with variance between treatment type. The result is referred to a value of F, the variance ratio.

Although similar patterns are seen between raw and screened data in the above results, the ANOVA was performed on the screened data set where effects are sharper.

Mill Feed

Table 8 - ANOVA Table
Mill Feed : Screened Data

Source of Variance	Sum of Squares	Degrees of Freedom	Mean Square
Between Treatments	2.46	3	0.82
Within Treatments	147.39	477	0.31
Total	149.86	480	0.31

From these calculations the F ratio for the test of significance is $0.82/0.31=2.65$. The critical value of F at the 10% level is approximately 2.1, and at the 5% level, approximately 2.6, so it may be concluded that the grinding treatment of the mill feed produces significantly different PGE grades at the 5% significance level.

Final Concentrate

Table 9 - ANOVA Table
Concentrate : Screened Data

Source of Variance	Sum of Squares	Degrees of Freedom	Mean Square
Between Treatments	8511.5	2	4255.8
Within Treatments	35063.6	123	285.1
Total	43575.1	125	348.6

From these calculations the F ratio for the test of significance is $4255.8/285.1 = 14.92$. The critical value for F at the 0.1% level is approximately 7.32, so the screened are significant at the 0.1% significance level.

Final Tailings

Table 10 - ANOVA Table
Tailings: Screened Data

Source of Variance	Sum of Squares	Degrees of Freedom	Mean Square
Between Treatments	0.400	3	0.133
Within Treatments	9.498	505	0.019
Total	9.898	508	0.152

From these calculations the F ratio for the test of significance is $0.133/0.019 = 7.08$.

The critical values of F for these test conditions are approximately 5.6, at the 0.1% level, so it may be concluded that the degree of grind of the tailings sample prior to fire-assaying significantly affects the grade of PGE estimated.

Proposed Mechanisms

Mill Feed and Tailings

Both mill feed and tailings samples show optimised PGE evaluation at the same grind, viz. 70% mass passing 75 microns. The effect is clearer for the tailings, probably because most of the recoverable PGE mineral hosts have been removed by flotation into the final concentrate.

It is suggested that this trend is consistent with the degree of liberation of the PGE-bearing minerals. The primary lead fusion stage of the fire-assay process is time- and temperature dependent, inferring that the kinetics of the reaction are sensitive to the particle size distribution and the degree of liberation of the minerals of interest. Inadequate liberation in mill feed and tailings samples would lead to incomplete recovery of the PGE into the lead button.

Final Concentrate

The difference in concentrate grade arising from prior pulverisation in clean silica, compared to analysis of the concentrate on an 'as is' basis, may have arisen from the heterogeneous nature of this sample material, *i.e.* that it carries PGE mineral hosts of varying grades and densities across several orders of magnitude. It is believed that the pulverisation process in transforms

these PGE minerals into a more homogenous form with a more symmetrical distribution of replicate values.

A cross-check on which of these two forms of sample preparation may be found in the result of Group C of this concentrate experiment. Here the discrete PGM, which are a high-density mineral group grading in the order of 40-50% PGE, were quantitatively removed using a laboratory scale superpanner prior to the fire-assaying process. This would result in the statistical stratification of the sample [Cochran, 1947] and a more reliable estimation of the mean grade of that concentrate sample.

Inspection of the results of Group C shows that the mean grade estimated is actually in closer agreement with the mean result of group B than Group A, suggesting that whether the concentrate sample is physically stratified or physically homogenised, a lower grade of PGE is estimated. This suggests that the result from Group A is the outlier and is a biased estimate.

Likely Biases

In collating these effects as they would appear to a production plant's metal accounting, it is necessary not only to examine the combined effects of robust mean grades, such as obtained in these exercises by large data sets, but also to examine the likely biases in estimating the mean grades of these samples from the small data sets which are typically used in daily production assaying. This is because skew and multimodal distributions are prone to misleading estimates of the mean when estimated from non-robust data sets.

Accepting in the first case that the estimates of the mean grades are more reliable after outlier removal at the 95% acceptance level, the following likely biases would develop, using typical plant grinding conditions and compared to the optimum state of sample preparation:

Table 11
Likely Biases Using Robust Screened Data

Sample	Grade at Production Grind	Grade with Optimised Sample Preparation	Difference % (Production Sample =100%)
Mill Feed	0.988	1.029	- 4.14
Concentrate	1.004	0.980	+ 2.29
Tailings	1.051	1.149	- 9.30

This analysis indicates that unless optimised sample preparation is used, mill feed grade will be understated by some 4.1%; tailings by some 9.3%, and concentrate overstated by some 2.3%. (A convention of negatively signed bias is used here to indicate underestimation of grade). When combined in a typical mass balance across the mineral process, these errors would amount to an overstatement of PGE recovery across flotation processing by some 5.2%, using typical mass balance data. Put differently, if PGE metal accounting were to be performed on a reconciled head basis, and a call factor calculated from the assayed head, an unaccounted gain of some 5.2% would be the typical result.

Accepting in the second case that the estimates of the means are robust to the presence of outliers, the following likely biases would develop, using typical plant grinding conditions and compared to the optimum state of sample preparation:

Table 12
Likely Biases Using Raw Data with Outliers Present

Sample	Grade at Production Grind	Grade with Optimised Sample Preparation	Difference % (Production Sample =100%)
Mill Feed	1.000	1.017	- 1.7
Concentrate	1.000	0.981	+ 1.29
Tailings	1.000	1.129	- 12.9

This analysis indicates that unless optimised sample preparation is used, mill feed grade will be understated by some 1.7%; tailings by some 12.9%, and concentrate overstated by some 1.29%. (A convention of negatively signed bias is used here to indicate underestimation of grade). When combined in a typical mass balance across the mineral process, these errors would amount to an overstatement of PGE recovery across flotation processing by some 2.5%, using typical mass balance data. Put differently, if PGE metal accounting were to be performed on a reconciled head basis, and a call factor calculated from the assayed head, an unaccounted gain of some 2.5% would be the typical result.

Geostatistical Considerations

The pattern of these biases appears to be grade-dependent, *i.e.* lower grades underestimated, and higher grades over-estimated. Earlier work in this regard, addressing the bias errors of gold evaluation in the Witwatersrand, found that generally this pattern held for original and check fire-assays performed for stope sampling [Krige, 1981; Sichel, 1947;]. Suitable quality controls dealing with outlier detection and deletion were also developed [Coxon and Sichel, 1959; Krige, 1962;]. This work showed that identification of grades outside the 95% limits of the distribution of mill feed grades was possible.

Conclusions

The investigation of evaluation bias errors of PGE in Rustenburg Section Merensky ore has shown that:

1. Lead collection fire-assaying, as practiced in the work performed for this paper, is not robust to the diverse mineralogical forms in which the PGE occur.
2. Mill feed and tailings samples show understatement biases of approximately 4.1% and 9.8% respectively. It is believed that the mechanism is by means of incomplete fusion of the incompletely liberated PGE minerals, thus the reason why finer grinding of these samples produces higher mean PGE grade estimates.
3. Concentrate sample material studied shows an overstatement bias of approximately 2.3%. It is believed that this mechanism is by means of the heterogeneous nature of the concentrate, *viz*, carrying PGE minerals across several orders of magnitude in grade, and of a range of PGE mineral densities. The pulverisation-in-silica method is likely to have homogenised this arrangement into an artificial set of minerals with less spread of specific grade.
4. By constructing technical exercises as described in this paper, the investigator is in a position to minimise these bias errors and obtain more reliable estimates of grade and recovery. It is possible that other parts of the Bushveld Complex would display similar bias effects, but with bias coefficients typical to the specific geological area in question. Wider ranges of grinding conditions should also be tested.

5. The overall effect of the bias errors documented for this section of the Bushveld Merensky is in the order of 5.2% expressed as either recovery or PGE call factor error.

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