A Quality Control Model for the Development of High-Confidence Flotation Test Data

A thesis submitted for the degree of
M.Sc.(Eng)(Chem)
at the University of Cape Town

Basis

All PGE Data are Indexed

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April 1995

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SUMMARY

This thesis addresses the problem of obtaining reliable laboratory scale flotation test data for the Merensky ore type found in the Bushveld Complex of South Africa. The complex nature of the platinum-group element (PGE) deportment in this ore renders the normally-practiced procedures inappropriate for this particular testwork. A more robust and thorough procedure is necessary because of the diverse mineralogical forms in which the PGE are found. The evaluation of the mass and value balances has accordingly to take these factors into account.

The major features of the evaluation of input and output errors across the laboratory scale flotation test are analysed. It is found that unless size-by-size variance of PGE in a conventionally crushed mill feed is taken into account the mill feed sample size is underestimated by some 176%. Further the preparation of a reference distribution of assayed head material is necessary to provide the 95% confidence limits of grade estimate.

The need for repeating flotation tests and compositing the adjudicated products is discussed, concluding that quintuplicates are suitable to achieve a desirable level of confidence in the built-up head grade.

The sample preparation of the flotation products has a critical role in minimising evaluation errors, as is the case with fire assaying of samples where minimum numbers of replicate determinations have been calculated.

An outlier rejection model for adjudication of the replicate built-up head grades is proposed, and a complete flowsheet of the quality control model is developed from first principles. The effect of this model on the PGE metal balance is analysed. It is concluded that workable controls are defined, since a metal balance with < 1% error has been achieved.

ACKNOWLEDGEMENTS

The author would like to acknowledge with thanks the support and encouragement given by the Technical Director, Consulting Engineers and Chief Consulting Metallurgist of Rustenburg Platinum Mines Limited during this study. In particular Mr. G.M. Wright - Chief Consulting Metallurgist, has shown his personal interest in this study, and his constructive advice has always been appreciated.

Certain persons are due their thanks for encouragement given to set up the proposal for this study. These include Messrs. J. Humphriss, C.J. Martin, G.M. Wright, H.C. Munro, W.S. McCarthy, Drs. E. Kirby, C.A. Hunt and T.J.B. Jones.

Dr. L.A. Cramer - Acting Consulting Engineer, was instrumental in setting up the necessary arrangements with the University of Cape Town for this study.

Mr. G.L. Frampton is due thanks for his interest shown during the study.

Messrs. E. Kinloch and W. Peyerl are due thanks for their mineralogical advice so readily given.

Mr. B. Bonthuys, at the Minerals Processing Research Laboratory Library, JCI, is due thanks for his assistance in making available most of the published literature used in the reference section. Mr. G. Chunnett, at the Amplats Geology Unit, kindly loaned a number of geostatistical references from his personal library, and gave encouragement to the author.

Professor C.T. O'Connor, University of Cape Town, is due special thanks for accepting the proposal to initiate this study at an academic level, and for taking on the job of supervisor of this thesis during a very busy year. His ongoing encouragement and objective view of the development of the theory has been most helpful.

Professor L. Minter, University of Cape Town, is due thanks for consulting on the first draft of the review paper.

My family, in particular my wife Heather, are due special acknowledgement for their patience and loyal support during the two years of this part-time study.

PUBLICATIONS

The following publications and congress papers emanated from this study:

Review of Evaluation Models for the Representative Sampling of Ore

In press, J. S. Afr. Inst. Min. Metall., 1995. SAIMM paper number 244/94 refers.

A Quality Control Model for the Development of High-Confidence Flotation Test Data

Read to the 1995 SME Annual Meeting and Exhibit, Denver, Colorado, USA, March 1995. Preprint number 95-40 refers.

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1. INTRODUCTION

1.1 Purpose of Thesis

A mineral process using milling and flotation as the primary separation treatments often relies on laboratory flotation tests to support the production optimisation programme, whereby weaknesses in the process are identified by means of surveying and of assessment of routine production performance. These process weaknesses are examined further at laboratory scale - where the economic risk is minimised - in order to evaluate and quantify potential improvements to the process.

The reliability of the laboratory scale test data in representing the production scale response is central to such an optimisation programme. Typical results of such test work are improved flotation recoveries resulting from matching production grind with the grinding requirements of the ore; or optimisation of reagent suite (activators, collectors or depressants), achievement of better grades and recoveries, etc. In all these affect the mineral process optimisation programme. Accordingly the use of quality control in laboratory scale test data has an important place in screening the raw data to assure a desired level of confidence in the conclusions drawn.

The complex nature of the platinum-bearing ores of Rustenburg Platinum Mines Limited makes the scrutiny of such usually-practiced laboratory scale flotation tests difficult, in that evaluation of the inputs and outputs requires a more thorough examination than normal in order to arrive at reliable conclusions. This problem stems from the complex and variable mineralogical nature of these ores, and the evaluation of mass and value balances has to take this into account.

A heuristic model to cater for this complexity in laboratory scale flotation tests was developed between 1988 and 1993 at Rustenburg Platinum Mines Limited [Lotter and Munro, 1994], using a measure of precision between replicate tests and analytical determinations as an adjudicating parameter for outlier rejection. The model inspected test data for outliers so as to exclude these from the decision-making data. This model enjoyed some success with regard to scale-up trials from laboratory test data. Sufficient potential was seen in the model to warrant further investigation.

1.2 Context of Merensky and UG2 Ores

The Pt-bearing Merensky and UG2 ores mined and processed by Rustenburg Platinum Mines Limited formed the subject of this study. These ores form the major reserve of platinum in South Africa, and are treated in several operating sections of the Bushveld, viz. at Rustenburg, Union and Amandelbult Sections.

The mineral process used to extract the platinum-bearing minerals, which is described in more detail in section 1.4, uses combinations of crushing, milling, classification and flotation to achieve the primary separation of these minerals from the host rock.

Secondary separation of the discrete PGM, or "metallics" minerals, is achieved using gravity recovery methods with James tables.

The mineral process therefore has the resemblance of a base metal sulphide operation together with a secondary gravity recovery process.

1.2.1 Geology and Mineralogy of the Ore Types Studied

The Bushveld Complex, or BC, is an extensive geological system in the Transvaal of South Africa, with Villa Nora as the northern limit, Rustenburg as the southern limit, and Atok as the north-eastern limit. Further, Bushveld Complex rocks are seen westwards in Botswana. The BC is well-known for its wealth and complexity of metals, amongst these platinum, palladium, gold, rhodium, copper, nickel, cobalt, tin, vanadium, iron and chrome.

Using Rustenburg Section of Rustenburg Platinum Mines as an initial example of the stratigraphy of the Critical Zone of the BC, Viljoen and Hieber [Viljoen and Hieber, 1982] described seven cyclical units, grading from pyroxenite with chromite through norite and anorthosite, together some 580m from top to bottom.

Two main features of economic significance in this system are the Merensky Reef and the UG2 Chromitite, both carrying platinum-group elements (PGE) in a complex array of platinum group minerals (PGM). Another economically significant layer is the LG6 Chromitite near the top of the pyroxenitic subzone of the Critical Zone.

The immediate floor of the BC in the Rustenburg area consists of rather irregular and dismembered masses of quartzite and hornfels with diabase and hybrid igneous rocks. These floor rocks are overlain by igneous rocks of the BC, dipping at some 10 degrees and commencing with pyroxenites, followed by norites and gabbros.

The Merensky Reef forms the base of the sixth cyclical unit, whilst at Rustenburg the UG2 chromitite is found some 140m below the Merensky Reef in the fifth cyclical unit.

Specifically the Merensky Reef Unit varies in width from 9 to 15 m. This unit starts with a pyroxenitic layer with associated thin chromitite layers, overlain by a differentiated suite of norite, anorthositic norite, spotted anorthosite and poikilitic, or mottled, anorthosite.

The Merensky Reef forms the lowermost part of the basal pyroxenite/chromitite assemblage of the Merensky Reef Unit. It commences with a thin 1 cm chromitite layer often resting directly 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 chromitite layer up to 1 cm thick 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 upper chromitite layer is generally immediately overlain by a medium-grained poikilitic pyroxenite, although in some cases this chromitite layer is not confined to this contact and can occur within the poikilitic overlying material. The poikilitic hangingwall is sometimes referred to as "Merensky Pyroxenite". The PGE values are concentrated chiefly in, or are associated with the pegmatoid. Lesser but economically significant PGM values are also found in this hangingwall, immediately overlying the Merensky Reef, and progessively decreases upward. This is also the case with the feldspathic footwall for cases where "contact reef" is found. Here the distance between the two chromitite seams is less than the typical 25 cm, and the feldspathic footwall can be enriched with PGM minerals.

The UG2 chromitite layer is found in the fifth cyclical unit of the upper group of the critical zone. It is found within pyroxenite and forms the uppermost of the substantial layers of chromitite within the BC. It is typically 70 cm thick and is underlain by 50 cm of coarse grained pegmatoidal feldspathic pyroxenite, similar in appearance to Merensky Reef. Approximately 8 m of feldspathic pyroxenite overlies this, containing up to three leader chromitite layers within the first metre of the main layer.

The regional trends in PG-mineralogy were reviewed by Kinloch [Kinloch, 1982], with description of the types, volume distribution and mode of occurrence of these. It is immediately apparent in this review that the PGM minerals of UG2 Chromitite and overlying Merensky Reef show close similarities in mineral types in a given area.

Examples given show Pt-Pd sulphides in Merensky Reef in one area being dominant in the PG mineral set, with similar Pt-Pd sulphide dominance in the underlying UG2. Similar trends are seen for ferroplatinum alloy, eg Pt_3Fe. The similarities in the distribution of these two categories of PG minerals, viz. Pt-Fe alloys and Pt-Pd sulphides, is one of the major features of the observed regional precious metal mineralisation trends of the Bushveld, while the distribution of yet another species of PGM, the Pt-Pd tellurides, shows no apparent pattern of significance in this Complex.

Pt-Fe alloy is a significant constituent of the western Bushveld ores to the north-east and south-east of the Pilanesberg Alkaline Complex. It is also developed in the UG2 layer in that immediate vicinity and in the dunite pipes of the eastern Bushveld. The Platreef on the farm Zwartfontein at Potgietersrus is also enriched in Pt-Fe alloys.

The other form of Pt-Fe occurrence is known to be blebs, or intergrowths, physically attached to base metal sulphides such as pyrrhotite.

The UG2 Chromitite layer throughout the Bushveld, in addition to Pt-Fe alloy and Pt-Pd sulphides, contains a discrete Rh sulphide not found in Merensky Reef. The latter contains Rh only in the form of solid solution in pentlandite. A definite correlation between laurite abundance, RuS_2, and chromite, is seen.

The overall forms of the discrete PG minerals were categorised and quantified by Kinloch as shown in Table 1.1.

The three main categories of the mode of occurrence are evident from this table, viz:

(i) PGE sulphides,
(ii) PGE alloys,
(iii) PGE tellurides.

These categories can be further subdivided into those PGM types either enclosed in, or attached to, base metal sulphides or silicates. Some correlation was seen between high Pt-Fe alloy content and platinum group minerals enclosed in gangue.

Equally, areas enriched in Pt-Pd sulphide or telluride mineralisation are associated with PG minerals in base metal sulphides. In the case of laurite definite association is seen with UG2, where it is commonly enclosed in chromite crystals.

Table 1.1
Categorisation of Discrete PG Minerals in the BC

Volume % Distribution per Locality							
PG Mineral Categories	Rustenburg		Union		Amandelbult		Bosch-Koppies
	M.R.	UG2	M.R.	UG2	M.R.	UG2	M.R.
Pt-Fe alloy	1,7	0,2	65,6	25,5	31,2	15,0	53,6
Pd alloys	0	0	tr	1,5	3,2	2,0	0
Electrum (AuAg)	3,3	0	tr	0	0,2	0	0
Sperrylite ($PtAs_2$)	6,0	1,2	7,0	1,4	9,2	0	1,1
Laurite RuS_2	5,2	10,2	20,2	35,4	17,5	25,0	18,6
Pt-Pd Sulphides	80,9	84,9	0,6	30,3	19,0	44,0	18,1
Rh Sulphides	0	3,3	tr	5,8	0	14,0	3,4
Pt-Pd tellurides	2,6	0,2	6,5	<0,1	19,6	0	5,2
PG Mineral Categories	Eastern BIC M.R.	UG2	Western Plats M.R.			Potgietersrus P.R.	
Pt-Fe alloy	0,7	5,5	6,1			10,4	
Pd alloys	0,8	2,1	0			6,2	
Electrum (AuAg)	0,6	<0,1	4,1			6,2	
Sperrylite ($PtAs_2$)	0	3,3	51,0			20,6	
Laurite RuS_2	14,1	16,6	0			1,5	
Pt-Pd Sulphides	53,4	51,6	17,3			19,1	
Rh Sulphides	0	20,0	0			0,9	
Pt-Pd tellurides	31,1	0,9	21,4			38,0	

It is believed by Kinloch [Kinloch,1982] that the formation of laurite in UG2 arose from initially high oxygen fugacity conditions, suitable for chromitite formation, causing chromite crystallisation along with the ferric oxide content of the magma. This resulted in a decrease of sulphur solubility and separation of immiscible sulphide liquid from which the high temperature Ru sulphide, laurite, crystallised at an early stage. The typical average sizes of the PG minerals in these three systems was found to be in the order of :

Merensky 26 microns,
UG2 9 microns,
Platreef 53 microns.

The association of PGE (platinum group elements) with base metal sulphides was documented by Kinloch to be the so-called latent fraction, where the PGE occur in solid solution with pentlandite, pyrrhotite and pyrite.

Although chalcopyrite is also present in this system, no PGE association of significance has been documented. A summary of typical concentrations was reported and is shown in Table 1.2.

These data show the interesting feature of high Pd concentration in pentlandite for locations known to have high concentrations of Pt-Fe (Table 1.1), however these also describe the tenor of association of PGE with base metal sulphides.

Table 1.2
Concentrations of PGE in Solid Solution in Pure Base Metal Sulphide Minerals

Location in BIC	Base Metal Sulphide	Pt ppm	Pd ppm	Rh ppm
Rustenburg Merensky Reef	Pentlandite Pyrrhotite Pyrite	11 11 15	140 0 34	23 3 9
Union Section Merensky Reef	Pentlandite Pyrrhotite Pyrite	10 47 15	416 0 0	74 3 0
Amandelbult Merensky Reef	Pentlandite	13	350	72
Driekop UG2	Pentlandite	36	93	13

Accordingly, an understanding of the latter will be influential in the understanding of the mineral process for PGE recovery. Equally Ni is known to substitute for Mg in serpentine. As Pd substitutes for Ni in pentlandite, it is likely that Pd and possibly Rh could substitute for Ni in silicates. This would identify a third category of PGE occurrence, in this case unrecoverable by flotation.

Kinloch postulates that the proximity of feeders to the formation of this geological system would have a profound effect on the magma, magmatic structures and especially on crystallising minerals. A relatively quiescent non-volatile system is proposed outside the zone of influence of these feeders. It was possible to model the mineralogy of a borehole core from knowledge of its distance from an area of volatile activity. This reasoning can be used to explain the geological differences in the sequences in different facies associated with sub-compartments in the complex, such as Amandelbult, Union and Rustenburg Sections.

This work therefore identified the following main categories of PGE in the BC:

(i) discrete PG minerals,
(ii) PG minerals present as discrete intergrowths with base metal sulphides,
(iii) PGE in solid solution with base metal sulphides,
(iv) PGE in solid solution with silicates.

It is expected that all but (iv) are amenable to successful flotation. This theme was pursued by Peyerl [Peyerl, 1983] and the following lists of minerals were identified:

Discrete PGE minerals

Pt-Fe alloy	Pt_3Fe
Cooperite	Pt>>PdNiS
Braggite	Pt>PdNiS
Vystotskite	Pd>PtNiS
Sperrylite	$PtAs_2$
Laurite	$Ru(Os,Ir)S_2$
Bismuthotellurides	Pt(Pd)BiTe
Pd alloys	Pd alloyed with Cu,Sn,Sb,Hg,Tl,Bi.

Of this group of minerals it is noted that Pt is the dominant form of the platinum group elements present. These "discrete" PGM are mostly of high specific gravity from 6 to 19 g/cc^3 and are amenable to recovery by gravity separation techniques. They were also reported to occur as intergrowths with the following base metal sulphides:

Chalcopyrite	$CuFeS_2$
Pentlandite	$(FeNi)_9S_8$
Pyrrhotite	FeS
Pyrite	FeS_2

These were investigated for the tenor of PGE in solid solution, Table 1.2, noting that chalcopyrite had no PGE association, and that the strongest association was with the Ni and Fe sulphides.

Detailed mineralogical studies, teamed with metallurgical testwork, showed that an increase in the level of serpentinisation of Merensky reef results in a decrease in Ni flotation recovery [Peyerl, 1983]. A correlation was found between the Ni recovery and the degree of "serpentinisation", the latter quantified by the total of serpentine, talc and chlorite in the sample of ore concerned. The inference that some PGE originally as solid solution in pentlandite follow the Ni into serpentine is significant. In summary, Peyerl proposed the modes of occurrence and metallurgical implications as shown in Table 1.3.

Table 1.3
Summary of the Modes of Occurrence of PGE in the BC and their Metallurgical Implications

Mode of Occurrence	Metallurgical Implications	Estimated Proportion of Total PGE %
"Discrete" PG minerals	Recoverable by flotation and gravity	45
Solid Solution in Sulphides	Recoverable by flotation	50
Solid Solution in Silicates and Oxides	Not recoverable	5-10

1.2.2 The Statistical Modelling of Value Distributions in an Orebody

A glossary of statistical and geostatistical terms and formulae is given in Appendix 1.

The errors in ore grade estimation made from chip sampling of ore in the Witwatersrand gold mines were first modelled by Sichel [Sichel, 1947], following earlier work by Watermeyer [Watermeyer, 1919]. The latter found that the distribution of gold in a certain block of ore followed a statistical law, and Sichel found that the error in the assay grade was grade-dependent.

The Percentage Error Distribution (PCE)

The frequency of the error q is given by F(q), where

$$F(q) = 1{,}25112(1 + (q/100))^{10}.e^{-0,1q} \qquad (1)$$

This model was transformed to:

The Transformed Percentage Error Distribution

By substituting

$z = (1+(q/100))$,

$$P(z) = 27557{,}3.z^{10}.e^{-10z} \qquad (2)$$

Using

t = the obtained assay value of the sample,
V = the true sample value,
q = the percentage error,

$$t = z.V \qquad (3)$$

The probability of an assay value t returning a true value V was then modelled as

$$p(t) = 27557{,}3.((t)^{10}/(V)^{11}).e^{(-10t/V)} \qquad (4)$$

From Equation 4, when t = V,

$$p(t) = 27557{,}3/V, \qquad V > 0 \qquad (5)$$

This would imply that lower grades have a greater probability of being returned with a larger error than do higher grades. This led to three observations from the PCE law:

(i) for the low grade group of assays, the number of probable assays is higher than the respective number of true values,
(ii) for the middle assay group, the number of probable assays is less than the number of true values,

(iii) for the higher assay group the number of probable assays is higher than the respective number of true values.

A grade-dependent error function was developed from the PCE, and with the Group Correction Factor, GCF, in order to correct for these biases in the sampling data. The True Sample Value model, or TSV, was developed to the form

$$\Phi(V) = 488,666.e^{-k} \qquad (6)$$

where

$$k = (-0,47190.\ln V + 0,76155)^2$$

Subsequently Sichel modelled the error in the assay grade of a sample as a function of sample mass. This error model showed that the error in the returned assay value of a sample was higher for small samples, in the order of < 20 Troy Ounces (approx. 622 g) than in samples heavier than 40 Troy Ounces (approx. 1244 g), and defined the axiomatic nature of reckoning with the sample size towards accurate assay results.

In a statistical analysis of borehole values in the Orange Free State goldfield, Krige showed that the lognormal frequency curve can be applied to the observed distribution of gold values in the main sector of the goldfield. It was shown that the frequency distribution of values could be normalised by the lognormal frequency plot [Krige, 1952].

It was shown by Krige [Krige, 1981] that the gold values of a given section of a mine, when collected in the form of a frequency distribution, had a positive skewness, with the mode to the left of the mean, and a long drawn-out tail to the right. By plotting such data on a logarithmic scale, the distribution becomes more symmetrical and approximates the normal distribution.

It was also shown that the same lognormal data becomes linear on probability paper, on which the x scale is probability of cumulative frequency and the y scale is the logarithm of cumulative grade. The straightness of this plot gives an indication of the degree to which the data fits a lognormal model.

A further development in this model was made for certain orebodies that did not produce a straight line plot as above, but showed curvilinear form, with departure from linearity in the low grade area of the data. In such cases it was proposed that a third term be added to the lognormal model:

$$\Phi(x) = [\sigma.(2\pi)^{0,5}]^{-1}\exp[-(1/2\sigma^2)(x-\alpha)^2] \qquad (7)$$

where

x = $\ln(z+a)$
α = mean of $\ln(z+a)$
a_2 = additive constant
σ^2 = variance of $\ln(z+a)$.

With regard to the sampling of lognormal populations, the arithmetic mean will be an unbiased estimate of the population mean if the population is regarded as an infinite number of individuals obtainable on repeated sampling following the same procedures. However the physical sampling can introduce a bias in relation to the population values; this is often the case in underground sampling. No mathematical treatment can correct for such a bias.

Having taken into account the error distribution, Krige concluded that the limits for the sampling mean and other estimators will be significantly skew, and this should be borne in mind in all ore evaluation procedures where lognormality applies.

Where small data samples are taken from such a lognormal distribution, the likelihood of such sampled data including a value from the high grade tail of the distribution and thus influencing the estimate of the mean should be considered. In this regard it was concluded that the geometric mean, after correction, should be used.

The quality control of mine sample assay data for gold showed that the lower grades of samples tended to display higher variances between original and repeat assay than did higher grades of samples. A method was proposed in the form of a control chart [Coxon and Sichel, 1959] that established checking limits for agreement between original and check assays. In their investigation it was found that the initial sample preparation was susceptible to bias due to inadequate riffling prior to reduction of sample size; and that the lower grade samples presented the most difficulty in arriving at an accurate assay because of the small prill mass from a standard fusion. This was later corrected by using a larger sample mass. A 10% inspection density of mine samples was established.

Subsequently Krige documented the statistical quality controls for mine valuation [Krige, 1962], listing three areas amenable to cross-checking, viz. mill values, assaying, and mine sampling.

Initially a plot of daily mill feed plus values over a 99-day period of production yielded a lognormal plot of frequencies. The distribution was cut off on either side at the 2½ % level, thus excluding only 5 % of the values in distribution.

The warning limits at the 5 % level were then overlaid on the plot of daily values, and the equivalent 1 % level limits also calculated and shown. The latter refer to action limits. The population mean was then estimated using a geometric formula. Such a graph provides an objective means of interpreting the significance of the nth mill feed value being within normal scatter.

A different form of graph was demonstrated for use with daily mill feed values plotted in a progressive form with 5% limits, as before, overlaid on the plot of daily values.

The quality control of assaying reported by Coxon and Sichel was used by Krige in the form of the fan chart which is a plot of routine mine assays on the y axis against the parted check assays for the same samples as the x axis. The controls are grade-proportional, i.e. more tolerant of differences at higher grades than lower ones. The correct control unit was found to be the ratio of original to check assay, and the limits calculated from large numbers of observed duplicate assays. The grade/error function documented, however, contradicted the above tolerance and found that lower value samples displayed higher variance between original and check assays. The graph modelled, Figure 1.1., used the data shown in Table 1.4.

Table 1.4

Comparison of Original and Check Assays for Routine Assays of Mine Samples
(Metricated Values)
Refer Fig. 1.1

Value	Assay Values, g/ton		
g/ton	Original	Check	Ratio O/C
> 214	231,09	197,49	1,17
171	185,83	157,02	1,18
137	149,16	124,30	1,20
103	112,45	93,26	1,21
69	75,60	61,37	1,23
34	38,91	29,65	1,31
17	20,14	14,14	1,42
14	16,30	11,13	1,47
10	12,48	8,09	1,54
7	8,61	5,10	1,69
3	4,65	2,22	2,09
2,6	3,69	1,47	2,50
1,7	2,64	0,77	3,42

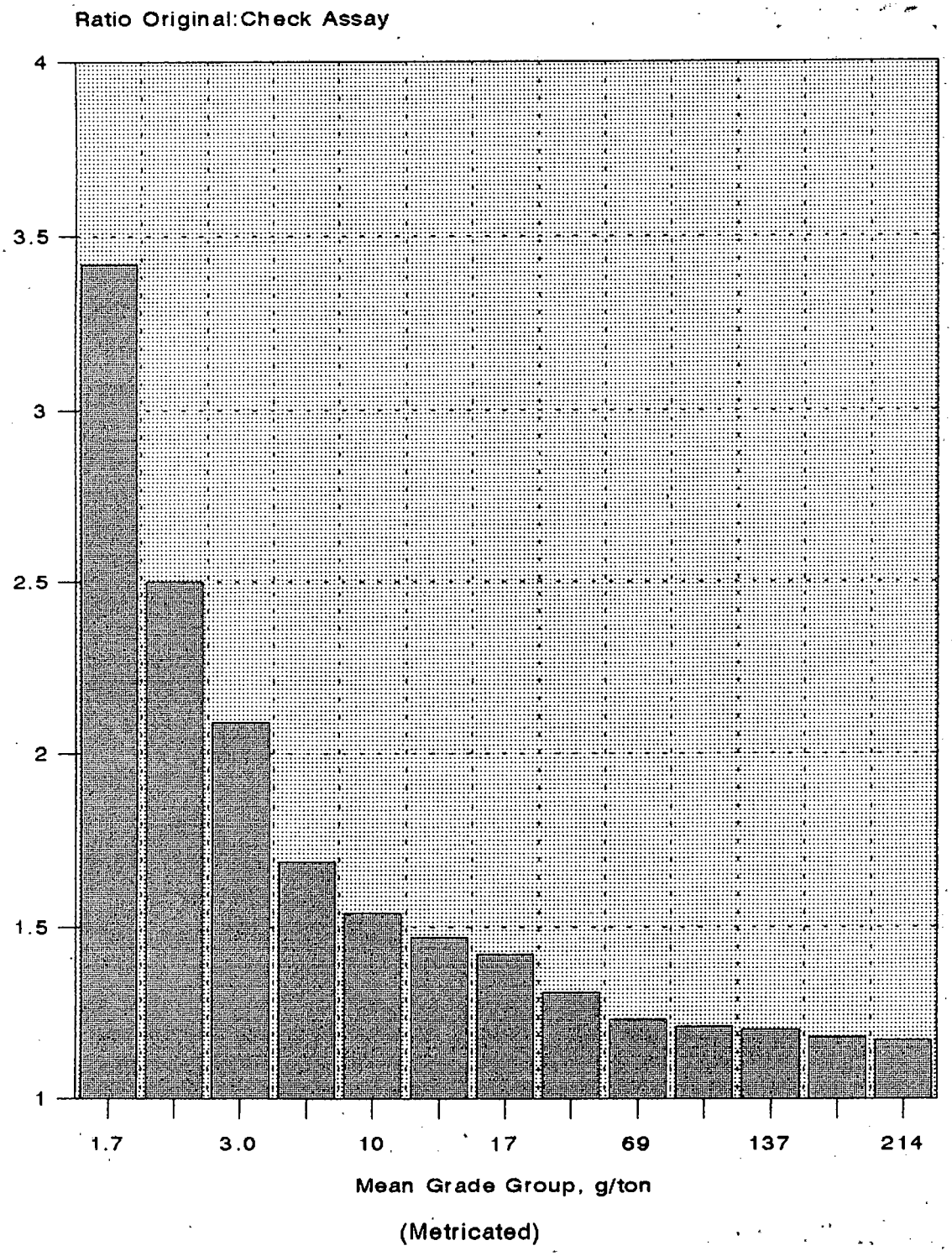


Figure 1.1
Comparison of original and check assays for routine mine samples (after Coxon and Sichel)

This led to the conclusion that generally, original assays overvalued the grade of samples, and particularly lower grade ore samples showed the largest variance in terms of the true grade, further that the error with which this was observed was inversely proportional to the grade.

In subsequent development of the concept of quality control in underground sampling and attendant assaying, St. J. Rowland and Sichel developed control charts to monitor duplicate assays on one sample and check samples taken from a working place in a stope [St. J. Rowland and Sichel, 1960]. It was shown that assay values, in dwt/ton, differed more at higher grades than lower, but the ratio of original to check assay was statistically the more correct unit of adjudication.

The stabilised standard deviation, calculated as the standard deviation derived from the ratio of the original to check values, and a distribution of standardised units was plotted after logarithmic transformation. It was shown that this distribution was not normal.

A control graph was constructed from this model, using upper and lower warning and action limits, to advise the investigator of the reliability of the sampling and assaying system for grade control purposes. These charts used a logarithmic Y-scale.

The issue of sampling from lognormal populations was reviewed by Sichel [Sichel, 1966], where the estimation of the population was attempted from small samples.

The use of a few boreholes in the estimation of an orebody experienced the problem of small data sets using arithmetic means instead of lognormal, yet had to account for the likelihood that the small data set included an observation from the right hand side of the skew distribution.

In the larger populations this is not as much a problem, since the data set is robust. In the case of the small data set, the arithmetic mean is misled by such high grades, and inclusion of the latter leads to overestimation of the mean value of ore grade.

In a case study of the Harmony Gold Mining Co., the database of 5170 values obtained from individual sample sections of underground faces drew an arithmetic mean of 618 inch. dwt. (2440,9 cm-g) and the data were subdivided on a random basis to 1034 subsets of 5 data.

In an extreme case, subset # 637 yielded the following data:

Observation #	Assay Value inch dwt.	Equivalent cm-g
1	329	1299,5
2	277	1094,1
3	111	438,4
4	189	746,5
5	5071	20028,9
Mean	1195	4719,9

The arithmetic mean of this subset does not agree well with the population mean of 618 inch. dwt; yet the value of 5071 inch. dwt. is valid and has to be included in the data.

If small data sets are to be used in the estimation of the mean grade of the orebody, neither the exclusion of the high value nor its inclusion provides an accurate estimate of the grade. The other 1033 subsets were not, however, reviewed.

Use of Sichel's t estimator reduces this estimate to 894 ins. dwt, and although closer to the population mean, is still +44,6% overstated, thus concluding that both the arithmetic mean and the Sichel t formulae do not derive accurate grade statements from a small population in this case.

Some advances on this theory were reported by Sichel [Sichel, 1987], indicating that for the typical case of 5 to 20 boreholes drilled for a particular orebody, the practice of the lognormal probability plot was not applicable as much larger quantities of data are necessary. This limits one from calculating the third lognormal parameter α in borehole data to correct for underestimation of low-grade sections of the orebody.

In such a case the robustness of the Sichel t estimate of the variance of the lognormal distribution forms the subject of a sensitivity analysis. It was concluded that for large variance and large shape factors, the t estimator was not robust.

Subsequently the typical lognormal theory was applied to risk analysis models for new mining ventures [Wainstein, 1975]. He stated that the lognormal model remains the most popular for the modelling of ore values, and that the t distribution, a central part of the theory, was dependent on the sample size n, which for borehole data on an undeveloped orebody is usually < 10. It was found that the t distribution was robust for values of the nuisance parameter σ^2 between 0,1 and 2,5, however use of σ^2 = 0,7 may be used with an error of ± 1%, which is acceptable for such calculations.

In essence

$$t = e^{\bar{x}}.\tau_n(V) \qquad (8)$$

where

$$\bar{x} = (1/n).\Sigma\, x_i \quad \text{for } i = 1 \text{ to } n$$

$$V = (1/n).\Sigma\, (x_i - \bar{x})^2 \quad \text{for } i = 1 \text{ to } n$$

$$\tau_n(V) = 1 + \Sigma\, (n-1)^r.V^r/(2^r.r!(n-1).(n+1)...(n+2r-3))$$

$$x_i = \ln(z_i + \alpha).$$

Later, Clark reported progress with the data processing of the Sichel t estimator, using personal computers instead of mainframe computers, and concluded that the tables of Sichel's t could be accurately calculated, obviating the need for linear interpolation between intervals of V [Clark, 1987].

This reference preferred the use of the natural logarithm rather than the base 10 logarithm, as the latter introduces an unnecessary constant. The overall conclusion of this work was that more accurate calculations resulted from the computed values of Sichel's t than from the older table and graph method.

New developments in borehole evaluation were reported by Krige and Assibey-Bonsu [1992], using a very large number of chip samples from a mined-out area of the Hartebeesfontein Mine. The data were subdivided into smaller units and evaluated in the form of simulated borehole data to represent the overall distribution of data. It was concluded that wherever possible, the three-parameter lognormal model should be used when the individual borehole values are available, leading to the Sichel t estimator.

In the case of very large data bases for an existing mine, however, there is little or no advantage to using the lognormal model instead of the normal model for calculating a mean grade.

The limitations of the lognormal model were shown for a specific case of Ventersdorp Contact Reef [Sichel et. al. 1992], where it is likely that a modification of the placer theory for the Witwatersrand system should be accounted for in considering the distributions of gold values in the orebody. As distances from the source of transportation increased, the ability to carry coarse gold reduced, resulting in the deposition of coarse gold nearer to the source. Thus there should be a relationship between the log-means and log-variances of the gold variable as a function of distance from the stream source.

Further, the erosional features of certain types of reefs invite the relocation of the high grade values, leaving sub-lognormality in the parent zone. Use of this approach reduced the bias of the gold grade estimate from < 5% for three-parameter lognormal to < 2,5% for compound lognormal models respectively. It was concluded that the plot of the empirical cumulative frequency distribution is very necessary as a test of departure from lognormality.

1.2.3 The Evaluation of a Mineral Process

In a summary of process evaluation in the dimension of time, the gathering of long-term process performance data for statistical and analytical purposes was addressed by Bartlett and Hawkins [1987]. These data are historical and are generated from the sampling and analysis of production process streams. The principal components of such an evaluation system are:

(i) sampling of process streams to produce representative samples,
(ii) sample preparation, usually dewatering, drying, crushing and pulverisation,
(iii) capture of mass balance data,
(iv) chemical analysis of samples,
(v) analysis of data to produce metallurgical balances.

Use was made of conventional statistics to calculate the mean and standard deviation, leading to the calculation of the coefficient of variation, as follows:

$$s^2 = (\Sigma x_i^2 - n\bar{x}^2)/(n-1) \qquad (9)$$

where
$\bar{x}$ = the mean value of the observations
x_i = the ith observation of x
n_2 = the number of observations
s^2 = the variance of the mean
s = the standard deviation of the mean.
The coefficient of variance, CV, is then given by

$$CV = (s/\bar{x})*100 \qquad (10)$$

This is also referred to as the relative standard deviation, or RSD.

The standard error of the mean is then given by $s/\sqrt{n}$. The upper and lower 95% confidence intervals are then given as $\pm 2s/\sqrt{n}$. It was observed that for sample sizes of ≥ 30, 68% of observations would fall within $x \pm s$, and 95% of observations would fall within $x \pm 2s$.

The limitation of the normal distribution to describe the distribution of gold values in a run-of-mine ore stream prior to any separation of gold was described, concluding that the lognormal distribution described the data more effectively.

As the normal distribution is symmetrical, the two key parameters in describing this symmetry are skewness and kurtosis, given by

$$\text{skewness} = m3/(s^3) \qquad (11)$$

$$\text{kurtosis} = m4/(s^4) \qquad (12)$$

where m3 and m4 are the third and fourth moments of the distribution respectively. These are calculated as

$$m3 = (1/n)\Sigma(x_i - \bar{x})^3 \qquad (13)$$

$$m4 = (1/n)\Sigma(x_i - \bar{x})^4 \qquad (14)$$

For normally distributed data the kurtosis = 3 and the skewness = 0.

The best known of the theoretical methods to determine the precision of sampling are reported by Gy [Gy, 1979a]. These sampling errors were listed by Bartlett and Hawkins [Bartlett and Hawkins, 1987] to be:

Type of Sampling Error	Origin
Fundamental Error	Particulate nature of ore
Group and Segregation Error	Inhomogenous mixing of ore
Weighting Error	Uneven flow of ore
Long Range Quality Fluctuation Error	Natural variability
Periodic Fluctuation Error	The quantities to be measured
Increment Delimitation Error	Incorrect cutter design
Increment extraction error	Incorrect cutter speed

The representative sample size was proposed to be calculated as Ms, where

$$Ms = c.l.f.g.d^3/(s^2) \qquad (15)$$

where

$$c = \tau/a \qquad (16)$$

τ = relative density of the precious mineral,

a = the grade of the precious mineral expressed as grams per ton of ore,

s^2 = the sampling variance.

$$f = V/(d^3) \qquad (17)$$

d = mean diameter of the ore particle,
V = volume of the ore particle.

It was recommended that f = 0,5 for most cases.

$$g = (1/(d^3M)) \cdot \sum_{i=1}^{n} M_i d_i^3 \qquad (18)$$

d = mean particle diameter in cm,
M = lot mass, grams,
d_i = the ith size fraction, expressed as particle diameter,
M_i = the ith fractional mass corresponding to d_i.

$$l = (d_l/d)^{\frac{1}{2}} \qquad (19)$$

d_l = liberation diameter of the precious mineral.

It was further proposed by Bartlett and Hawkins that the sampling variance was indirectly proportional to the cube of the top particle size in the distribution of sizes being sampled. As a cross-check, Gy's 50 - piece experiment was reviewed, where 50 or more specimens of rock are taken from a conveyor belt carrying run-of-mine ore. The solids density and assay value of each are determined.

The results of this experiment are transposed with the following equation to determine the relationship between the fundamental variance and the sample mass:

$$f_v = (1/M_s).((gv/a^2M).\sum_{i=1}^{n}((a_i-a)^2/V_i).M_i^2 \qquad (20)$$

The acceptable fundamental variance f_v is defined by the investigator, e.g. 8%, and Eq. 20 reduces to

$$M_s = K/f_v \qquad (21)$$

where K = a real number derived from the calculation of g, v, a, and M.

The resulting value of M_s is sensitive to observations of grade with high values. This results in increasing the M_s value by large amounts. The value of M_s is also sensitive to size-dependent value concentration into fine fractions, thus if only large ore specimens are taken for the experiment, the lower grades found in the coarser sizes will understate the value of M_s.

The combined effect of grade and particle size were reported by Laplante to be very significant in the calculation of M_s [Laplante, 1991], and a further enhancement in the M_s model was suggested for alluvial tin deposits where M_s should be divided by the fractional mass of tin finer than the topsize. This indicates that the model would underestimate M_s unless the size-related distribution of values are taken into account. A similar approach was suggested by Laplante for gold or silver in solid solution or finely disseminated in sulphides.

Laplante further interpreted Gy's equation as a form of the Poisson distribution, where events have a low probability of success but a large number of trials. One property of this law is that if an average of n successes is calculated, given a certain sample size, then the standard deviation of the average will be $\sqrt{n}$. Thus if a sample contains 100 chalcopyrite particles on average, 100 ± 10 particles will be encountered, yielding an RSD of 10%. Gy's model therefore may provide an estimate of the order of minimum sample size, but has its limitations.

A cross-check was suggested by use of the d_{95} of a particle size distribution in the case of a mill feed. Here the Poisson distribution is used, since by calculating the mass per topsize particle in the distribution, and requiring that 100 particles of topsize are polled, the representative mass of topsize is calculated. Thus the total sample mass required, ie inclusive of the whole size distribution, is the representative sample mass calculated as M, where:

s = specific gravity of ore, t/m^3,
S = topsize in particle size distribution, cm,
m = mass of topsize particle, g,
r = radius of topsize particle, cm,

$$m = s.((4/3).\pi.r^3) \qquad (22)$$

For 100 topsize particles to be included, the mass of topsize to be sampled will be 100m, and the total mass of sample M will then be (100/5).100m.

The construction and use of the spherical semivariogram after Matheron was reviewed by Clark [Clark, 1993], initially in the spatial dimension for the analysis of relationships between sample values in a borehole drilling programme. There are several versions of the semivariogram for special cases, however the spherical model is regarded as the simplest and most convenient in the first analysis.

The semivariogram value $\tau(h)$ is calculated for different spacings of data, i.e. $\tau(1)$ is calculated as

$$\tau(1) = (1/2N)*\sum_{i=1}^{N} (g_i - g_j)^2 \qquad (23)$$

where
i and j are data separated by 1 distance unit.
g_i = the grade at location i
N = the number of data.

Equally $\tau(2)$ is calculated from Eq. 23 but using 2 distance units between i and j. The successive results of $\tau(h)$ are plotted on the y - axis and the corresponding h value, on the x - axis. A typical format of this version is shown in Fig. 1.2, with the following construction:

$$\begin{aligned} \tau(h) &= C_o && h = 0; \\ \tau(h) &= C[(1{,}5*h/a) - (0{,}5*(h^3/a^3))] + C_o && 0 < h \leq a; \\ \tau(h) &= C + C_o && h > a. \end{aligned} \qquad (24)$$

The overall sample data variance should be equivalent to the sill value $C+C_o$. The semivariogram may also be used in the time dimension, as for a production mineral process where a crushed mill feed is conveyed to a primary milling step. Apart from the necessary rules of the probability of inclusion for any particle to enter the sample increment, the semivariogram informs of the minimum time between sample increments as well as the overall sample data variance.

It is possible, therefore, in setting up an evaluation system for a mineral process, to determine the fineness to which a mill feed should be crushed prior to assaying. This could be done by using a series of intermediate crushing steps with semivariograms at each stage in the dimension of replicates to determine the minimum number of assay determinations required for that size distribution.

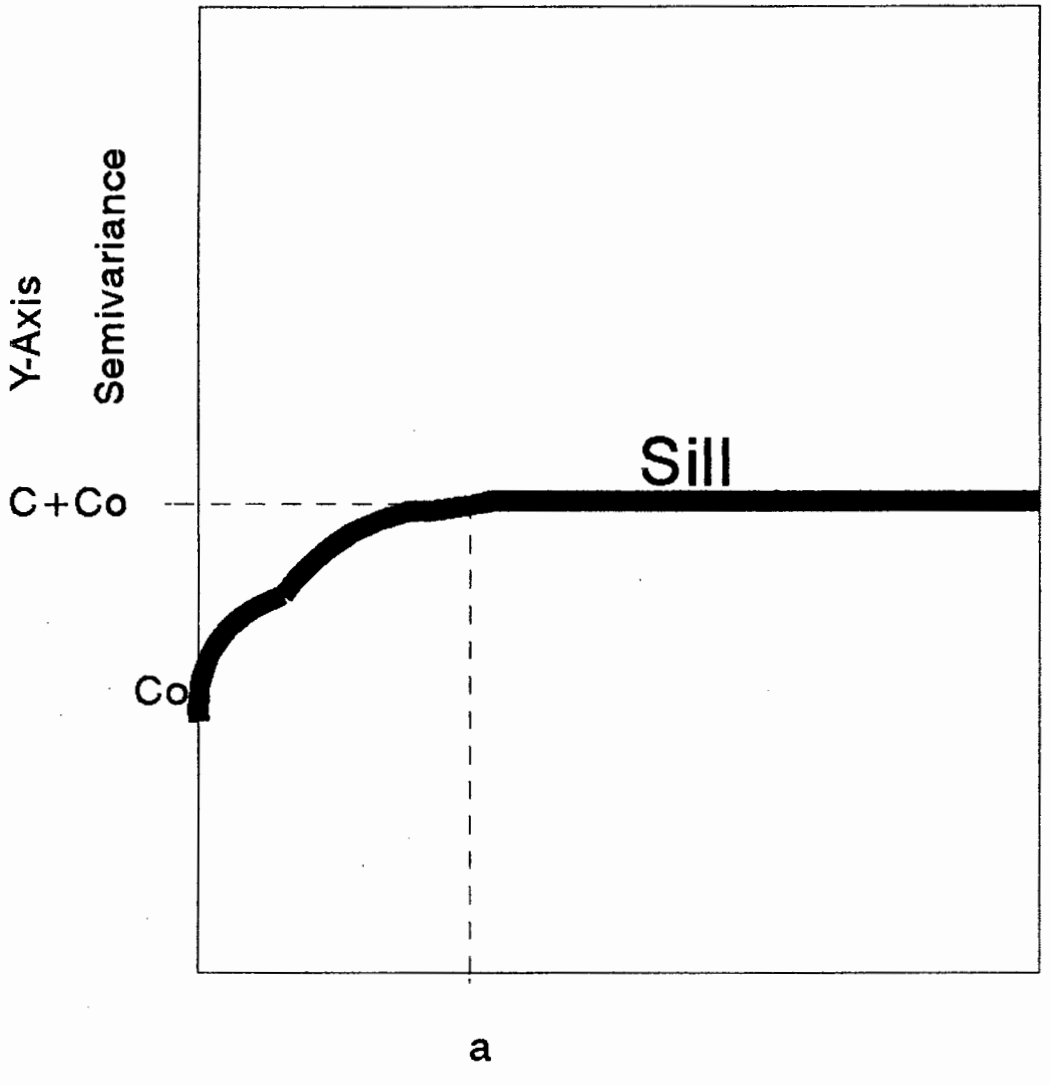


Figure 1.2

The Spherical Semi-Variogram
(after Matheron)

In such a case the recommended method is to take a complete belt cut per increment, i.e. to stop the conveyor belt and place a metre long frame over the belt, thereafter scoop the entire contents of the conveyor belt between the two frame limits into a sample bucket. This includes all the material on the belt between the limits of the frame.

The crushing of the sample prior to subdivision is limited by the reduction ratio of the sample crushing process. The percentage cut into the subsample is related to the topsize of the original sample, d_1, and the subsample topsize d_2 by

$$M_1/M_2 = (d_1^3/d_2^3) \qquad (25)$$

where

M_1 = mass of sample, kg,
M_2 = mass of subsample, kg.
d is quoted in mm.

The crushing and blending of a representative ore sample, however, has the advantage of reducing whatever skewness may exist in the primary sample value distribution [Bartlett and Hawkins, 1987], therefore it is likely that for the time dimension, viz. samples of a production mill feed from month to month, many of the spatial constraints of skew distributions mentioned above will have a minimal effect. The critical parameter in time would then be a structured method of systematic crushing, blending and subsampling followed by inspection of replicate head samples for scatter in terms of RSD.

This is a procedure for which control graphs should be applicable.

1.2.4. Conclusions

This review has examined available literature on the characteristics of evaluation for mostly gold ores, with some reference to the complex mineralogy of the Merensky and UG2 orebodies of the BC.

1.2.4.1 Sampling Ore in Time

In the common case of preparing to sample a production mill feed from a conveyor belt to provide a flotation laboratory with representative material, four checks are necessary:

(i) the 50-piece experiment, taking note of the consequence to sample size of mill feed topsize and of variance between grades of metal in the topsize,

(ii) the fundamental sample size equation of Gy to cross-check the sample size,

(iii) the possibility of Laplante's proposed method to poll 100 pieces of topsize in the sample using an interpretation of the Poisson distribution, thus arriving at a sample size through knowledge of the d_{95} of the particle size distribution,

(iv) the semi-variogram to check on sampling interval between increments so as to minimise short-term scatter effects in the sampling variance.

Further checks on scatter between replicate subsamples of crushed and blended ore prior to flotation are advisable. This is to confirm whether significant skewness in the value distribution between replicates has in fact been reduced to inconsequential levels.

1.2.4.2 Sampling of Ore in Space

The extension of flotation tests to support an exploration programme for a new orebody encounters many of the problems identified by Sichel, Krige and others, in that small sample populations from a borehole drillcore programme in the order of n ± 5-10 may be drawn from a lognormally distributed population. In this regard certain exercises in Merensky and UG2 will be provided for in the form reported by Sichel [Sichel,1966] and Krige et al. [Krige et. al. 1992]. If the lognormality is proven in such an exercise then the use of Sichel's t estimator for recoverable grades as well as assay head grades may be applicable; further the three-parameter distribution model of Krige may be valid. Should lognormality not apply then the procedures for sampling in time for this programme will be re-examined for use in the spatial dimension.

1.3 Review of the Published Literature

Since the beginning of this century, the importance of representative sampling ahead of any ore evaluation, be it analysis or performance testing, was recognised [Taggart, 1945]. The recommendation then was to perform size fractionation of the sample and analyse each bound size further by density group. The overall assay result of the sample was recommended to be the weighted mean. This was later confirmed by Laplante [Laplante, 1991], details of which appear in section 1.2.3.

1.3.1 Particle Size Distribution

Presentation of the size distributions of samples was reviewed. The Rosin-Rammler function to linearise the particle size distribution was identified as suitable. Taggart's view was reported to be

$$w_r = 100\exp(-(D/a))^b \qquad (25)$$

where

w_r = cumulative % mass retained
D^r = mean partice size diameter
a,b = constants.

This form of the equation is unwieldy and is better linearised in the form

$$y = mx + c \qquad (26)$$

where

m = gradient
c = intercept

This may be arranged as

$$Ln[Ln(1/R_i)] = nLn[x_i] + nLn[k] \qquad (27)$$

where

R_i = cumulative mass retained on size i, as a decimal fraction
x_i = the size i
n^i = the gradient
k = the intercept for R_i = 0,3679 (equivalent to the $d_{63,21}$ of the distribution)

This form was reported by Allen [Allen, 1990a].

The linearised form by Allen has the advantage of being amenable to a least mean squares regression procedure to attest to the consistency of the particle size distribution (psd) data, in that a correlation coefficient may be calculated to quantify the agreement of reported % mass per size with the overall data.

Such a procedure for arithmetical values was reported by Moroney [Moroney, 1982] to use the following format:

$$m = \left(\left(\sum_{i=1}^{n} x_i y_i - \left(\sum_{i=1}^{n} x_i\right)\left(\sum_{i=1}^{n} y_i\right)/n\right)\right) / \left(\left(\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2/n\right)\right) \qquad (28)$$

where

m = the gradient
n = the number of observations
x = the independent variable
y = the dependent variable

$$c = \left(\left(\sum_{i=1}^{n} x_i\right)\left(\sum_{i=1}^{n} x_i y_i\right) - \left(\sum_{i=1}^{n} y_i\right)\left(\sum_{i=1}^{n} x_i^2\right)\right) / \left(\left(\sum_{i=1}^{n} x_i\right)^2 - n\left(\sum_{i=1}^{n} x_i^2\right)\right) \qquad (29)$$

c = the intercept

The covariance of x and y is now calculated as an intermediate step to the correlation coefficient:

$$Cov(x,y) = (1/n)\left(\sum_{i=1}^{n} (x_i - \bar{x})(y_i - \bar{y})\right) \qquad (30)$$

where

Cov(x,y) = covariance of x and y.

The correlation coefficient r is then calculated using the covariance of x and y as

$$r = (Cov(x,y))/(\sqrt{(s^2_x)(s^2_y)}) \qquad (31)$$

The value of r is bound within

$$1 \geq r \geq -1 \qquad (32)$$

i.e. r=1 implies perfect positive correlation; and r=-1 implies perfect negative correlation. Theoretically r=0 implies that no association of significance can be found between x and y.

By using the transform

$$y = Ln[Ln(1/R_i)]$$

and

$$x' = nLn[x_i]$$

$$Ln[Ln(1/R_i)] = nLn[x_i] + nLn[k] \qquad (33)$$

becomes

$$y = nx' + nc;$$

Therefore a plot of $Ln[x_i]$ as x' against $Ln[Ln(1/R_i)]$ should give a straight line plot of gradient n and intercept $nLn[k]$.

This should be a suitable check on grading data at whatever size used in the flotation laboratory.

1.3.2 Laboratory Grinding

The use of a laboratory scale ball mill to treat samples of crushed ore and produce simulated flotation feed material for testing is common, as often the study material as ore is only available in this form. However certain limitations apply to this format as the laboratory scale grinding is in batch format, as opposed to continuous arrangement in a production mill. This results in the laboratory mill producing a wider size distribution than obtained in production closed-circuit grinding [Wills, 1987a]. In particular the overgrinding of high-density sulphides is underestimated by this procedure due to the inverse classification effect in a closed-circuit production mill. To an extent this is paradoxical, as the batch format of the laboratory mill will also encourage overgrinding of fine sulphides due to the absence of a continuous release of fines during grinding [Wright, 1994].

Wills further observes that the data interpretation from this format is the more accurate if the performance is normalised in terms of the actual mineral size (presented to flotation), assuming that the same fineness of the valuable mineral will give the same flotation results from both formats regardless of the size distributions of the other minerals present.

A further extension of this reasoning indicates that testwork should be carried out over a range of grinding sizes, in conjunction with flotation tests, in order to determine the optimum mesh of grind.

This view is confirmed by equivalent research at production scale which is configured to optimise the grinding circuit so as to maximise the amount of minerals produced in floatable sizes [McIvor et al, 1990]. The grinding and flotation operations at whatever scale thus need to be viewed as a combined process.

Work reported by Dawson indicated that the one-kilogram sample size for such testwork remained the optimum, in preference to one-gram or one-ton sizes [Dawson, 1978], and was the most vital link in the chain for the development of ore resources. This view is shared by Casquina in the testwork programme for selective flotation of a Cu/Pb/Zn ore [Casquina, 1990]. It was observed that ultra-small laboratory cells do not provide a practical basis for scale-up [Coleman, 1980],however in none of the aforementioned references is any clear statement made about how may times the flotation test should be repeated to verify the reproducibility of the response.

Other investigations into the grinding media used showed that the effect on the flotation electrochemistry as a result of media selection could have a significant influence on the floatability of certain minerals [Martin et. al., 1991], therefore the most practical beginning would be to use similar media in both production and laboratory milling, or in the case of a new mine, to experiment with various media and document the reponses so as to optimise the flotation performance.

In terms of calibrating the laboratory mill to correlate grinding time with mesh of grind produced, it is common to perform several test grinds on subsamples of equal mass of a batch of crushed ore, using different trial grinding times. The milled product is tested for particle size distribution and a plot of grind versus grinding time is made [Lotter and Munro, 1993].

This procedure heuristically relies on the selection function in selection and breakage theory [Austin et. al., 1984a], and produces a curvilinear plot which is drawn empirically between points. Longer grinding times produce more curvature in the relationship, suggesting that the rate of grinding slows down as finer grinds are attempted.

Examination of the batch grinding equations by Austin et. al. shows that, in the first instance, a $\sqrt{2}$ test sieve series is essential to obtaining reliable particle size distribution (psd) data. Further, the selection function S_j describes the rate of breakage of material out of parent size. This function is summarised as

$$w_j^*(t) \;/\; w_j^*(0) = \exp(-S_j t) \qquad (34)$$

where

$w_j^*(t)$	=	weight of size j present after milling t minutes
$w_j^*(0)$	=	weight of size j present prior to the commencement of milling, ie t=0 minutes
S_j	=	the specific rate of breakage of size j, in units of min^{-1}
t	=	the milling time, in minutes.

Experimentally this first-order hypothesis has been shown to be an excellent approximation for many materials in several types of mill. The description of the progeny of such breakage is made by the breakage function, summarised as

$$B_{i,1} = \Phi_1 (x_{i-1}/x_1)^{\tau} + (\Phi - 1)(x_{i-1}/x_1)^{\beta} \qquad (35)$$

where

Φ_1	=	breakage coefficient of the ore
τ, β, z	=	parameters specific to the ore
x_{i-1}	=	Size i-1
x_1	=	Size i

Φ_1 is further modified by size using

$$\Phi_1 = \Phi_{i-1}(x_i/x_{i-1})^{-z} \qquad (36)$$

This allows for estimation of the mass produced per size i below the breaking, or parent, size.

It has been shown that the above parameters are relatively independent of milling conditions and are characteristic of the ore.

Under conditions using long milling times in a single-stage fine grind it is likely that the coarser sulphides liberated early on in the grinding are rebroken and overground. This leads to the concept of staged grinding at this scale to float the coarse sulphides off in a rougher float prior to commencement of the subsequent finer grinding. In such a case it is advisable to filter the rougher tails off the coarse float and reconstruct the slurry rheology to a suitable density rather than simply transfer the tailings as a flotation slurry into the mill. In this way the calibration of the grinding curve is kept more consistent, and a more accurate picture of the likely metallurgical response obtained. The benefit of staging grinds for flotation performance benefits was noted by Dawson [Dawson, 1978].

It is concluded from the foregoing that, in principle, batch milling of ore subsamples from a common bulk blended sample can reliably be done on a time-based control. Empirical calibration of the time-based relationship between grinding time and fineness of grind remains to be proven in confirmatory tests. Check gradings of the milled product would, however, be advisable.

1.3.3 Flotation Tests

The theory of froth flotation is an incomplete science, but utilises the differences in physico-chemical surface properties of the particles of various minerals. Once suitable reagents are added to a milled slurry of ore, the differences in surface properties become apparent and the floatable particles, attached to collector reagents, are transported vertically through to an aerated froth by air bubbles [Wills, 1987].

This process relies on the hydrophobic nature of the collected particle. The contact angle formed by the interaction of the mineral solid surface, the air bubble and the water determines the floatability.

For sulphide flotation it is common to use sulphydryl collectors such as xanthates and dithiphosphates. These are heteropolar and have an alkyl chain for the hydrophobic character, and an ionised group containing sulphur. The latter reacts with water.

Under certain redox conditions both the xanthate and dithiophosphate form their respective dimers, viz. dixanthogen and phosphatogen, the latter less readily [M.C.Fuerstenau, 1978a].

In the case of xanthate,

$$2X^- = X_2 + 2e \quad (37)$$

$$\tfrac{1}{2}O_{2(ads)} + H_2O + 2e = 2OH^- \quad (38)$$

Overall

$$2X^- + \tfrac{1}{2}O_{2(ads)} + H_2O = X_2 + 2OH^- \quad (39)$$

where

X^- = xanthate ion
e = electron
X_2 = dixanthogen.

Reaction 39 is pH-dependent and tends to favour the reactants over products at high pH values, i.e. more dixanthogen is produced at acidic pH than from alkaline pH. In particular pyrite depends on dixanthogen to float.

pH is a significant factor in flotation, firstly because it affects the balance of xanthate/dixanthogen present in solution, and secondly, because of the free hyroxyl ions OH^- present at higher pH incurring the likelihood of sulphide surface coating.

The mechanism of adsorption of sulphydryl collectors may occur by chemisorption, by van der Waal's bonding betweeen hyrocarbon chains, and by physisorption [M.C. Fuerstenau, 1978b]. Studies conducted in the virtual absence of oxygen found that the adsorption is independent of pH and that adsorption density is less for longer-chained xanthates. The further conclusion was that the adsorbed species was xanthic acid, using a chemisorption mechanism which exchanges hydroxyl ion on the sulphide mineral surface with xanthate ion.

In the presence of oxygen, the semiconductor characteristics of a floatable sulphide mineral play a significant role. It was shown that oxygenation of a flotation pulp affects particularly the flotation of pyrite and pyrrhotite, further assists the activating effect of copper sulphate [Huout and Duhamet, 1990]. The importance of oxygen in xanthate/pyrrhotite interaction was compared to depression of pyrrhotite by flotation in pure nitrogen, concluding that oxygen played a significant role [Rao and Finch, 1991]. An alternative reaction to the formation of dixanthogen in the adsorption mechanism, as well as recognition of the role of oxygen, was reviewed by Martin et. al., concluding from several references [Martin et. al., 1991] that:

$$MS + 2X^{-} = MX_2 + S + 2e \qquad (40)$$

where

MS = metal sulphide
MX_2 = metal xanthate

(i) the mineral must be conducting or semi-conducting to permit the flow of electrons in reactions 37 and 40 to 38.
(ii) dissolved oxygen must be present in solution to act as the electron accepting element. (Reaction 39).

This review also noted that the presence of too much oxygen presents disadvantages to collector adsorption. The pyrite/pyrrhotite group encounter surface oxidation as a result, and very seldom is enough collector used to cover the entire mineral surface.

The effect of oxidation on pyrrhotite flotation was noted by Iwasaki. Infrared spectra of reaction products from known mineral surfaces showed either dixanthogen or metal xanthate. The nobility of the mineral surfaces in relation to the xanthate/dixanthogen couple determines whether dixanthogen or metal xanthate will form. Minerals nobler than the couple encourage dixanthogen formation; metal xanthate forms on minerals less noble than this couple [Iwasaki, 1988].

Electrochemical investigations into the flotation chemistry of pentlandite and pyrrhotite to describe process water and xanthate interactions showed that certain inorganic ions

such as Ca^{++}, $S_2O_3^{2-}$ and SO_4^{2-} were

significantly active at normal pH, and would influence the extent of adsorption of X^{-} by the sulphide minerals and retard the development of hydrophobicity [Hodgson and Agar,

1989]. It was further shown that Ca^{++} and $S_2O_3^{2-}$

competed for adsorption on pentlandite but Ca^{++} increased the xanthate dosage requirements of pyrrhotite.

In terms of xanthate species, xanthate chemisorption was identified for pentlandite, whilst pyrrhotite showed none of this behaviour but became hydrophobic in the presence of dixanthogen.

The floatability of a mineral is then a balance between hydrophobic and hydrophylic sites on the mineral surface. Flotation is an oxygen-dependent process, and the condition of the mineral surface also influences the amenability of collector interaction.

The above interaction is further complicated by the corrosion of steel grinding media used in milling. The floatability of pyrrhotite was reported to be higher when produced from stainless steel or porcelain media than from "carbon steel"[mild steel] media [Pavlica and Iwasaki, 1982]. A form of galvanic interaction was discussed, concluding that

Pyrrhotite Electrode (Cathode):

$$FeS + \tfrac{1}{2}O_2 + H_2O + 2e = Fe(OH)_2 + S^{2-} \qquad (42)$$

Mild Steel Electrode (Anode):

$$Fe^{o} = Fe^{2+} + 2e \qquad (43)$$

Ferrous iron released by the pyrrhotite upon dissociation reacts with the hydroxyl ion produced by the cathodic reduction reaction between pyrrhotite and oxygen, producing initially an iron hydroxide that is stable under the relevant conditions.

The standard oxidation potentials ϵ^{o} of the half reactions of Fe/Fe^{2+} and Fe^{2+}/Fe^{3+} [Hepler, 1964] were reported to be :

$$Fe^{o} = Fe^{2+}_{(aq)} + 2e \qquad \epsilon^{o} = 0,44\ v \qquad (44)$$

$$Fe^{2+}_{(aq)} = Fe^{3+}_{(aq)} + e \qquad \epsilon^{o} = -0,77\ v \qquad (45)$$

These potentials show that metallic iron is a moderately strong reducing agent, and that the ferric ions are oxidising agents. Because of the smaller size and higher charge of the trivalent Fe^{3+} compared to the divalent Fe^{2+}, ferric ions are much more hydrolysed in aqueous solution.

For the first step in the hydrolysis,

$$Fe^{3+}_{(aq)} + H_2O = Fe(OH)^{2+}_{(aq)} + H^{+} \qquad (46)$$

for which $K = 5 \times 10^{-5}$, shows that increasing concentration of H^+, i.e. lower pH, shifts the equilibrium, by Le Chatelier's principle, to the left hand side. Equally as the pH is increased, $Fe(OH)^{2+}_{(aq)}$ and more complicated species containing more than one atom of Fe per ion are formed; addition of base forms a precipitate of hydrous ferric oxide, represented by $Fe_2O_3.nH_2O$.

Incipient oxidation of pyrrhotite generates elemental sulphur on its surface leading to collectorless flotation as well as improved flotation with xanthate. Further oxidation retards the floatability due to the formation of iron hydroxide coating that masks the elemental sulphur coating. The grinding media now influence the floatability of pyrrhotite in two ways [Iwasaki, 1988; Adam et.al., 1984]:

(i) Abraded mild steel particles interact magnetically with pyrrhotite and lower the rest potential of pyrrhotite, making the production of dixanthogen difficult,

(ii) Galvanic interaction between pyrrhotite and grinding media produces iron hydroxide, coating the pyrrhotite surfaces and making collector attachemnt difficult.

In conclusion of the potential effects of electrochemistry on flotation, it is seen that redox conditions overall are very influential in assisting the stability and concentration of suphydryl collectors in their various forms, and that depending on which mineral is to be floated by which collector form, the range of redox suitable for the operation must be identified.

The overall use of this approach was recently documented by Fleay and Lane in a study on the effects of grinding environment on the flotation of Kambalda ores [Fleay and Lane, 1994], where EDTA titrations of mill product water, and E_h measurements were able to pinpoint the cause of unsuccessful flotation of ores from certain sources in the ore reserve. The model so constructed was able to configure laboratory ball mill conditions suitable in terms of redox for simulation of a future autogenous grinding retrofit at the concentrator.

The use of copper sulphate activator in the flotation of these base metal sulphides was investigated by Perry in the context of fine particle flotation. It was concluded that for the pH range 6-9 the use of copper sulphate activated the base metal sulphides but also activated certain gangue minerals, in particular pyroxene.

This was explained by the predominance of the copper species $CuOH^+$ and $Cu_2(OH)^{++}$ in the pH range 6-8, and that copper sulphate caused the zeta potentials of the gangue minerals to be reversed in sign at $6 < pH < 9$. Under these conditions the zeta potentials of these gangue minerals are reversed as the metal ions in solution begin to hydrolyse to their repective hydroxy complexes. This reversal in sign of zeta potential was attributed to the adsorption of these metal hydroxy cations on the mineral surfaces [Perry, 1980]. From his analysis of the soluble forms of copper in this system, the following equilibria were suggested by Perry:

$$Cu^{++} + H_2O = CuOH^+ + H^+ \qquad (47)$$

$$K_2 = 2{,}51 \times 10^{-8}$$

$$Cu(OH)_2(s) = Cu^{++} + 2OH^- \qquad (48)$$

$$K_{sp} = 1{,}6 \times 10^{-19}$$

$$2Cu^{++} + 2H_2O = Cu_2(OH)_2^{++} \qquad (49)$$

$$K_3 = 1{,}58 \times 10^{-11}$$

From these equilibria the role of pH on the state of equilibrium is seen. Clearly acidic pH favours production of Cu^{++} in equation 47. Equally, high pH favours the production of $Cu(OH)_2(s)$ in equation 48.

Copper activation of sulphide minerals [Wills, 1987] is described in its simplest form as a cementation reaction of the type

$$MS + Cu^{++} = CuS + M^{++} \qquad (50)$$

where

M = divalent metal
MS = metal sulphide.

The free availability of Cu^{++} to drive equation 50 to the right-hand side is therefore a factor in determining the degree of cementation of copper on the metal sulphide surface; this in turn is - for copper sulphate - dependent on lower rather than higher pH.

The more metallic copper surface on the metal sulphide is now more amenable to adsorption of collector, rendering the metal sulphide particle hydrophobic.

In his discussion of test results, Perry observed that, in the absence of copper sulphate, use of collectors over a range of pH values yielded optimum recoveries at the lowest pH values (in the order of 3-4), with pyrrhotite showing the worst response at high pH. When copper sulphate is used ahead of collector dosage, the recovery of all three minerals improved at the higher pH values, but particularly pentlandite.

It is concluded at this stage that copper sulphate has been documented as an activator for the flotation of chalcopyrite, pentlandite and pyrrhotite, however the response is complex and pH-sensitive; and activation of gangue minerals is also likely.

The principal effects of particle size in flotation were identified by Trahar, finding that the minimum degree of hydrophobicity for particle flotation depended on particle size. The use of size-by-size metal or mineral recovery stemmed from this as a diagnostic tool for process optimisation [Trahar, 1981]. Entrainment was identified as a contributing mechanism for fine particle flotation, when coupled with a low flotation rate for fine sizes, substantially accounted for the observed behaviour of fine particles.

Trahar proposed three size categories for flotation:

(i) < 5-10 microns : difficult to float and more difficult to separate,
(ii) 10-70 microns : ideal size range,
(iii) > 70 microns but with an undefined upper size, where flotation may be easy or difficult depending on mineral type.

The exact limits to the above performance groups were shown to be case-specific by mineral type and operation. A generic correlation between particle size and flotation rate was modelled, suggesting the following proportional form:

$$k \alpha d^n \qquad (51)$$

where

k = a suitable measure of flotation rate,
d = particle diameter
n = a constant, in the order of 1,5-2,0.

In data collection for this model, k was calculated over the first minute of flotation. Under such conditions, entrainment was regarded as insignificant.

Entrainment was also modelled by size as a normalised function expressing the recovery of mineral to that of water, and a correlation was found. Entrainment was most significant for fine particles. This infers that the fines recovered by both entrainment and slow flotation will be found in concentrates further down the sequence of the mineral process.

The rate of flotation is also an important parameter to describe. A well-known rate model for flotation was developed in the following form [Dowling et.al., 1984] :

$$R(t) = R_e[(1-1/kt)(1-\exp(-kt)] \qquad (52)$$

where

R_e = Equilibrium Recovery at $t = \infty$

$R(t)$ = Recovery of the floating mineral at time t, minutes

k = the flotation rate, min^{-1}.

This model, called the First Order Model with Rectangular Distribution of Flotabilities, was selected from 13 different model forms. The study material was a bulk sample from Newmont's Pinto Valley chalcopyrite/pyrite orebody. The test format was a rougher float using soda ash in the milling to depress the pyrite, and potassium amyl xanthate for the Cu flotation. Results estimated k to be in the order of 6,5 for a grind of some 42,7 % passing 75 microns, and in the order of 14,7 for a coarser grind of 31,6 % passing 75 microns. From this investigation two important observations are made:

(i) The value of k is dependent on grind. Coarse grinding produces a faster flotation rate than finer grinding.

(ii) The value of k=6,5, together with the R_e value,

models 86,5% recovery at t=3 minutes, which is rapid flotation. No discussion was offered for slower floating minerals, for example the pyrite which was being depressed during these tests.

This reference, however, made two more important observations. Statistical analysis of the calculated head values across the entire range of tests reported a mean head grade of 0,534% Cu with a standard deviation of 0,003.

The authors used this observation to conclude that considerable confidence in the assay values and the sampling technique could be assumed. Further, the flotation tests were repeated in triplicate and the products of each replicate analysed to attest to the reproducibility of the results for the given test conditions.

1.3.4 Conclusions

In summary of this section of review, the following conclusions have been drawn:

(i) Particle size distribution, an essential part of the vocabulary of flotation tests, may be linearised using the Rosin- Rammler model, and least squares regression formulae are applicable to the checking of the test data.

(ii) Batch-scale laboratory grinding to produce test flotation feed may be controlled on the basis of time to produce similar replicate grinds, using trial grinding times to calibrate the mill. However a check grading is advisable amoungst these replicates to confirm the grind produced.

(iii) The media used in the mill are influential in configuring flotation conditions similar to those prevailing in the production plant, therefore it is advisable to match laboratory media to plant media.

(iv) Electrochemical conditions leading from (iii) continue into flotation. In particular pH is identified as influential, also the dissolved inorganic cations such as Ca^{++}. This infers that selection of process water from the plant is more realistic for flotation tests than tap water. Oxygen is seen in a key role in the process, as collector attachment is a redox process.

(v) Copper sulphate has a complex influence on the flotation of pentlandite and pyrrhotite in particular. Its effect is pH- dependent and under neutral to moderately alkaline conditions it also activates pyroxene, making the latter amenable to collector adsorption.

(vi) Particle size plays a central role in flotation rate and recovery, and three main performance groups have been identified, the limits of which would advisably be locally determined for a particular ore and operation.

1.4 Evaluation of the Mineral Process, with Particular Reference to Fire Assay Techniques

The objectives of mineral process evaluation, which were described in § 1.2.3, defined certain requirements of minimum sample mass, and how a production mill feed could be sampled representatively using a stop-belt technique. The rules of minimum sample mass calculation are applicable to concentrator products. Further rules concerning the reduction ratio necessary in crushing such a sample, followed by blending prior to subsampling a 10% cut for investigation or analysis, were noted.

The further rules relating to representative sampling of the products of a mineral process, and subsequent preparation for analysis, also require examination.

1.4.1 Sampling of Mineral Process Products

In the context of flotation, it is common for mineral process streams to be handled in the form of a slurry. These slurries are often exposed as falling streams from an inclined launder, therefore Gy's rules of the sampling of a falling slurry [Gy, 1979b] apply.

Presentation of Production Stream to Sampling

A U-shaped launder whose transported stream discharges through a well delimited area is suitable. Extreme turbulence in this stream is to be avoided as transport through the delimited area becomes problematic. Such turbulence may be corrected for by installing a series of metal pipes in the last few metres of the launder, such that the top surface of the turbelent stream flows through the top layer of pipes.

Increment Delimitation Error

The sample cutter has the correct geometry only when the cutter edges are parallel, regardless of the angle with the stream. The increment actually delimited through the stream is an inclined parallelogram. In such a case the cutter geometry is correct as all the elements of the stream are intercepted during the same length of time. The sampling probability for all particles is uniform for all particles regardless of their position in the stream cross-section. The cutter must, however, traverse the stream such that the stream is intercepted by the middle third of the cutter length.

Correctness of Cutter Speed and Cutter Width

The cutter velocity should remain constant during its traverse of the falling stream, further should be constant between traverses. For an intermittent electrically driven

electrically driven cutter, a timer sets the cutter in motion from an "idle" position, which is outside the domain of the falling stream. The cutter then enters the stream at a constant speed and takes an increment. At the end of its travel, the cutter drive is automatically switched off and the carriage braked to a standstill. On the later return trip the motion is reversed. It is recommended that the cutter speed should be 0,6 m/sec according to

$$V = (1+n)0,3 \quad m/sec \qquad (53)$$

where

V = cutter velocity, m/sec
n = the cutter width expressed as a multiple of minimum cutter width W_o

The correct minimum cutter width W_o is calculated as

For particle topsize > 3mm:

$$W_o = 3d \qquad (54)$$

For particle topsize ≤ 3mm:

$$W_o = 10mm \qquad (55)$$

The most economic sampling is obtained with n=1, in which case $W = W_o$ and V = 0,6 m/sec.

1.4.2 Sample Preparation Prior to Analysis

The steps involved in sample preparation are amenable to Gy's preparation errors [Gy, 1979c], and care is necessary in configuring the methods, physical conditions and selection of equipment to perform this task.

Filtration

Samples taken from a production concentrator are frequently in the form of a slurry contained in a bucket. It is essential that these buckets use lids that prevent loss of sample and contamination by foreign material, further that the buckets are colour-coded by grade of sample to limit their use to specific sampling applications [Lenehan and de L. Murray-Smith, 1986a]. The sample is best filtered using a pressure filter, consisting of a stainless steel drum sitting on a filter cloth covered by suitable filter paper. Compressed air is introduced from the air-tight top of the filter.

Drying

The most reliable drying oven uses a thermostatically controlled regime. The consequences of uncontrolled high temperature during drying tend to induce sulphur dioxide emission from samples containing sulphide minerals, particulary chalcopyrite, pentlandite and pyrrhotite, as these become pyrophoric at certain grades. Generally 105-110^{o}C is a good temperature range for ore and tails samples; this is cooled to less than 80^{o}C for concentrates.

Blending

It is vital that the bulk dry sample be rigorously blended prior to any subdivision for whatever purpose. A V-blender is popular in this duty. A pair of steel cylinders join at an angle to form a V, arranged vertically. A sump of cylindrical steel is joined at the base of this V. The arrangement is driven horizontally by an electric motor. The top ends of the V have lockable lids to allow discharge of blended sample after the operation.

Such a unit has three efficiency parameters: rotational speed, volume of charge and mixing time.

A 100 litre V-blender would handle a sample charge of

300-500 cm^{3} in 10-30 minutes at a rotational speed of 20 rpm.

Splitting of Samples

Several authors refer to the spinning riffler as the most reliable way of subdividing a sample [Lenehan and de L. Murray-Smith, 1986b; Spedden, 1985; Allen, 1991b]. The alternative use of the Jones riffler to subdivide samples may be acceptable but the method of operation is critical to producing an unbiassed subsample [Gy,1978d].

The relative efficiencies of five common sampling procedures reviewed by Allen, using a binary sand mixture, quantified the effect of these on sampling error. The results clearly show that the spinning riffler is the superior device [Allen, 1990b]:

Table 1.5
Reliability of Selected Sampling Methods

Method	Std Dev %	Variance	Estimated Maximum Sample Error %
Cone and Quarter	6,81	46,4	22,7
Scoop Sampling	5,14	26,4	17,1
Table Sampling	2,09	4,37	7,0
Chute Riffling	1,01	1,02	3,4
Spinning Riffler	0,125	0,016	0,42
Random Variation	0,076	0,0058	0,25

It was concluded that very little confidence could be placed in the first three techniques; and that the spinning riffler was so superior to all other methods that it should be used wherever possible.

Further work with this device showed that a minimum of 35 presentations was necessary to obtain best results. The device was later recommended in ASTM Standard F577B-78. Further remarks by Allen state that this is the correct equipment to reduce the bulk sample not only to the intermediate "laboratory" size sample of ca. 1 kg, but also down to the analysis sample.

1.4.3 The Fire Assay Process

Fire assaying is commonly practiced in the determination of trace concentrations of precious metals. This is because it pre-concentrates the precious metals from a large sample into a lead button, which may be processed further in the domain of higher precious metal tenor. This process minimises the sampling error arising from the discrete particle deportment within the sample, otherwise termed the nugget effect. The purpose of the process is to quantitatively separate the precious metals in an ore or metallurgical process sample into a lead button, and the other material in the sample, into a slag [Lenehan and de L. Murray, 1986b].

The flux used consists principally of sodium carbonate, litharge (lead oxide), and borax. A reducing agent is also necessary to react with the litharge and produce lead metal and carbon dioxide.

Weighed sample and flux are mixed and placed in a crucible. The crucible is heated in a furnace, the rate of heating and final temperature of which are important controls. Initial furnace temperature is 1100°C. As the batch of crucibles is loaded this falls to 800-900°C, and rises to 1100°C over a period of ca. 45 minutes. On completion of the fusion the crucible is removed from the furnace and its contents poured into a mould, commonly of inverse conical shape. Here the fusion products settle and solidify as they cool. Thereafter the mould is inverted and the lead button removed from the slag. The former may now proceed to further treatment such as cupellation, where the lead is separated from the precious metal at high temperature, leaving a precious metal prill suitable for gravimetric determination.

Categorisation of Ores for Fire Assay

Ore types are classified for this process into acidic, neutral and basic. The majority of South African ores are acidic due to their high silica content in the order of 60%. Certain anologies between aqueous chemistry and pyrochemistry are made. These relate to the acidic/basic properties of the ions in aqueous solution, and those of the oxides in pyrochemistry. These analogies are summarised in Table 1.6.

A common acidic oxide in South African ores is SiO_2, with B_2O_3 an important separate feature where flux preparation is concerned.

Basic oxides in these ores are the oxides of bivalent metals, viz. FeO, MgO, CaO, PbO. Of lesser importance are the trivalent forms of chrome and iron, Cr_2O_3 and Fe_2O_3.

A relevant example of an amphoteric oxide in ore is Al_2O_3.

Beacuse of their naturally high silica content, most South African ores are acidic, and self-fluxing to a certain extent. In the case of evaluation samples from a sulphide mineral process, the presence of metal sulphides such as pyrite introduce a reducing agent into the fusion, which complicates the aforementioned initially convenient arrangement.

Table 1.6
Analogies Between Aqueous and Pyrochemical Acid/Base Reactions

Solution Chemistry	Pyrochemistry
Hydrogen ions impart acidic properties to a solution.	Non-metal oxides impart acidic properties to a slag.
Hydroxyl ions impart basic properties to a solution.	Metal oxides impart basic properties to a slag.
Acids and bases in solution react to produce salts and water.	Acidic oxides and basic oxides combine by fusion to form slags (or salts).
A solution is classified as acid, neutral or basic by the ratio of concentrations of hydrogen to hydroxyl ions.	A slag is classified by the type of silicate or borate produced, viz. by the ratio of the oxygen in the base to the oxygen in the acid, before fusion.
An acid reacts with sodium carbonate to form a salt and water, with liberation of carbon dioxide.	An acidic oxide will fuse with sodium carbonate to form a slag (salt), liberating carbon dioxide.

Solution Chemistry

$$2HCl + Na_2CO_3 = 2NaCl + H_2O + CO_2$$

Pyrochemistry

$$SiO_2 + Na_2CO_3 = Na_2SiO_3 + CO_2$$

Principles of Fluxing

Some reactions relevant to this process are summarised as follows:

$FeS_2 + 5PbO = FeO + 2SO_2 + 5Pb$

$2Fe_2O_3 + C = 4FeO + CO_2$

$2Cr_2O_3 + C = 4CrO + CO_2$

$FeS_2 + Fe = 2FeS$

The production of bivalent metal oxides is central to the creation of a suitable slag. In the reaction of pyrite FeS_2 with metallic iron, a soluble form of the sulphide is produced which remains in the slag.

The fluxing of samples containing amphoteric oxides involves a two-stage reaction:

$Al_2O_3 + Na_2CO_3 = 2NaAlO_2 + CO_2$

$2NaAlO_2 + 3SiO_2 = NaAlSi_3O_8$

Fluxing Practice

The characteristic properties of the major flux constituents are as follows:

Sodium Carbonate: Na_2CO_3

A powerful basic flux. Readily forms alkali silicates.

Melts at 850°C. Sulphates are formed in the presence of air; for this reason it may be considered as a desulphurising agent.

$FeS_2 + 7PbO + 2Na_2CO_3 = FeO + 7Pb + 2Na_2SO_4 + 2CO_2$

At 950°C it dissociates partly and evolves carbon dioxide:

$Na_2CO_3 + Na_2SiO_3 = Na_2SiO_4 + CO_2$.

Silica: SiO_2

Silica is a strong acidic flux. It combines with metallic oxides to form silicates, fundamental to most slags.

$PbO + SiO_2 = PbSiO_3$

Borax: Anhydrous Sodium Borate $Na_2B_4O_7$

Borax melts at 741°C to form a viscous glass, and is fluid at elevated temperatures. It is a strongly acidic reagent that readily dissolves most metal oxides. This occurs in two stages:

$$Na_2B_4O_7 = Na_2B_2O_4 + B_2O_3$$

$$B_2O_3 + ZnO = ZnB_2O_4$$

In addition to being an excellent solvent for base metal oxides, borax lowers the fusion temperatures of all slags by significant amounts.

Litharge: PbO

A readily fusible basic flux. It melts at 883°C and, together with reductant such as maize meal (a source of carbon), produces metallic lead for precious metal collection. Litharge also has a strong affinity for silica, therefore the silica content of the sample should be known so as to prevent crucible wall damage.

Red Lead Oxide: Pb_3O_4

This may be used as an alternative source of lead, however has an oxidising action

$$2Pb_3O_4 = 6PbO + O_2.$$

Nitre (Potassium Nitrate): KNO_3

Nitre is a powerful oxidising agent. It melts at 339°C

and decomposes at 400°C, releasing oxygen. In the presence of silica this effect is enhanced.

$$2KNO_3 = 2KNO_2 + O_2$$

$$4KNO_3 + 2SiO_2 = 2K_2O.SiO_3 + 2N_2 + 4O_2$$

Nitre has an important oxidising effect on base metal sulphides. This is critical to effective slagging of base metals in the primary fusion so as to prevent scoria problems in later cupellation.

$$4FeS_2 + 10KNO_3 = 4FeO + 5K_2SO_4 + 3SO_2 + 5N_2$$

Reducing Agents

The purpose of reduction in a fire assay is to reduce lead oxide to metallic lead. Carbon is the most effective reductant.

This is sourced from maize meal, a common agricultural product in South Africa; alternatively flour is suitable. The process of reduction produces either CO or CO_2 depending on temperature:

$PbO + C = Pb + CO$ (high temperature)

$2PbO + C = Pb + CO_2$ (low temperature)

Metal Collectors

Lead is commonly used for gold collection. It is less effective for platinum group metals, where nickel as nickel sulphide forms the basis of the case-specific "nickel collection" method for PGE. This has, however, the disadvantage of lower gold recovery. Silver is sometimes added to the flux to assist lead in PGE collection.

Cupellation Practice

The purpose of cupellation is to separate the lead from the precious metals. This is done by placing the lead button from the primary fire assay fusion on a preheated cupel,

made of calcined magnesite, in a furnace at 900- 1100^{o}C under oxidising contions.

Cupels have a particular geometry. Circular in plan cross-section, the top surface of the cupel must have a concave shape, and depth 0,6 times the radius. The cupel mass must be sufficient to absorb all the lead oxide formed during the process. Some lead (2%) is volatilised. The molten precious metals are not absorbed by the cupel since they do not oxidise as readily as the lead, and have a higher surface tension.

The condition of the prill produced is critical to accurate gravimetric determination. Base metal oxides, if present to a certain extent in the lead button, cause scoria, or oxide encrustation of the prill. This reduces the recovery of precious metals to the prill, since the latter are partly soluble in the molten oxides, therefore a low result will be obtained. This behaviour defines the need to completely oxidise the base metal sulphides present in the original sample, so as to collect them in the slag phase in the primary fusion. Nitre plays a central role in this regard.

Temperature control during cupellation is critical to accuracy of the analysis, for the following reasons. The melting points of gold, silver and litharge are 1064, 961, and 886^{o}C respectively. If the cupellation furnace temperature falls below 900^{o}C, litharge freezes and "feathers" of crystallised litharge form, preventing complete cupellation.

Precious metal losses during cupellation occur as the temperature increases much above 1000^{o}C, mostly due to a decrease in surface tension, encouraging absorption by the cupel. Minor volatile losses occur. In the case of gold, this may be inhibited to a certain extent by addition of silver to the primary fusion. Such losses are inversely grade-proportional. In the case of PGE, this technique is helpful in collecting the former in very low grade samples. In such cases the final prill is not gravimetrically determined but by acid dissolution and either spectrophotometric or inductively coupled plasma methods. Losses during cupellation have therefore to be corrected for by a calibration process.

1.4.4 Fire Assaying for PGE

Case-Specific Factors

The fire assaying process described in § 1.4.3 undergoes certain alterations to cater for the complexities inherent in Bushveld igneous ores such as Merensky and UG2. Although when added together to the collective grade term "PGM+Au" commonly used in the metal accounting and control of the mineral process, the total grade may be workable in terms of detection limits, the individual grades of Pt, Pd, Au and Rh (called the 4-element set "4E") are in their own lower grade groups that are more difficult to determine on an individual basis.

A further complication is inherited from the four different mineralogical forms in which the 4E are borne (refer § 1.2.1), as well as the wider forms of major silicates and oxides in the host rock, viz. pyroxenite, feldspar, serpentine, chromite.

Fusion of such samples incurs the need for higher primary fusion temperatures in the order of 1000-1150^{o}C for 60 minutes [Lenehan and de L. Murray-Smith, 1986c], as well as recalculation of primary flux composition for the following reasons:

(i) The high CaO and MgO content of the ore, in the order of 5-7 and 15-17% respectively, imparts more basic properties to the fusion slag.
(ii) The base metal sulphides chalcopyrite, pentlandite, pyrrhotite and pyrite impart reducing characteristics to the primary fusion, as well as interfering with cupellation by scoria formation unless correctly slagged. This effect is grade-proportional.
(iii) The presence of chromite requires reducing conditions to form the CrO slag-soluble form.
(iv) The presence of alumina in significant amounts (ca. 5%) introduces amphoteric behaviour and incurs the need for the two-stage formation of $NaAlSi_3O_8$ suitable for slag formation.

The standard lead fusion flux for Merensky ore samples thus consists of sodium carbonate, borax, maize meal and silica, with the optional addition of nitre in higher grade samples where base metal sulphides become significant. Less maize meal is needed in this application than in gold ores, as the sulphides in Merensky add to the reducing nature of the charge.

The cupellation of the lead button from PGE-bearing samples undegoes two stages, a low-temperature (LT) and a high-temperature (HT) stage. The low-temperature stage is performed at 900-1000^{o}C,as for gold ores, but the second stage is done at 1300-1350^{o}C.

Various procedural options emerge from this analysis. In the case of very low-grade samples such as tailings from the mineral process, silver is added to to the primary fusion, and only LT cupellation is done. The additional silver "masks" the 4E from volatilisation and/or absorption by the cupel, gravimetric determination is waived and the silver button dissolved in acid prior to instrumental analysis.

More commonly both steps of cupellation are used, and the three volatile PGE, Ru, Os, Ir, are quantitatively burnt off the prill as their tetroxides in the HT process. This however incurs secondary but significant losses of the 4E Pt, Pd, Au and Rh, and correction factors become necessary. Again the order of this loss is inversely grade-proportional because of the surface area to unit mass aspect of the prill by grade, but is in the order of 1-30%.

For this reason calibration tests are necessary for a given mining area or concentrator. Such a test is available in an alternative primary fusion method called nickel collection.

Correction Factors and Nickel Collection

The method of nickel collection for PGE determination originates in South Africa and was first prototyped by Munro in 1969 [Munro, 1994] with other workers developing the theme in publications [Robert et. al., 1971, Palmer et. al., 1971, Dixon et. al., 1975, Robert et. al., 1975]. Other work was reported in Russia [Kuztnetzov et.al., 1974].

Nickel sulphide has several advantages as a collector:

(i) a low melting point, 800^{o}C,
(ii) the method is amenable to samples containing significant amounts of nickel and copper,
(iii) the high density of nickel sulphide encourages efficient matte separation,
(iv) the matte buttons are easily ground for further treatment,
(v) the ground matte is easily treated by acid.

Alterations to the primary fusion flux are made so as to form a slightly acidic slag. A charge of nickel carbonate is added to the flux. Nickel collection flux therefore uses no litharge and consists of borax, sodium carbonate, elemental sulphur, nickel carbonate and copper sulphate.

The sulphur and nickel carbonate produce the nickel sulphide, which collects the PGE during fusion. The copper sulphate corrects for the initial inadequacy of the nickel collection to quantitatively collect gold, since it reduces to copper sulphide. The borax and sodium carbonate perform the conventional slagging function.

Fusion temperature is 1250^{o}C for 60 minutes.

The mass of the matte button produced can be accurately calculated from the mass of sulphur and nickel carbonate in the flux charge, giving a cross-check on the method prior to the dissolution step.

The matte button separates easily from the slag when the fusion conditions are correct, and proceeds through pulverisation in a ring pulveriser to primary leach in

HCl/NH_4Cl. This starts an exothermic reaction

generating H_2S gas, and the reaction is quenched as

the gas generation ceases. The leach residue is washed on a millipore filter and finally dissolved in aqua regia, made up to volume and the solution read on either atomic absorption or inductively coupled plasma spectrophotometers.

As this method does not use cupellation, and since the PGE are more readily recovered in the nickel sulphide matte than in metallic lead, it does not incur losses in the order of 1-30%, as is the case with lead collection. The correction factor for the particular ore type or mineral process product is then determined by performing a minimum of 12 each of lead fusions and nickel collections on one blended composite sample. The mean grade of each is calculated, and the factor CF as

$$CF = Ni_{coll}/Pb_{fus} \qquad (56)$$

where

Ni_{coll} = mean PGE grade from Ni collections,

Pb_{fus} = mean PGE grade from Pb fusions,

PGE grade = sum of individual grades of Pt, Pd, Au and Rh.

If the prills from the replicate lead fusions are kept after weighing and taken to a prill split technique, where the quadruplicate sets of prills from the determinations are dissolved in aqua regia and the individual PGE determined in a manner similar to the leached nickel matte button, then individual correction factors for Pt, Pd, Au and Rh are obtainable with little extra effort.

1.4.5 Conclusions

This section of review has drawn the following conclusions:

(i) The sampling of an operating mineral process is amenable to Gy's rules of sampling a falling slurry. Increments taken towards the bulk sample must observe the rules of probable inclusion.

(ii) The bulk sample must be effectively contained and labelled during transport to filtration. Principal grade groups of sample must be separately handled.

(iii) Drying of the sample must be temperature-controlled according to grade group, in order to prevent decomposition of sulphides prior to analysis.

(iv) After blending, the dried bulk sample should be subdivided only using a spinning riffler. Other methods of subsampling incur unnecessary error.

(v) The fire assay process for precious metals may be used for Bushveld ore evaluation, provided that certain alterations to flux and furnace procedure are made. Although the modified lead collection method incurs losses of PGE prior to gravimetric determination, these may be corrected for by use of the nickel collection method, which is also applicable to individual PGE.

1.5 Specific Objectives of this Research

The specific objectives of this research programme are therefore:

(i) To study replicate grading analyses within a natural standard and provide a checking method to certify grading tests on unknown samples, especially laboratory ball mill products.

(ii) To construct value frequency distribution models for samples of ore and mineral process products.

(iii) To use probability plots as a foundation to characterise the ore studied.

(iv) To assess tailored sample preparation methods that will correct the value frequency distribution prior to calculation of mean grade in whatever sample.

(v) To determine representative mill feed sampling standards to ensure that reliable samples are obtained for flotation tests. To examine flotation test products in the same light.

(vi) To formulate reliable estimates of grade in the mill feed sample. This may involve prior grinding of the ore sample before analysis, and calculation of how many replicates are necessary in analysis.

(vii) To quantify the acceptable expectations of agreement between replicate assay determinations, using the accuracy of correlation between reconciled and assayed head grades as the criterion.

(iix) To construct a quality control model, to decide whether to accept or reject certain tests on the basis of their values being outside Sichel's 95% limits about the mean.

(ix) To compare the assayed head grade data on the same basis, and to reconcile the metal account within the 95% confidence limits.

2. EXPERIMENTAL PROCEDURE

2.1 Particle Size Distribution

The true particle size distribution of a mineral process stream is never known; estimates of the distribution may be made, but there will always be an error in its estimation. This is because the probability of obtaining a sample which perfectly represents the parent distribution is remote. Further, several samples of the same stream may differ even if these are representative [Allen, 1990c]. One can only attempt to characterise a process stream within a certain margin of error; herein lies a practical approach of stating what constitutes acceptable error, and what equivalent confidence level accrues from such a statement.

The following experiment was constructed from this argument in order to demonstrate and quantify what agreement between determinations of particle size distribution, within a blended composite sample of flotation tailings, could be obtained.

This exercise in precision started with a bulk sample of Waterval Concentrator Final Tailings. The sample was taken over a week of production using the rules of incremental sampling and blending described in § 1.4.1. A total sample of 200 kg was collected. This large mass was to provide reference natural standard material for a suitable period.

The bulk sample was then subsampled into more workable representative units by operating the spinning riffler (§ 1.4.1 Splitting of Samples) by using an "odds and evens" technique. Increments of ca. 10kg. of the bulk sample were run through the spinning riffler into 10 equal and proportional subsamples, each captured in their own subsample vessel.

The subsample vessels were numbered 1-10. At the end of each run, the contents of all the odd numbered vessels were emptied into a clean dry drum of capacity 200 litres, labelled "Odd". Equally all the even numbered vessels were emptied into a separate drum of the same capacity, labelled "Even".

At the end of this module of subsampling the drum labelled "Odd" was rerun through the spinning riffler to again halve the sample size. Two subsamples in the order of 1 kg each were taken for test purposes. The same was done to the drum labelled "Even".

The four quarters of this sample, each in the order of 50 kg, were stored. Initial grading tests were performed on 200 g subsamples of the 1 kg lots from the odd and even sets to obtain an idea of the size distribution. The results were presented to the minimum sample mass formula for gradings [Allen, 1990c] according to

$$M_s = 0,5[S_s/\theta^2]\{(1/w_i)-2)d_i^3.1000 \qquad (56)$$

where

M_s = the limiting mass in grams

S_s = the powder density, in g/cm^3

θ = the variance of the tolerated error

w_i = the fractional mass of the coarsest size sampled

d^3 = the arithmetic mean of the cubes of the extreme diameter in the size class, in cm^3.

The grading method whereby these primary data were obtained used a wet-screening technique that removes the finest fraction in the series prior to dry-shaking the remainder of the weighed sample [Allen, 1990d].

The catchweight of approximately 200 g of tailings was slurried in water using a stirrer. The slurry was then loaded in batches of ca. 50 g on a 38 micron test sieve mounted in a Fritsch wet-screen vibrator and the motor energised. Fines were washed through the 38 micron screen during vibration. The next 50 g increment was added when the fines underflow pipe showed insignificant quantities of solids being removed from the sample.

At the end of this batchwise procedure the +38 micron remainder of the aliquot was dried in a sample oven at

106^oC. The dried product was then shaken over a stack of test sieves using sizes 425, 300, 212, 150, 106, 75, 53 and 38 microns. The shaking time was 10 minutes.

The sized products were weighed cumulatively on a toploader Mettler balance with 0,1 g divisions. A check was made on the presence of any minus 38 micron material in the fines sump underneath the test sieves, in order to confirm that the wet-screening had been effective.

The minimum sample mass for size distribution having been calculated from these initial gradings, replicate grading tests were performed over a period of several weeks, using different operators at random. The results were pooled and a distribution of the results, based on cumulative % mass passing 75 microns, was calculated.

The 95% control limits of any subsequent determination of grading on a subsample of this natural standard were then calculated as $\bar{x} \pm 2s$, ie within two robust standard deviations of the sample mean.

This enabled the use of the natural standard to certify the reliability of any grading on an unknown tailings or float feed sample. A typical use is calibrating the laboratory ball mill for test grinds to produce a desired grind. Such a test grind procedure used the time-based approach of grinding time (§ 1.3.2).

The reason for this check is twofold:

(i) The sieve becomes blinded with intersize particles, reducing the percentage open area of the sieve, this leading to the incomplete sieving error ϵ [Austin et al, 1984b; van Niekerk, 1989]. A similar effect is seen with short shaking time during the grading trial, also with too large a sample.

(ii) The sieve develops a hole larger than the test aperture; this is either a puncture in the sieve mesh or, more commonly, a failure of the solder at the rim of the sieve between the mesh and the sieve frame. In such a case the fineness of the sample is overstated.

A trial grading on the natural standard producing a result outside the 95% confidence limits thus indicates that the test sieve deck requires inspection and cleaning or replacement, and that the trial data on the unknown samples in test grinding are unreliable and must be repeated.

2.2 Value Frequency Distribution (VFD) and Probability Plot in Ore

It was shown in § 1.2 that basic studies to characterise the VFD of a particular ore would be an informative start to good evaluation. In particular, the degree of skewness in such a VFD will incur certain requirements in terms of the calculation of mean grade and 95% confidence limits about the mean. This remark is particularly intended towards the taking of small samples ($4 < n < 10$) from such a distribution, as laboratory scale flotation tests are in the latter domain.

It is accepted that in terms of the Central Limit Theorem, large data increments will yield a robust estimate of the population statistics by using the arithmetical average [Box et. al., 1978].

Three production shafts in a particular mining area of Merensky ore were identified for this study. Two are located in the opposite extremes of the mining lease area, and one in the centre.

Survey chip sampling of these shafts is undertaken on a routine basis for purposes of grade control and mine call factor. The hangingwall, reef and footwall are each sampled and analysed for PGE as a 4-element total of Pt, Pd, Au and Rh.

Using the method of Krige to pool the data for a particular shaft, ie treating the particular shaft as an ore reserve [Krige, 1981], the fire assay data were collected over several months. Separate records for hangingwall, reef and footwall were kept. Grade is expressed as the mass of PGE per ton of ore.

The final data were then sorted from lowest to highest grade for each ore type, and the frequency of observation per grade group, or bin, counted. The frequency was then normalised as a percentage of total observations per bin.

Three tables of such data were thus obtained for each shaft. The frequencies by grade group were then plotted as y values, with grade group as x values, on arithmetic scale graph paper. This constructs the *Value Frequency Distribution,* or VFD.

The VFD data were then transformed into probability plot format by calculating the cumulative frequencies with the cumulative grades, starting from the lowest grade of the distribution and working systematically towards the highest grade in the distribution. The transformed data were then plotted on logarithmic probability paper to test for lognormality. This paper uses a probability scale for the x-axis (based on inverse Gaussian format) with a logarithmic y-axis for cumulative grade [Krige, 1951; Clark and Garnett, 1974]. This plot should form a straight line if the data are lognormal.

Should more than one straight line appear in such a plot, this is regarded as evidence of multiple mineralisation phases [Clark and Garnett, 1974].

2.3 Description of the Mineral Process, and Exercises in Value Frequency Distributions of Mineral Process Streams

The mined ore described in section 2.2. is delivered to a concentrator, where initial jaw crushing and screening allows the hydraulic separation of an ultrafine size range of ore by means of a rake classifier, which works on a Stokes' law principle. This material bypasses the primary and secondary milling operations and proceeds directly to rougher flotation after thickening.

This leads to subsequent finer cone crushing of the topsize fraction of the jaw crusher product, leading to a fine mill feed being presented to conventional ball milling. Refer Figure 2.1. There are two main process inputs to the conventional Merensky concentrator:

(i) Conventionally crushed and screened ore for presentation as primary ball mill feed,
(ii) Ultrafine Crusher Classifier Overflow (CCO) for presentation directly to rougher flotation.

The value frequency distributions for these primary circuit inputs were gathered from 13 months of production data. Probability plots were prepared in the same manner as in § 2.2.

Figure 2.2 shows the mill feed undergoing a staged grinding and flotation process. Only the primary stages are shown here. Cleaner flotation (not shown) finally produces a concentrate of base metal sulphides with smaller quantities of discrete metallic minerals such as ferroplatinum, cooperite, braggite, etc., the bulk of the latter having been recovered by gravity processing of the primary concentrates.

The fact that the separation of metallic minerals to the gravity concentrate is imperfect causes the PGM+Au values in the final flotation concentrate to be carried in two intrinsic forms, viz. in solid solution with the BMS, and as discrete but fine-grained PGM. As these two categories in their pure mineral forms represent two different grade domains, it is necessary to investigate their value frequency distributions prior to accepting a standard method for their evaluation.

The production data for Frank Concentrator Final Concentrate were collected over a 13-month production period. The equivalent data for Waterval Concentrator were also collected. Value frequency distributions and lognormal probability plots were prepared in the same manner as § 2.2.

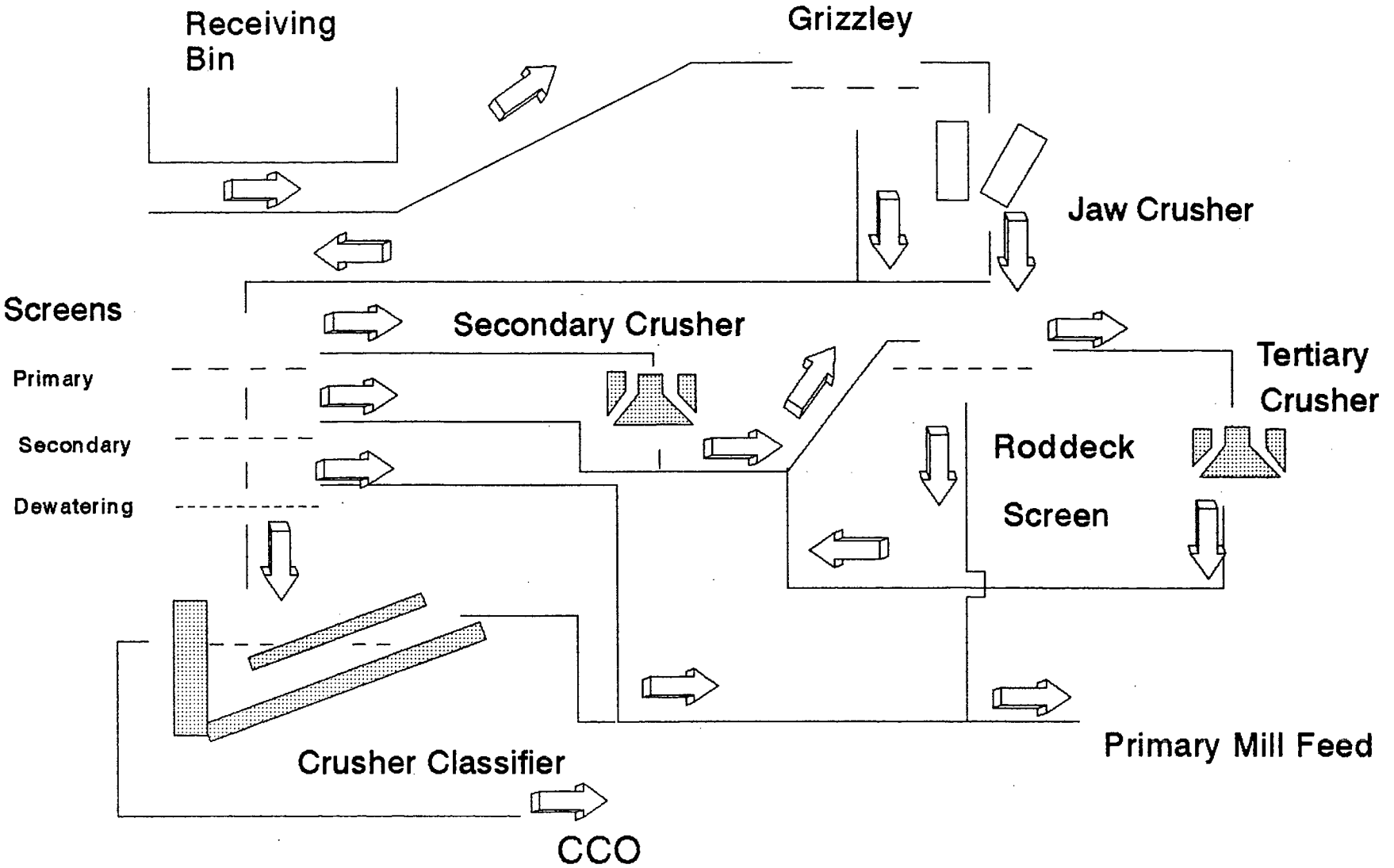


Figure 2.1
Waterval Concentrator Crushing
and Screening Flow Schematic

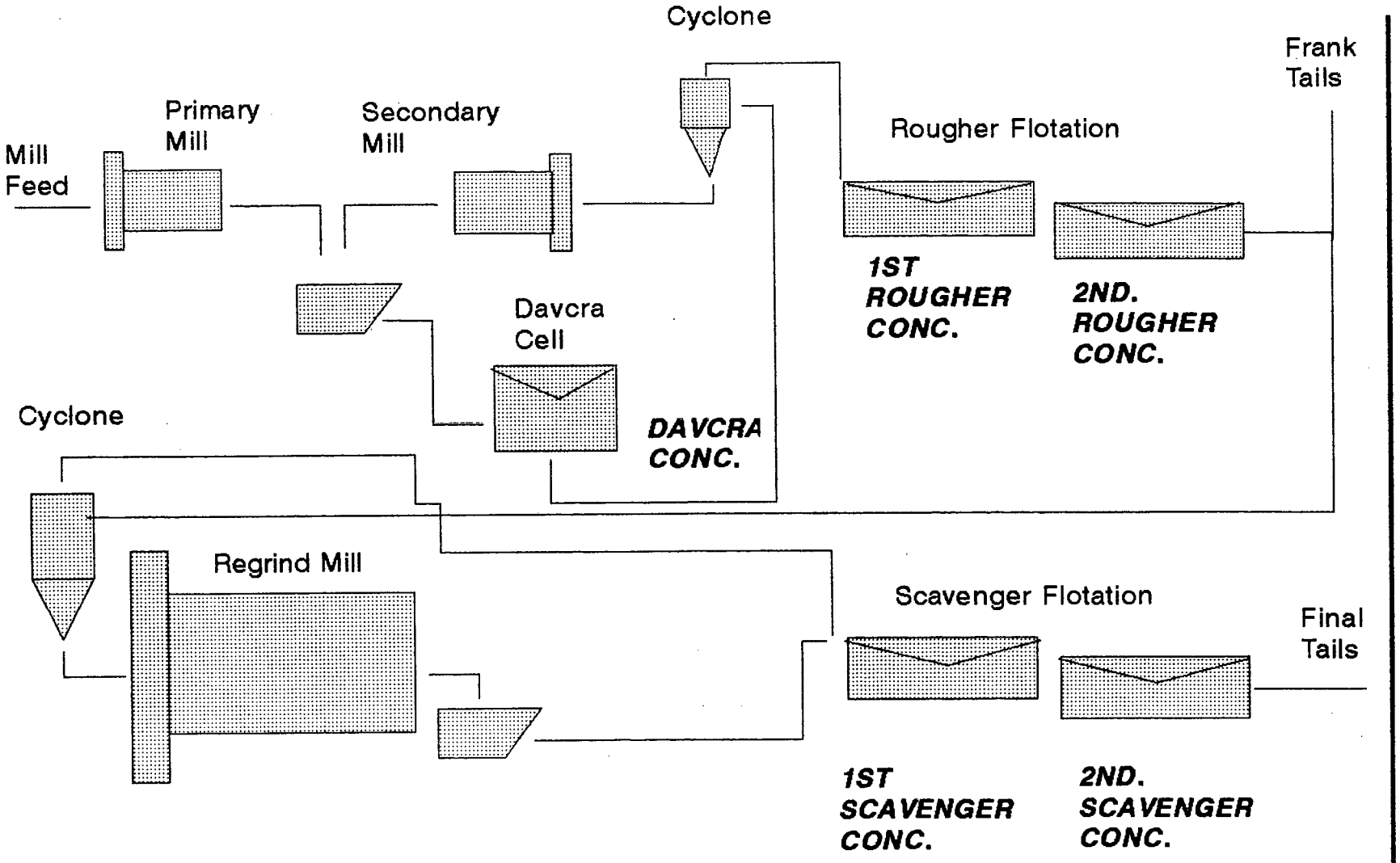


Figure 2.2
Waterval Concentrator Milling and
Flotation Flow Schematic

In order to describe the equivalent VFD and probability plot for the gravity concentrate (called "metallics"), where a higher proportion of discrete PGM will be present, the Davcra Concentrate and Final Metallics data for Waterval Concentrator were gathered over a 13-month production period. Data preparation was done in a similar manner to § 2.2.

This set of exercises will produce with time VFD and probability plots for the following mineral process streams:

Waterval Mill Feed
Waterval CCD Float Feed
Frank Final Concentrate
Waterval Final Concentrate
Waterval Davcra Concentrate
Waterval Metallics Concentrate

From these results the appropriate observations may be made with regard to the nature of the VFD.

2.4 Assessment of Errors Inherent in Sample Processing and Analysis

2.4.1 Mill Feed

Size Distribution and Size-by-Size Analysis: Preparation for the Calculation of Representative Sample Size

A bulk representative sample of Waterval Mill Feed was taken using the technique described in § 1.2.3. After drying at 106°C it was blended using the spinning riffler odds and evens method described in §2.1, and a preliminary grading performed in quadruplicate on the following sieve sizes:

Size, mm
9,50
6,70
4,75
3,35
2,36
1,70
1,18
0,850
0,600

The mean particle size distribution of these data was plotted on Rosin-Rammler graph paper in order to examine the linearity of the plot. The d80 size of the sample was then obtained by inspection of this graph, using the 20%

cumulative mass retained as the mark. The bulk sample was then reblended and subsampled using the odds and evens method to release a 10kg subsample for size-by-size analysis. The 10kg subsample was sytematically fractionated into the above sizes using increments of ca. 200 g loaded on to the top of the test sieve deck. After each pass the separated fractions were put into identified sample buckets for later use.

These fractionated sizes of mill feed were then analysed for 4E PGE grade and dry solids specific gravity.

This test was repeated using a larger 50 kg subsample of the mill feed, this time subsampling the sized fractions into six samples per size. The results of PGE and specific gravity analysis from this exercise would lead into an interpretation of Gy's 50-piece experiment for mill feed sample size.

A further 50 kg of the bulk sample of mill feed was then passed over the test sieve size closest to the d80 value found by the preliminary gradings. The fines were discarded and all the material coarser than the sieve size was accumulated into a sample bucket. This was blended and subsampled into lots of ca. 300 g, each subsample pulverised using a ring pulveriser. These were analysed for 4E PGE, and would form the basis of data for Laplante's interpretation of the Poisson Distribution as a means of calculating mill feed sample size.

Analysis of the Variance of PGE grades within a Sample of Mill Feed: State of Grind and Determination of the Minimum Number of Assay Replicates Required for a 95% Confident Statement of Grade

A further bulk sample of mill feed was taken for the purpose of a combined study of "assay heads" and "built-up heads". Because flotation was to be used as part of the trial, the bulk sample was first sun-dried on a clean concrete surface, rather than in the drying oven at 106°C. An initial grading was done on a subsample of this bulk, thereafter it was crushed in a laboratory cone crusher to minus 3 mm and processed in the spinning riffler in the odds and evens method to produce subsamples. The bulk sample weighed approximately 200 kg. The mill feed subsamples were made up into 2,5 kg lots for test purposes. This was done with the spinning riffler by relying on the proportionality of the method to produce tenths of sample mass, thus by loading 25 kg of mill feed into the feed hopper, each subsample will be close to 2,5 kg in mass. The final adjustments of less than 25 g were made manually whilst the raw 2,5 kg sample was on top of the laboratory scale.

Test lots of 2,5 kg were milled one at a time in a laboratory mill using a 40 kg charge of high-chrome 40 mm steel balls and one litre of water. The mill operates at 85% of critical speed. Trial grinding times of exactly 12, 20, 27 and 35 minutes were used, and on each occasion the mill was stopped on termination of the trial grinding time. These times were chosen to produce grinds of approximately 40, 50, 60, and 70% mass passing 75 microns in milled product. The milled product was discharged as a slurry into a bucket and filtered using a pressure filter. The filter cakes were dried in the drying oven at 106°C. A grading was done on each of these milled products using 525, 300, 212, 150, 106, 75, 53 and 38 micron size test sieves. The method for the grading is described in § 2.1.

The gradings were summarised into terms of cumulative % mass passing 75 microns, and this value plotted on the y-axis of arithmetic graph paper opposite the grinding time as the x-axis. A curvilinear fit was made to the data, and modelled grinding times for exactly 40, 50, 60, and 70% passing 75 microns inferred from the graph.

Thereafter triplicate repeats of this procedure were done using the now-determined grinding times so as to produce 7,5 kg of milled ore at each of these known grinds. The overall 7,5 kg lot of ground ore per grind was then blended using the spinning riffler and subsampled into 200 g unit masses. This produced 37 replicate samples per grind, with 100 g to spare in each case. Each of these was submitted for fire assay to determine the 4E PGM+Au grades. The analyst was advised to allow natural scatter in the determinations. Each sample provided 4 fusions.

These results will be used to determine the effect of grind on the characteristics of the VFD, particularly the scatter, for crushed and blended ore. Further, they will provide a means of calculating the minimum number of assays necessary to arrive at a 95% confident estimate of the mean grade of the ore sample, called the "assay head". Here the t-distribution will be used [Moroney, 1982b]. Calculation of the mean and 95% confidence limits are discussed in § 2.8.

The remainder of this ore sample was reserved for later use in flotation tests.

2.4.2 Flotation Concentrate

A representative bulk sample of Waterval Final Flotation Concentrate was taken, observing the rules noted in § 1.4.1. This was oven-dried at 80°C and blended using the spinning riffler odds and evens method.

The sample was divided into three equal portions, A, B and C. Portion A was again subdivided into sixty replicate subsamples, and each analysed for 4E PGM+Au. Portion B was also subsampled to sixty replicates, but in this case exactly 50 g of sample was pulverised in a sieb mill with exactly 50 g of clean silica for 1 minute. These pulverised products were then sent as if concentrate samples for analysis. The thinking behind this treatment was based on the premise that discrete PGM present as metallics in the concentrate will distort the view of the VFD within a composite sample because of the nugget effect they are known to have. By pulverising the sample in clean silica, an artificial "solid solution" of metallics in silica is obtained, hopefully with less nugget effects in the VFD. The remaining portion C was used for laboratory scale superpanning tests to remove the metallics as a heavies concentrate, leaving a base metal concentrate as a superpanner tailing. This separation was done 12 times in order to obtain a reliable estimate of the metallics content of the sample.

The method used for superpanning the 12 replicate concentrate samples was as follows:

(i) An 80 g catchweight of the concentrate sample was slurried in 250 ml water using a stirrer. The slurry was presented to the superpanner in 50 ml aliquots so as not to overload the unit.

(ii) The superpanner was started and the aliquot separated into crude heavies concentrate and light superpanner tailings by the shaking motion of the unit. The primary separation was regarded as complete when the gravity concentrate had travelled the entire length of the superpanner.

(iii) The crude heavies concentrate was removed by pipette into a 500 ml glass beaker containing some 100 ml of water.

(iv) The superpanner tailings were removed by vacuum into a Buchner flask.

(v) After the 5 batches of this treatment, the original slurry sample was quantitatively in two products. The crude heavies were returned to the superpanner and cleaned in a secondary superpanning treatment. The heavies are silver in colour, and are easily identified opposite the yellow colour of chalcopyrite, which demarcates the separation.

(vi) The cleaner tailings were added to the rougher tailings, and the overall tailings sample filtered, dried and weighed.

(vii) The cleaned heavies concentrate was removed by pipette and released on to a weighed Whatman #541 hardened ashless filter paper in a glass funnel. The funnel rested on top of a 250 ml glass beaker, and the filtrate drained through the filter into this vessel.

(iix) The wet filter paper with the cleaned metallics concentrate was dried in a drying oven at 106°C, thereafter weighed.

(ix) The mass of the metallics concentrate was calculated as the difference between the dry mass of filter paper plus metallics, and dry filter paper.

(x) The dry filter paper plus metallics were then prepared for analysis by pulverisation in clean silica. The mass of silica used was calculated as 50 g minus the estimated mass of metallics; the 0,5 g of filter paper will burn during the fire assay to an insignificant mass of ash.

At the end of all these analyses, the following may be determined:

a) The sample mean, standard deviation and RSD of the unprocessed flotation concentrate (group A). A probability plot of the VFD will derive a statement of normalisation.

b) The sample mean, standard deviation and RSD of the pulverised concentrate (group B), also with a probability plot. Note that here the raw assays must be multiplied by 2 to obtain the true grade of sample.

c) The mean metallics potential of the concentrate sample, to check opposite any diagnostic feature of the group A probability plot.

d) A conclusion regarding the best method of sample preparation prior to analysis of flotation concentrate.

2.4.3 Flotation Tailings

A bulk sample of flotation tailings was taken from Frank Concentrator, using the methods described in § 1.4.1. It was dried at 106°C in a sample drying oven and blended using the spinning riffler and the odds and evens method. A total of 200 kg sample was collected over a week of production.

A grading was done on 4 subsamples of ca. 1 kg produced from this procedure. The remaining bulk of the sample was then spin-riffled into 2,5 kg test lots and stored in 10-litre buckets with sealed lids. It was important to seal the buckets to minimise the likelihood of sample contamination, as tailings are generally much lower grade than ore and concentrate.

Twelve of these test lots were again split down to unit size of 1 kg, then analysed for 4E PGM+Au without further sample preparation as thirty samples. The analyst was requested to allow natural scatter in the replicate determinations.

Three more blocks of thirty samples, each 1 kg, were prepared for analysis by successively finer grinds, 50, 60 and 70% passing 75 microns. This was done using the test grind and milling method described in § 2.4.1.

At the end of this exercise the VFD of tailings may be constructed for each state of grind; if there is no significant difference the data may be pooled and the number of replicate determinations required to arrive at a 95% confident estimate of the mean grade will be calculated. If state of grind is shown to have a significant effect on the scatter in the VFD then an optimum grind will be selected as a standard for use in preparation of tailings samples prior to analysis.

2.5. A Quantitative Graphical Calculation of the Amount of Discrete PGM "Metallics" Present in Flotation Concentrate

If discrete PGM and solid solution PGE are in two different grade domains, or orders of magnitude, then the likelihood exists that the probability plot of a VFD describing the PGE grades within a sample will infer how much of each form is present. If superpanning tests have also been done on the sample to empirically derive a statement of metallics potential, then the proposed graphical method after Clark and Garnett will apply.

As an easy start to this theory, the primary concentrate known from production experience to carry the most metallics, Davcra Concentrate, was selected. A bulk sample of ca. 50 kg was taken using the rules and methods of representative sampling described in § 1.4.1. The sample was dried and blended as for concentrate (§ 2.4.2) and split into the replicate subsample groups A, B and C.

The ABC treatments of § 2.4.2 were repeated for this sample, producing:

(i) Sixty Group A subsamples without pulverisation,

(ii) Sixty Group B subsamples with pulverisation in an equal mass of clean silica,

(iii) Twelve sets of Group C superpanner concentrates (pulverised in clean silica up to a known total sample mass of 50,0 g) and tailings.

The sample sets A, B and C were analysed for PGE using fire assay.

Value frequency distributions and probability plots for A and B were constructed using the methods described in § 2.2, and the metallics potential of C determined as follows:

For each pair of superpanner products "concentrate" and "tailings":

a) Mass of superpanner tailing, g = M_t

b) Assay grade of superpanner tailing, g/t PGE = A_t

c) Mass of superpanner concentrate (pulverised in silica) g = 50,0

d) Assay grade, g/t PGE, of pulverised (50 g) concentrate = A_{sil}

e) Mass of superpanner concentrate before pulverisation with clean silica, g = M_c

f) Inferred primary grade, % PGE, of superpanner concentrate prior to pulverisation in clean silica = A_c

g) PGE content of superpanner tailings, mg PGE = PGE_{tl}

h) PGE content of superpanner concentrate, mg PGE = PGE_{con}

The PGE contents, in mg, of the superpanner products are calculated as

$$PGE_{con} = (50/(10^6)).\ A_{sil}.10^3 \qquad (57)$$

$$PGE_{tl} = (M_t/(10^6)).A_t.10^3 \qquad (58)$$

The grade, in % PGE, of the superpanner concentrate prior to pulverisation in clean silica, A_c, is calculated as

$$A_c = ((PGE_{con}/(10^3))/M_c).10^2 \qquad (59)$$

The metallics potential, MP, in g/t of PGE, of the Davcra Concentrate sample was then calculated as

$$MP = 10^6.(PGE_{con}/10^3)/(M_t+M_c) \qquad (60)$$

Stoichiometric calculation of the order of magnitude of PGE grade in discrete PGM (metallics) showed the following results:

PG Mineral	Formula	Approximate Pure Mineral Grade, % PGE
Ferroplatinum	Pt_3Fe	91,29
Sperrylite	$PtAs_2$	56,56
Braggite	Pt<PdNiS	41,47

Although each of these metallics minerals varies in composition - as naturally occurring mineral species - the order of PGE concentration is clearly in percentages, not parts per million as seen in solid solution data (§ 1.2.1).

A probability plot of PGE values from Group A replicate subsamples of the Davcra Concentrate was made. At the point where the plotted points departed from the original line (starting from the low-grade values) and started another straight line, a mark was made and labelled A [Clark and Garnett, 1974].

The arithmetic mean of the VFD in Group A was calculated using the formula shown in Appendix 1, § 1: Basic Statistics. The result, x, was in units of g/t.

The horizontal x-axis intercept corresponding to point A in the probability plot was obtained by vertical extrapolation of A to the x-axis. The result, A_1, was in units of cumulative % frequency.
As the value of A_1 represents the cumulative frequency to the left of A_1, ie the lower grades of the VFD, the metallics are represented by the higher grades to the right of A_1, so the cumulative % frequency of metallics in the distribution must be $100-A_1$.

The metallics potential of this sample by graphical method must therefore be MP_{graph} where

$$MP_{graph} = x.(100-A_1)/(100) \qquad (61)$$

A numerical comparison of MP_{graph} and MP was made in order to test the possible agreement between the two methods.

A further benefit from this exercise was to compare the VFD models and probability plots for Groups A and B subsamples of Davcra Concentrate. In later work if significant quantities of metallics are to be floated in common with base metal sulphides carrying solid solution PGE, the sample preparation prior to analysis should be considered. The scatter about the mean, and the possible improvement in linearity in the Group B probability plot as a result of the pulverisation, shall be adjudicating features.

2.6 Thirty Replicate Standard Flotation Tests: Construction of Mean and 95% Confidence Limits in Reconciled Head Grade and Recovery

In order to quantify the justifiable limits within which reproducible flotation tests are possible, a set of thirty replicate standard flotation tests was established, using the remainder of the bulk ore sample used for the second exercise in § 2.4.1.

The remainder of the ore sample was already in 2,5 kg subsamples and had been crushed to a suitable size for laboratory scale ball milling. The test grind curve was also already known, so all that remained was to define at what grind the standard flotation tests would be done, and to list a standard procedure.

A one-step grind of 70% passing 75 microns was selected as this corresponds to suitable liberation of the PGE. The test grind graph was consulted and a grind time of 33,5 minutes was found for this grind.

The following standard procedure was repeated thirty times, and the test products from each replicate test kept separate:

2,5 kg of crushed ore was loaded into a clean laboratory ball mill with 40,0 kg of high-chrome 40 mm diameter steel ball charge. One litre of water was added to the mill contents. The mill door was closed and locked, then the mill placed horizontally on driver rolls on a mill table. The power to the driver rolls was switched on and a stop-watch started at the same time. The mill was stopped at a milling time of 33,50 minutes and the stop-watch reset.

The mill was removed from the driver rolls and was stood vertically so as to orientate the mill door at the top of the mill. The mill door was unlocked and its inside surface washed into the rubber lined mill sump below, which had a retaining plate drilled with 10 mm holes to allow the slurry from the mill to fall into a plastic bucket below.

The rest of the mill contents were discharged in a similar manner into the mill sump, and clean wash water used to remove the remaining slurryfrom the mill. After a final wash, the steel ball charge was returned to the mill for the next grind. The milled slurry was quantitatively collected in the plastic bucket during this process, and was transferred to the laboratory scale Denver flotation cell. The unaerated slurry was made up to exactly 4,0 litres total volume by addition of clean water.

The flotation cell was loaded into the Denver flotation machine assembly and the rotor lowered into the operating position. The following flotation treatment was then applied:

Time, mins	**Activity**	**Reagent Dosage g/t**
	Start Flotation Machine Rotor Speed 900 rpm.	
0	Conditioning #1 Add $CuSO_4.5H_20$	80
	Add Sasfroth	8±1
7	Conditioning #2 Add Xanthate X-222	40
	Add Dithiophosphate Senkol 5	40
10	Commence Flotation Aeration Rate 7,5 l/min Collect 1st Rougher Concentrate	
13	Collect 2nd Rougher Concentrate	
25	Stop Flotation Stop Aeration Stop Flotation Machine, wash rotor Reset Stop Watch.	

Concentrate collection was done in a stainless steel rectangular pan of depth 50 mm, width 15 cm and length 25 cm.

During flotation, specific arrangements of the froth scraping were used. The froth scraper was custom made, in the T-shaped form of a depth gauge. The outer extremities of the T sat on the flotation cell wall so that the bottom section of the scraper worked at a constant froth depth of 10 mm relative to the overflow launder. A second, simpler froth paddle was used to recover froth from the rear of the cell. The froth scraping procedure used was at intervals of 15 seconds, with primary froth scraping from the rear of the cell, followed by depth-gauge scraping to remove the froth from the cell and capture it in the concentrate pan below.

Make-up water was added to the flotation cell as necessary during flotation, so as to ensure a consistent froth depth in line with the above scraping practice. To achieve this, a mark was made on the inside wall of the cell to show the 4-litre level with the flotation rotor in the slurry.

The two concentrates were separately filtered using a vacuum filter and Whatman # 541 hardened ashless filter paper. The tailings were decanted into a large pressure filter and filtered.

Concentrates and tailings were then separately dried at 80^{o} C and 106^{o} C respectively.

The dried flotation test products were then weighed for each replicate test, and analysed for 4E PGE using the fire assay method reported in § 1.4.3.

For each replicate flotation test, therefore, the following set of samples and sample data were obtained:

Sample	**Mass** g	**Grade** g/t 4E PGE
Conc 1	M_1	G_1
Conc 2	M_2	G_2
Tailing	M_3	G_3
Generic form	M_i	G_i

Subscript i denotes the array within the particular flotation test, and is bound for this exercise between $1 \leq i \leq 3$.

The mg PGE content of each of these products was calculated as follows:

$$Cont_1 = 10^3.G_1.(M_1/10^6) \quad (61)$$

$$Cont_2 = 10^3.G_2.(M_2/10^6) \quad (62)$$

$$Cont_3 = 10^3.G_3.(M_3/10^6) \quad (63)$$

The built-up head (or reconciled head) grade was then calculated as BH for the replicate flotation test, where

$$BH = 10^{-3}.(\sum_{i=1}^{3} Cont_i)/(\sum_{1=1}^{3}(M_i/10^6)) \quad (64)$$

The recovery of PGE in the test is calculated as RPGE where

$$RPGE = 100.(\sum_{i=1}^{2} Cont_i)/(\sum_{1=1}^{3} Cont_i) \qquad (65)$$

The set of built-up heads BH_j and recoveries $RPGE_j$ for j=30 replicate tests was then gathered using Eqs. 61-65.

It was then possible to calculate the mean and standard deviation of built-up head grade and recovery of PGE.

These measures of PGE grade, estimating grade of ore and recoverable grade, form a foundation of later quality control. This axiom is based on the conservation of matter, since provided that the handling of the flotation test products incurred no significant loss of material, the built-up head, or reconciled head grade should theoretically have a true mean and acceptable confidence limits about that mean. It remains to be shown how these may be estimated.

2.7 Construction of the Mean and 95% Confidence Limits in Reconciled Head Grade and Recovery

On the assumption that all thirty replicate flotation tests were conducted under exactly the same conditions, one is entitled to expect that similar reconciled head grade values will be obtained, also that similar recoveries will be recorded. This assumption is bound within the following three influences:

(i) the natural scatter of these two variables arising from non-homogeneous distribution of PGE minerals in the unmilled ore sample of 2,5 kg,

(ii) the measurement error derived from measuring the dry masses of each of the flotation test products,

(iii) the analytical error accruing from the determination of PGE grade in each flotation test product.

Initially the means and standard deviations of the set BH_j and $RPGE_j$ were calculated, using the formulae set out in Appendix 1. The standard errors, s_ϵ, of the means, were also calculated as

$$s_\epsilon = s/\sqrt{n} \qquad (66)$$

where

s = the sample standard deviation

n = the number of observations.

Thereafter the Sichel t estimator was used to calculate the means and confidence limits for a skewed distribution (§ Appendix 1, Section 3).

The 95% confidence limits of reconciled head grade and PGE recovery are thus given as

Arithmetical Basis

Table 2.1
Nomenclature of Arithmetical Estimates for the Mean and 95% Confidence Limits of 30 Replicate Flotation Tests

Parameter	Reconciled Head Grade g/t PGE	PGE Recovery %
Mean	BH	RPGE
Standard Deviation	s_{BH}	s_{RPGE}
Standard Error	$s_{\epsilon}(BH)$	$s_{\epsilon}(RPGE)$
95% Confidence Limits:		
of Mean	$BH \pm 2s_{\epsilon}(BH)$	$RPGE \pm 2s_{\epsilon}(RPGE)$
of Data	$BH \pm 2s_{BH}$	$RPGE \pm 2s_{RPGE}$

Skewed Basis

Table 2.2
Nomenclature of Geometric Estimates for the Mean and 95% Confidence Limits of 30 Replicate Flotation Tests

Parameter	Reconciled Head Grade g/t PGE	PGE Recovery %
Mean Log	t_{BH}	t_{RPGE}
Variance	V_{BH}	V_{RPGE}
95% Confidence Limits of Mean		
Upper	$UCL_{95}(BH)$	$UCL_{95}(RPGE)$
Lower	$UCL_{95}(BH)$	$UCL_{95}(RPGE)$

The 95% confidence limits of the skewed data (equivalent to the $BH \pm 2s_{\epsilon}$) will be investigated with the data analysis.

2.8 Construction of the Mean and 95% Confidence Limits in Assayed Head

Starting from the Null Hypothesis that the state of grind in an assay head sample has no affect on the estimation of the mean grade, all replicate determinations of grade at 40, 50, 60 and 70% passing 75 microns may be pooled, giving a robust data set of 37 x 4 = 148 mean determinations. The fact that each mean determination has up to 4 replicates within that mean infers that we have 148 x 4 = 592 observations of the assayed head grade.

Initially the 148 determinations, then the 592 at analytical replicate level, will undergo the same calculations as shown in Tables 2.1 and 2.2. This will generate the estimated mean and 95% confidence limits of the assayed head grade.

The calculation will then simplify into the subsets at known grinds, using the 37 assayed means and the 148 analytical replicates to repeat the estimation of the mean and 95% confidence limits of assayed head grade, again using the calculations indicated in Tables 2.1. and 2.2.

The Null Hypothesis will then be tested on these data to determine whether state of grind should be known or is an influential factor in determining assayed head grade.

In both cases of assayed and reconciled head grade, the number of determinations necessary to arrive at a 95% confident estimate of each will be calculated using the t-distribution as follows:

$$t = (|X - \bar{x}|/s)*\sqrt{n} \qquad (67)$$

with

X = the robust estimate of mean grade
$\bar{x}$ = the estimate of mean grade from the small data set
s = the robust estimate of the standard deviation
n = the minimum number of determinations needed to arrive at a 95% confident estimate of X.

2.9 Testing for Minimum Replicates Using Semivariograms

For both the assayed head and reconciled head data, the semivariogram will be used as a possible form of determining the minimum number of replicates required for each to arrive at a reliable estimate of the mean grade. Here it is assumed that there will be a short-term relationship between replicates, and that an estimate of the nugget effect C_o may be made.

The method of calculation is set out in Appendix 1, Section 4.

3. RESULTS

3.1 PREPARATION OF MILL FEED SAMPLE

3.1.1 Measurement and Control of Particle Size Distribution

3.1.1:a Initial Grading Tests on the Natural Standard Sample (Refer § 2.1)

The initial four grading tests on the natural standard tailings sample are shown in Table 3.1.

Table 3.1
Natural Standard Sample
Waterval Final Tailings

Size, microns		Replicate Number				Mean
		1	2	3	4	
+	-	% Fractional Mass				A
425		0	0	0,1	0,1	0,05
300	425	0,3	0,4	0,3	0,3	0,33
212	300	1,6	1,5	1,6	1,6	1,58
150	212	4,9	5,1	4,5	4,7	4,8
106	150	12,0	12,0	11,8	11,9	11,88
75	106	14,5	14,3	15,4	15,3	14,88
53	75	15,7	15,2	15,1	15,3	15,33
38	53	8,5	8,5	9,2	9,2	8,85
0	38	42,1	43,0	42,0	41,6	42,17

Use of Allen's minimum sample mass formula for gradings now finds M_s for the fractions indicated:

$S_s = 3,1\ t/m^3$ $\qquad\qquad$ $\theta = 0,05$

w_i = Per Column A /100

$$M_s = 6,2 \times 10^5\ ((1/w_i)-2)((d_u^3 + d_l^3)/2)$$

The following critical masses are indicated at the 5% error level, assuming that the bound size shown is the "topsize" of the distribution:

Size, microns		M_s
+	-	
d_l	d_u	g
300	425	9863
212	300	694
150	212	75,3
106	150	9,1
75	106	2,4
53	75	0,8
38	53	0,6

The catchweight of ca. 200 g for grading tests therefore meets with the requirements of sizes finer than 212 microns. From Table 3.1 this covers more than 95% of the size distribution, however infers that a size-related grading error function will develop in the database, with coarser sizes showing higher RSD between replicates tha finer sizes.

3.1.1:b Development of the Database Reference Sample

The database developed totalled 87 replicate gradings at the laboratory, with a further 40 independent checks done at the chemistry laboratory. Refer to Appendix 2. Initially these blocks of data were kept separate to check that they were similar enough to pool as a control. The VFD of this natural standard is shown in Table 3.2, and is based on cumulative % mass passing 75 microns.

Table 3.2
Value Frequency Distribution
Reference Grading Sample

Grading Group % -75 microns	Freq. % DML	Freq. Cum. % DML	Freq. % PFR Lab	Freq. Cum. % PFR Lab
60	0	0	0	0
60,5	0	0	0	0
61	0	0	0	0
61,5	0	0	0	0
62	0	0	0	0
62,5	1,15	1,15	0	0
63	0	1,15	0	0
63,5	0	1,15	0	0
64	0	1,15	2,5	2,5
64,5	3,45	4,60	7,5	10,0
65	11,49	16,09	7,5	17,5
65,5	10,34	26,43	15,0	32,5
66	24,14	50,57	22,5	55,0
66,5	25,29	75,86	20,0	75,0
67	19,54	95,4	5,0	80,0
67,5	1,15	96,55	10,0	90,0
68	2,30	98,85	10,0	100,0
68,5	0	98,85	0	100,0
69	0	98,95	0	100,0
69,5	1,15	100,00	0	100,0

Statistics (Sample Data)		
Observations	87	40
Mean % - 75 microns:	65,99	65,98
Standard Deviation % -75 microns	0,92	1,00
RSD %	1,40	1,52
Skewness	-0,24	+0,15
Kurtosis	7,84	2,41

The VFDs for these data are shown in Figure 3.1.

The variance ratio test is first performed to test the likeness of the two sample sets for comparable scatter:

$F = (1,00)^2/(0,92)^2 = 1,18$

For (40-1) and (87-1) degrees of freedom repectively, at the 95% confidence level $F_c = 1,55$, therefore the variances are comparable enough to warrant comparison of means [Box et. al., 1978b].

The likeness of the two mean values is tested using a pooled estimate of variance (PEV) to formulate an overall standard error, thus calculate t as the ratio of the difference in means to the standard error:

$PEV = ((n_1-1)(s_1^2)+(n_2-1)(s_2^2))/(n_1+n_2-2)$

Values of Variables used

n_1 : 87; n_2 : 40 $\qquad$ s_1^2: 0,846; s_2^2: 1,00

PEV = 0,894

The variance of the difference of the means is now

$s^2_{y1-y2} = 0,894((1/87)+(1/40))^{0,5} = 0,170$

$t = (65,99-65,98)/0,170 = 0,09$

Referring to tables of the t-distribution t_c(125 df;95% confidence level) is approximately 2,00, therefore it may be concluded that the mean values of replicate gradings between the two laboratories are similar.

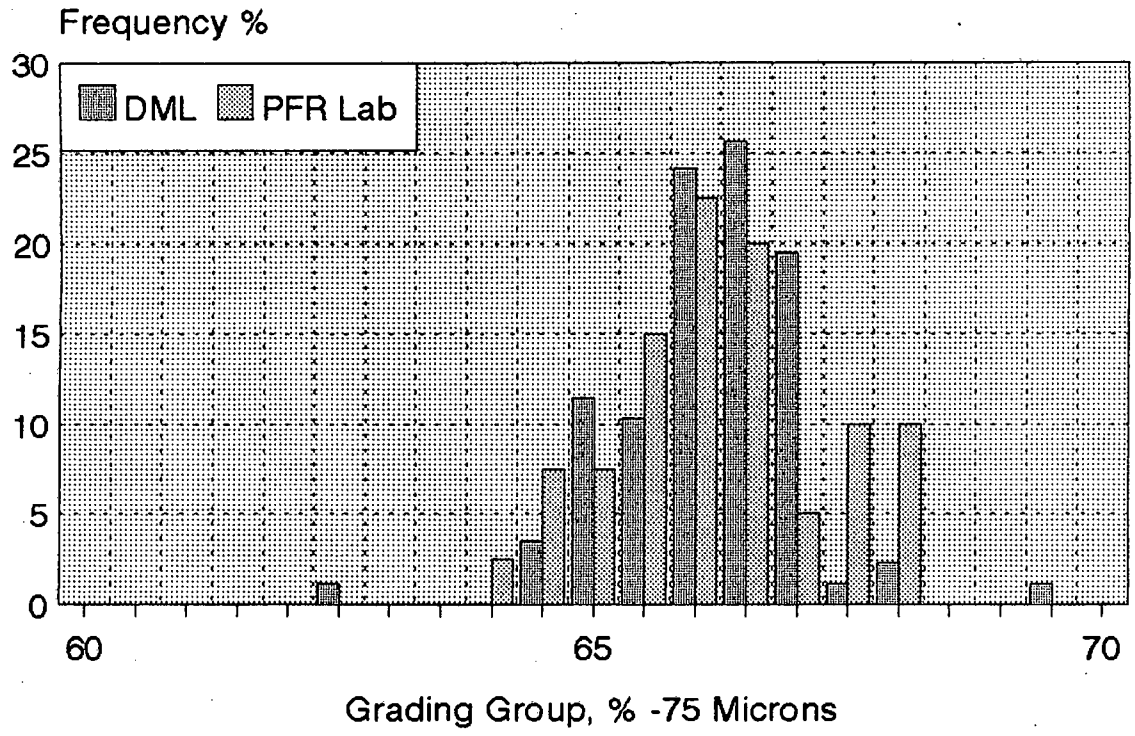


Figure 3.1 : Reference Grading Sample Value Frequency Distribution

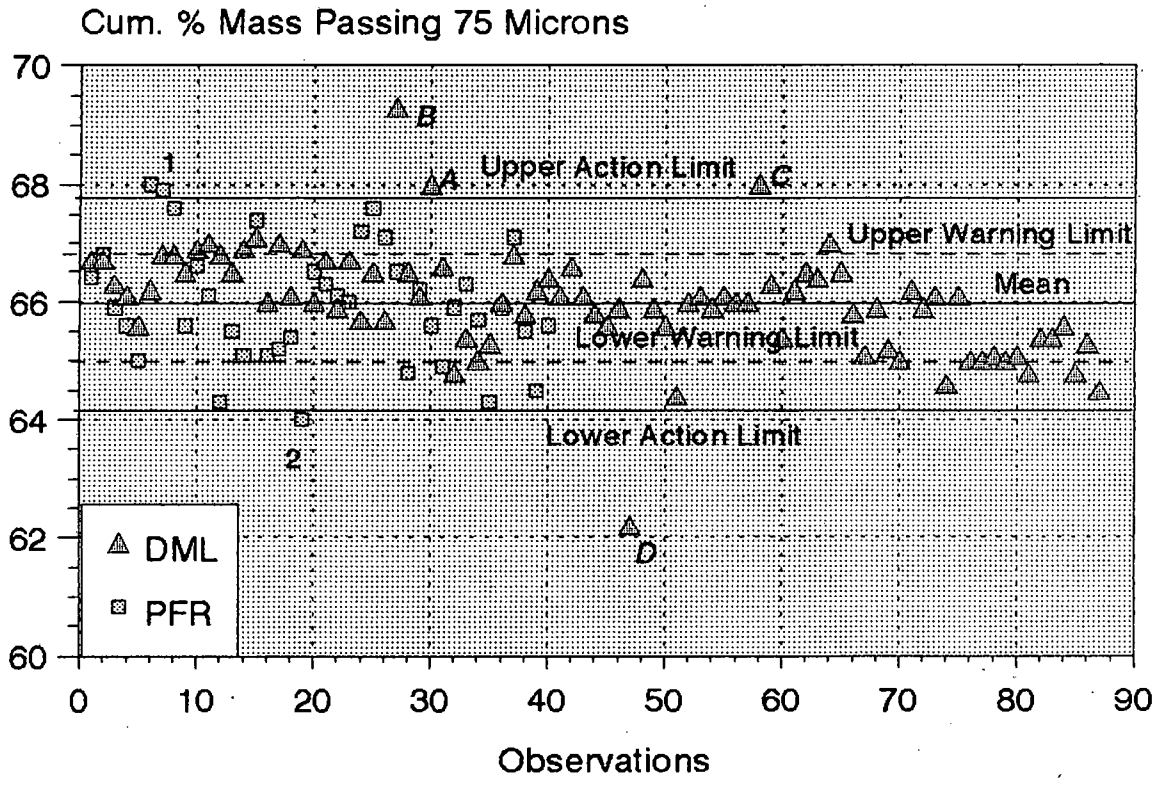


Figure 3.2 : Reference Grading Sample Control Chart

The appropriate control graph for this natural standard is shown in Figure 3.2. Warning limits are set at $\bar{X}\pm s$, action limits at $\bar{X}\pm 2s$, using the DML data set for these calculations. Inspection of this graph shows that for both laboratories the occurrence of "out of control" data is real. Examples are marked A-D for DML, and 1-2 for PFR Laboratory, however account for a small percentage of the overall data. Inspection of the means and standard deviations of the individual fractions in this reference sample yielded the following results in terms of scatter:

Table 3.3
Reference Grading Sample
Analysis of Size Fractions

Size Fraction +	-	Mean % Mass	Std Dev	RSD%	Std Error
425		0,03	0,048	192	0,005
300	425	0,274	0,062	22,6	0,007
212	300	1,617	0,063	3,9	0,007
150	212	4,703	0,265	5,63	0,028
106	150	11,264	0,495	4,39	0,053
75	106	15,771	0,860	5,45	0,092
53	75	15,885	1,515	9,53	0,162
38	53	8,62	1,154	13,4	0,123
0	38	41,42	0,99	2,39	0,106

The above data suggest that, in terms of RSD, the two coarsest sizes > 300 microns endure much higher scatter between replicate determinations than do the finer sizes. This is to be expected in terms of Allen's formula, which earlier indicated that several kilograms of sample per grading test would be necessary to obtain an error of 5%. If the normalised standard error is used per size fraction, viz. $(s/\sqrt{n})*100/\bar{x}$ per size fraction, a closer measure of Allen's error will be obtained. This is shown in Table 3.4.

Table 3.4
Reference Grading Sample
Normalised Standard Error per Size

Size Fraction +	-	Std Error	Rel.Std Error %	Size Fraction +	-	Std Error	Rel.Std Error%
425		0,005	16,7	75	106	0,092	0,6
300	425	0,007	2,6	53	75	0,162	1,0
212	300	0,007	0,4	38	53	0,123	1,4
150	212	0,028	0,6	0	38	0,100	0,3
106	150	0,053	0,5				

The above calculations show that errors of < 5% were attained in all sizes finer than the topsize 425 microns. A further examination of this theme in terms of cumulative % mass data, which is in keeping with production management vocabulary, derived equivalent data for two typically quoted sizes, 75 and 150 microns. These results are shown in Table 3.5:

Table 3.5
Mean, Scatter and Errors in Cumulative Sizes

Size: Microns		Mean Cum. %	Std. Dev.	Std. Error	RSD %	Relative Std. Error %
+	-					
150		6,59	0,29	0,031	4,3	0,47
	75	65,99	0,92	0,098	1,4	0,15

Inspection of these data indicates that the normalised standard error is lower for cumulative data than for the average sizes constituting that cumulative size group. In particular a figure of less than 5% error is noted. The Rosin-Rammler size distribution model for this grading reference sample is drawn in Figure 3.3, and indicates that good linearity is attainable for these data.

3.1.1:c Conclusions : Particle Size Distribution

It is concluded from this exercise that quantitative control is possible for grading data, for the following reasons:

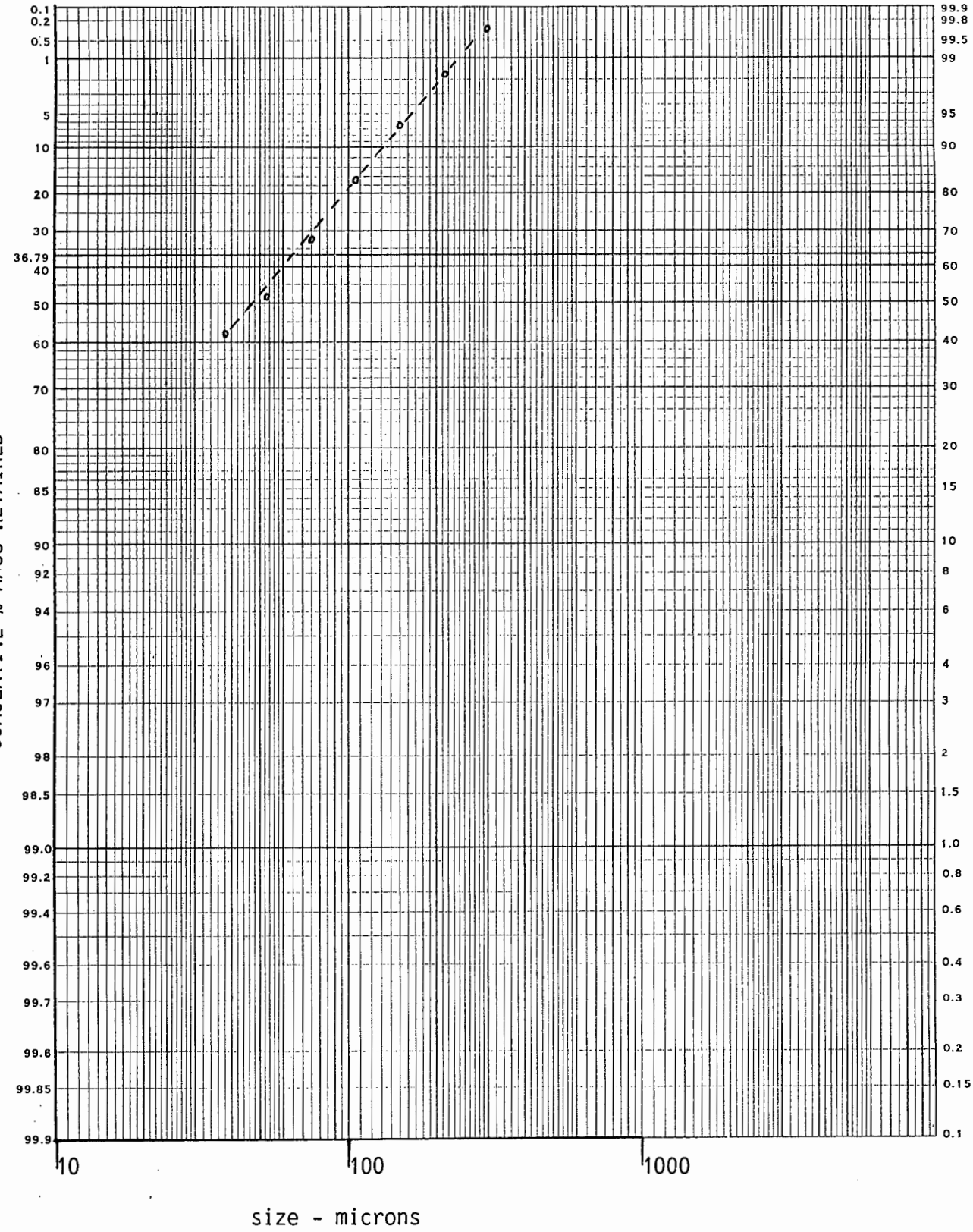
(i) The precision of the grading test may be checked against a database of replicate determinations. 95% confidence limits can be calculated therefrom and test-by-test measurement on the control graph performed to attest to the credibility of a particular result. Observations outside the action limits were found, indicating that the two types of sieving errors (§ 2.1) do exist and are identifiable. These are sieve blockage and sieve rim welding failure repectively.

(ii) Independent measurement of subsamples from this reference sample by another laboratory arrived at a similar result.

(iii) Catchweight test samples in the order of 200 g provide acceptable accuracy in the critical sizes. The topsize group, which account for less than 5% of the sample mass, incur higher errors, consistent with Allen's formula.

Figure 3.3
Reference grading sample : Plot of cumulative % mass retained by size on Rosin-Rammler model format

n : 1,17
k : 67,2
r^2 : 0,99



(iv) When cumulative % mass is described in terms of passing 75 and retained on 150 micron sizes respectively, the normalised errors are < 5%.

3.1.2 Chip Sampling of Merensky Ore, and Test for Lognormality

In order to gain an understanding of the evaluation characteristics of the Rustenburg Merensky Reef, data were collected from the routine chip sampling activities in underground mining for three shafts in Rustenburg Section of RPM. These were assembled into value frequency distribution models: refer Tables 3.6 - 3.8. Units of grade are 4E PGE, viz. the sum of Pt,Pd,Au and Rh, in grammes per ton. The distributions of PGE values per grade fraction are shown in the following Figures. The grade groups are indexed but proportional, starting from lowest grade. For example, grade group 10 is twice the grade of grade group 5:

Figure	Description	
3.4	Frank Shaft	Footwall
3.5	Frank Shaft	Reef
3.6	Frank Shaft	Hanging Wall
3.7	Townlands Shaft	Footwall
3.8	Townlands Shaft	Reef
3.9	Townlands Shaft	Hanging Wall
3.10	Turffontein Shaft	Footwall
3.11	Turffontein Shaft	Reef
3.12	Turffontein Shaft	Hanging Wall

The cumulative frequency probability plots for these data are shown in the following Figures:

Figure	Description
3.13	Frank Shaft
3.14	Townlands Shaft
3.15	Turffontein Shaft

Inspection of these graphs shows the following features:

3.1.2:a Frank Shaft (Refer Figure 3.13)

All three fractions of the stope cut demonstrate lognormal distribution, however in two sections for each, as if there is a bimodal distribution. The points of inflection A, B and C for hanging wall, reef and footwall respectively indicate the loci of these inflections. It is suggested that the above phenomenon is due to the bimodal deportment of PGM+Au values in solid solution and as discrete PG minerals. The base metal sulphides carry low grades of PGE in the order of grams per tonne [Kinloch, 1982] whilst the other major carriers of PGM+Au are minerals such as ferroplatinum, cooperite, braggite etc., and represent grades in percentages (§ 2.5).

Table 3.6
Value Frequency Distribution Data
Rustenburg Section Merensky Ore
Frank Shaft

1. 4E: Sum of Pt,Pd,Au,Rh: Relative Grades are Proportional

Rel. Grd.PGE (4E)[1]	Footwall		Reef		Hanging Wall	
	Freq %	Freq Cum%	Freq %	Freq Cum%	Freq %	Freq Cum%
1	0	0	0	0	0	0
2	52,01	52,01	2,05	2,05	32,03	32,03
3	11,46	63,47	4,68	6,73	6,54	38,57
4	10,22	73,69	12,28	19,01	9,15	47,72
5	6,50	80,19	9,06	28,07	7,84	55,56
6	4,33	84,52	5,56	33,63	7,84	63,40
7	5,88	90,40	7,60	41,23	5,23	68,63
8	2,79	93,19	6,73	47,96	4,58	73,21
9	2,48	95,67	2,05	50,01	5,88	79,09
10	1,86	97,53	4,68	54,69	4,58	83,67
11	0,93	98,46	4,09	58,78	2,61	86,28
12	0,31	98,77	4,09	62,87	1,31	87,59
13	0,31	99,08	1,46	64,33	1,96	89,55
14	0,93	100,0	2,34	66,67	0,65	90,20
15	0		2,05	68,72	2,61	92,81
16			2,34	71,06	1,31	94,12
17			2,34	73,4	0	94,12
18			3,22	76,62	1,31	95,43
19			2,05	78,67	0,65	96,08
20			2,92	81,59	0	96,08
21			3,51	85,1	0,65	96,73
22			2,05	87,15	0,65	97,38
23			2,05	89,20	0	97,38
24			2,05	91,25	0	97,38
25			0,58	91,83	0	97,38
26			1,75	93,58	0,65	98,03
27			0,58	94,16	0	98,03
28			1,17	95,33	0	98,03
29			1,46	96,79	0	98,03
30			0,29	97,08	0	98,03
31			0,88	97,96	0	98,03
32			0,29	98,25	0	98,03
33			0,29	98,54	0	98,03
34			0	98,54	0	98,03
35			0	98,54	0	98,03
36			0,58	99,12	0	98,03
37			0	99,12	0	98,03
38			0	99,12	0	98,03
39			0,58	99,17	0	98,03
40			0	99,17	0,65	98,68
41			0	99,17	0,65	99,33
42			0	99,17	0,65	99,98
43			0	99,17		
44			0	99,17		
45			0,29	99,99		

Table 3.7
Value Frequency Distribution Data
Rustenburg Section Merensky Ore
Townlands Shaft

1. 4E: Sum of Pt,Pd,Au,Rh: Relative Grades are Proportional

Rel. Grd.PGE (4E)[1]	Footwall Freq %	Footwall Freq Cum%	Reef Freq %	Reef Freq Cum%	Hanging Wall Freq %	Hanging Wall Freq% Cum%
1	0	0	0	0	0	0
2	53,25	53,25	2,68	2,68	62,26	62,26
3	5,88	59,13	0	2,68	5,66	67,92
4	4,95	64,08	2,01	4,69	9,43	77,35
5	4,33	68,41	2,01	6,70	1,89	79,24
6	6,19	74,60	0,67	7,37	2,83	82,07
7	2,79	77,39	2,68	10,05	2,83	84,90
8	4,95	83,34	2,68	12,73	4,72	89,62
9	3,41	85,75	4,03	16,76	0	89,62
10	1,24	86,99	5,37	22,13	0	89,62
11	2,79	89,78	5,37	27,50	2,83	92,45
12	2,17	91,95	4,70	32,20	0	92,45
13	1,24	93,19	5,37	37,57	2,83	95,28
14	1,55	94,74	4,70	42,27	0,94	96,22
15	0,93	95,67	4,03	46,30	1,89	98,11
16	1,24	96,91	4,70	51,00	0,94	99,05
17	1,24	98,15	2,01	53,01	0	99,05
18	0	98,15	3,36	56,37	0	99,05
19	0,31	98,46	4,03	60,40	0	99,05
20	0	98,46	5,37	65,77	0	99,05
21	0,62	99,08	2,68	68,45	0	99,05
22	0	99,08	2,68	71,13	0,94	99,99
23	0,31	99,39	4,03	75,16	0	97,38
24	0	99,39	2,01	77,17	0	97,38
25	0	99,39	2,01	79,18	0	97,38
26	0,31	99,70	1,34	80,52	0	98,03
27	0	99,70	1,34	81,86	0	98,03
28	0	99,70	2,68	84,54	0	98,03
29	0	99,70	1,34	85,88	0	98,03
30	0	99.70	0,67	86,55	0	98,03
31	0	99,70	0,67	87,22	0	98,03
32	0,31	100,01	0,67	87,89	0	98,03
33			0	87,89	0	98,03
34			0,67	88,56	0	98,03
35			1,34	89,90	0	98,03
36			0,67	90,57	0	98,03
37			0,67	91,24	0	98,03
38			0,67	91,91	0	98,03
39			1,34	93,25	0	98,03
40			0	93,25	0,65	98,68
41			0	93,25	0,65	99,33
42			1,34	94,59	0,65	99,98
43			2,68	97,27		
44			0	97,27		
45			0	97,27		

Table 3.8
Value Frequency Distribution Data
Rustenburg Section Merensky Ore
Turffontein Shaft

1. 4E: Sum of Pt,Pd,Au,Rh: Relative Grades are Proportional

Rel. Grd.PGE (4E)[1]	Footwall Freq %	Footwall Freq Cum%	Reef Freq %	Reef Freq Cum%	Hanging Wall Freq %	Hanging Wall Freq% Cum%
1	0	0	0	0	0	0
2	64,17	64,17	4,55	4,55	48,36	48,36
3	5,83	70,00	0	4,55	6,56	54,92
4	4,17	74,17	0,65	5,20	2,87	57,79
5	2,08	76,25	3,25	8,45	4,92	62,71
6	4,58	80,83	6,49	14,94	9,02	71,73
7	3,33	84,16	2,60	17,63	4,51	76,24
8	1,67	85,83	1,95	19,58	5,74	81,98
9	2,92	88,75	2,60	22,18	5,74	87,72
10	2,08	90,83	3,25	25,43	3,69	91,41
11	2,08	92,91	3,25	28,68	2,46	93,87
12	1,25	94,16	2,60	31,28	1,64	95,51
13	1,25	95,41	4,55	35,83	2,46	97,97
14	1,25	96,66	0,65	36,48	0	97,97
15	0	96,66	1,30	37,78	0	97,97
16	0	96,66	2,60	40,38	0,82	98,79
17	0,42	97,08	3,90	44,28	0,41	99,20
18	0	97,08	1,30	45,58	0	99,20
19	0	97,08	5,19	50,77	0	99,20
20	0,42	97,50	3,90	54,67	0	99,20
21	0	97,50	3,25	57,92	0	99,20
22	0,83	98,33	5,19	63,11	0,41	99,61
23	0,42	98,75	1,95	65,06	0	99,61
24	0	98,75	1,95	67,01	0	99,61
25	0,42	99,17	4,55	71,56	0	99,61
26	0,42	99,59	1,30	72,86	0	99,61
27	0	99,59	1,95	74,81	0	99,61
28	0	99,59	3,25	78,06	0	99,61
29	0	99,59	2,60	80,66	0	99,61
30	0	99,59	2,60	83,26	0	99,61
31	0,42	100,0	0,65	83,91	0	99,61
32	0	100,01	0,65	84,56	0	99,61
33	0		0	84,56	0	99,61
34	0		2,60	87,16	0,41	100,0
35	0		2,60	89,76		
36	0		0	89,76		
37	0		0	89,76		
38	0		0	89,76		
39	0		1,30	91,06		
40	0		1,30	92,36		
41	0		1,30	93,66		
42	0		1,95	95,61		
43	0		0,65	96,26		
44	0		0	96,26		
45	0		0	96,26		

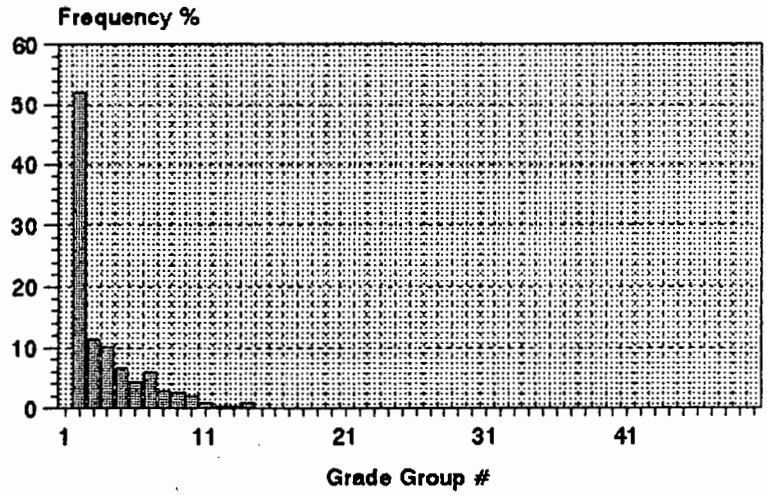


Figure 3.4 : Frank Footwall Value Frequency Distribution

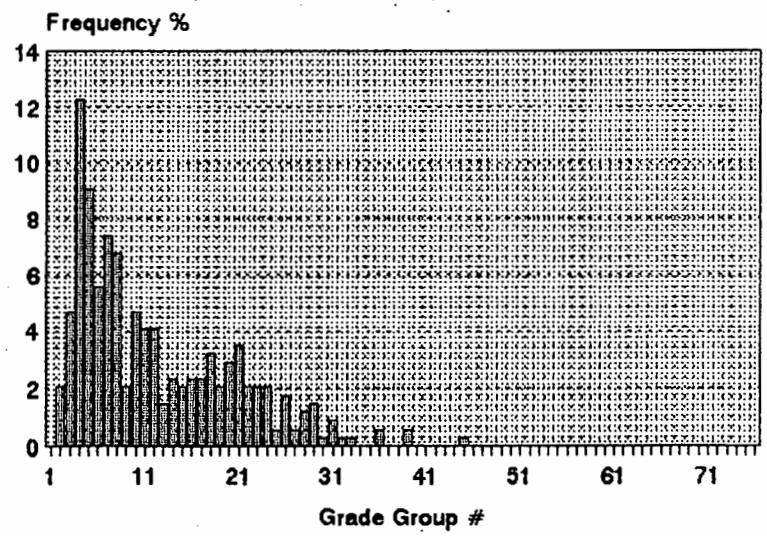


Figure 3.5 : Frank Reef Value Frequency Distribution

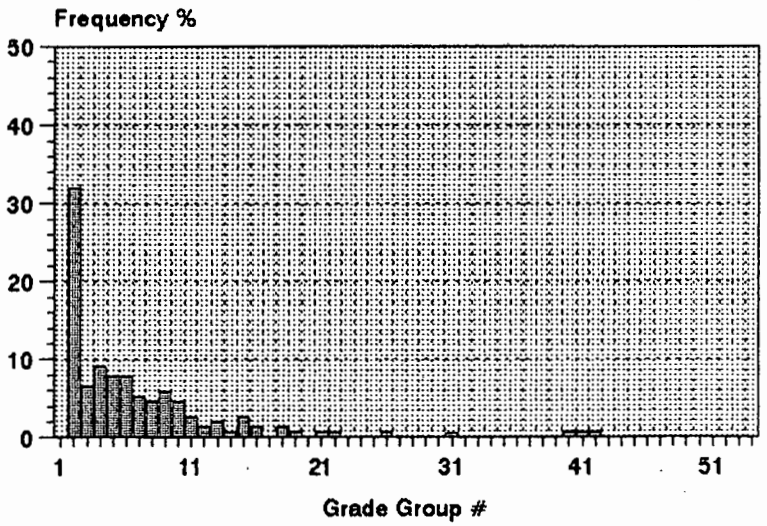


Figure 3.6 : Frank Hanging Wall Value Frequency Distribution

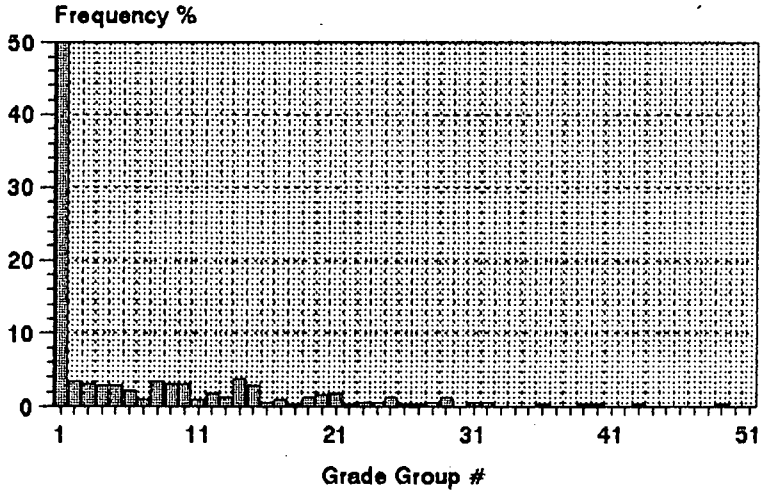


Figure 3.7: Townlands Footwall Value Frequency Distribution

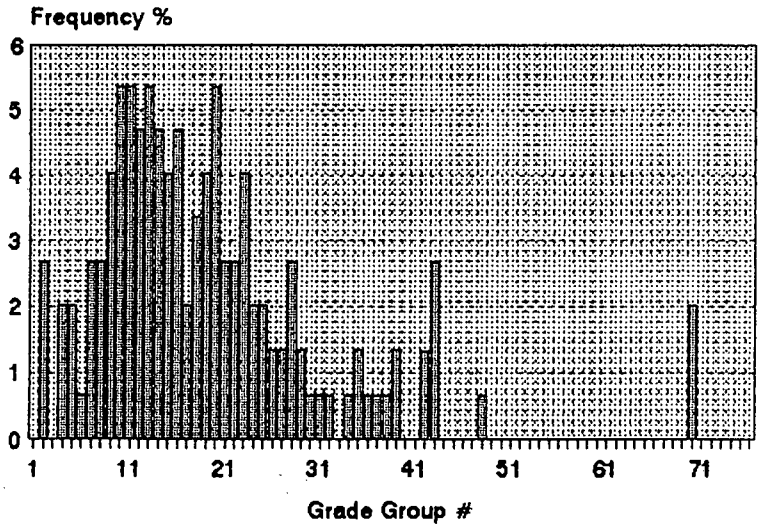


Figure 3.8 : Townlands Reef Value Frequency Distribution

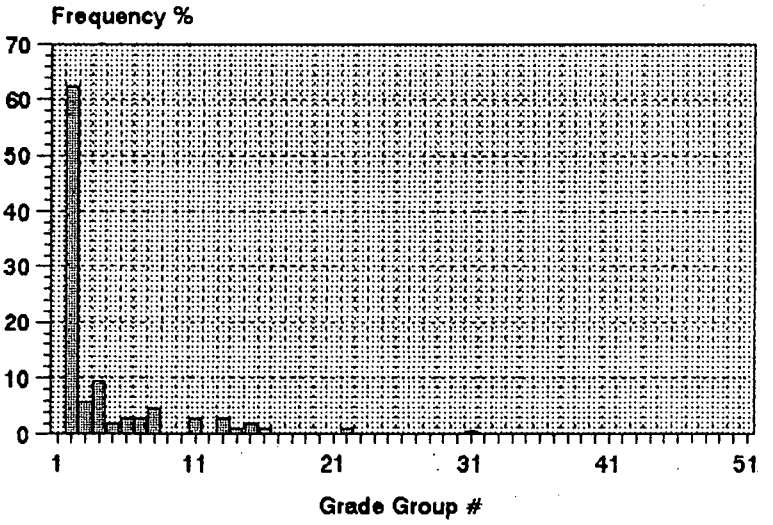


Figure 3.9 : Townlands Hanging Wall Value Frequency Distribution

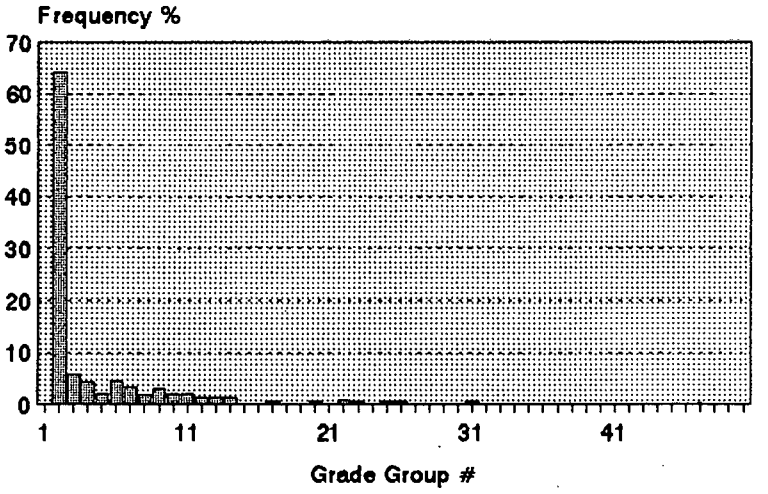


Figure 3.10 : Turffontein Footwall Value Frequency Distribution

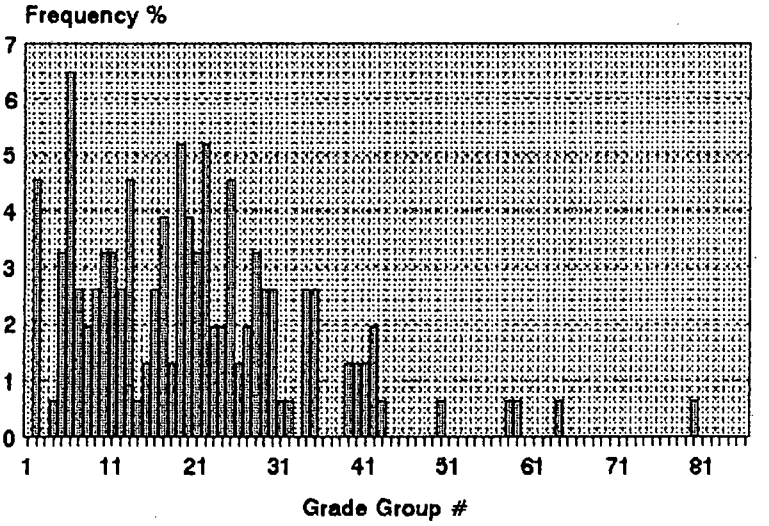


Figure 3.11 : Turffontein Reef Value Frequency Distribution

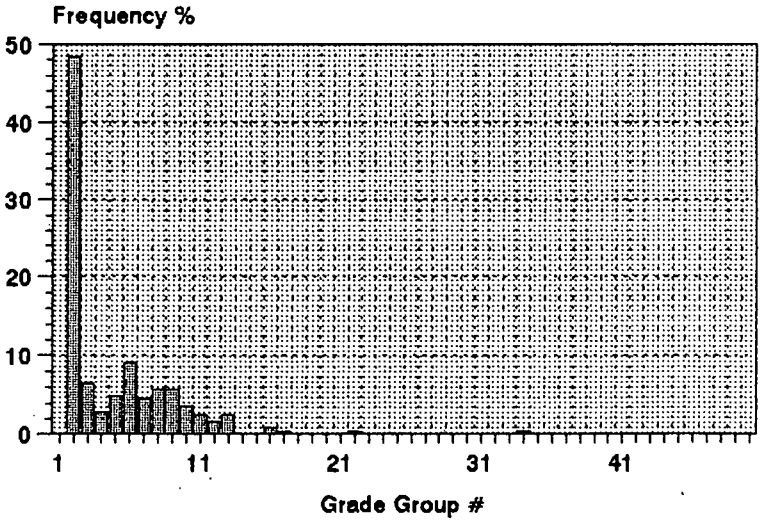


Figure 3.12 : Turffontein Hanging Wall Value Frequency Distribution

Thus the values to the right of A,B or C probably represent the metallics-associated PGM, whilst those to the left, those associated with BMS. Such an interpretation was made for other ores by Clark [Clark, 1974] where the multiple mineralisation phases carried different grades of metal.

Another feature of Figure 3.13 is the apparent overstatement of frequency observed for the lowest grade in the distributions of PGM values for hanging wall and footwall. The modelled frequencies are extrapolated to D and E repectively. Table 3.9 summarises the initial statistics of these observations.

Table 3.9
Frank Shaft
Interpretation of VFD Data
from Logarithmic Probability Plot

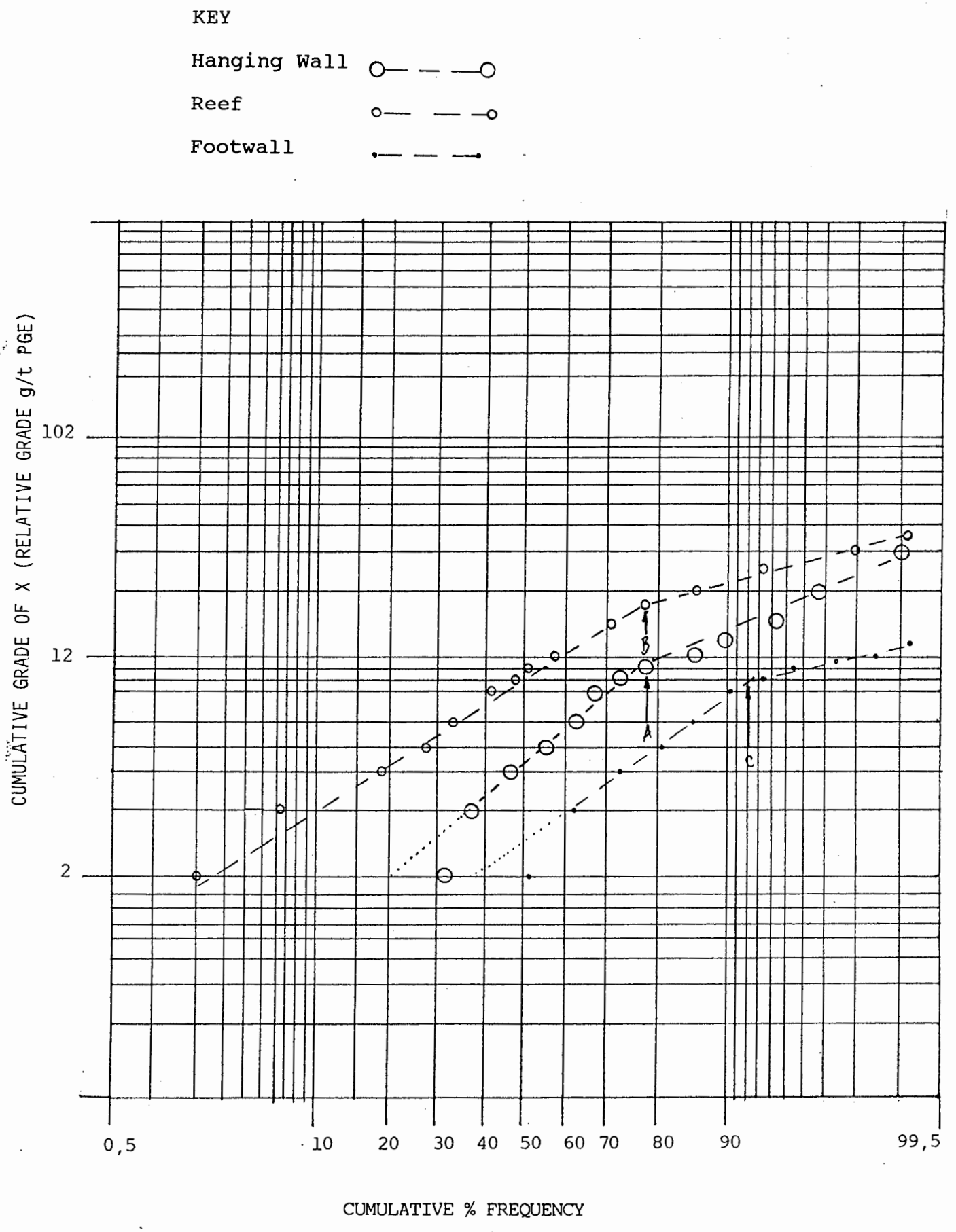
Detail	Hanging Wall	Reef	Footwall
Mean Grade gpt PGM+Au	0,504	1,000	0,210
RSD %	127,6	79,4	79,4

Table 3.10 subdivides the above basic data into the indicated categories.

Table 3.10
Frank Shaft
Use of Logarithmic Probability
Plot to Subdivide Values

Detail	Hanging Wall	Reef	Foot Wall
Point of Inflection			
Cum Fi %	A	B	C
	79,09	78,67	92,0
Inferred %			
Above Inflection	20,91	21,33	8,0
Inferred Metallics Recoverable Grade gpt PGM+Au	0,105	0,22	0,02
Remainder of PGM+Au gpt	0,399	0,78	0,19
Overstatement of Low Grade Section Cum Fi%	12,3	0	14,01

Figure 3.13
Logarithmic Probability Plot
Frank Shaft



It is debatable whether the overstatement of frequencies at the low grade end of the hanging wall and footwall distributions can be reliably interpreted as unrecoverable PGE; rather it is more likely that the overstated percentage is due to waste rock above or below the stope being sampled as ore.

3.1.2:b Townlands Shaft (Refer Figure 3.14)

The points of inflection A,B and C in hanging wall, reef and footwall for Townlands Shaft are shown in Figure 3.14.

These reflect bimodal distributions as for Frank Shaft, however the loci are different. Table 3.11 summarises the initial statistics of these data.

Table 3.11
Townlands Shaft
Interpretation of VFD Data
from Logarithmic Probability Plot

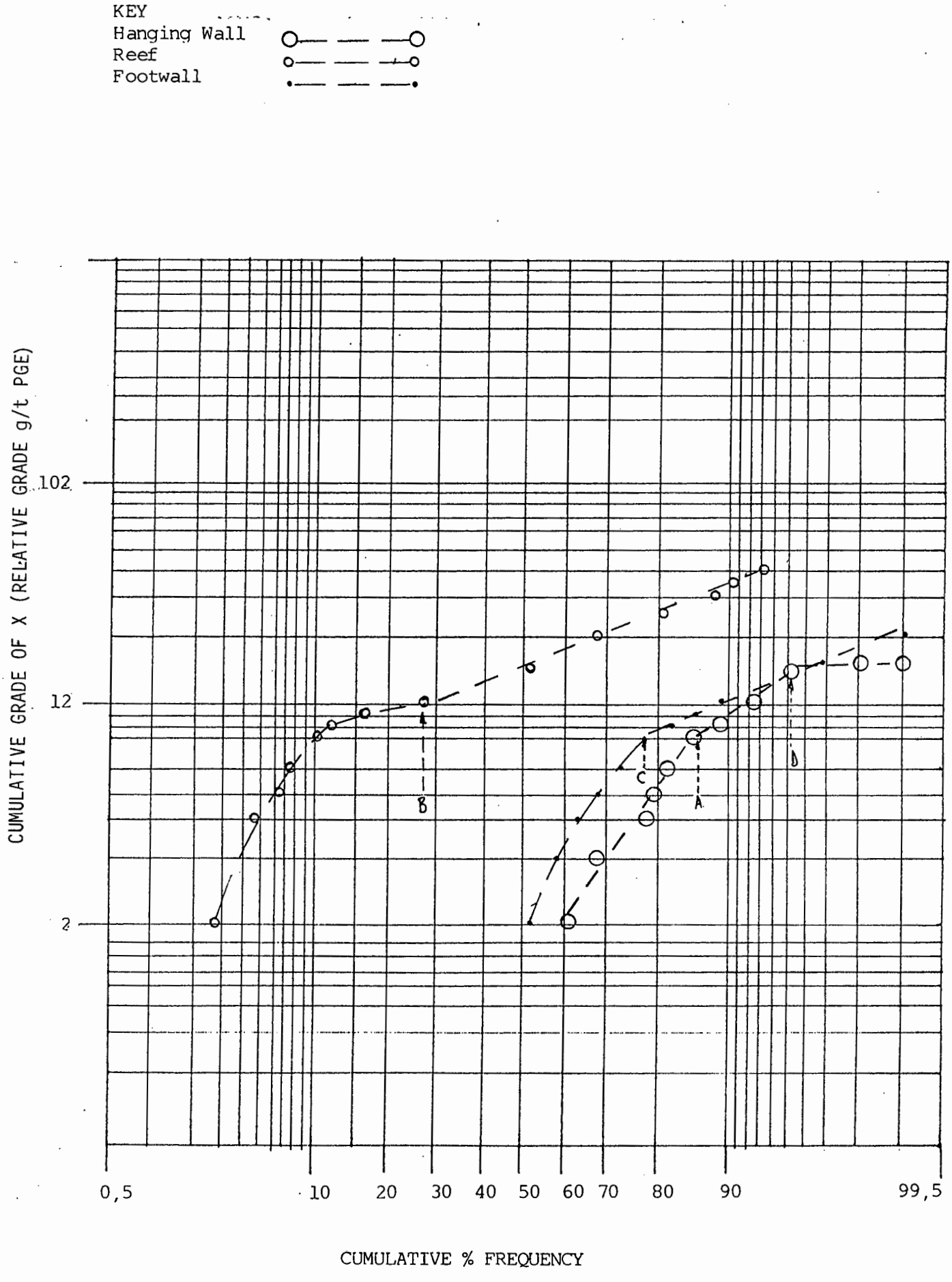
Detail	Hanging Wall	Reef	Foot Wall
Mean Grade gpt PGM+Au	0,146	1,000	0,197
RSD%	150,5	69,39	13,28

Table 3.12 subdivides the above basic data into the indicated categories.

Table 3.12
Townlands Shaft
Use of Logarithmic Probability
Plot to Subdivide Values

Detail	Hanging Wall	Reef	Foot Wall
Point of Inflection	A	B	C
CumFi%	84,90	27,50	77,3
Inferred % Above Inflection	15,10	72,50	22,6
Inferred Metallics			
Recoverable Grade gpt PGE	0,022	0,72	0,16
Remainder of PGM+Au gpt	0,124	0,28	0,16
Overstatement of			
Low Grade Section Cum Fi %	0,00	0,88	8,25

Figure 3.14
Logarithmic Probability Plot
Townlands Shaft



3.1.2:c **Turffontein Shaft** (Refer Figure 3.15)

The points of inflection A, B and C in hanging wall, reef and footwall for Turffontein Shaft are shown in Figure 3.15, reflecting bimodal distributions as for Frank Shaft, however the loci are different. Table 3.13 summarises the initial statistics of these data.

Table 3.13
Turffontein Shaft
Interpretation of VFD Data
from Logarithmic Probability Plot

Detail	Hanging Wall	Reef	Foot Wall
Mean Grade			
g/t PGE	0,179	1,000	0,146
RSD%	118,4	73,9	164,7

Table 3.14 subdivides the above basic data into the indicated categories.

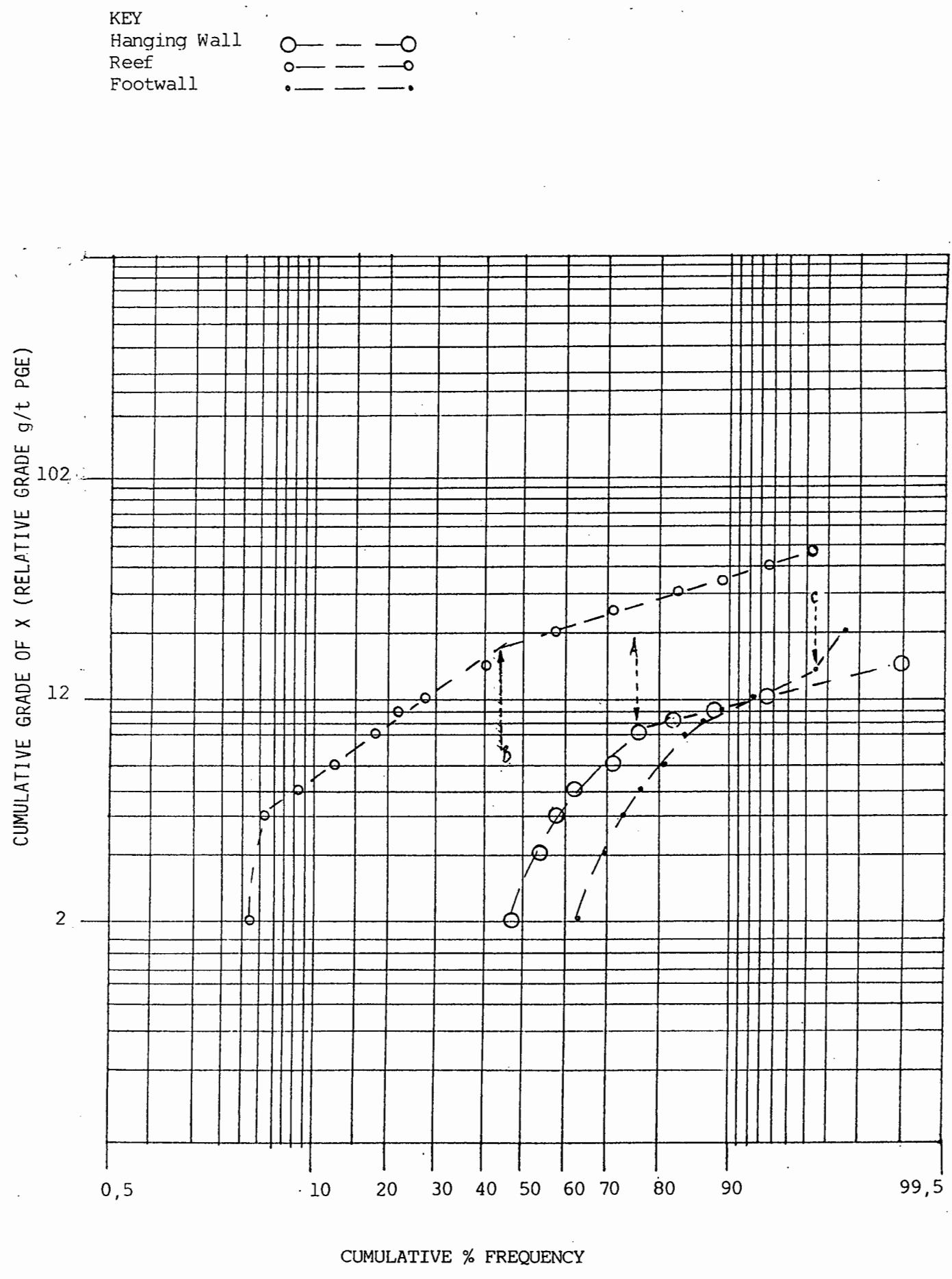
Table 3.14
Turffontein Shaft
Use of Logarithmic Probability
Plot to Subdivide Values

Detail	Hanging Wall	Reef	Foot Wall
Point of Inflection	A	B	C
Cum Fi %	76,24	44,00	96,66
Inferred %			
Above Inflection	23,76	56,00	3,34
Inferred Metallics			
Recoverable Grade			
gpt PGM+Au	0,043	0,56	0,005
Remainder of PGM+Au			
gpt	0,136	0,44	0,141
Overstatement of			
Low Grade Section			
Cum Fi %	10,36	4,05	0,0

3.1.2:d **Conclusions**

In conclusion, it is seen that the chip sampling in Rustenburg Merensky does not immediately follow the two- or three- parameter lognormal distribution model, but

Figure 3.15
Logarithmic Probability Plot
Turffontein Shaft



rather follows a bimodal distribution format, either two-parameter in the case of reef, or three-parameter, as in the cases of hanging wall and footwall. Further, the biomodal format of VFD diagnosed by the probability plots is an indicator of significantly different grade groups that probably correspond to solid solution PGM in base metal sulphides, and to discrete metallics minerals such as ferroplatinum, cooperite and braggite. Later technical tests will investigate the validity of this likelihood. It may be the case that this bimodal distribution characteristic has implications for the calculation of mean grade of sample; if proven this could affect the metal balance of the flotation tests to be conducted.

3.1.3 Value Frequency Distributions and Probability Plots of Mineral Process Feeds and Products

3.1.3:a Waterval Primary Circuit Feeds

The following primary circuit feeds were identified in § 2.3 for investigation:

(i) Conventionally crushed and screened ore for presentation as primary ball mill feed, with a typical d80 size of some 5mm,

(ii) Ultrafine Crusher Classifier Overflow (CCO), with a typical d80 size of 80 microns, for presentation directly to rougher flotation.

The value frequency distributions for these primary circuit inputs were gathered from 13 months of production data. These are summarised in the following Tables 3.15 and 3.16 for mill feed and CCO respectively. The VFD data are plotted for mill feed and CCO in Figures 3.16 and 3.17, and probability plots, in Figure 3.18. Units of grade are 4E, viz. the sum of Pt,Pd,Au and Rh, in grammes per ton. The grade groups are indexed but proportional, starting from the lowest grade. Inspection of these indicates a compound distribution effect in both products, with points of inflection marked as shown. The basic features of these data are summarised in Table 3.17.

A further feature of the probability plot in Figure 3.18 is a second main point of inflection at D, equivalent to 47,83 % cumulative frequency. This could demarcate the changeover from pentlandite-PGE to pyrrhotite-PGE; in which case the distribution of PGE between metallics, pentlandite and pyrrhotite will be 8,95:43,22:47,83% respectively. The second point of inflection in Figure 3.18 for mill feed is reflected in CCO at C, equivalent to 16,72 % cumulative frequency. This could indicate the distribution of PGE between metallics, pentlandite and pyrrhotite will be 22,09:61,19:16,72% respectively.

Table 3.15
Value Frequency Distribution Data
Waterval Mill Feed

1. 4E: Sum of Pt,Pd,Au,Rh: Relative Grades are Proportional

Rel. Grd. g/t 4E [1]	Frequency %	Cumulative Frequency %
1	0,26	0,26
2	0,51	0,77
3	0,77	1,53
4	1,79	3,32
5	3,32	6,65
6	3,32	9,97
7	5,88	15,86
8	5,88	21,74
9	7,93	29,67
10	12,02	41,69
11	6,14	47,83
12	9,21	57,03
13	8,95	65,98
14	6,65	72,63
15	7,16	79,80
16	6,14	85,93
17	5,12	91,05
18	3,84	94,88
19	1,79	96,68
20	0,51	97,19
21	0,77	97,95
22	0,51	98,47
23	0,51	98,98
24	0,26	99,23
25	0,51	99,74
26	0,0	99,74
27	0,0	99,74
28	0,0	99,74
29	0,0	99,74
30	0,26	100,00

Table 3.16
Value Frequency Distribution Data
Waterval CCO

1. 4E: Sum of Pt,Pd,Au,Rh: Relative Grades are Proportional

Rel. Grd. 4E g/t [1]	Freq %	Cum Freq %	Value Group g/t PGE	Freq %	Cum Freq %
18	0,30	0,30	50	5,07	64,18
19	0,0	0,30	51	4,78	68,96
20	0,30	0,60	52	4,78	73,73
21	0,0	0,60	53	4,18	77,91
22	0,30	0,90	54	4,48	82,39
23	0,0	0,90	55	1,19	83,58
24	0,60	1,49	56	3,28	86,67
25	0,90	2,39	57	2,39	89,25
26	0,60	2,99	58	1,19	90,45
27	0,0	2,99	59	1,19	91,64
28	0,0	2,99	60	0,90	92,54
29	0,30	3,28	61	0,60	93,13
30	0,60	3,88	62	0,60	93,73
31	1,19	5,07	63	0,90	94,63
32	0,30	5,37	64	0,90	95,52
33	1,19	6,57	65	0,90	96,42
34	0,60	7,16	66	0,0	96,42
35	2,09	9,25	67	0,90	97,31
36	1,79	11,04	68	0,0	97,31
37	2,69	13,73	69	0,90	98,21
38	2,99	16,72	70	0,0	98,21
39	1,79	18,51	71	0,30	98,51
40	4,18	22,69	72	0,30	98,81
41	3,58	26,27	73	0,60	99,40
42	2,69	28,96	74	0,0	99,40
43	3,28	32,24	75	0,0	99,40
44	2,69	34,93	76	0,0	99,40
45	4,18	39,10	77	0,0	99,40
46	4,48	43,58	78	0,0	99,40
47	4,48	48,06	79	0,30	99,70
48	5,97	54,03	80	0,30	100,0
49	5,07	59,10			

Table 3.17
Interpretation of Logarithmic Probability Plot
Waterval Primary Circuit Feeds

Detail	Mill Feed	CCO
Mean Grade g/t PGE	1,00	1,73
RSD%	8,92	11,46
Inflections	B	A
Cum. Fi%	91,05	77,9
Metallics Potential gpt	0,09	0,38

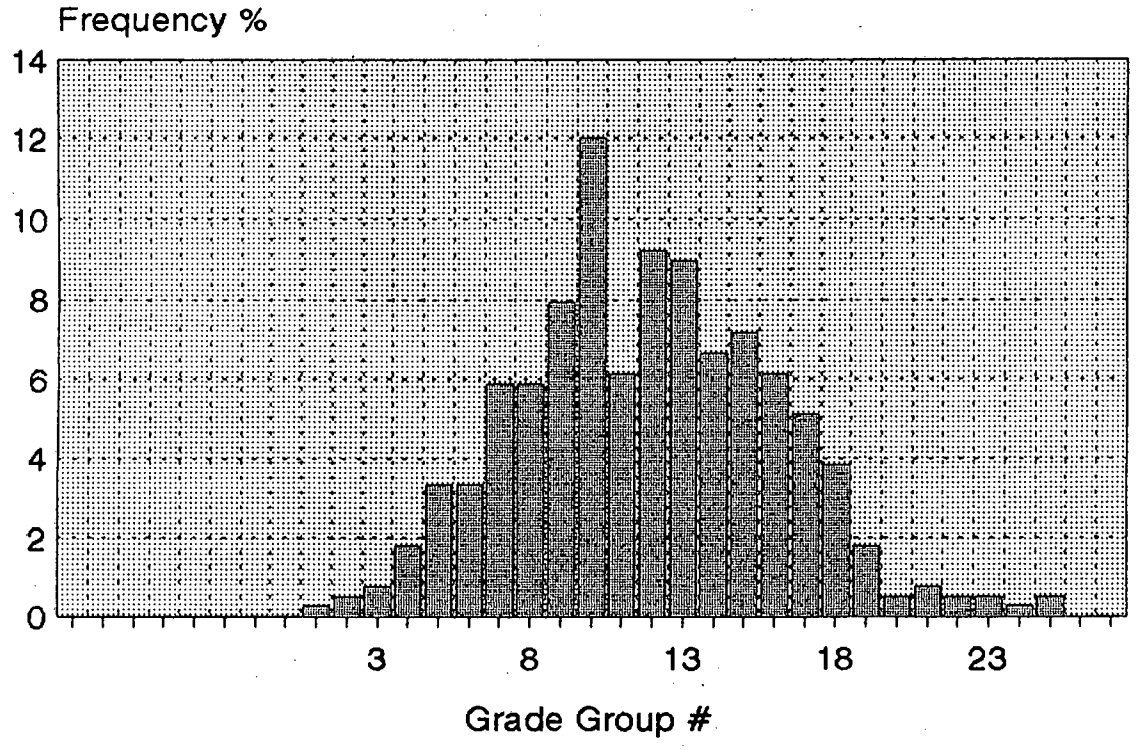


Figure 3.16: Waterval Mill Feed Value Frequency Distribution

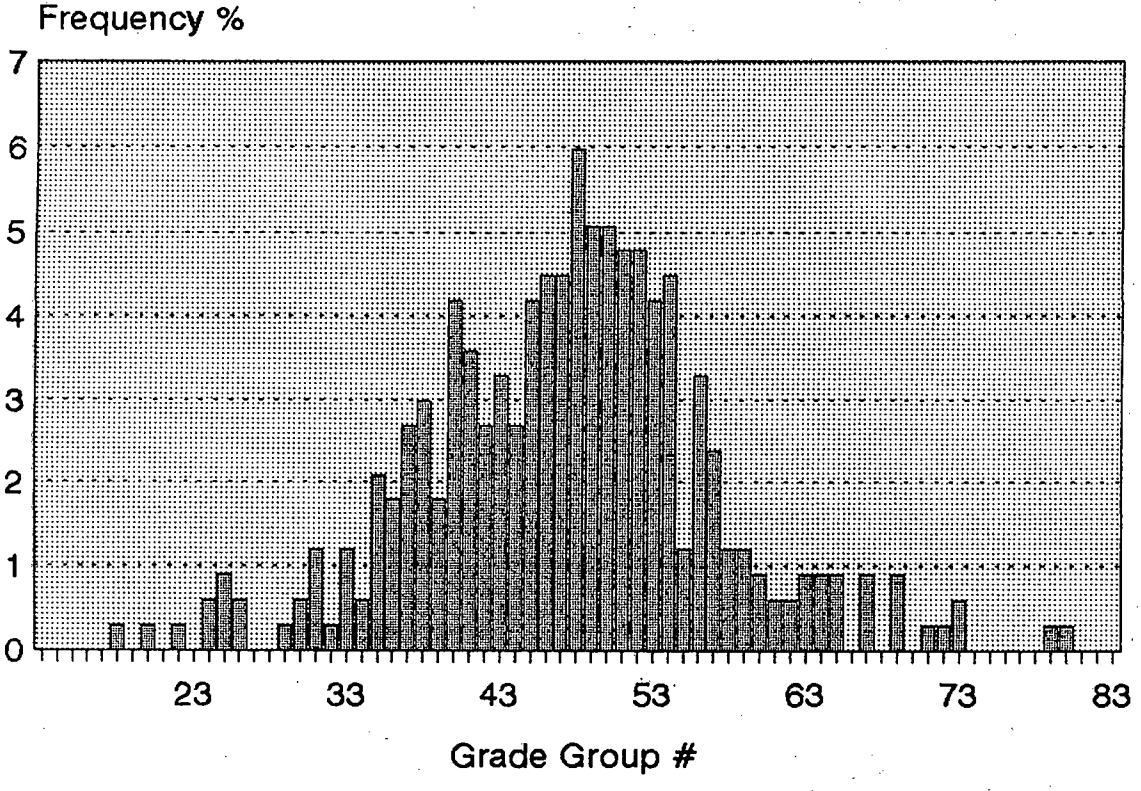
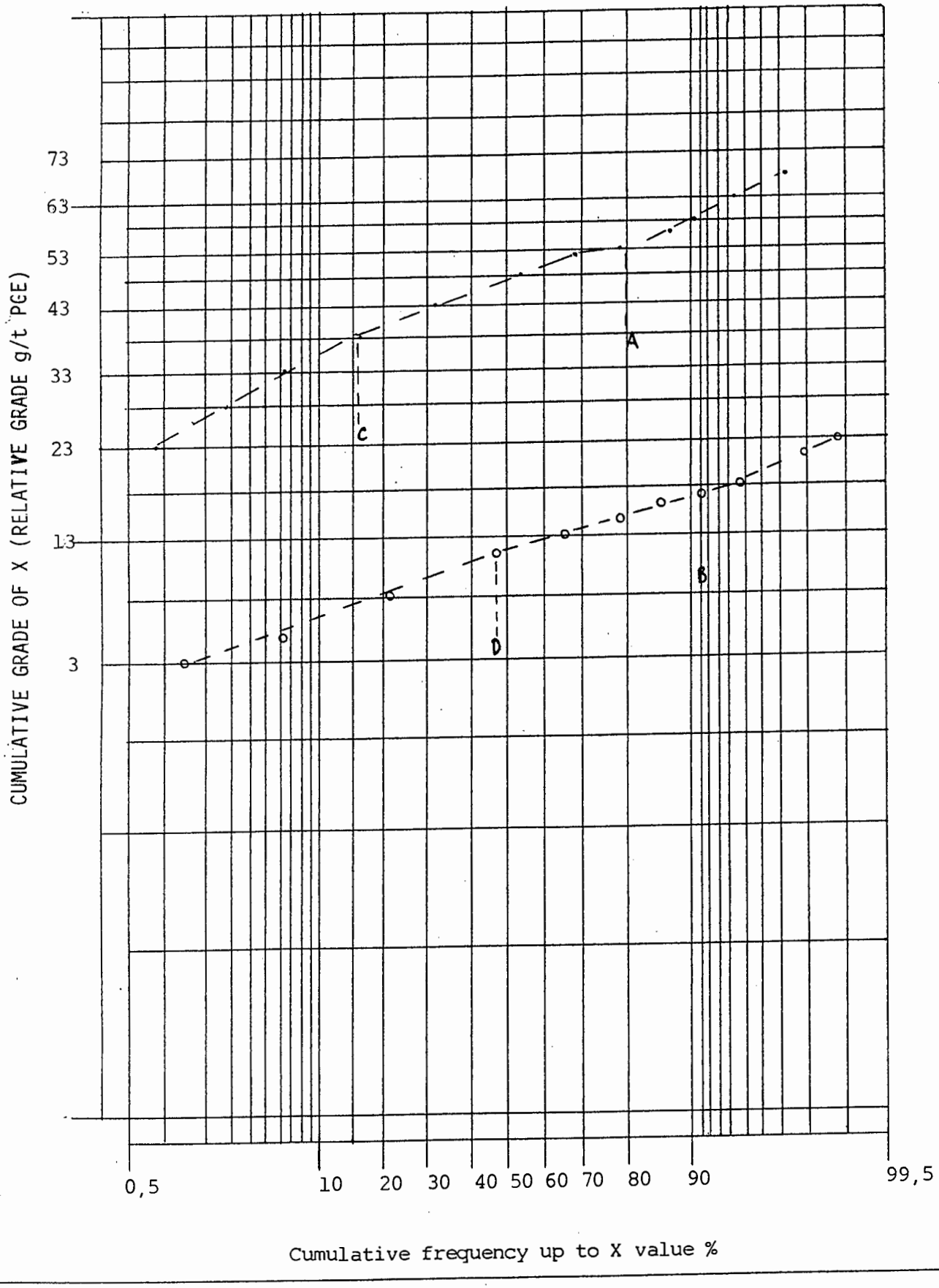


Figure 3.17: Waterval CCO Value Frequency Distribution

Figure 3.18
Logarithmic Probability Plot
Waterval Mill Feed and CCO



3.1.3:b Mineral Process Products

The following primary circuit products were identified in § 2.3 for investigation:

Frank Final Concentrate
Waterval Final Concentrate
Waterval Davcra Concentrate
Waterval Metallics.

The VFD data for these are shown in Tables 3.18-3.21 and Figures 3.19-3.22 respectively. Data are indexed as before (§ 3.1.3:a). The probability plots for these are shown in Figures 3.23-3.25.

Table 3.18
Frank Final Flotation Concentrate
Value Frequency Distribution Data

1. 4E: Sum of Pt,Pd,Au,Rh: Relative Grades are Proportional

Rel. Grd. g/t 4E [1]	Freq. %	Cum. Freq %	Grade Bin # PGE 4E	Freq %	Cum. Freq. %	Grade Bin # PGE 4E	Freq. %	Cum Freq. %	Grade Bin # PGE 4E	Freq. %	Cum. Freq. %
1	0,0	0,0	21	1,29	13,66	41	0,77	64,69	61	0,00	98,45
2	0,26	0,26	22	3,09	16,75	42	3,87	65,56	62	0,00	98,45
3	0,26	0,52	23	2,32	19,07	43	5,15	73,71	63	0,26	98,71
4	0	0,52	24	2,58	21,65	44	3,61	77,32	64	0,00	98,71
5	0	0,52	25	4,12	25,77	45	4,12	81,44	65	0,26	98,97
6	0	0,52	26	2,84	28,61	46	3,35	84,79	66	0,00	98,97
7	0	0,52	27	2,32	30,93	47	3,09	87,89	67	0,00	98,97
8	0,52	1,03	28	1,29	32,22	48	1,03	88,92	68	0,00	98,97
9	0	1,03	29	2,06	34,28	49	2,84	91,75	69	0,00	98,97
10	0,52	1,55	30	1,29	35,57	50	1,80	93,56	70	0,77	99,74
11	2,06	3,61	31	2,58	38,14	51	1,03	94,59	71	0,00	99,74
12	0,52	4,12	32	2,58	40,72	52	1,03	95,62	72	0,26	100,0
13	0,77	4,90	33	3,61	44,33	53	1,03	96,65			
14	1,03	5,93	34	1,80	46,13	54	0,26	96,91			
15	0,52	6,44	35	2,58	48,71	55	0,26	97,16			
16	0,77	7,22	36	3,87	52,58	56	0,00	97,16			
17	0,77	7,99	37	2,58	55,15	57	0,00	97,16			
18	1,29	9,28	38	2,32	57,47	58	0,26	97,42			
19	1,29	10,57	39	3,35	60,82	59	0,26	97,68			
20	1,80	12,37	40	3,09	63,92	60	0,77	98,45			

The points of inflection in the probability plot for Frank Final Concentrate (Figure 3.23) marked A, B, and C indicate possible demarcation between subdistributions of a compound distribution; and an exercise to subdivide and examine each was done. The straight line form of most of these offers the opportunity to extrapolate to lines DE, FG and HI to describe the overlap of the tails of the subdistributions [Parker, 1991].

Table 3.19
Waterval Final Flotation Concentrate
Value Frequency Distribution Data

Rel. Grd. g/t 4E	Freq. %	Cum. Freq %	Grade Bin # PGE 4E	Freq %	Cum. Freq. %	Grade Bin # PGE 4E	Freq. %	Cum Freq. %	Grade Bin # PGE 4E	Freq. %	Cum. Freq. %
1	0,26	0,26	21	0,77	97,69	41	0,00	99,74	61	0,00	99,74
2	1,03	1,29	22	0,77	98,46	42	0,00	99,74	62	0,00	99,74
3	0,77	2,06	23	0,26	98,71	43	0,00	99,74	63	0,00	99,74
4	2,31	4,37	24	0,00	98,71	44	0,00	99,74	64	0,00	99,74
5	2,31	6,68	25	0,00	98,71	45	0,00	99,74	65	0,00	99,74
6	4,11	10,82	26	0,26	98,97	46	0,00	99,74	66	0,00	99,74
7	6,17	16,97	27	0,00	98,97	47	0,00	99,74	67	0,00	99,74
8	5,40	22,37	28	0,26	99,23	48	0,00	99,74	68	0,00	99,74
9	11,31	33,68	29	0,26	99,49	49	0,00	99,74	69	0,00	99,74
10	10,03	43,70	30	0,00	99,49	50	0,00	99,74	70	0,00	99,74
11	10,03	53,63	31	0,00	99,49	51	0,00	99,74	71	0,00	99,74
12	12,34	66,07	32	0,00	99,49	52	0,00	99,74	72	0,00	99,74
13	11,57	77,63	33	0,00	99,49	53	0,00	99,74	73	0,00	99,74
14	7,46	85,09	34	0,26	99,74	54	0,00	99,74	74	0,00	99,74
15	4,11	89,20	35	0,00	99,74	55	0,00	99,74	75	0,00	99,74
16	2,83	92,03	36	0,00	99,74	56	0,00	99,74	76	0,00	99,74
17	1,29	93,32	37	0,00	99,74	57	0,00	99,74	77	0,26	100,0
18	1,03	94,34	38	0,00	99,74	58	0,00	99,74			
19	1,03	95,37	39	0,00	99,74	59	0,00	99,74			
20	1,54	96,92	40	0,00	99,74	60	0,00	99,74			

Table 3.20
Value Frequency Distribution Data
Waterval Davcra Concentrate

Rel. Grd. g/t 4E	Freq %	Cum Freq %	Grade Bin # PGE 4E	Freq %	Cum Freq %	Grade Bin # PGE 4E	Freq %	Cum Freq %	Grade Bin # PGE 4E	Freq %	Cum Freq %
1	0,00	0,00	15	7,20	66,76	29	0,28	94,18	43	0,28	99,45
2	0,00	0,00	16	5,82	72,58	30	0,83	95,01	44	0,00	99,45
3	0,83	0,83	17	2,49	75,07	31	1,11	96,12	45	0,00	99,45
4	0,00	0,83	18	4,16	79,22	32	1,11	96,12	46	0,00	99,45
5	0,28	1,11	19	2,77	81,99	33	0,28	97,51	47	0,00	99,45
6	0,28	1,39	20	1,66	83,66	34	0,28	97,78	48	0,00	99,45
7	0,55	1,94	21	2,49	86,15	35	0,28	98,06	49	0,00	99,45
8	4,99	6,93	22	1,94	88,09	36	0,00	98,06	50	0,00	99,45
9	5,54	12,47	23	1,11	89,20	37	0,28	98,34	51	0,28	99,72
10	8,86	21,33	24	0,83	90,03	38	0,28	98,61	52	0,00	99,72
11	9,14	30,47	25	0,55	90,58	39	0,00	98,61	53	0,00	99,72
12	11,63	42,11	26	1,11	91,69	40	0,55	99,17	54	0,00	99,72
13	9,97	52,08	27	0,83	92,52	41	0,00	99,17	55	0,00	99,72
14	7,48	59,56	28	1,39	93,91	42	0,00	99,17	56	0,28	100,0

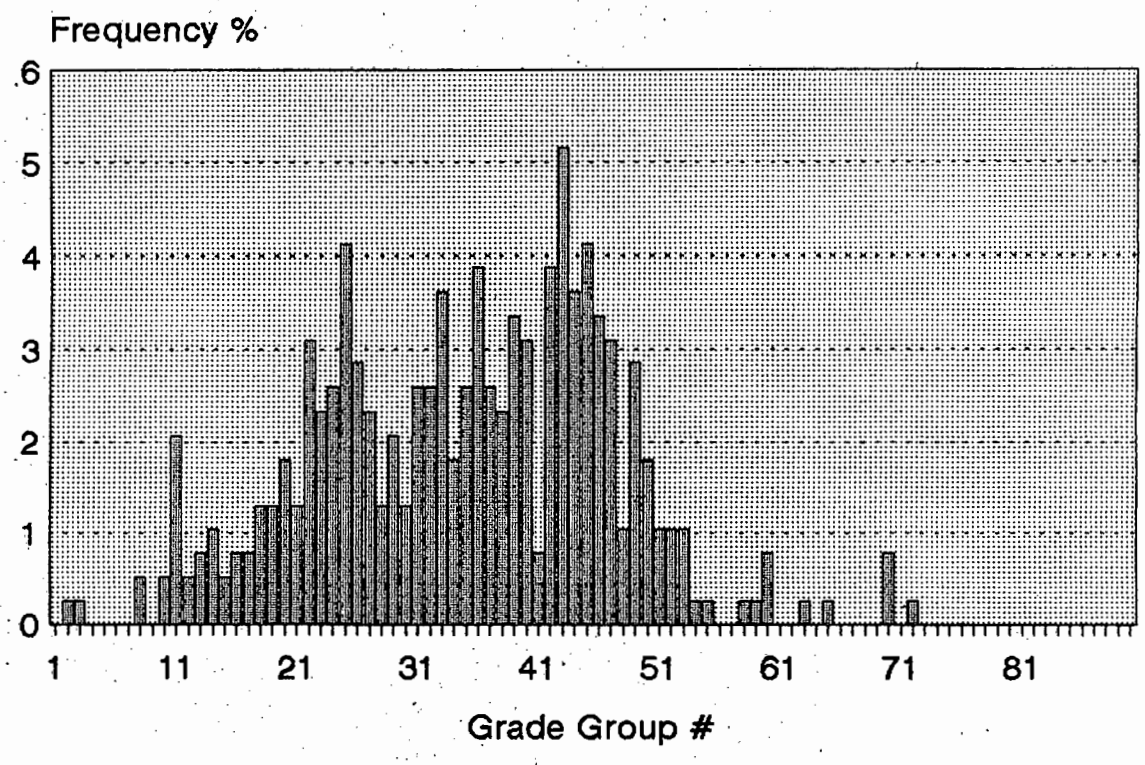


Figure 3.19: Frank Final Concentrate Value Frequency Distribution

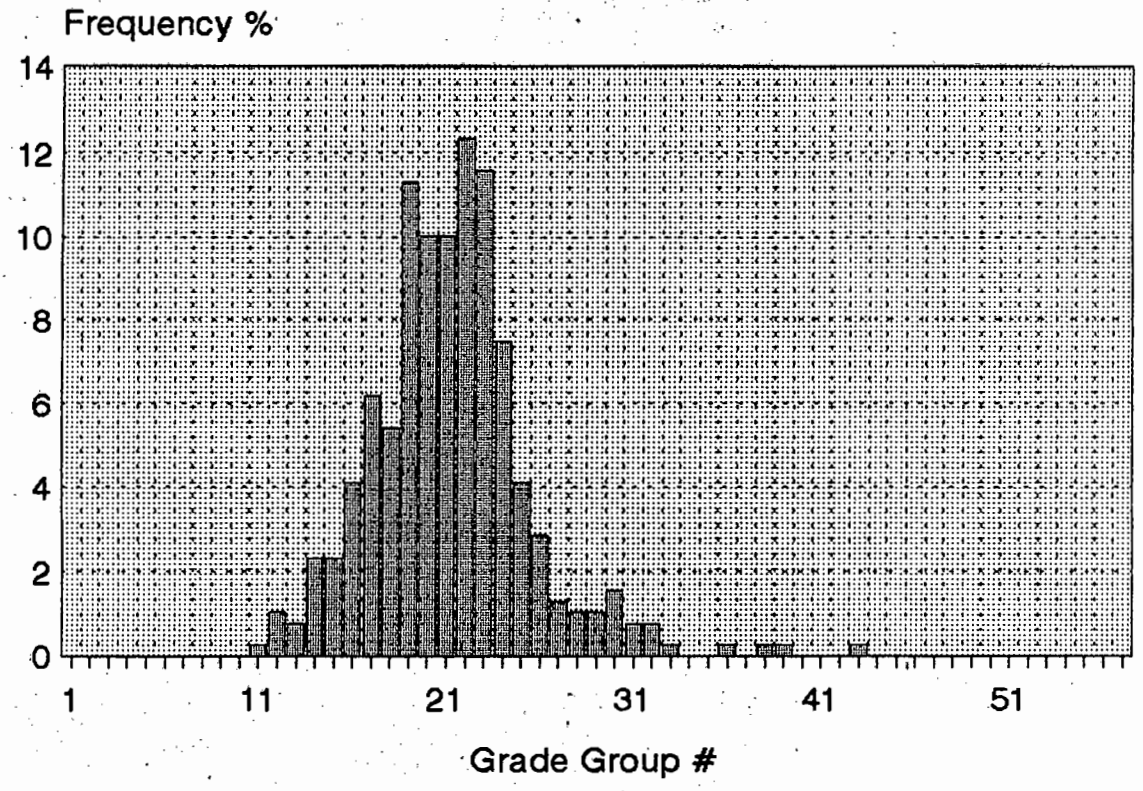


Figure 3.20: Waterval Final Concentrate Value Frequency Distribution

Table 3.21
Value Frequency Distribution Data
Waterval Metallics

Rel. Grade % 4E	Freq %	Cum Freq %	Grade Bin # PGE 4E	Freq %	Cum Freq %
1	0,00	0,00	9	10,52	82,52
2	7,01	7,01	10	8,77	91,19
3	5,26	12,27	11	3,51	94,70
4	8,77	21,04	12	3,51	98,21
5	5,26	26,30	13	1,75	99,96
6	19,29	45,59	14	0,00	99,26
7	19,28	64,87	15	0,00	99,26
8	14,03	71,90	16	0,00	99,26

This decomposition of the Frank Concentrate VFD proved viable for the first two subdistributions aforementioned, however the further subdivision of the remainder of the parent distribution about C using line HI proved untenable as it produced negative frequencies in the remaining subdistribution. Therefore it was concluded that the parent distribution to the left of B was actually one subdistribution, not two, and was probably three-parameter lognormal. The VFDs for each subdistribution, Series 1, 2 and 3, were then modelled using the extrapolated lines DE and FG. The subdistributions were subtracted from highest grade to lowest, with the results shown in Table 3.22:

Table 3.22: Modelled VFD: Subdistributions of Frank Final Concentrate

Rel. Grade g/t 4E	% Frequency			Rel. Grade g/t 4E	% Frequency		
	Series 1	Series 2	Series 3		Series 1	Series 2	Series 3
11	1,9	0,0	1,7				
13	2,6	0,0	0,8	43	2,5	13,71	7,0
15	5,0	0,0	0,8	45	1,5	7,73	6,0
17	6,5	0,0	1,6	47	1,0	6,45	4,0
19	8,0	0,0	2,1	49	2,0	3,86	10,0
21	9,0	0,0	2,8	51	1,0	2,84	10,0
23	8,0	0,0	0,4	53	0,65	2,06	1,5
25	8,0	0,8	5,8	55	0,51	0,0	2,5
27	8,0	1,3	2,0	57	0,0	0,0	0,0
29	6,0	2,9	9,0	59	0,52	0,0	0,0
31	8,0	10,0	3,0	61	0,77	0,0	0,0
33	3,0	5,0	6,3	63	0,26	0,0	0,0
35	6,0	10,0	1,7	65	0,26	0,0	0,0
37	3,0	11,0	6,0	67	0,0	0,0	0,0
39	3,0	14,0	6,0	69	0,0	0,0	0,0
41	2,0	5,0	3,0	71	0,77	0,0	0,0

Figure 3.23
Logarithmic Probability Plot
Frank Final Concentrate

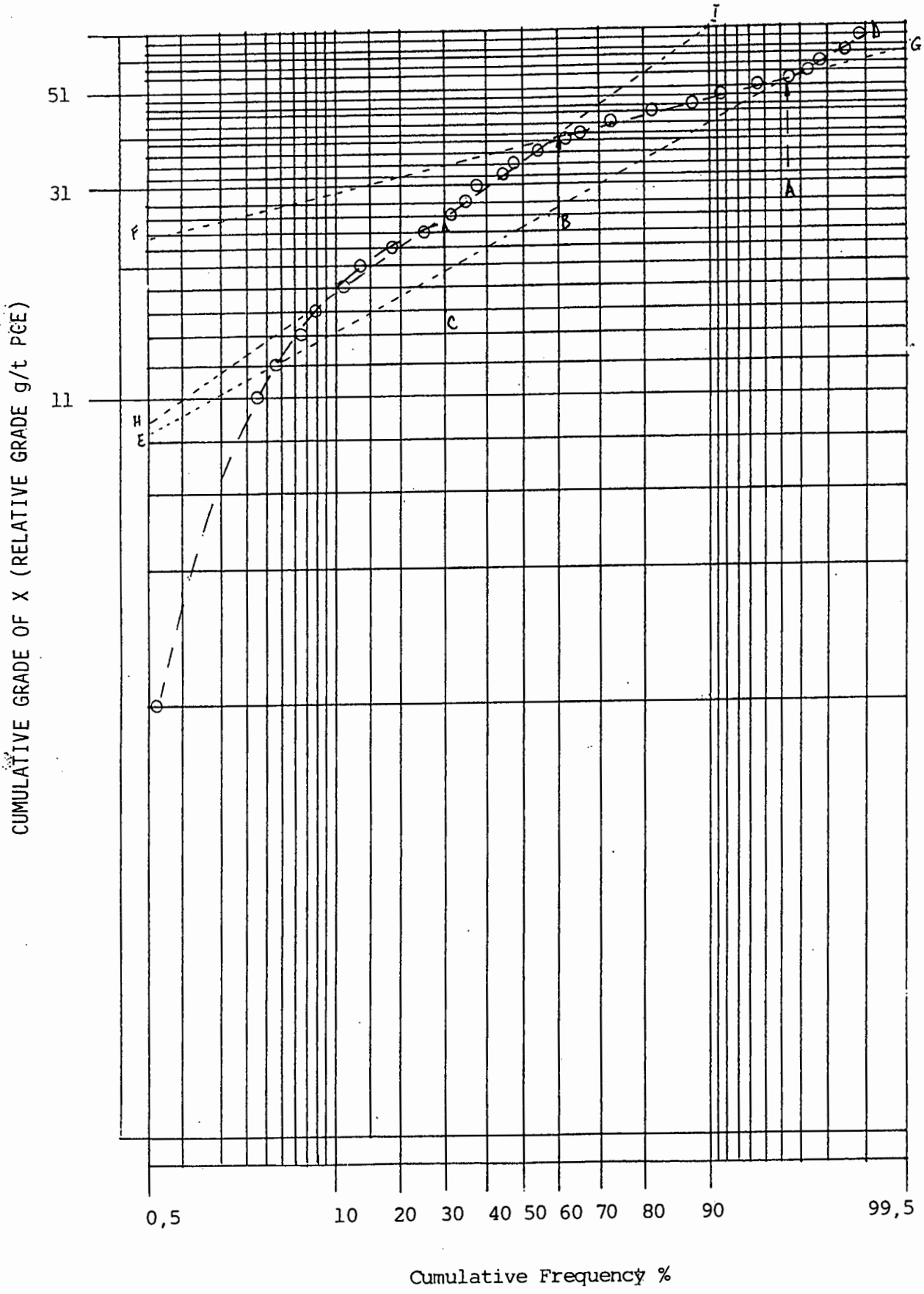
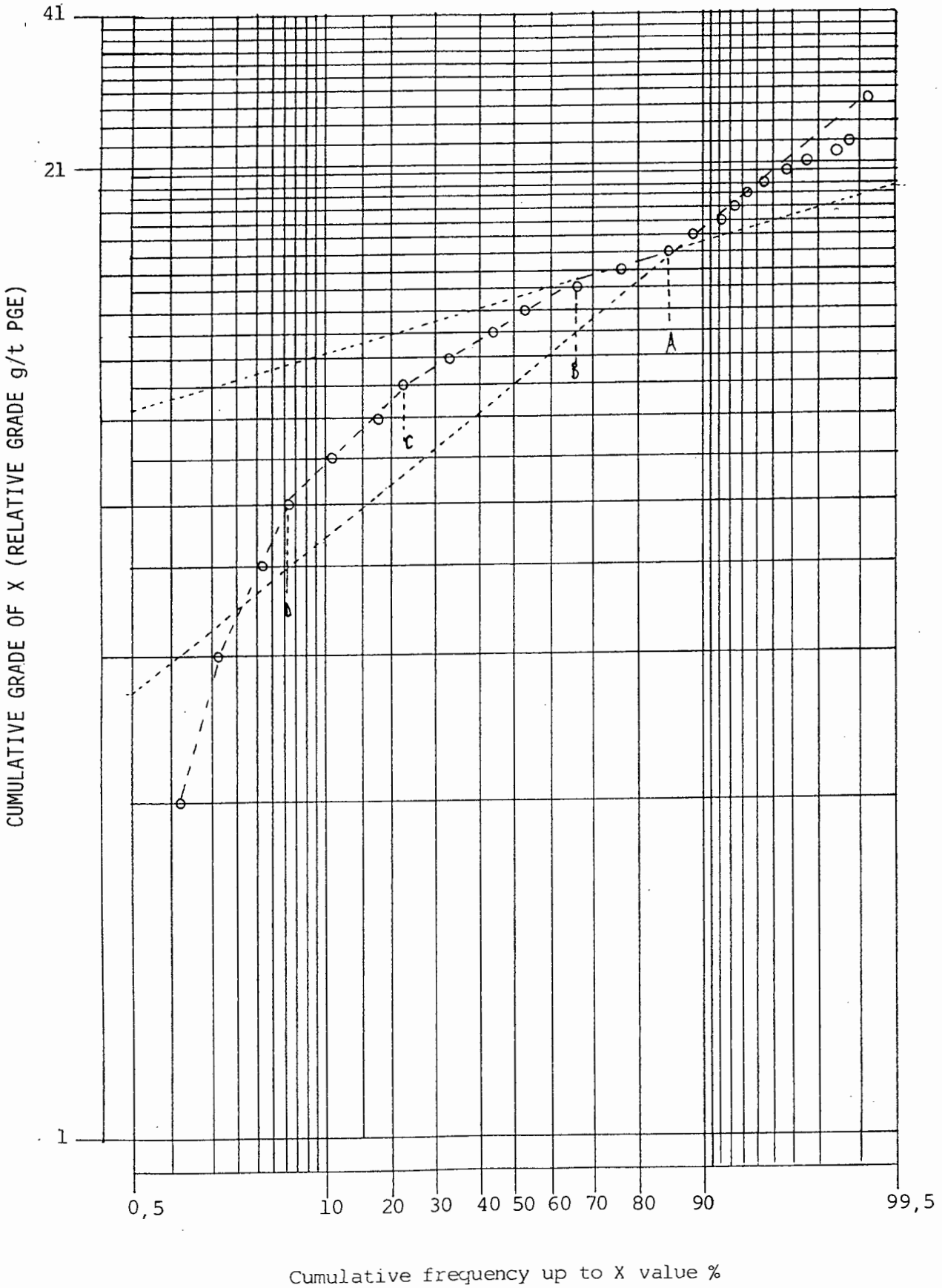


Figure 3.24
Logarithmic Probability Plot
Waterval Final Concentrate



The value frequency distributions of these three series are shown in Figure 3.27, and their relative weights in the overall distribution, in Table 3.23.

Table 3.23
Relative Weights in Compound Distribution
Series 1-3, Frank Final Concentrate

Inflection Cum Fi%	Series 1	2	3	Other
A	96,3			
B		60,0		
C			30,0	
Relative % Weight in Compound Distribution	3,7	36,3	30,0	30,0

Thus approximately 3,7% of the overall PGE value in Frank Final Concentrate would appear to be present as residual metallics in this stream.

The equivalent data for Waterval Final Concentrate were examined using the same approach, in this instance using two indicated points of inflection A and B (Figure 3.24), and producing three estimated subdistributions from the parent compound distribution. These are summarised in Table 3.24, and plotted in Figure 3.28.

Table 3.24
Modelled VFD: Subdistributions of
Waterval Final Concentrate

Grade Group g/t PGE	% Frequency Series 1	Series 2	Series 3
1	0,0	0,0	0,0
5	12,5	0,0	0,0
9	43,5	10,0	0,0
13	25,0	67,6	27,0
17	11,5	20,4	22,29
21	4,0	1,5	6,87
25	1,8	0,0	2,52
29	0,0	0,0	1,28
33	0,0	0,0	0,0
37	0,0	0,0	0,25
41	0,0	0,0	0,0

The relative weights of these series in the overall distribution are shown in Table 3.25.

Table 3.25
Relative Weights in Compound Distribution
Series 1-3, Waterval Final Concentrate

Inflection Cum. % Fi	Series 1	2	3	Other
A	85,09			
B		43,70		
C			22,37	
Relative % Weight in Distribution	14,91	41,39	21,33	22,37

Thus approximately 14,91% of the overall PGM+Au value in Waterval Final Concentrate would appear to be present as metallics in this stream.

Inspection of Figures 3.21 and 3.25: Waterval Davcra Concentrate, indicates a simpler arrangement of a compound distribution than shown in the final concentrates. Points of inflection A and B for 72,58 and 15,00% cumulative frequency respectively demarcate possible step changes in the grade of PGE in the overall distribution. It appears that the Davcra Concentrate carries more metallics than final concentrate, since (100-72,58)= 27,42% of the PGE is found to the right of A. This result should be expected since the Davcra Concentrate is treated by a gravity recovery process prior to being released as final concentrate.

Inspection of Figures 3.22 and 3.26: Waterval Metallics, indicates only one point of inflection, A, located at 30% cumulative frequency. This would imply that (100-30)=70% of the PGE in metallics are metallics, and 30% are in some other form such as a high-density base metal sulphide.

3.1.3:c Conclusions

This section of results has shown that the value frequency distributions of Merensky ore, both sampled underground and as presented to the milling process in a crushed form, demonstrate complex lognormality in mixtures of two- and three-parameter lognormal models. These inferences have been made by visual inspection of the probability plots, as suggested by Clark and Garnett for preliminary investigation [Clark and Garnett, 1974]. The pattern is further seen in mineral process products, where the possibility of modelling subdistributions belonging to certain forms of association by grade group has been noted.

Figure 3.25
Logarithmic Probability Plot
Waterval Davcra Concentrate

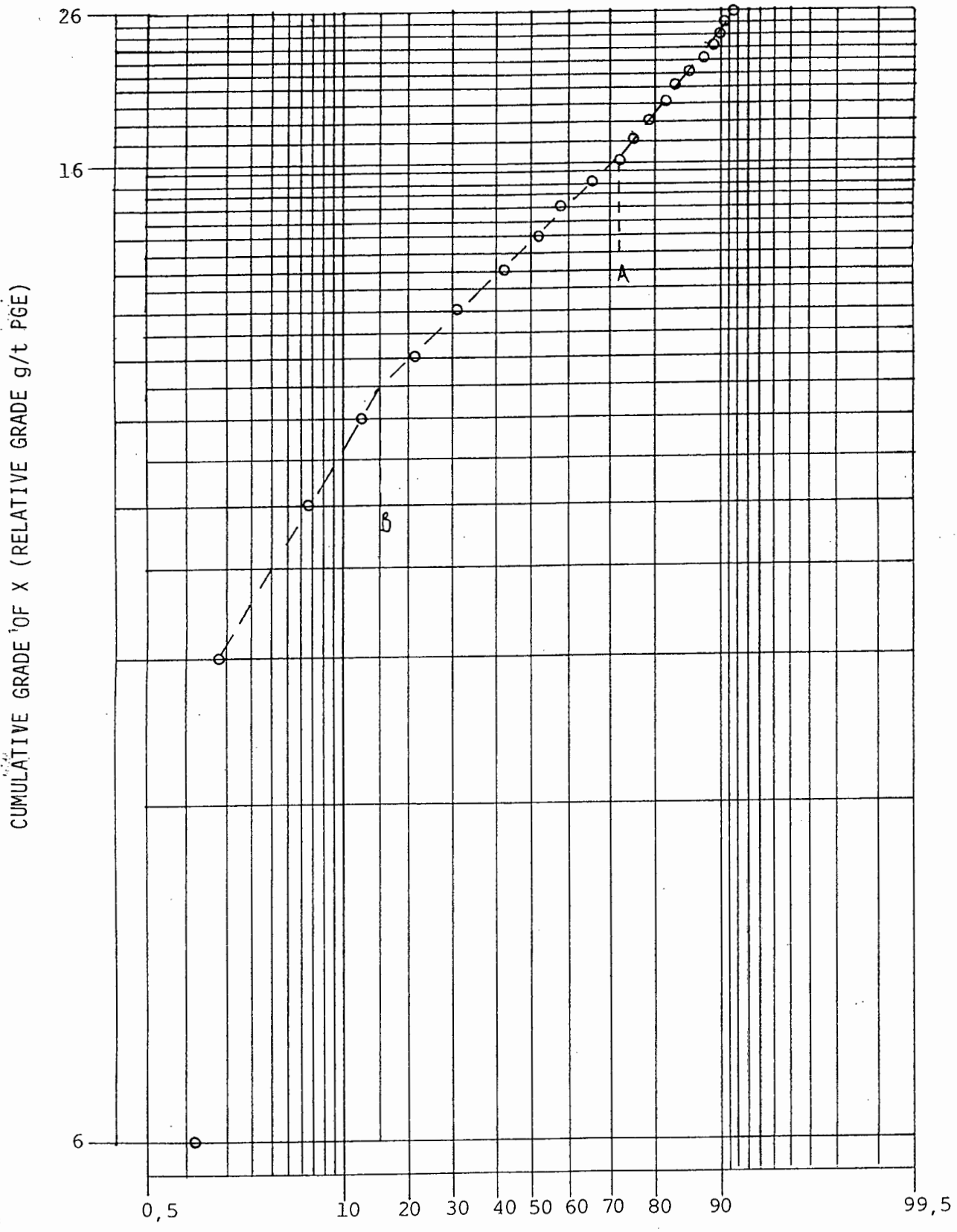
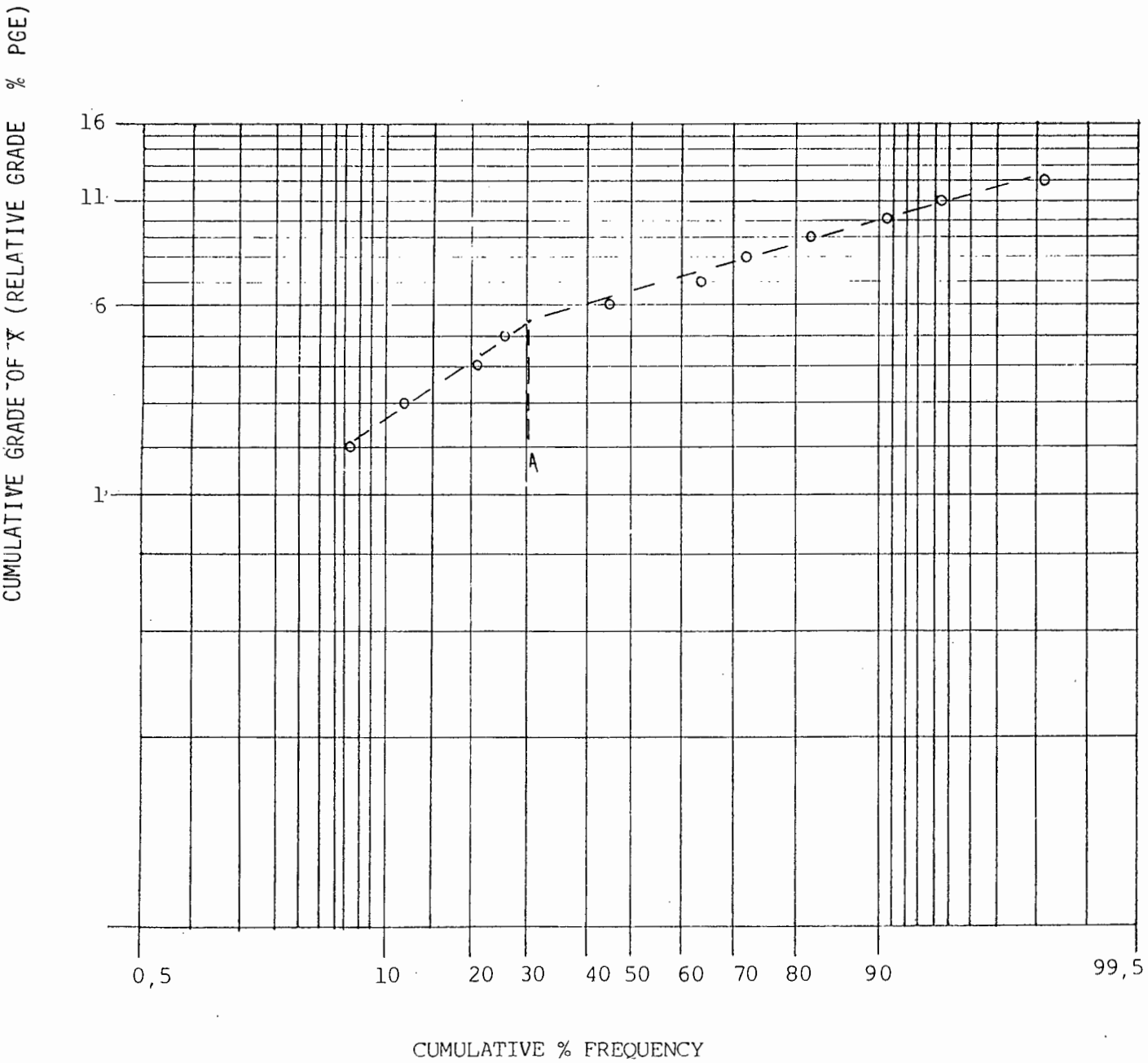


Figure 3.26
Logarithmic Probability Plot
Waterval Metallics



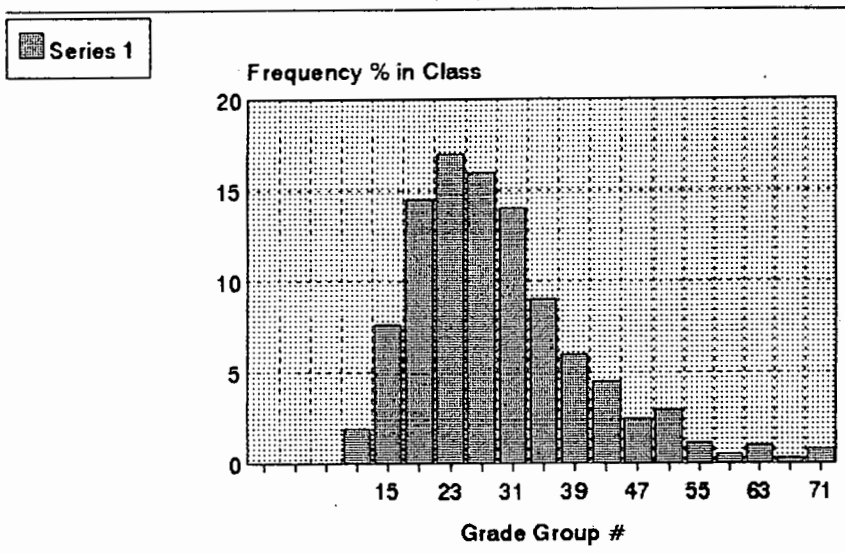


Figure 3.27: Frank Final Concentrate Value Frequency Subdistribution 1

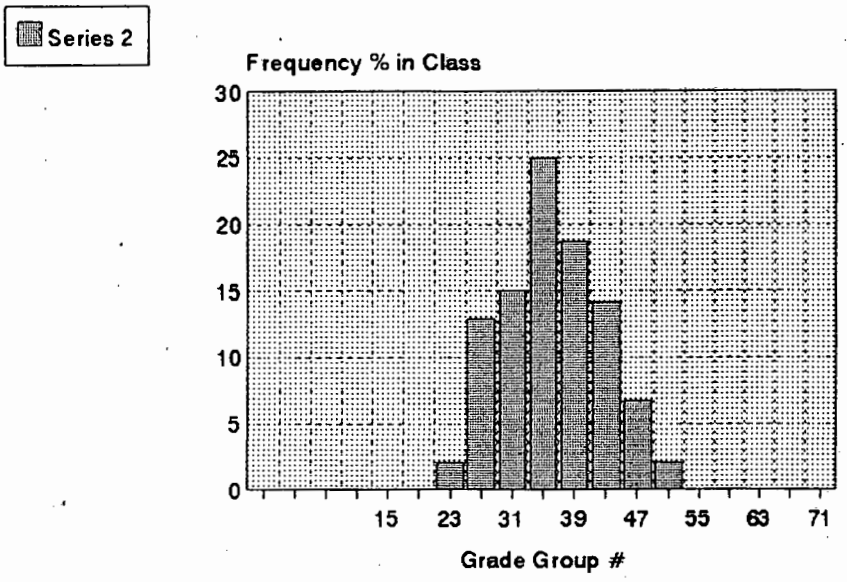


Figure 3.27: Frank Final Concentrate Value Frequency Subdistribution 2

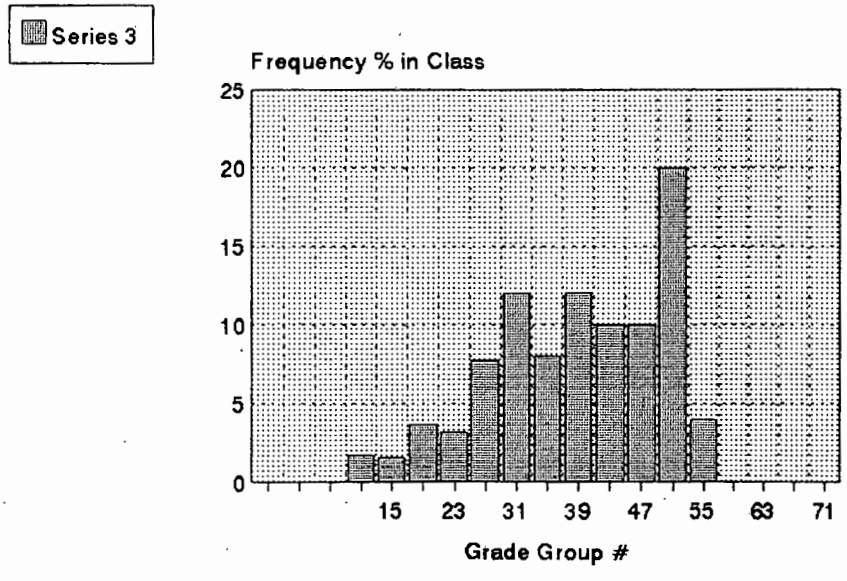


Figure 3.27: Frank Final Concentrate Value Frequency Subdistribution 3

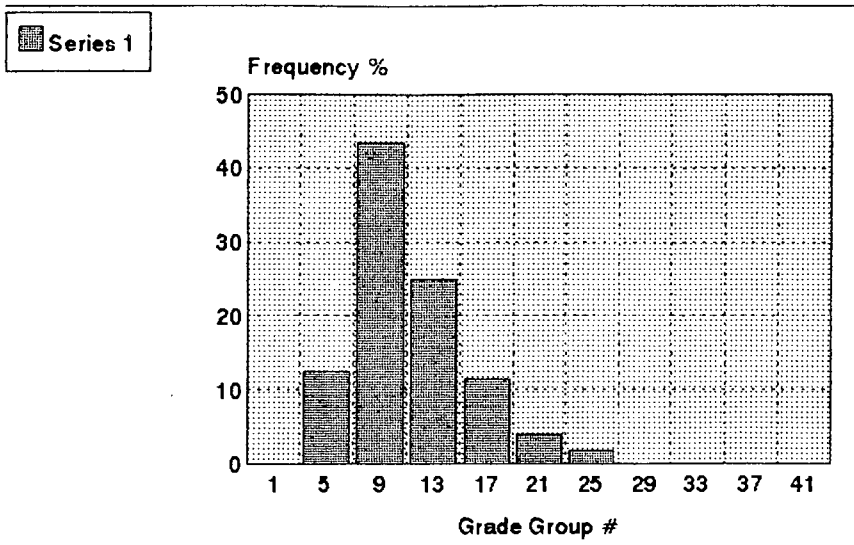


Figure 3.28: Waterval Final Concentrate Value Frequency Subdistribution 1

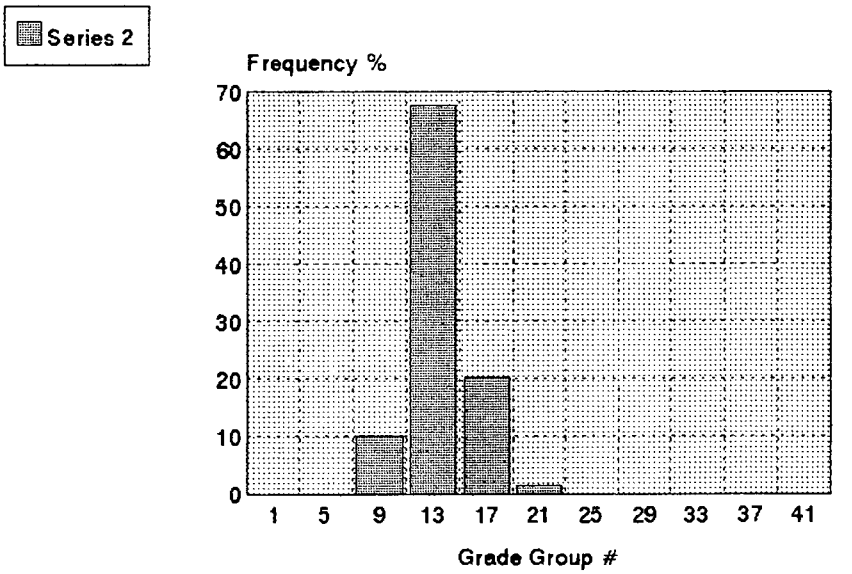


Figure 3.28: Waterval Final Concentrate Value Frequency Subdistribution 2

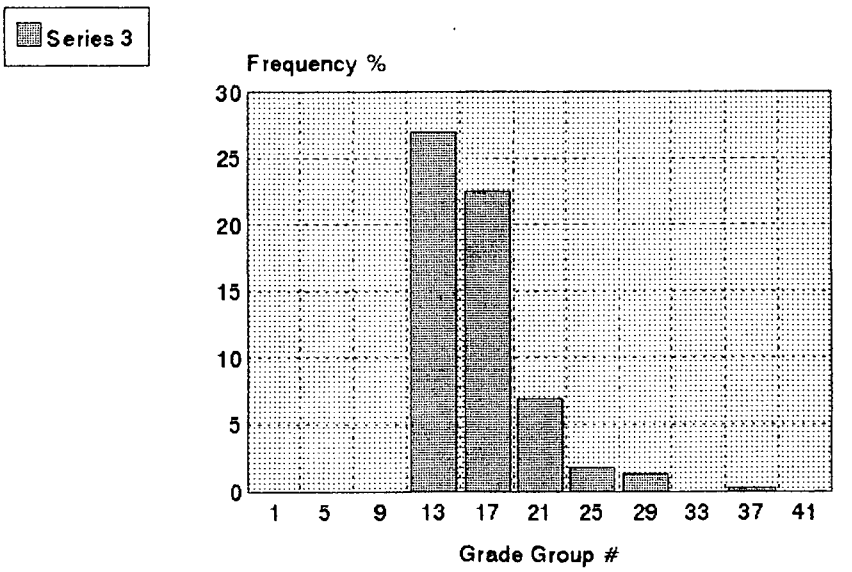


Figure 3.28: Waterval Final Concentrate Value Frequency Subdistribution 3

The major feature of all VFDs studied, however, seems to be the point of inflection in the right-hand section of the probability plot, which will be shown in § 3.1.4 and § 3.2.1 to be equivalent to the step change in grade from base metal sulphide association of PGE to discrete metallics occurrences. If this typical point of inflection in a probability plot of PGE values for a mineral process stream represents the demarcation between base metal sulphide and discrete metallics forms of deportment, validation exercises should follow, since this could be the basic cause of bias errors in the evaluation of these samples.

3.1.4 The Estimation of Discrete PGE as Metallics in Davcra Concentrate Using Probability Plots

The procedural treatment of a technical grade sample of Waterval Davcra Concentrate (§ 2.5) yielded the results shown in Table 3.26. A technical grade sample is one taken with due regard for the rules of sampling as set out in § 1.2.3.

Table 3.26
Summarised VFD Data
Waterval Davcra Concentrate

Grade Group g/t PGE	A As is	B Pulv.	C Superpanned
1	1,67	0	0
2	0	0	0
3	1,67	0	0
4	5,00	1,67	0
5	6,67	1,67	0
6	1,67	8,33	18,18
7	10,00	8,33	18,18
8	5,00	5,00	9,09
9	11,67	20,00	18,18
10	1,67	16,67	9,09
11	6,67	8,33	9,09
12	10,00	8,33	0
13	15,00	5,00	0
14	3,33	5,00	9,09
15	1,67	5,00	0
16	1,67	0	0
17	5,00	0	0
18	1,67	0	0
19	5,00	1,67	0
20	1,67	1,67	9,09
21	1,67	0	0
22	1,67	0	0
23	0	1,67	0
24	0	1,67	0
25	0	0	0

The basic statistics of these results are shown in Tables 3.27 and 3.28.

Table 3.27
Waterval Davcra Concentrate
(Technical Sample)
Basic Statistics

Attribute	A As is	B Pulverised	C Superpanned
Observations	60	60	11
Mean Grade gpt PGE	1,000	0,994	0,987
Std. Error of Mean, g/t PGE	0,62	0,50	1,26

Table 3.28
Waterval Davcra Concentrate
(Technical Sample)
Subsample Group C: Superpanning

Test #	Metallics Potential % of Total PGE [1]
1	20,11
2	24,07
3	21,76
4	24,55
5	21,98
6	19,75
7	27,75
8	17,88
9	26,62
10	26,62
11	21,65
Mean Value	22,97
Std. Deviation	3,19
RSD %	13,88

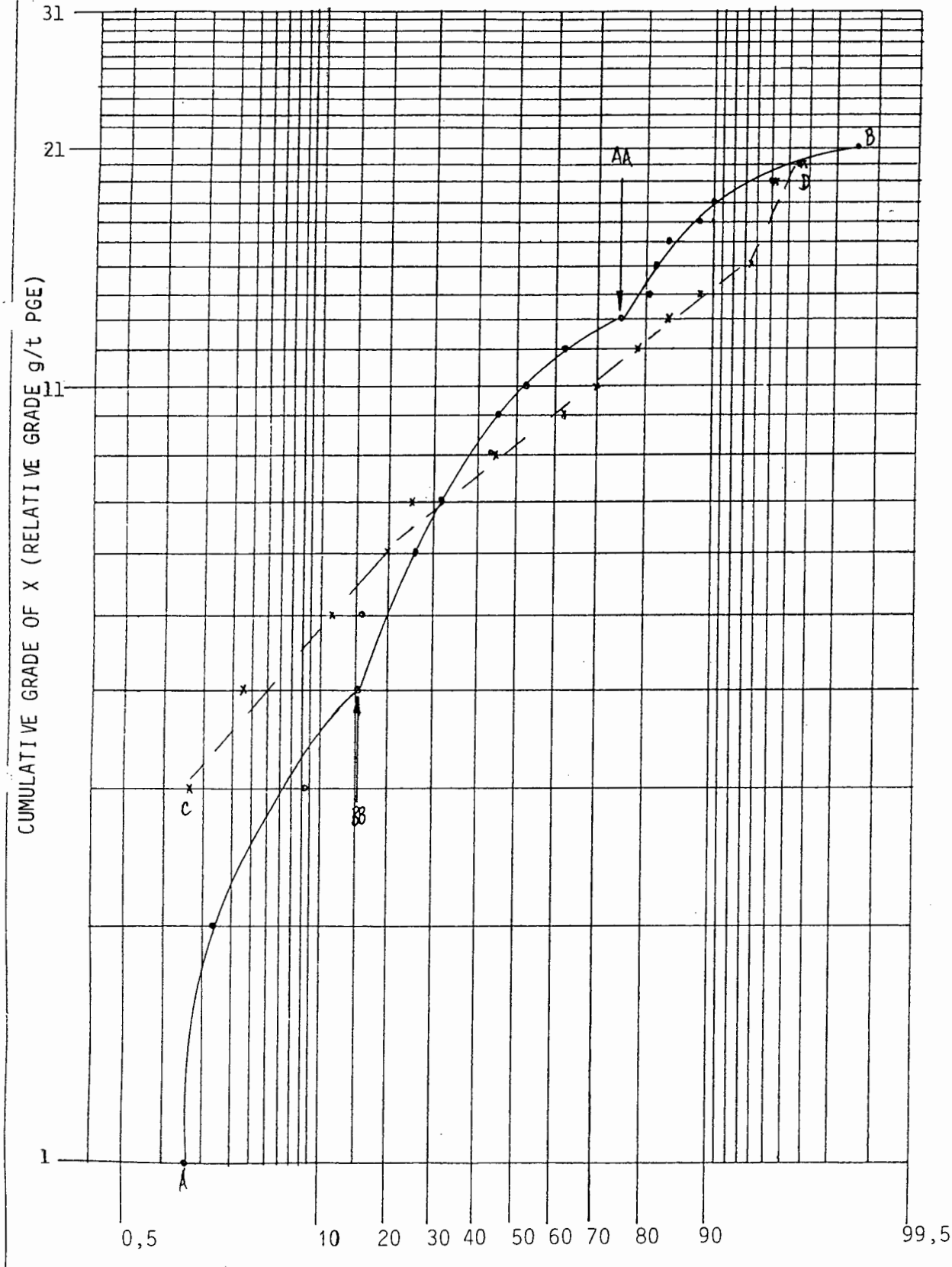
[1] : Metallics Potential per Eq. 60 § 2.5. This is an empirical method that quantifies the amount of 4E PGE present as discrete PGM, or "metallics"

The probability plots of the A and B groups of data are shown in Figure 3.29. Inspection of this graph shows that the Group A data (curve AB) show a definite point of inflection at AA, with a secondary point at BB. The cumulative frequency corresponding to AA is 76,67%, implying that (100-76,67)% = 23,33% of the PGE are present as metallics.

Figure 3.29
Logarithmic Probability Plot
Waterval Davcra Concentrate
Groups A and B

Key

o—— o Curve AB, Group A
x– –x Curve CD, Group B



The empirical superpanner test result and the indicated graphical method result for metallics potential on this sample are compared in Table 3.29.

Table 3.29
Comparison of Estimated and Actual
Metallics Potential of Davcra Concentrate
(Composite Sample)

Detail	Metallics Potential g/t PGE
Empirical (Table 3.28)	22,97
Estimated (Graphical Method)	23,33
Difference % PGE	+0,36
Error %	+1,56

The above comparison indicates good agreement between the two estimates, suggesting that the interpretation of logarithmic probability plots for multiple mineral host forms such as proposed by Clark is workable [Clark, 1974]. Further exercises on this A-B-C basis are necessary to extend the concept into final flotation concentrate.

The possibility of skewness in the distribution of metallics potentials per Table 3.28 misleading the calculation of mean value was tested by a mathematical exercise using the mean and 95% confidence limits in both normal and Sichel models [Sichel, 1966; Clark, 1993]. The results are shown in Table 3.30.

Table 3.30
Means and Confidence Limits
Actual Metallics Potentials per Table 3.28
n = 11

Attribute	t Statistics	Sichel Model	Notes
Estimated Mean gpt PGE	22,97	23,00	1. $\tau_n(V) = 1,00985$
Std. Error	0,96		
UCL (95%)	24,89	25,65	2. $\Phi_{(V,n)} = 1,1152$
LCL (95%)	21,04	21,49	3. $\Phi_{(V,n)} = 0,9343$

The multiple of standard errors Sε for 10 degrees of freedom in the distribution of t is 2,228 at the 95% confidence level [Box et. al., 1978c]. Inspection of Table 3.30 indicates agreement between the two estimates of the mean, suggesting that skewness may not be a significant factor in the estimation of the mean in this case. The two sets of confidence limits, however, differ slightly. Of significance, however, is the fact that the modelled metallics potential derived from the proposed graphical method falls within both the 95% confidence limits of the actual mean estimates; viz. the 35,00 g/t metallics potential derived from Figure 3.23 falls within the 30,86-36,28 (t) and 32,1-37,4 (Sichel) g/t limits. This proves that the graphical method provides an estimate of the metallics potential of the Davcra Concentrate within the 95% confidence limits of the empirically derived figure.

3.1.5 Representative Sampling of Mill Feed

The first results from the exercise detailed in § 2.4.1 are shown in Table 3.31, listing the size-by-size PGE grades and densities together with the masses per fraction. As a result of the actual topsize and other masses per size the top and bottom sizes were simplified, or grouped.

Table 3.31
Waterval Mill Feed
Size-by-Size Data

Size, mm	Mass g	%	PGE Dist'n %	Density g/cc	Dist'n %
6,7	4981,0	9,71	11,54	3,03	9,49
4,75	6432,7	12,54	12,48	3,33	13,47
3,35	4965,0	9,68	10,40	3,04	9,49
2,36	4219,0	8,23	6,52	3,11	7,26
0,85	3967,0	7,20	6,71	3,10	7,20
0,60	3779,0	7,37	3,71	3,10	7,36
0	15538,0	30,29	35,06	3,08	30,08
Weighted Mean				3,10	

Use of the individual prill results to calculate the variance per size and thus the weighted variance, using metal distribution as a weighting factor, produced the following results:

Weighted std. deviation 0,807 g/t
Weighted standard error 0,292 g/t

The sampling constant C was defined by Gy to be

$C = c.l.f.g$

where

c = mineralogical composition factor	$0,775 \times 10^6$
l = liberation factor	0,214
f = particle shape factor	0,5
g = size range factor	0,25

The values found are as indicated, and lead to an estimate of C = 20730. The estimation of fundamental variance is now made by

$$\sigma^2(FE) = Cd^3/M_s$$

$$= (20730)(0,75)^3/51292,7$$

$$= 0,17$$

Thus σ = 0,41 g/t, equivalent to a 9,1% RSD in the mean mill feed value.

Extension of this reasoning, as referred in §1.2.3, reduces the $M_s:\sigma^2(FE)$ relationship to

$$M_s = K/\sigma^2(FE),$$

thus $K = Cd^3 = 8745,47$. From this relationship it is now possible to estimate the representative sample mass equivalent to a given fundamental variance. Table 3.32 summarises these calculations.

Table 3.32
Estimation of Representative Sample Mass
Waterval Mill Feed
After Gy

Fundamental Variance %	K	Sample Mass Kg	Sample Mass Kg*
20	8745,47	43,73	48,31
15	8745,47	58,30	64,42
10	8745,47	87,45	96,62
5	8745,47	174,91	193,25

* Corrected for metal distribution finer than topsize (after Laplante)

The cross-check calculation for M_s using the size-by-size variance by Bartlett and Hawkins was performed on the same data. The following salient features refer.

$$f_v = (1/M_s).\ ((gv/a^2M).\sum_{i=1}^{n}((a_i-a)^2/V_i).M_i^2 \qquad (20)$$

$$g = (1/(d^3M))\ .\ \sum_{i=1}^{n} M_i d_i^3 \qquad (18)$$

M = 51292,7 g = 3,378
V = 0,027115
a = 4,274

From these calculations fv = 1703220/M_s, using fv as a percentage (Gy uses the decimal fraction). The estimation of M_s using the size-by-size metal variance is shown in Table 3.33.

Table 3.33
Waterval Mill Feed
Estimation of M_s after
Bartlett and Hawkins

Fundamental Variance	K	Sample Mass Kg
20	1703220	85,1
15	1703220	113,5
10	1703220	170,3
5	1703220	340,6

A direct comparison of M_s values from the two different approaches shows that unless size-by-size variance of metal values is taken into account, the calculation will underestimate M_s by approximately 176% at the 10% variance level. Accordingly it is concluded that the method by Bartlett and Hawkins is advisable.

3.1.6 Evaluation of Mill Feed: State of Grind, and Number of Determinations

The exercise on replicate assay head determinations within an overall mill feed composite sample (§2.4.1) to determine the overall confidence limits yielded the following results. The influence of grind on the assay head, the VFD and estimation of mean are studied. Initially all prill values were pooled on assumption that the Null Hypothesis would be true, viz. the variances of the subset means were similar

enough to warrant pooling of values as if they belonged to the same distribution. This produced the results shown in Table 3.34. The raw data forms a statistically robust population with 501 observations.

Table 3.34
Waterval Mill Feed
Pooled Data: VFD

Grade Bin # PGE	Raw Data Freq. %	Screened Data Freq%
1	0,20	0
2	0	0
3	0	0
4	0,40	0
5	1,00	0
6	0,60	0,42
7	2,99	3,13
8	14,97	15,66
9	37,13	38,83
10	26,95	28,18
11	9,38	9,81
12	3,39	3,55
13	0,6	0,42
14	1,00	0
15	0,20	0
16	0,20	0
17	0	0
18	0	0
19	0	0
20	0	0
21	0	0
22	0	0
23	0,20	0
24	0	0
25	0	0
26	0	0
27	0	0
28	0	0
29	0	0
30	0	0
31	0	0
32	0	0
33	0,20	0
Mean Grade g/t PGE	1,008	1,006
RSD %	19,51	10,99
n	501	479

One is entitled to estimate the standard deviation from these and define the 95% confidence limits of the data at $\bar{x}$ ± 2s. This was done and resulted in the second VFD shown in Table 3.34.

Inspection of Table 3.34 shows that whereas the means of the two VFDs are close, the standard deviations are very different, with the screened data set showing approximately half the value of the raw data. Only 22 data were rejected in the process, or 4,4%. This is initial evidence of the effect of outlier data on the estimation of the standard deviation and RSD, and leads to the initial inference that mill feed ground to the domain of flotation sizes can work at approximately 10% RSD when screened to the 95% confidence limits of data. The effect of this quality control is shown in Figure 3.30. The analysis of this effect at each grind is shown in Tables 3.35 - 3.36.

Table 3.35
Waterval Mill Feed
Analysis of VFD by Grind
Raw Data

Parameter	Grind, % Passing 75 Microns			
	40	50	60	70
Mean Grade g/t PGE	1,000	0,997	1,025	1,017
RSD %	20,0	15,2	26,0	15,6
n	143	143	107	108

Table 3.36
Waterval Mill Feed
Analysis of VFD by Grind
Screened Data

Parameter	Grind, % Passing 75 Microns			
	40	50	60	70
Mean Grade gpt PGE	0,988	1,002	1,010	1,019
RSD %	13,3	10,9	8,8	14,7
n	139	140	105	107
Rejection Rate %	2,8	2,1	1,9	0,9

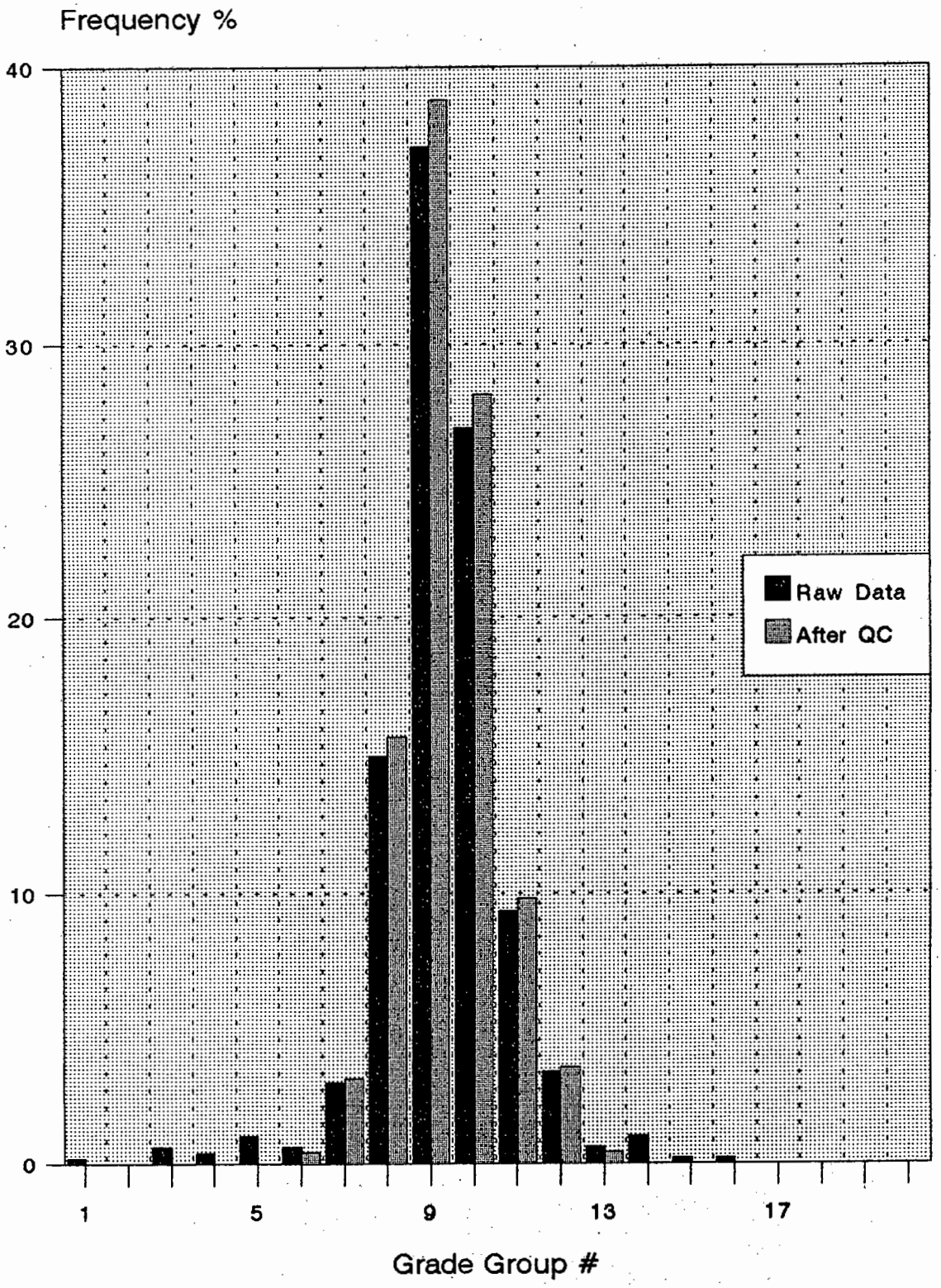


Fig 3.30: Pooled Data
Waterval Mill Feed: Value Frequency Distribution

These results show two main features, viz:

(i) The RSD of the estimated mean grade is reduced significantly by the rejection of data outside x ± 2s, in fact rejection of no more than 3% data in any case reduces the RSD from 19,2 to 11,9% on average.

(ii) The RSD of screened data shows a response to grind, in that the RSD approaches a minimum at 60% passing 75 microns. This may be related to liberation sizes of PGE-bearing minerals, and would indicate that assay head determinations would be best performed at this grind.

The calculation of the minimum number of replicates necessary to produce a stable mean estimate was addressed for the 60% grind. The accepted 105 prills from this test were assembled into a sorted sequence from lowest to highest grade. The semivariance was calculated through the data in the dimension of replicates. The results of this calculation are shown in Figure 3.31. Rejection of the two outliers at ± 2s was advisable in terms of work reported by Krige [Krige and Magri, 1982], in that the semivariogram is not robust to the presence of outliers. Inspection of the sorted semivariogram shows that the sill shows definite formation at 13 replicates or more, with any less replicates not reflecting the full semivariance. It is thus concluded for this sample of ore that 13 replicate prills are necessary to provide a stable estimate of the mean grade of assay head. Use of Student's t-distribution to confirm the result of the semivariogram now calculates the error term in the estimated mean grade arising from n replicate determinations. Since $t = ((X-\bar{X})\sqrt{n})/s$, and from Table 3.36 at the 60% grind, X = 1,010 and s = 0,09 gpt respectively with n = 105. At the 95% confidence level t tends towards a value of 2,00 for large n, so it is possible to formulate X ± $\bar{x}$ as a function of n replicates in a one-tailed error model per Table 3.37.

Table 3.37: One-Tailed Error Distribution: Mill Feed at 60% Passing 75 Microns

n	Error Term g/t	Upper Conf. g/t	Lower Conf. g/t	Error Term %	n	Error Term g/t	Upper Conf g/t	Lower Conf g/t	Error Term %
2	0,125	1,125	0,875	12,51	12	0,052	1,051	0,948	5,10
3	0,098	1,102	0,907	10,21	13	0,049	1,049	0,951	4,90
4	0,088	1,088	0,912	8,84	14	0,048	1,047	0,952	4,72
5	0,080	1,079	0,920	7,90	15	0,046	1,045	0,954	4,57
6	0,073	1,072	0,927	7,22	16	0,045	1,044	0,955	4,47
7	0,067	1,066	0,933	6,67	17	0,043	1,042	0,957	4,28
8	0,063	1,062	0,937	6,25	18	0,042	1,041	0,958	4,16
9	0,059	1,058	0,941	5,89	19	0,041	1,040	0,959	4,04
10	0,056	1,055	0,944	5,59	20	0,040	1,039	0,960	3,94
11	0,054	1,053	0,946	5,32					

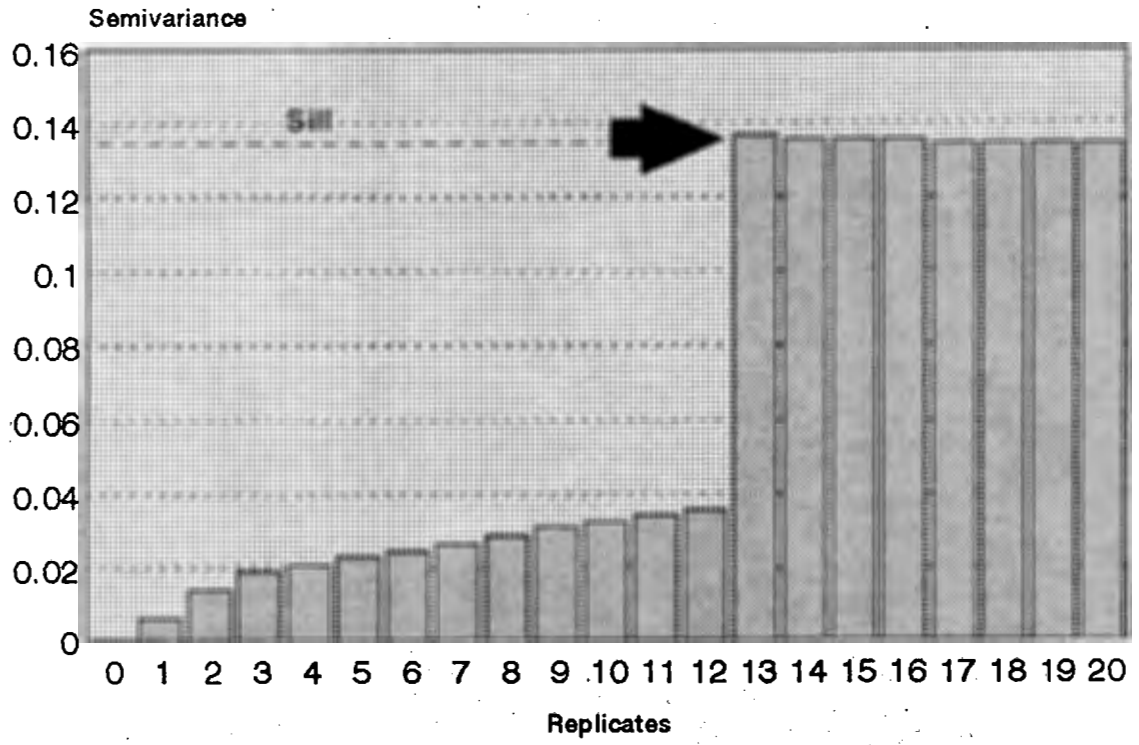


Figure 3.31: Waterval Mill Feed Semivariogram for Replicates

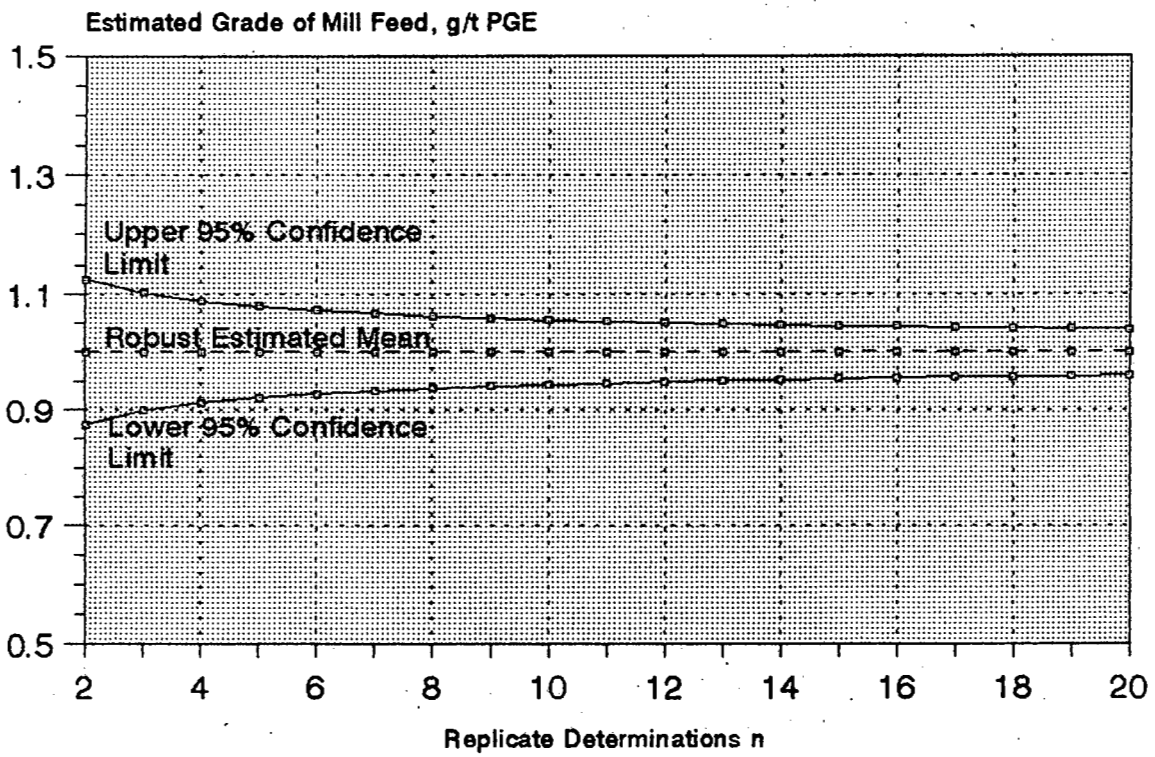


Figure 3.32: Waterval Mill Feed Number of Replicate Determinations for Mill Feed Ground to 60% Passing 75 Microns

The 13 replicates identified by the semivariogram would then equate to a one-tailed error of 4,9% in the estimated mean grade, or 9,8% overall. This is in the same order as the robust 8,8% RSD shown in Table 3.36 for the 95% confidence data limits of the 105 replicates at this grind.

A plot of these confidence limits in the mean with increasing n is shown in Figure 3.32.

It is concluded from this section of the investigation that:

(i) The state of grind of a mill feed sample influences the scatter between replicate determinations, and a minimum standard deviation is attained at a grind of 60% mass passing 75 microns,

(ii) Within this specified grind, rejection of data outide $\bar{x} \pm 2s$ on a robust estimation basis reduces the scatter from ca. 26 to 8% RSD by rejecting less than 5% of the data,

(iii) It is therefore justifiable to establish control limits on mill feed analysis for such a state of grind at RSD <> 8% about the mean,

(iv) A minimum of 13 replicate prills are necessary within these conditions to obtain an accurate estimate of the mean grade. At this point the error limits in the estimate are in the order of 9,8%.

3.2. FLOTATION TESTWORK

3.2.1. Evaluation of Final Concentrate: Waterval High-Grade Concentrate using the ABC Method

The analysis of mill feed sampling with its attendant bias errors having been attended to, attention will now be given to the bias errors of flotation test products. These products contain artificially concentrated forms of known PGE-bearing minerals. Such concentration will accentuate the difficulty of accurate evaluation; accordingly these errors need to be identified, quantified and treated. The procedural treatment of a technical grade sample of Waterval High-Grade Concentrate (§ 2.4.2) yielded the results shown in Table 3.38. A technical grade sample is one taken with due regard for the rules of sampling as set out in § 1.2.3.

Table 3.38
Summarised VFD Data: Waterval High Grade Concentrate

Rel. Grd.[1] g/t PGE	A As is Freq.%	B Pulv. Freq. %	C Panned Freq. %	Rel. Grd.[1] g/t PGE	A As is Freq. %	B Pulv. Freq. %	C Panned Freq. %
1	1,67	0,00	0,0	9	11,67	19,64	33,33
2	1,67	0,00	0,0	10	28,33	8,93	8,33
3	0,00	0,00	0,0	11	21,67	10,71	0,00
4	0,00	1,79	8,33	12	6,67	1,79	0,00
5	0,00	5,36	0,00	13	6,67	1,79	0,00
6	1,67	8,93	0,00	14	1,67	0,00	0,00
7	6,67	21,43	25,00	15	0,00	0,00	0,00
8	11,67	19,64	25,00	16	0,00	0,00	0,00

[1]: Grades are indexed but proportional, in units of 4E (Pt,Pd,Au,Rh).

The basic statistics of these results are shown in Tables 3.39 and 3.40.

Table 3.39
Waterval High-Grade Concentrate: (Technical Sample)
Basic Statistics

Attribute	A As is	B Pulv.	C Panned
Observations	60	56	12
Mean Grade g/t PGE	1,000	0,981	0,973
RSD %	3,06	2,66	2,14
Std. Error of Mean, %	0,39	0,35	0,61

Table 3.40
Waterval High-Grade Concentrate
(Technical Sample)
Subsample Group C: Superpanning

Test #	Metallics Potential % of Total PGE	Test #	Metallics Potential % of Total PGE
1	31,10	6	25,93
2	29,93	7	28,88
3	32,09	8	30,66
4	33,15	9	33,97
5	26,24	10	28,33
6	25,93	11	32,26
7	28,88	12	28,92

Mean Value 30,12
Std. Deviation 2,57
RSD % 8,53

The probability plots of the A and B groups of data are shown in Figure 3.33. Visual inspection of this graph shows that the Group A data (curve AB) show a point of inflection at AA, with a secondary point at BB. The cumulative frequency corresponding to AA is 63,33%, implying that (100-63,33)% = 33,67% of the PGE are present as metallics.

The empirical superpanner test result and the indicated graphical method result for metallics potential on this sample are compared in Table 3.41.

Table 3.41
Comparison of Estimated and Actual
Metallics Potential of
Waterval High-Grade Concentrate
(Composite Sample)

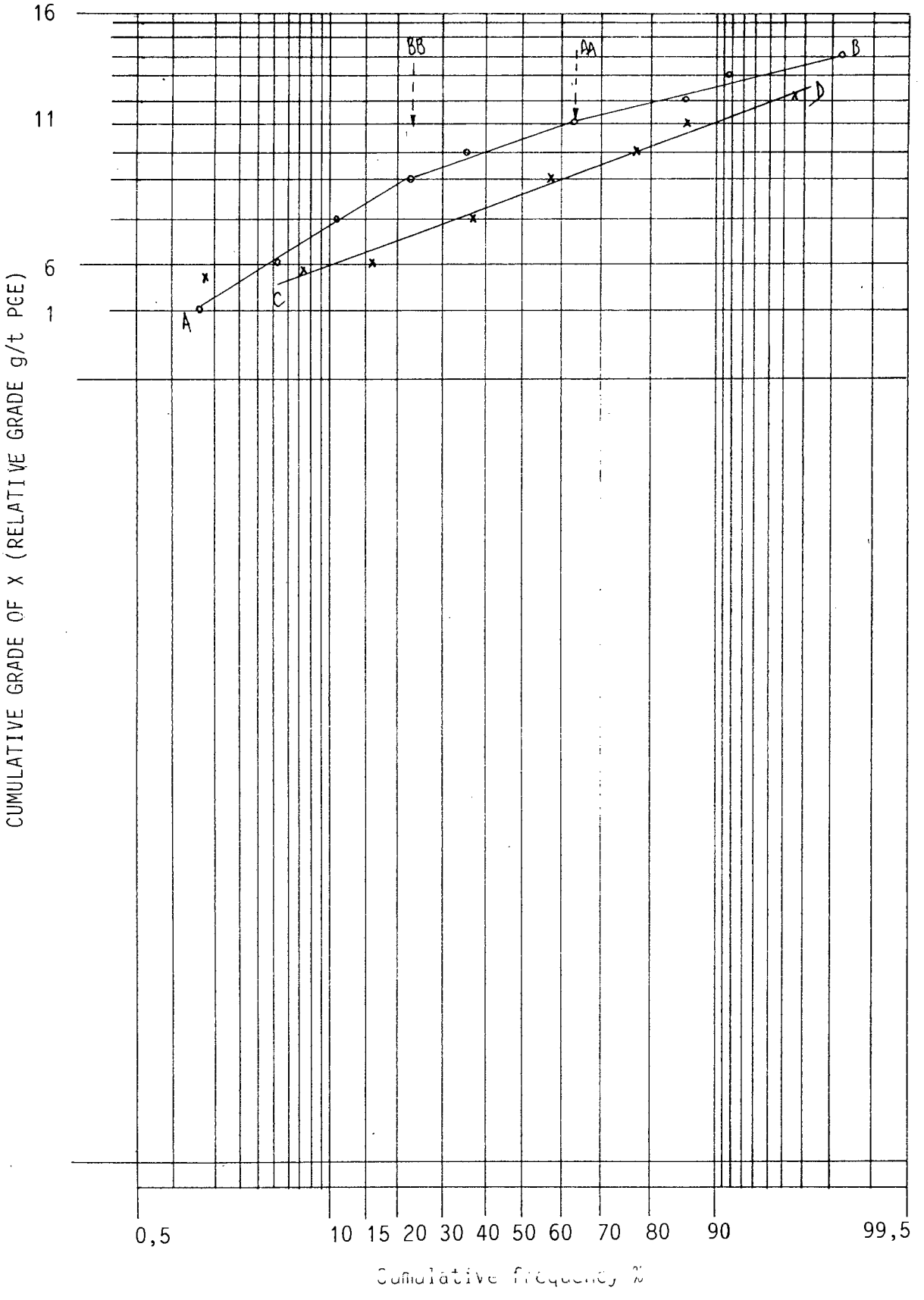
Detail	Metallics Potential % of Total PGE
Empirical (Table 3.40)	30,12
Estimated (Graphical Method)	33,67
Difference gpt PGM+Au	+3,55
Error %	+11,78

Figure 3.33

Waterval High-Grade Concentrate

Probability Plot

Group A and B



The above comparison indicates agreement between the two estimates within 12%. Further exercises on this A-B-C basis are necessary to improve the accuracy using numerical regression methods to identify the points of inflection more accurately are worthwhile listing for future attention. Such methods have been suggested by Clark [Clark, 1977]. Nevertheless it was shown in § 3.1.4 that the error of this estimation method could be as low as 1,56% for Davcra Concentrate; the error may be dependent on the size of the metallics present. This would be logical as the metallics present in final concentrate are residual, i.e. have not been captured by the James Table operation treating Davcra Concentrate. It is known that James Tables do not perform efficiently at fine sizes [Wills, 1987b]. If the total error in this case is ascribed to inefficient superpanning, particularly the very fine sizes, the inferred efficiency of the superpanning operation for this duty would be approximately 88,2%.

The possibility of skewness in the distribution of metallics potentials per Table 3.40 misleading the calculation of mean value was tested by a mathematical exercise using the mean and 95% confidence limits in both normal and Sichel models [Sichel, 1966; Clark, 1993], the results of which are shown in Table 3.42.

Table 3.42
Means and Confidence Limits
Actual Metallics Potentials per Table 3.40
n = 12

Attribute	t Statistics	Sichel Model
Estimated Mean % of PGE	30,12	31,05
Std. Error	0,74	
UCL (95%)	31,60	32,13
LCL (95%)	28,63	30,39

Notes
$\tau_n(V)$ = 1,03427
$\Phi_{(V,n)}$ = 1,0350
$\Phi_{(V,n)}$ = 0,9879

The upper 95% confidence limits of the mean metallics potential for both the t-distribution and the Sichel t-estimator do not include the graphical method figure of 33,67%. The further work suggested by Clark is therefore worth pursuing but beyond the scope of this thesis.

The main purpose of this exercise, being the reduction of scatter between replicates in a high-grade concentrate sample, is best investigated from Table 3.39.

The unprocessed concentrate shows an RSD of 3,06%, with reduction to 2,66% by prior pulverisation in silica, further to 2,14% by superpanning and separate evaluation of superpanner products. The other observation from Table 3.39 is the slightly lower mean grade estimate as a result of these treatments. In particular comparison of the mean grades of A and B groups suggests a difference of 1,9% of total PGE. This would infer that the state of pulverisation or not affects the grade estimate. If so, then the consequence in metal balance deserves investigation. To attest the significance of this observation, the following statistics were extracted using the F and t tests [Box et. al., 1978d]. First the F test, or variance ratio test, compares the scatter between the two sample groups in order to confirm that these are similar enough to warrant comparison of means. This is shown in Table 3.43.

Table 3.43
Waterval High-Grade Concentrate
F Test on Groups A and B

Estimate	Group	
	A	B
Mean Grade	1,000	0,981
n	60	56
Variance	1,37	1,00

Variance Ratio F 1,37

The critical value of F at 59 and 55 degrees of freedom is approximately 1,53 at the 95% level, so the two samples are similar enough in variance to warrant comparison of mean values. The standard error of the pooled data is now calculated in preparation for the t-test. This is done using

$$s^2 = \left(\sum_{i}^{Na} (Xa_i - Xa)^2 + \sum_{i}^{Nb} (Xb_i - Xb)^2\right)/(Na+Nb-2)$$

where

s^2 = the pooled estimate of variance,
Xa = the raw data for group a
Xb = the raw data for group b
Xa,Xb = the respective sample means of groups a and b.

Table 3.44 summarises the salient features of the calculations.

Table 3.44
Waterval High-Grade Concentrate
Calculation of t for Groups A and B

Element	Value
Pooled Estimate of Variance	21,01
Degrees of Freedom	114
Diff. in Means	14,26
Standard Error	3,90
t	3,65

The critical value of t at the 95% level for 114 degrees of freedom is approximately 2,00, so it is concluded that the state of pulverisation reduces the estimated mean grade of Waterval high-grade concentrate by some 1,94%, and that this difference is significant at the 95% confidence level. The effect of this finding on the reconciliation of built-up head grade and assayed head grade may be responsible for bias errors. The calculation of the number of replicates (n) necessary to arrive at a reliable estimate of the mean grade is now performed in a similar manner to that of mill feed. This is done in Table 3.45 for the A and B groups of sample.

Table 3.45
Waterval High-Grade Concentrate
Calculation of Number of Replicates

n	Group A	Group B	n	Group A	Group B
	Error %*	Error %*		Error %*	Error %*
2	4,32	3,76	6	2,50	2,17
3	3,53	3,01	7	2,31	2,01
4	3,06	2,66	8	2,16	1,88
5	2,74	2,38	9	1,93	1,78
			10	1,93	1,69

* One-tailed error

Inspection of this table shows that Group A requires 6 replicates, and Group B, 5 replicates, to arrive at an estimate of $\bar{x}$ within ± 2,5% (5% two-tailed) error.

These convergences are compared in Figures 3.34 and 3.35.

It is accordingly concluded that high-grade flotation concentrates require pulverisation in an equal mass of clean silica prior to analysis (§ 2.4.2); that failure to observe this preparation will result in overstatement of the mean grade of the sample by nearly 2%; and that 5 replicate fusions are necessary to arrive at an estimated mean grade of this concentrate with a two-tailed error of less than 5%.

3.2.2. Evaluation of Final Concentrate: Waterval Low-Grade Concentrate using the ABC Method

The procedural treatment of a technical grade sample of Waterval Low-Grade Concentrate (§ 2.4.2) yielded the results shown in Table 3.45. The basic statistics are shown in Table 3.46. The probability plots of the A and B groups of data are shown in Figure 3.36. Inspection of this graph shows that the Group A data (curve AB) show a point of inflection at 1, with a secondary point at 2. The cumulative frequency corresponding to 1 is 86,2%, implying that (100-86,2)% = 13,8% of the PGE are present as metallics.

Table 3.46
Summarised VFD Data
Waterval Low Grade Concentrate

Grade Group g/t PGE	A As is	B Pulv.	C Panned	Grade Group g/t PGE	A As is	B Pulv.	C Panned
1	1,72	0,00	8,33	15	13,79	8,62	8,33
2	0,00	0,00	0,00	16	6,90	8,62	8,33
3	0,00	1,72	0,00	17	8,62	12,07	25,00
4	0,00	0,00	0,00	18	3,45	13,79	0,00
5	0,00	0,00	0,00	19	1,72	12,07	0,00
6	0,00	0,00	0,00	20	0,00	3,45	0,00
7	3,45	3,45	0,00	21	0,00	5,17	0,00
8	1,72	1,72	0,00	22	0,00	5,17	8,33
9	1,72	0,00	0,00	23	0,00	0,00	0,00
10	1,72	0,00	8,33	24	0,00	1,72	0,00
11	3,45	3,45	8,33	25	0,00	3,45	0,00
12	6,90	5,17	8,33	26	0,00	0,00	0,00
13	27,59	5,17	0,00	27	0,00	0,00	8,33
14	17,24	5,17	8,33				

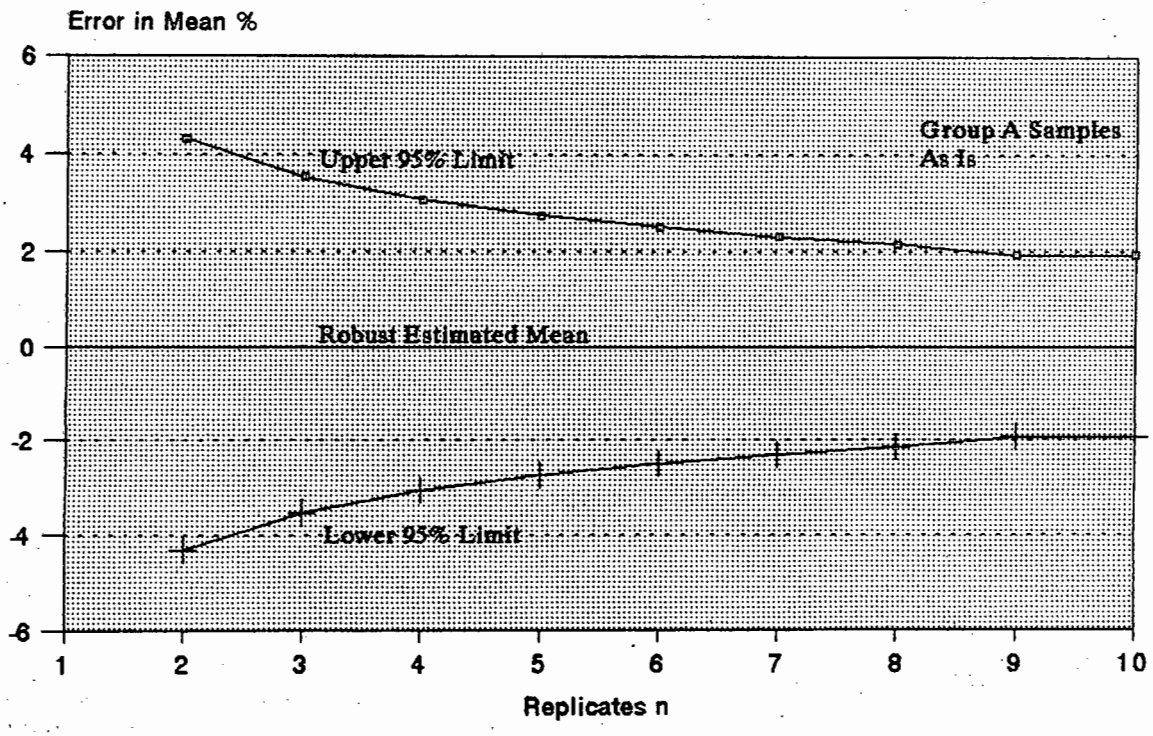


Figure 3.34: Waterval High-Grade Concentrate
Convergence on Robust Mean Grade with Increasing Replicates
Group A: Sample As Is

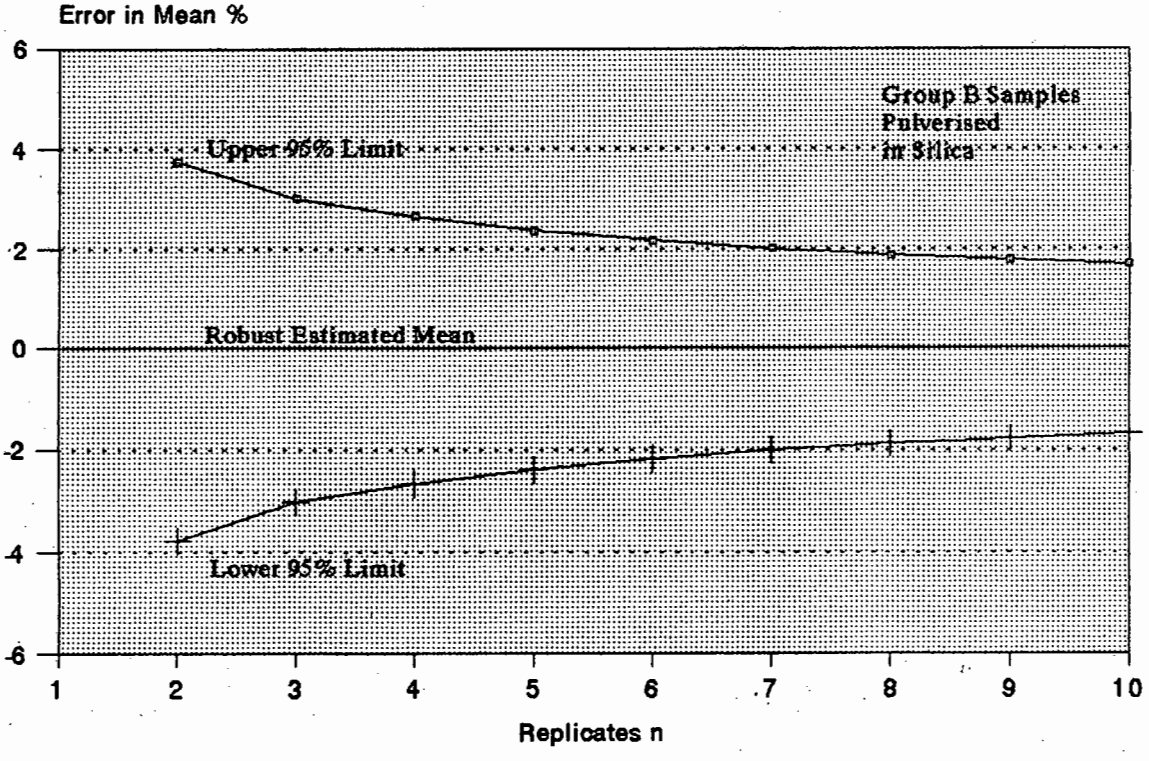


Figure 3.35: Waterval High-Grade Concentrate
Convergence on Robust Mean Grade with Increasing Replicates
Group B: Sample Pulverised in Silica

Figure 3.36

Waterval Low-Grade Concentrate

Probability Plot

Group A and B

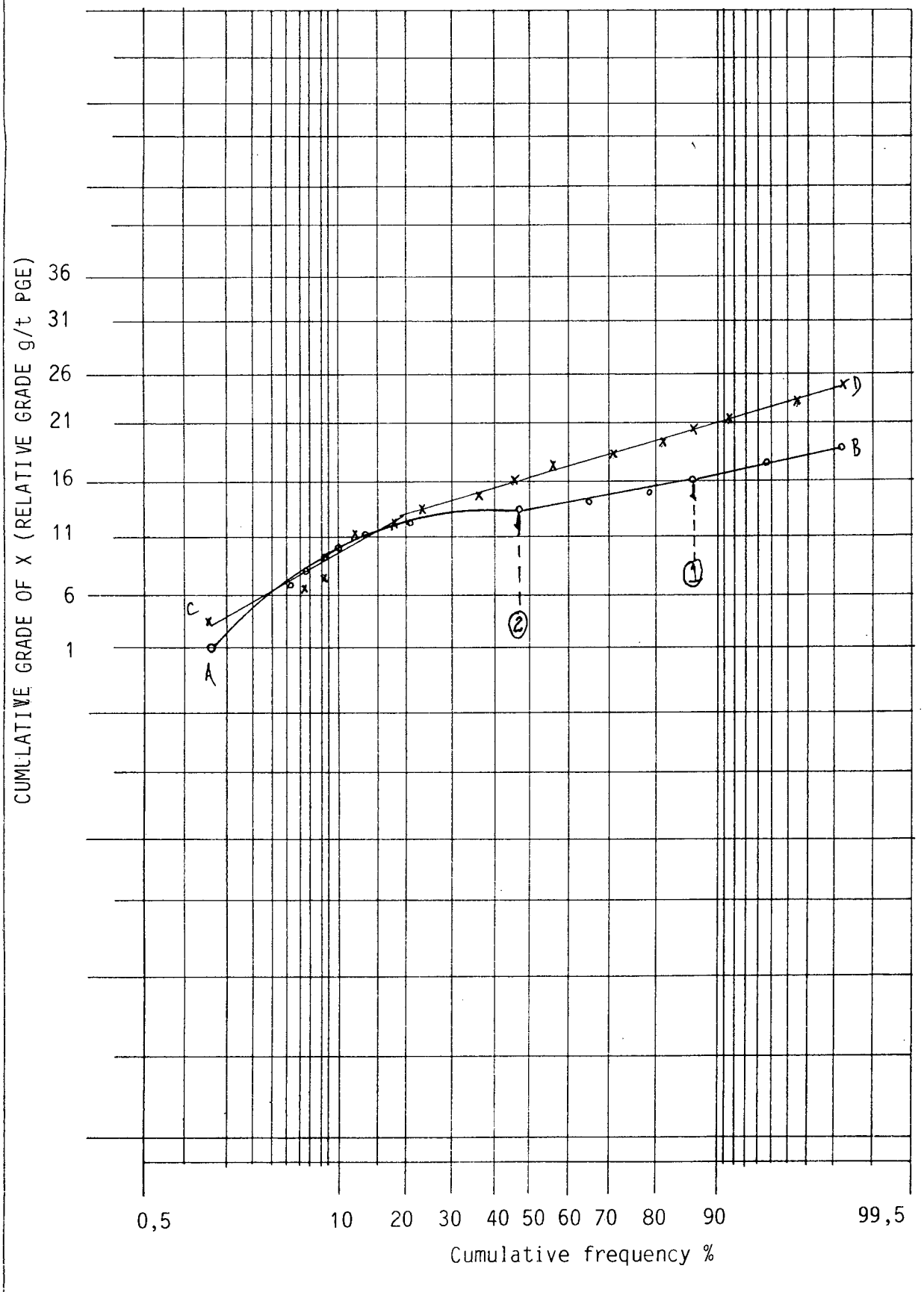


Table 3.47
Waterval Low-Grade Concentrate
(Technical Sample)
Basic Statistics

	As is	Pulv.	Panned
Observations	58	58	12
Mean Grade gpt PGM+Au	1,000	1,017	1,004
RSD %	2,05	2,84	4,69
Std. Error of Mean,%	0,27	0,37	1,34

The graphically determined metallics potential of the Group A sample was 13,8% of total PGE, from Figure 3.36.

The empirical superpanner test result and the indicated graphical method result for metallics potential on this sample are compared in Table 3.48.

Table 3.48
Waterval Low-Grade Concentrate
(Technical Sample)
Subsample Group C: Superpanning

Test #	Metallics Potential % of Total PGE	Test #	Metallics Potential % of Total PGE
1	10,56	7	12,92
2	11,30	8	10,44
3	11,13	9	10,98
4	14,18	10	12,52
5	12,82	11	8,74
6	11,53	12	10,49

Mean Value	11,46
Std. Deviation	1,44
RSD %	12,56
Estimated (Graphical Method)	13,80
Difference % of Metallics Potential	+2,34
Error %	+20,4

Table 3.49
Means and Confidence Limits
Actual Metallics Potentials per Table 3.48
n = 12

Attribute	t Statistics	Sichel Model
Estimated Metallics		
% of Total PGE	11,46	11,47
Std. Error	0,42	
UCL (95%)	12,3	12,36
LCL (95%)	10,62	10,94

Notes
$\tau_n(V)$ = 1,00746
$\Phi_{(V,n)}$ = 1,0775
$\Phi_{(V,n)}$ = 0,9539

The graphically estimated metallics content of the sample does not fall within the 95% confidence limits of the empirically obtained mean metallics content, both from the t-distribution and the Sichel estimator, although as pointed out earlier the method will probably improve when the calculation is supported by ROKE. The graphical method produces a result some 20,4% higher than does the superpanning. The 11th datum of the latter, with a value of 8,74%, would seem to be a dubious member of the test data, however, cannot be rejected as an outlier. The nature of the sample tested, ie recovered from downstream of the regrind circuit, could be the basic cause of the disagreement between the estimates of metallics potential. In this area of the circuit the residual metallics are likely to be very fine-grained with certain attachments to base metal sulphides, impairing the superpanner separation technique. This would underestimate the actual metallics potential of the sample. The main purpose of the exercise is to examine the effect of sample preparation method on the estimation of the mean grade of concentrate. In this vein the mean grades of sections A and B of the Waterval Low Grade Concentrate are compared.

Table 3.50
Waterval Low-Grade Concentrate
F Test on Groups A and B

Estimate	Group	
	A	B
Mean Grade	1,000	1,017
n	58	58
Variance	1,97	1,00

F = 1,97

The critical value of F at 59 and 55 degrees of freedom is approximately 1,53 at the 95% level, so the two samples are too different in variance to warrant comparison of mean values in a simple t- distribution.

The only conclusion to be drawn in this regard is that the pulverisation method produced too much scatter in the data to be accepted as a recommended method of sample preparation at this grade. The linearisation of the probability plot of values was noted, consistent with the previous result in high-grade concentrate. Possibly a repeat of the test at a more advanced level of test design, such as analysis of variance, may yield more certain conclusions. This will be listed in further work.

The calculation of the number of replicates necessary to arrive at a reliable estimate of the mean grade is now performed in a similar manner to that of mill feed. This is done in Table 3.51 for the A group of sample.

Table 3.51
Waterval Low-Grade Concentrate
Calculation of One-Tailed Error
and Number of Replicate Determinations

Replicates n	Group A Sample One-Tailed Error %	Replicates n	Group A One-Tailed Error %
2	2,90	7	1,55
3	2,37	8	1,45
4	2,05	9	1,37
5	1,83	10	1,29
6	1,67		

The above analysis shows that a two-tailed error of less than 5% in the estimation of the mean grade of low-grade concentrate is obtainable with 3 or more replicate fusions, using the sample in an unpulverised form.

The convergence of the 95% confidence limits of the estimated mean grade with increasing replicates on this basis is shown in Figures 3.37 and 3.38.

It is accordingly concluded that low-grade flotation concentrates do not require pulverisation in an equal mass of clean silica prior to analysis; and that 3 replicate fusions are necessary to arrive at an estimated mean grade of this concentrate with a two-tailed error of less than 5%.

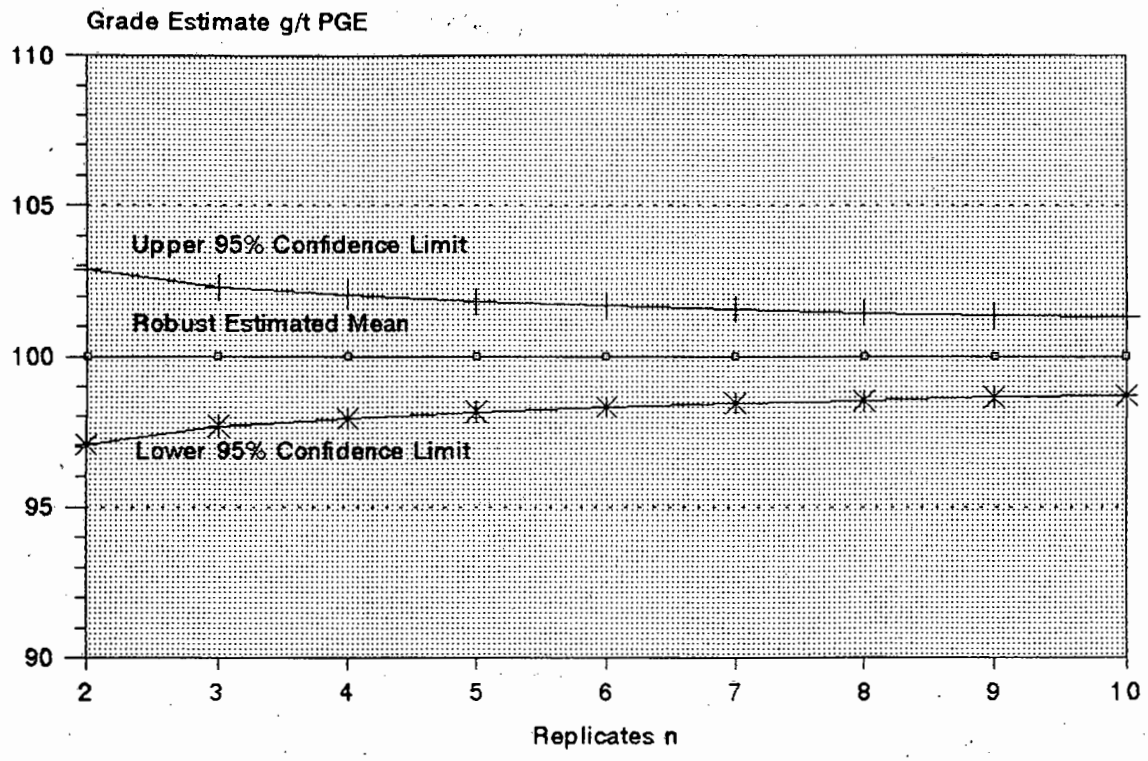


Figure 3.37: Waterval Low-Grade Concentrate
Convergence of 95% Confidence Limits with Replicate Determinations

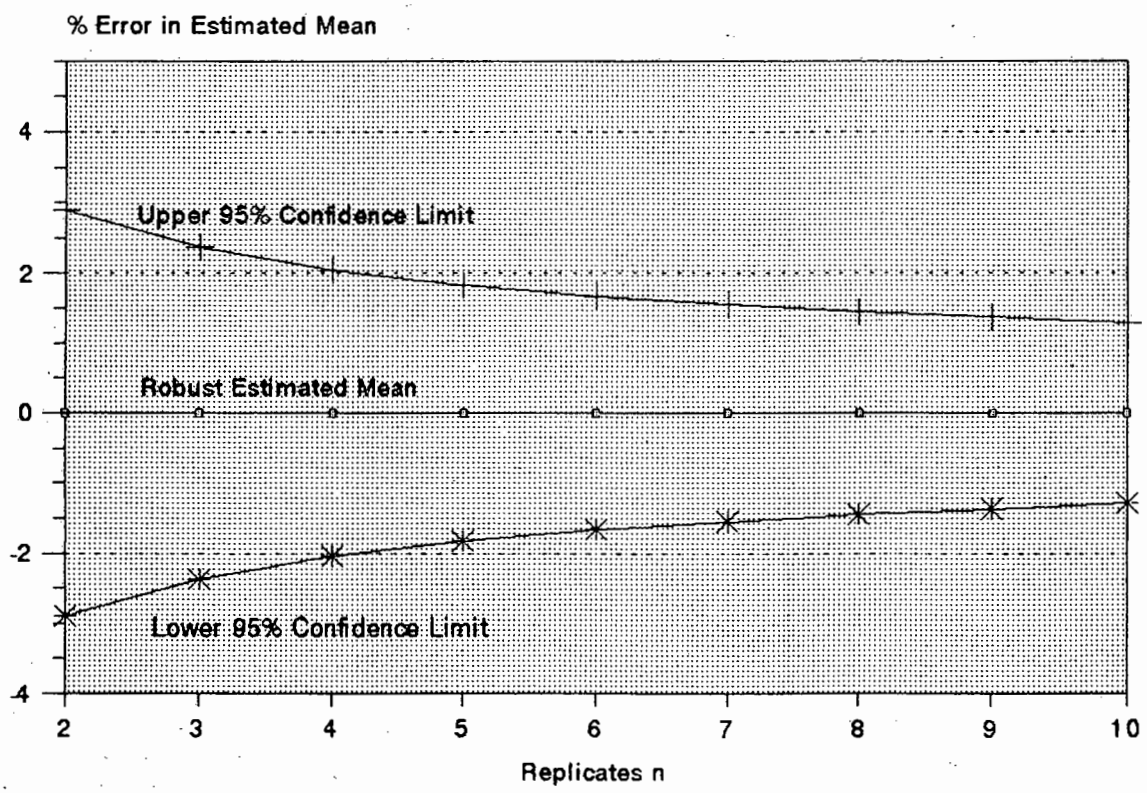


Figure 3.38: Waterval Low-Grade Concentrate
Convergence of 95% Confidence Limits with Replicate Determinations

3.2.3 Evaluation of Tailings: State of Grind, and Number of Determinations

The results of § 2.4.3., examining the evaluation errors inherent in tailings evaluation, are summarised for a technical grade sample of Frank Rougher Tailings in Table 3.52. Data screening was performed at $\bar{x} \pm 2s$.

Table 3.52
Frank Rougher Tailings : Summary of Evaluation Data

Element	State of Grind: % Passing 75 Microns 40	50	60	70
Raw Data				
Mean	0,876	0,920	0,954	0,966
RSD %	20,4	14,7	20,0	18,8
n	168	135	103	135
Screened Data				
Mean	0,907	0,936	0,991	1,000
RSD %	14,2	9,3	12,9	11,4
n	158	127	97	126

Inspection of this table suggests that the state of grind of the tailings sample has an effect on the estimated mean grade, further that the screening of data outside two robust standard deviations and recalculation of the mean using screened data increases the estimated mean grade. Assuming that the grade estimate is converging between the two grinds 60 and 70% passing 75 microns, the 95% confidence limits of the estimated mean may be calculated as before using the t-distribution. These are shown in Table 3.53.

Table 3.53
Frank Tailings : Evaluation at 70% Passing 75 Microns
Calculation of Number of Replicates

Replicates n	Error %	Replicates n	Error %
2	15,9	6	9,2
3	13,0	7	8,5
4	11,3	8	7,9
5	10,0	9	7,5
		10	7,1

The value frequency distributions of these data are shown in Figures 3.39-42.

Inspection of these figures indicates a negative skewness in the distributions, not noted in any of the other VFD studies for chip sampling of ore or mineral process streams.

A possible explanation for this observation is sought in either or both of the following:

(i) Incomplete fusion of sample during lead collection, possibly as a result of coarse material in the sample reacting more slowly with the melt, leaving unaccounted PGE losses entrained in the fusion slag. A similar effect would be noted if the fusion temperature was too low,

(ii) The mineralogical likelihood that the tailings contain most of the unfloatable PGE of the ore treated. In such circumstances the explanation would only be valid if the PGE tenor in silicate solid solution is much lower than for pentlandite or pyrrhotite. Refer § 1.2.

For either reason, the negative skewness potentially affects the estimation of the mean grade of the sample when small data sets are used. The latter are not robust to this skewness and will underestimate or overestimate the mean according to which of (i) or (ii) is true.

The effects of grind on estimated mean tailings grade, and of increasing replicate determinations on the converging 95% confidence limits of the estimate, are shown in Figures 3.43 and 3.44 respectively.

An analysis of variance between the measurements of grade at each grind of tailing sample was performed [Box et. al., 1978e], yielding an F value of 10,75, concluding with a probability of better than 99% that the grade estimate increases as a result of the finer grind.

Accepting that the tailings sample must be milled to a known degree of fineness prior to analysis, inspection of the 70% grind data still shows a negative skewness. This is the reason for the high standard deviation and must form part of the diagnosis of improved resolution between replicate determinations.

It is reasoned that incomplete fusion cannot be playing a significant role in this skewness at the finer grinds. The analysis of the sample by grind showed that the mean grade increased only between the 40, 50 and 60% grinds, whereafter the increase as a result of the 70% grind was not much more. In other words, the increment in grade estimation per grind increment converges to a limit as the correct state of grind is approached.

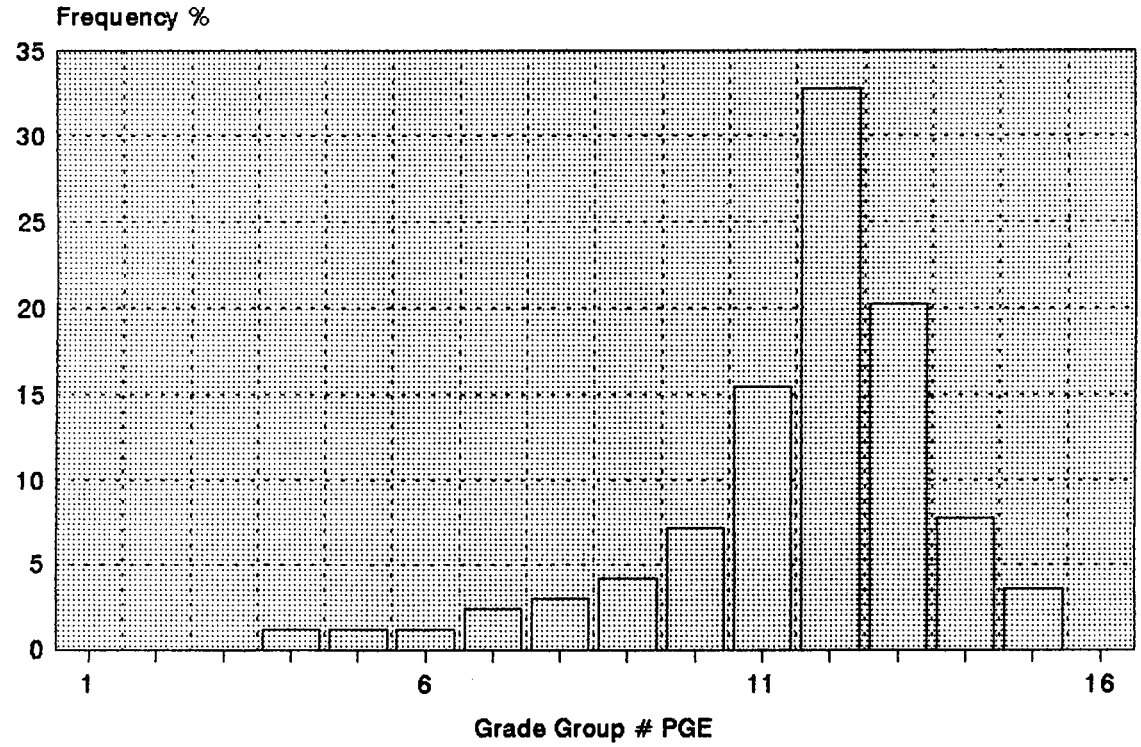


Figure 3.39: Frank Rougher Tailings
Value Frequency Distribution: 40% Passing 75 Microns

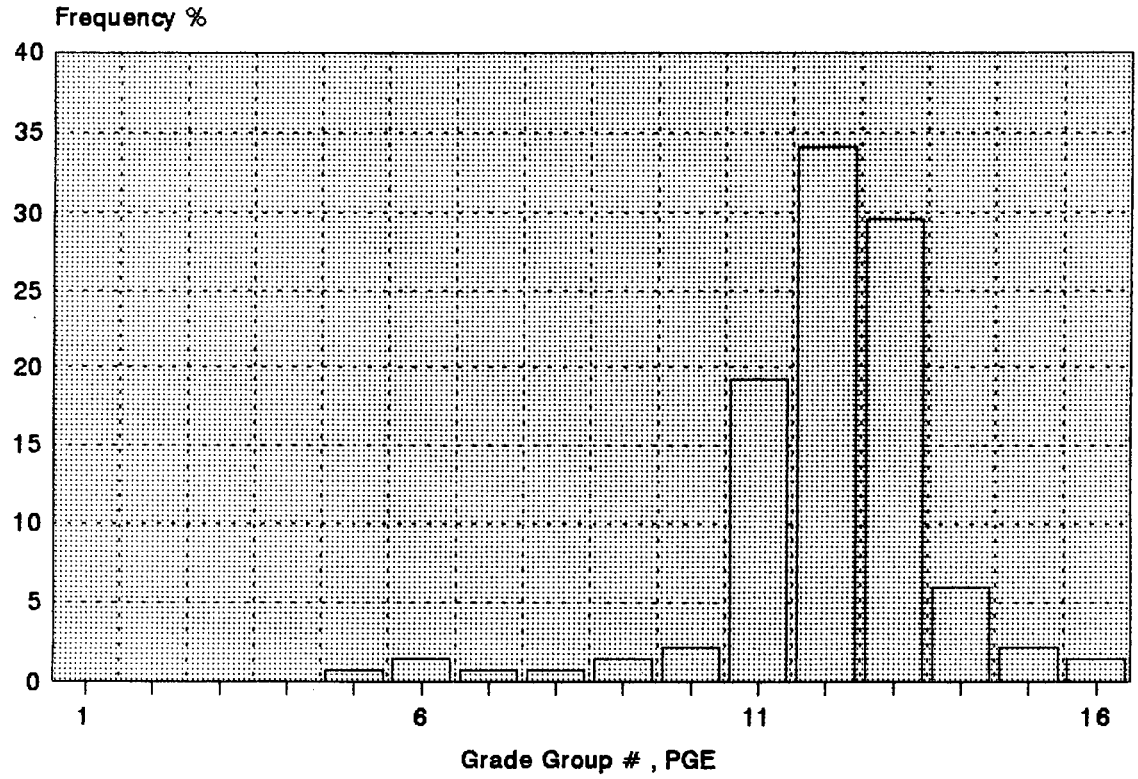


Figure 3.40: Frank Rougher Tailings
Value Frequency Distribution: 50% Passing 75 Microns

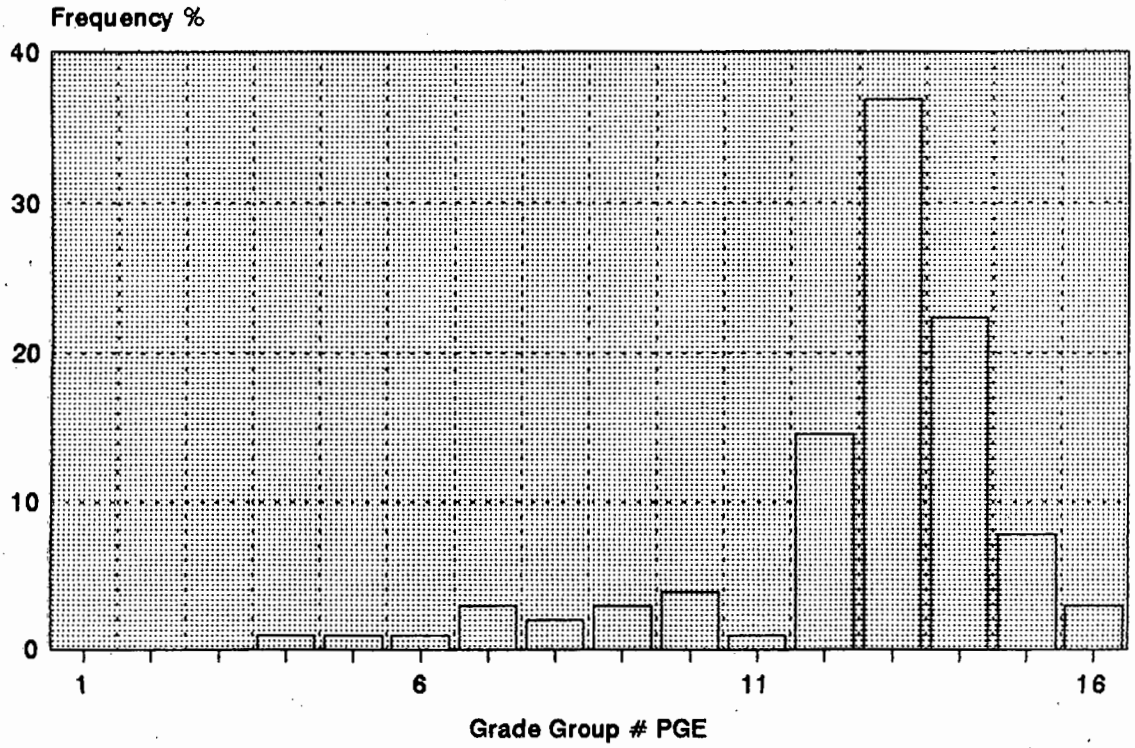


Figure 3.41: Frank Rougher Tailings
Value Frequency Distribution: 60% Passing 75 Microns

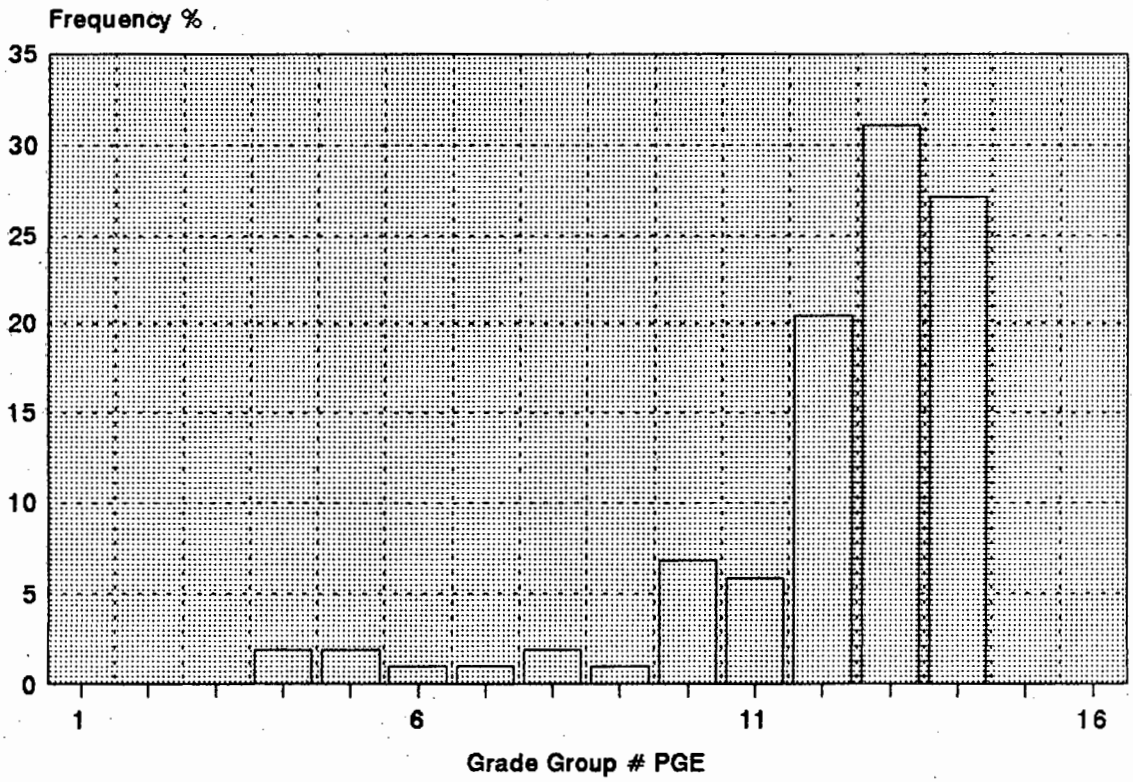


Figure 3.42: Frank Rougher Tailings
Value Frequency Distribution: 70% Passing 75 Microns

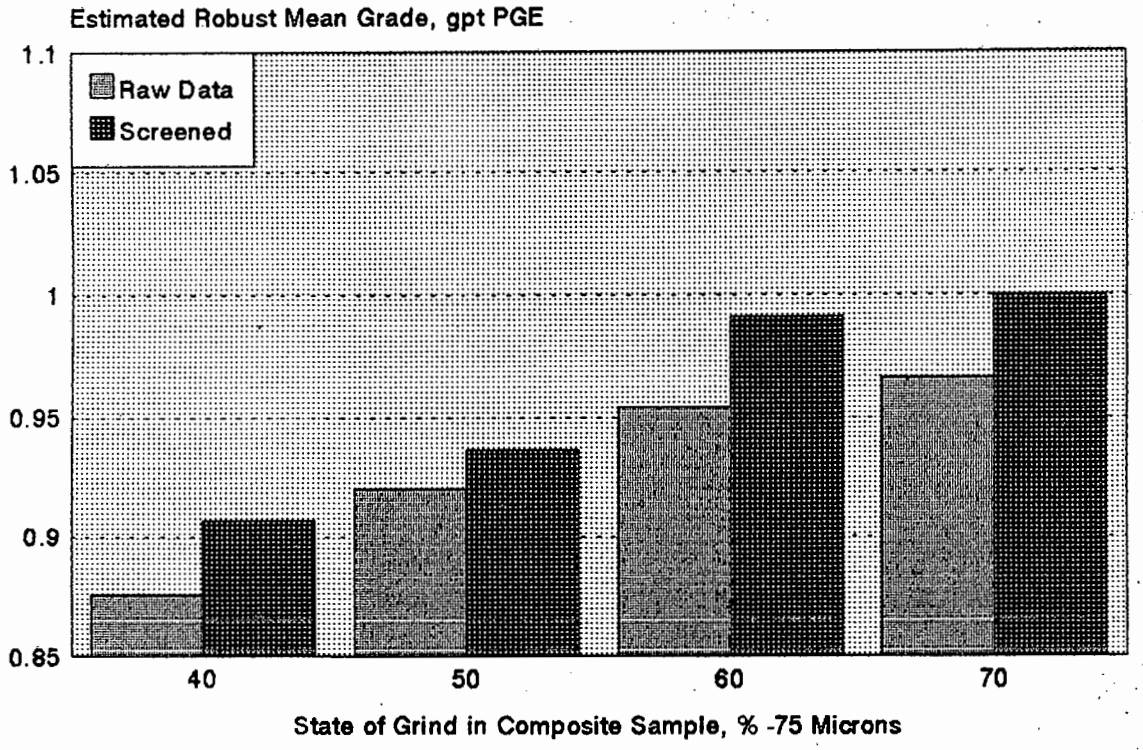


Figure 3.43: Frank Rougher Tailings
Effect of Grind on Grade Within Composite Sample

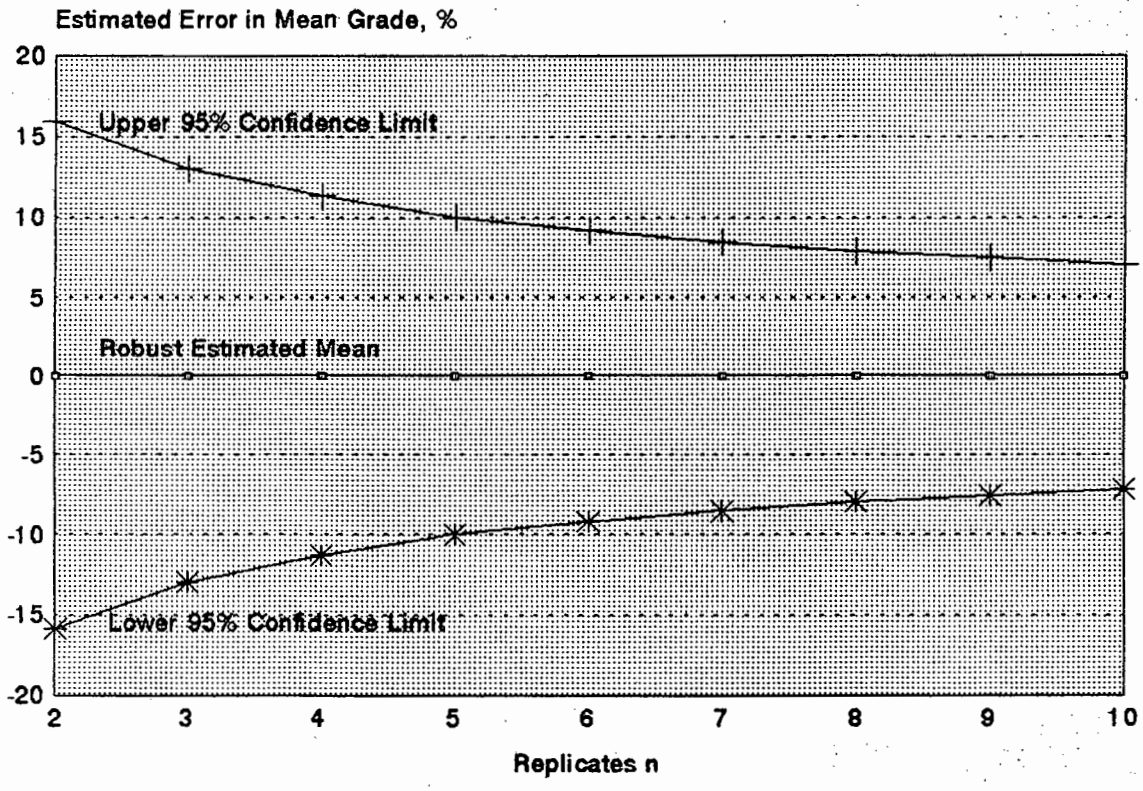


Figure 3.44: Frank Rougher Tailings
Convergence of 95% Confidence Limits With Replicate Determinations

An understanding of the shape of the distribution at 70% passing 75 microns enlightens this argument. The VFD of these data are shown in Table 3.54, and the probability plot, in Figure 3.45.

Table 3.54
Frank Rougher Tailings: 70% Passing 75 Microns
Value Frequency Distribution

Grade Group gpt PGE	Freq. %	Cum Freq. %
1	0	0
2	0	0
3	0	0
4	1,94	1,94
5	1,94	3,88
6	0,97	4,85
7	0,97	5,83
8	1,94	7,77
9	0,97	8,74
10	6,80	15,53
11	5,83	21,36
12	20,39	41,75
13	31,07	72,82
14	27,18	100,0

Examination of Figure 3.45 shows that the raw data, AB, divide into two subdistributions CE and BD about a point of inflection marked 1. The cumulative frequency at 1 is 11,0%.

The medians and limits of these distributions are summarised in Table 3.55.

Table 3.55
Frank Rougher Tails
Subdistribution Parameters

Parameter	Subdistribution CE	BD
Median g/t	5,0	1,15
99,5% Limit	106	1,60
0,5% Limit	0,16	0,80

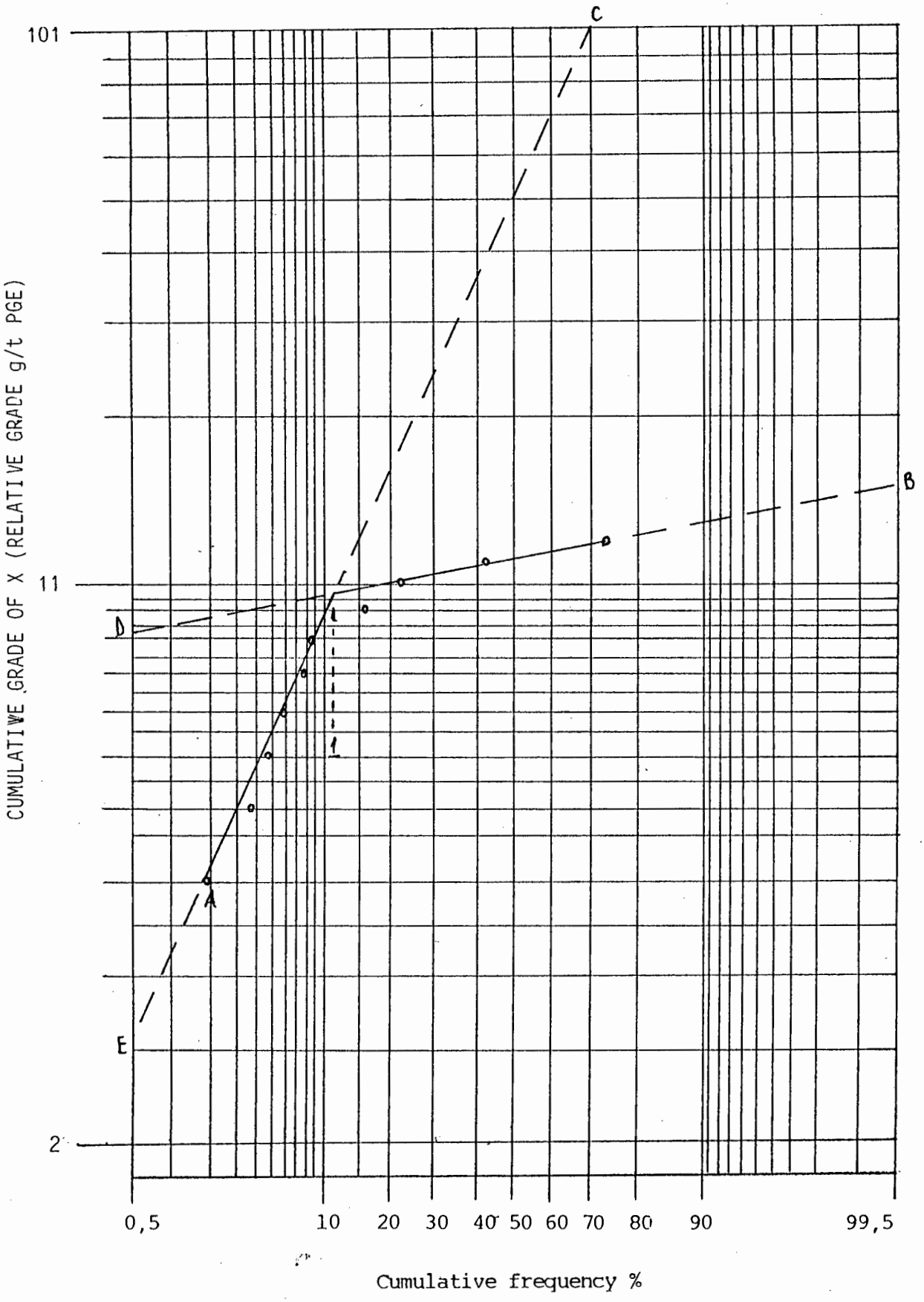
Lognormal Probability Plot

Figure 3.45

Frank Rougher Tailings

Probability Plot at 70%

Passing 75 microns



Two observations stem from this analysis:

(i) Subdistribution CE must be the PGE associated with incompletely recovered sulphides, since the median grade is equivalent to mill feed grade, and the upper limit of the distribution is equivalent to low-grade concentrate,
(ii) Subdistribution BD must be PGE in some other mineralogical form, possibly silicates and oxides, since the upper limit does not equate to mill feed grade.

It can be argued that the tailings from a sulphide flotation process treating Merensky ore should contain less PGE associated with sulphides than PGE associated with silicates and oxides, since by design the process is recovering the former, albeit imperfectly, by flotation. Therefore the point of inflection at 1 in Figure 3.45 is reflecting the population of unrecovered sulphide-PGE as well as their parent solid solution tenor, which appear to have a cleaning potential of 106 g/t PGE. By this it is meant that should these unfloated sulphides be retreated in a release analysis test to clean and reclean them to their near-pure mineral form, the highest grade attainable would be 106 g/t PGE. This interpretation is consistent with the use of probability plots in that the higher gradient plot is associated with higher grade and variance [Rendu, 1981].

In terms of practical diagnosis of replicate fire assay fusions, however, the chemist is presented with the problem of attesting the attributes of likeness between four to six values from a sample. Here a lower rejection limit genuinely resulting from incomplete fusion must be defined, and a figure of 0,16 g/t PGE is suggested from Table 3.54. The acceptable scatter between replicate fusions cannot be as tight as found in earlier mineral process samples for this reason. Examination of the t-distribution in Table 3.52 shows that the distribution of errors converges at approximately 7% at 8-10 fusions. A two-tailed error of 14% results in this section of the evaluation.

Further argument for this interpretation is made from the fact that subdistribution CE fits into a definite pattern on the probability plot. If these values were produced as a result of incomplete fusion, it could not be expected that they would fit a pattern but would be more random.

It is accordingly concluded that more scatter between replicates in tails samples should be allowed than is the case for concentrates and mill feeds; that the VFD of rougher tailings is bimodally distributed, and that such samples should be milled to 70% passing 75 microns prior to analysis, where 8-10 replicate fusions be performed. A lower rejection limit of 0,16 g/t PGE is suggested.

3.2.4. The 30 Replicate Flotation Tests

The results of tests conducted per § 2.6 were first gathered in terms of mass balance. These are summarised in Table 3.56.

Table 3.56
30 Replicate Flotation Tests
Mass Balance Data
(n = 30)

Detail	Mean g	Std Dev. g	Mass %
Conc 1	105,1	5,48	4,22
Conc 2	133,7	5,63	5,37
Tails	2249,4	6,99	90,40
Built-up Head	2488,2	2,14	100,0

The mean mass balance, based on the weighed 2500g of mill feed per test, therefore shows a nett loss of -11,8 g or -0,47%. The distribution of this error is shown in Table 3.57. It is reasoned that the consistent mass loss must be a result of filtration losses during handling of the flotation test products, in particular the concentrates as these carry enriched quantities of friable base metal sulphides. The latter are prone to ultrafine breakage from milling and the choice of filter paper may have been too porous.

Table 3.57
Distribution of Mass Loss
30 Replicate Flotation Tests

Mass Loss Group %	Freq. %	Cum. Freq. %	Mass Loss Group %	Freq %	Cum Freq%
-0,65	0	0	-0,45	33,33	66,66
-0,60	10,00	10,00	-0,40	10,00	76,66
-0,55	10,00	20,00	-0,35	10,00	86,66
-0,50	13,33	33,33	-0,30	13,33	100,00

An alternative explanation for the consistent mass loss of -0,47% could be the residual moisture present in the "dry" mill feed at the time of weighing. Production mill feed commonly has a moisture content of ca. 5%, and with the climatic conditions that occur in Rustenburg the dried sample might retain 0,47% moisture.

A test of which of the two factors is causing this loss might be seen in the metal balance. If an unaccounted loss of PGE occurs, then the loss of mass must be in filtration. If a reasonable metal balance is obtained, then the error must be in residual moisture present in the mill feed sample at weighing the 2500 g sample prior to flotation testwork. The reconciled, or built-up, head grades from these replicate flotation tests produced the following basic statistics:

Table 3.58
30 Replicate Flotation Tests
Built-up Head Grades

Detail	Units	Raw Data Value	Screened Data Value
Mean BUH	g/t PGE	1,018	1,010
Std. Dev.	g/t PGE	0,066	0,051
RSD	%	6,55	5,06
Std. Err.	g/t PGE	0,012	0,009
n		30	29

The 95% confidence limits of the raw data are $\overline{x} \pm 2s$, so the rejection limits about this estimated mean are 1,149 and 0,884 g/t. Inspection of the raw data in Table 3.59 shows one outlier at 1,214 g/t (test 26). Rejection of this datum and recalculation of the mean and standard deviation is shown in Table 3.58 as screened data. The concentrates in these tests did not require pulverisation in silica, as their grades were not high enough to warrant this treatment. The tailings, however, were already at a grind of 60% passing 75 microns so further grinding prior to analysis was unnecessary.

The number of flotation tests required to produce an estimated mean grade of ore with the defined error in the estimate was calculated using the t-distribution as before. The results are shown in Table 3.60.

This convergence is shown in Figure 3.46, and indicates that 5 tests are necessary to produce an estimated mean built-up head grade with ca. 5% one-tailed error. This would equate to 90% confidence limits level in the mean of ± 0,05 g/t PGE.

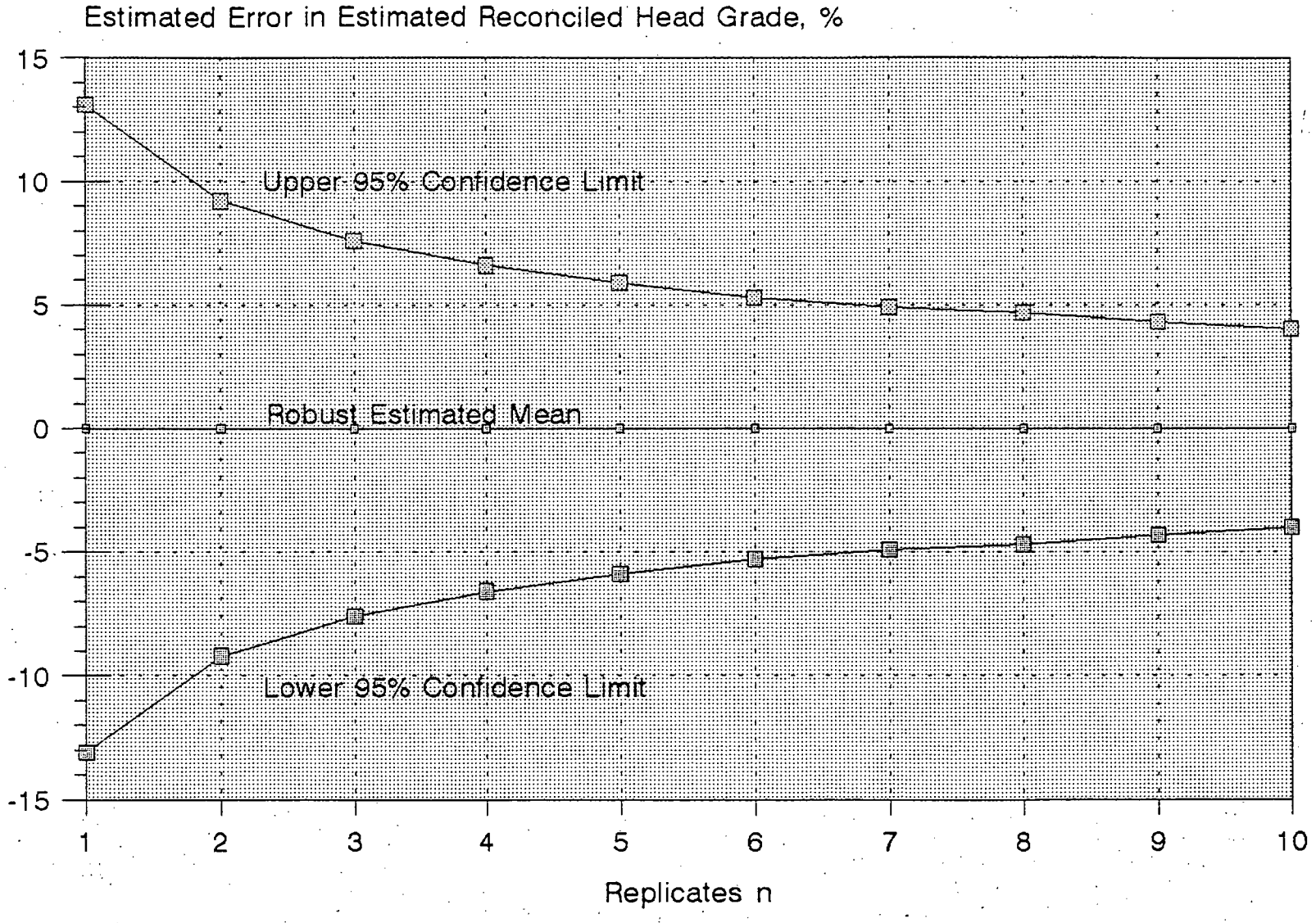


Figure 3.46: Replicate Flotation Tests: Convergence of 95% Confidence Limits With Replicate Determinations

Table 3.59
30 Replicate Flotation Tests
Raw Data: Built-up Head Grades

Test #	g/t PGE	Test #	g/t PGE	Test #	PGE
1	0,981	11	0,914	21	1,025
2	1,030	12	0,951	22	0,924
3	0,999	13	0,965	23	1,206
4	1,167	14	0,864	24	0,902
5	0,964	15	1,032	25	0,945
6	0,965	16	1,046	26	1,214
7	1,013	17	0,993	27	1,023
8	0,972	18	0,930	28	0,951
9	0,936	19	0,959	29	1,044
10	1,054	20	0,961	30	1,068

Table 3.60
Calculation of the Required
Number of Replicate Flotation Tests
(Using All Raw Data)

Replicates n	Error %
1	13,1
2	9,2
3	7,6
4	6,6
5	5,9
6	5,3
7	4,9
8	4,7
9	4,3
10	4,0

It will be recalled that the equivalent number of replicates necessary in assay heads to produce this result was 13, reflecting the ability of the flotation test to attain accurate statements of grade more readily than the direct analysis of mill feed. This is to be expected, as the flotation test is artificially homogenising the PGE- bearing minerals by selective recovery of PGM, and of PGE in base metal sulphides, to a concentrate, making the task of evaluation easier.

Statistically this is because the flotation test is separating the PGE from mill feed into more homogeneous subsamples that are easier to evaluate, a procedure called stratified sampling [Hunt, 1994].

On this basis, viz. that the built-up head grades from the flotation tests require only 5 replicates to arrive at an estimated mean grade within 5% error, the raw data from Table 3.58 were averaged in blocks of 5, in the order observed, to arrive at a short set of simulated built-up head grades that would result from compositing quintuplicate replicates prior to evaluation. These results are shown in Table 3.61.

At this stage of assessment the 95% confidence limits of the robust mean built-up head grade must be compared to the above estimates in order to validate the calculation that quintuplicate replicates are necessary.

Table 3.61
Averaged Sets of 5 Replicates
Flotation Tests

Set #	Sets of 5 Mean g/t PGE	Assayed Head g/t PGE
1	1,020	
2	1,020	
3	0,975	
4	1,009	
5	0,991	
6	1,094	
Data Mean	1,018	1,00
Std. Dev.	0,041	0,0647
RSD%	4,01	6,47
UCL	1,075	
LCL	0,974	

The upper and lower confidence limits are drawn from the robust population (Table 3.57). Data set 6 contains the outlier value of 1,214 g/t PGE, which was rejected at the 95% data confidence limits in the raw data. It will be further noted that the RSD of the composited quintuplicates is less than 5%. It will also be noted that the RSD of the composited quintuplicate built-up head grades is 3,13%, lower than the 6,71% of the individual data. This is a characteristic of averaging in terms of the Central Limit Theorem [Box et. al., 1978a].

To conduct the PGE metal balance, subsamples of the same ore sample used in the flotation test were milled to a grind of 60% passing 75 microns and blended using the spinning riffler odds and evens method to prepare a reference distribution of assay heads. This procedure deliberately took advantage of the findings of § 3.1.6. A total of 50 assay subsamples were presented for analysis. The results are shown in Table 3.62.

Table 3.62
Reference Distribution
Assay Head Samples

Detail	Units	Raw Data Value
Mean Grade	g/t PGE	1,000
Std. Dev.	g/t PGE	0,065
n		50
Std. Error	g/t PGE	0,009
RSD	%	6,49
UCL (95%)		1,129
LCL (95%)		0,871

The PGE metal balance is thus the mean built-up head divided by the mean assayed head, expressed as a percentage. From Table 3.61 this value is 101,8 %, indicating that loss of concentrate during filtration cannot be occurring to any significant extent.

It is accordingly concluded that replicate flotation tests produce a mass balance with a mean loss of -0,47%, and that the loss is likely to be error in moisture content of "dry" ore. This loss is insignificant as the metal balance shows an unaccounted gain of +1,8%. Such a balance is regarded as acceptable for initial results.

3.2.5. Trial of Sichel's t-Estimator on Results

The 30 replicate flotation tests described in § 3.2.4. were analysed in terms of the Sichel t-estimator. The same was done to the replicate assay head samples. The results are summarised in Table 3.62.

Table 3.63
Summary of Sichel t Estimator Results
Replicate Flotation Tests

Detail	Sample Basis Floats BUH	Assay AH (1)	Assay AH (2)
Observations n	6	7	49
Sichel t	1,019	1,000	1,000
UCL (95%)	1,033	1,006	1,008
LCL (95%)	1,013	0,998	0,994

$r_n(V)$	1,00065	1,00024	1,002
$\phi(V,n)$	1,0070	1,0051	1,008
$\phi(V,n)$	0,9938	0,9978	0,9939

Notes:

1. Blocks of 7 duplicate fusions
2. Blocks of duplicate fusions

A call factor of 101,9% is found from these mean grades.

It is also noted that the 95% confidence limits of the assayed and built-up head do not overlap at the upper and lower limits respectively. This suggests a problem with the data and precludes reporting of recovery until the heads reconcile.

Comparison of the 95% confidence limits about the Sichel t value of built-up head grade reveals an important feature: the upper 95% limit (1,033 g/t PGE) suggests that test 6 (1,094 g/t PGE) falls outside the confidence limits of the estimate and should accordingly be rejected from the calculation of the mean grade. Equivalent argument at the lower confidence limit of 1,013 g/t PGE rejects test 3 (0,975 g/t PGE), test 4 (1,009 g/t PGE), and test 5 (0,991 g/t PGE). This matter will be discussed further in § 4.2.

Alternatively the small difference in grades may stem from another error source in sample preparation or analysis not yet identified in the present investigation. Further work in this regard may be profitable.

4. DISCUSSION

4.1. MILL FEED SAMPLING

Reflection on § 3.1 shows that large samples are needed for reliable study material, notwithstanding the fact that the ore is a crushed production stream with d80 = 5 mm. Although it was expected from the Gy and Bartlett models that the topsize would determine the order of magnitude of the sample mass, further that the 5 mm d80 value would reduce M_s considerably, for a 10% fundamental variance the various M_s models estimated 87,45 (Gy), 96,62 (Laplante) and 170,3 (Bartlett). As size-by-size variance is not included in the Gy version of M_s, it should be expected that M_s will be underestimated since the data in Table 3.31 clearly show that the various sizes carry markedly different quantities of PGE. In particular the fine size -600 micron affected the calculation.

The consequences of underestimating M_s from the above argument are shown using the Bartlett model in Figure 4.1. Inspection of this figure shows that large variances in the primary sample stem from such underestimation of sample mass. The practice over several years from heuristics has been to start with a primary sample in the order of 500-600 kg, which locates the investigation in a more favourable variance area.

The M_s values for crushed Merensky are still manageable compared to run-of-mine sampling. Other calculations suggest for such a size of material the order of M_s is 1-2 tons [Kloppers, 1994], so the convenience of working on a ball mill feed is clear.

For practical purposes the sampling of ball mill feed of this size at 500 kg per batch will assure a fundamental variance less than 4%. Such a quantity of ore will provide 200 subsamples of 2,5 kg at laboratory scale, of which some 50 will provide the basic charcteristic data for internal reference purposes, leaving 150 for experimentation. This is convenient especially if factorial designs are the investigation format, since advice by suitable reference [Box et al, 1978f] suggests that the most perfect test design is the one calculated after all the testwork is complete.

In cases of borehole drill core, the problem enters a different domain. At the density noted (3,1 g/cc), a 50 mm diameter drill core will yield some 6,09 kg of ore per metre. The minimum mass necessary for a standard flotation test would be 20 kg, or 3,28 m of core. The fundamental variance of the sample is indeterminate at this stage, but using the crushed mill feed as a start, this would be ca. 43%.

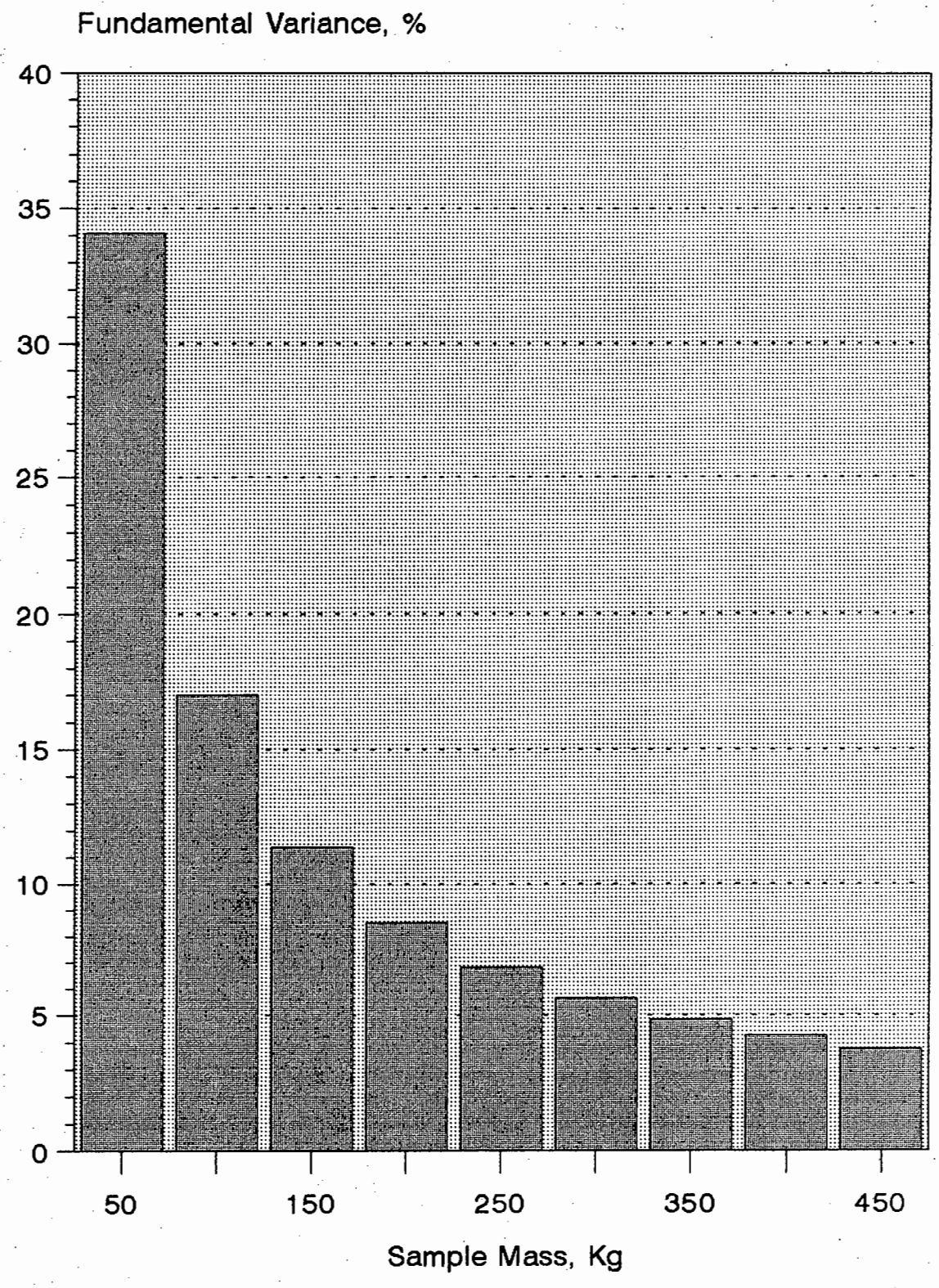


Figure 4.1: Waterval Mill Feed
Effect of Sample Mass on Sample Variance (Bartlett and Hawkins Model)

This problem diminishes once a pilot shaft has been sunk, in which case one may revert to the above mill feed model. This area will be worthwhile further investigation as drill core is at times the only study material available for an ore reserve estimation.

4.2. FLOTATION TEST PRODUCTS

Reflection on § 3.2.4 suggests that the unaccounted gain, although minor, separates the confidence limits of the assayed and built-up, or reconstituted, head. Ideally these should merge. An overview of the tailing analysis showed bimodal distribution which would not respond to correction by sample preparation. The chemistry practice of tailings analysis is to reject low-grade prills as these do not appear to fit the main pattern of fusions.

It is worthwhile, therefore, to reconstruct the metal balance of the 30 replicate floats using a recalculated tails value for each test. Such a recalculation will use all the observations > 0,16 g/t PGE; recall that this figure is the defined 0,5 % cumulative frequency limit of the distribution.

These results are shown in Table 4.1.

Inspection of Table 4.1 shows that a similar mean built-up head grade results from this recalculation as from the original data. It is concluded from this comparison that the unaccounted gain cannot lie with the tailings analysis, rather an examination of the justifiable expectations of replicate built-up heads will reveal the presence of an outlier that is causing overestimation of the mean BUH grade.

The heuristic model [Lotter and Munro, 1993] developed the practice of outlier rejection in small data sets on the basis that the sample standard deviation, or crude standard deviation when an outlier is present, would overestimate the standard deviation in the presence of an outlier, and rejection of replicates at one crude standard deviation would identify such an outlier and halve the scatter in the short data set.

Such a standard deviation is calculated in the normal manner but the Bessel Correction is used to modify the estimate of s as small data sets underestimate this parameter [Moroney, 1975b].

Table 4.1
Recalculation of Built-Up Heads
Using Raw Tailings Data

Detail Quintuplicate Floats	Original BUH	Recalculated BUH
1	1,019	1,020
2	1,020	1,022
3	0,975	0,974
4	1,009	1,010
5	0,991	0,994
6	1,094	1,096
Data Mean	1,019	1,019
Std. Dev.	0,041	0,042
RSD%	4,01	4,09

Taking the grouped raw data from Table 3.63, this calculation is performed as follows:

Quintuplicate #	BUH
1	1,020
2	1,020
3	0,975
4	1,009
5	0,991
6	1,094
Mean	1,014

Corrected Standard Deviation = 0,041

Thus the initial heuristic rejection limits about the sample mean 1,019 are 1,060 and 0,978 g/t PGE. Accordingly the observation 1,094 g/t (test 6) is rejected; as test 6 is known to have an outlier replicate in its composition (the proof was shown using robust statistics at 2 standard deviations), the heuristic model has accurately identified the outlier, and the shortened data set has a reduced sample standard deviation. The low head grade in test 3 is a

borderline case; past heuristic practice was to accept these in the data. Of further interest is the new metal balance; the short BUH data set now has a mean of 1,002 g/t PGE opposite the robust assayed head mean of 1,000 (Table 3.60), giving a metal balance of 100,2 %. It is reasoned that the 1 % unaccounted gain in the initial quintuplicate data had its origins in the inclusion of an outlier relicate at compositing the 5 test floats. Recalculation of the Sichel t-estimator on the built-up heads was done with the following results:

Table 4.2
Summary of Sichel t Estimator Results
Replicate Flotation Tests
(Outliers Rejected)

Detail	Sample Basis Floats BUH	Assay AH (1)	Assay AH (2)
Observations n	5	7	49
Sichel t	1,020	1,00	1,00
UCL (95%)		1,006	1,008
LCL (95%)		0,998	0,994
$\tau_n(V)$	1,0003	1,00024	1,002
$\Phi(V,n)$		1,0051	1,008
$\Phi(V,n)$		0,9978	0,9939

Notes:

1. Blocks of 7 duplicate fusions
2. Blocks of duplicate fusions

Inspection of this table shows that the 95% confidence limits of the assayed head do not include the built-up head grade, indicating that exclusion of the data on this basis did not improve the metal balance.

The Sichel rejection of outliers in the BUH set now gives 101,98 %, which is a worse metal balance than before. This suggests that the Sichel t-estimator is limited in such an application as possibly the logvariance of the built-up heads is too small to draw meaningful values in the table of multipliers. It did, however, estimate the mean grade of ore in agreement with the arithmetic mean. A comparison of the two outlier rejection systems in terms of their effects is made in Table 4.3. Inspection of this table shows that the heuristic and Sichel models arrive at different results; the latter shows a call factor of 101,98 % PGE.

Table 4.3
Comaprison of Effects:
Heuristic and Sichel Outlier Rejection Models

Detail	Heuristic	Sichel
Raw Data		
Robust		
Assayed Head	1,00	1,00
BUH		
Mean g/t PGE	1,015	1,019
Std. Dev. g/t PGE	0,036	
RSD%	3,62	
Screened Data		
BUH		
Mean g/t PGE	1,002	1,020
Std. Dev. g/t PGE	0,024	
RSD %	2,44	

This comparison shows that the Sichel t-estimator does not improve the metal accountability as the heuristic model does. An objective test of such a model to identify outlier data should be found in the metal balance, accordingly reference to appropriate literature was made. An outlier detection system was reported in the form of a t-distribution [Grubbs, 1969] and was recommended in another reference [Taylor, 1990b]. The Grubbs test consists of ranking the test data in order from lowest to highest, and calculating the difference between the ith datum and the mean. This difference is divided by the standard deviation to arrive at a multiple of standard deviations that separate the ith datum from the mean. Critical values of Grubbs' estimator are given for various sample sizes, in particular $3 < n < 10$. If the Grubbs estimator value exceeds the critical value, then the datum is rejected as an outlier. An extract of these critical values at the 5% level is shown in Table 4.4.

Table 4.4
Critical Values of Grubbs' Outlier Test

n	Critical Value at 5% Level	n	Critical Value at 5% Level
3	1,155	7	1,938
4	1,463	8	2,032
5	1,672	9	2,110
6	1,822	10	2,176

Trial of this outlier rejection method on the built-up head grades per Table 3.60 is shown in Table 4.5. The mean and standard deviation of the raw data are 1,018 and 0,041 g/t PGE respectively.

Table 4.5
Grubbs Outlier Test on Raw Data
Simulated Quintuplicate Tests

Order # by Grade	BUH g/t PGE	Mean BUH g/t PGE	Difference g/t PGE	Grubbs T
1	0,975	1,019	-0,044	1,06
2	0,991	1,019	-0,028	0,68
3	1,009	1,019	-0,010	0,24
4	1,020	1,019	+0,001	0,02
5	1,020	1,019	+0,001	0,03
6	1,092	1,019	+0,074	1,84

The critical value for the Grubbs test for n=6 is 1,82, so the value 1,092 g/t PGE is rejected at the 95% confidence level. This shortens the data set to 5 and changes the built-up mean head grade to 1,002 g/t PGE. The metal balance improves as a consequence of this outlier rejection to 100,2 %. This is in agreement with the heuristic model. The impressive feature of the Grubbs test is the allowance of the sample size in calculating the critical value of the rejection limit. It is accordingly concluded that the heuristic practice of adjudicating replicate built-up heads by means of the crude standard deviation estimate is workable, however should for the future take account of sample data size in the form of the Grubbs test to determine the multiple of standard deviations at which outliers should be rejected. The effect of this practice on the PGE metal balance seems to be effective.

4.3 COMPARATIVE ERRORS

Mill Feed

It has been shown in § 4.1 that mill feed sampling could easily be an underestimated factor in presenting representative study material to the flotation laboratory. Apart from the obvious extraction errors pointed out in § 1.2.3, provided that the probable inclusion rule is observed by using the procedure described to obtain an unbiassed sample, the gross potential error is clearly in sample mass. Further the use of manual subsampling methods have been reported in the literature to introduce as gross a set of errors. These two factors, viz. mill feed sample size and subsampling procedure, potentially dominate the base variance of the flotation test data as follows:

Table 4.6
Ball Mill Feed
Comparative Potential Errors
(Expressed as RSD%)

Error Source	Sample Mass	Sample Subdivision
Underestimation of Sample Mass		
Mass : 50 kg	5,83	
: 500 kg	1,84	
Subsampling		
Cone and Quarter		6,81
Spinning Riffler		0,13

Provided that errors are independent of each other, their effect is additive [Box et. al., 1978g]. On this basis the following combinations are possible:

Combination	Effect as RSD % in Mill Feed	Combination	Effect as RSD% in Mill Feed
Low Sample Mass Cone and Quarter	12,64	Adequate Sample Mass Cone and Quarter	8,65
Low Sample Mass Spinning Riffler	5,96	Adequate Sample Mass Spinning Riffler	1,97

The above information may be used to estimate the tolerance limits about the head grade in question to demonstrate the effect of these combinations. Since the robust assayed head grade in this study was 1,00 g/t, and since for 6 replicates the Grubbs T is 1,82 at the 95% confidence level, Table 4.7 summarises the comparative resolutions that would result from the stated combinations of error.

This effect is shown in Figure 4.2. Clearly the combination of adequate sample mass and the use of a spinning riffler minimises the tolerance limits, i.e. achieves the tightest resolution.

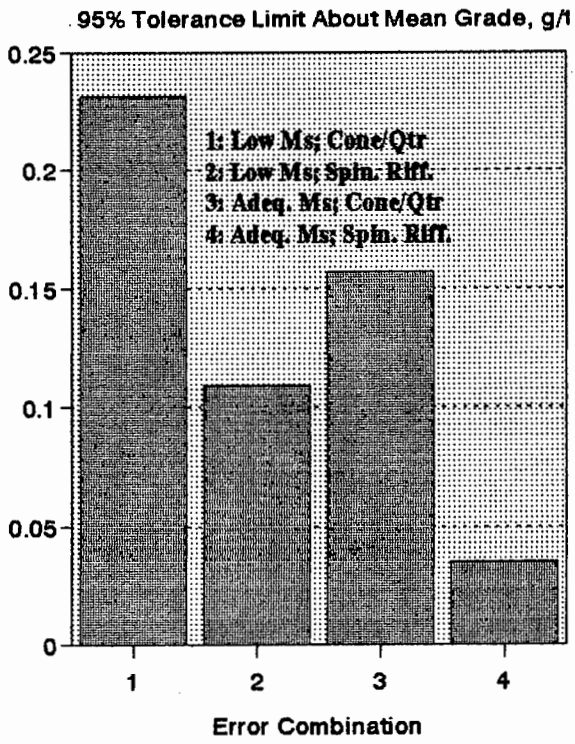


Figure 4.2 : Conventional Ball Mill Feed
Comparative Errors, Expressed as Tolerance Limits About Head Grade
Head Grade = 1,000 g/t PGE

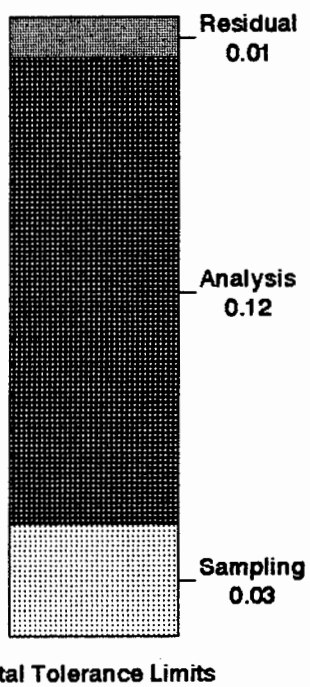


Figure 4.3: Conventional Ball Mill Feed
Breakdown of Comparative Errors Expressed as Tolerance Limits About Head Grade
Head Grade = 1,000 g/t PGE

Table 4.7
Ball Mill Feed Evaluation
Comparative Errors as Tolerance Limits
(Based on Assay Head Reference Distribution Mean)

Error Combination	RSD%	Est. Std. Dev. g/t PGE	Est. Tolerance by Grubbs T g/t PGE
Low Sample Mass Cone and Quarter	12,64	0,13	± 0,231
Low Sample Mass Spinning Riffler	5,96	0,06	± 0,109
Adequate Sample Mass Cone and Quarter	8,65	0,09	± 0,157
Adequate Sample Mass Spinning Riffler	1,97	0,020	± 0,035

Another level of resolution may be possible beyond adequate sample mass and use of the spinning riffler. Review of § 3.1.6 shows that with adequate sample mass and spinning riffler subsampling, the state of grind affects the RSD about the estimated mean grade. In particular the 60% passing 75 micron grind achieves the minimum RSD. The unwary investigator could incur the following changes to the 95% tolerance limits about the assayed head mean:

State of Grind % Passing 75 Microns	**RSD%**	**Est. Std Dev. g/t PGE**	**Est. Tolerance Limits by Grubbs g/t PGE**
40	13,3	0,14	0,25
50	10,9	0,11	0,20
60	8,8	0,09	0,16
70	14,7	0,15	0,27

The 0,16 g/t PGE tolerance limits for the 60% grind may be further tightened by use of the t-distribution to calculate the one-tailed error per number of replicate determinations. This was shown to reduce from 12,5 % for 2 fusions to 4,90 % for 13 fusions (§ 3.1.6.). At n = 13 the Grubbs T is 2,33, so the tolerance limits at 95% would be ± 0,12 g/t PGE for analysis error. A residual error of 0,16-(0,03+0,12) = 0,01 g/t PGE is obtained. This effect is shown in Figure 4.3.

Mass Balance Error

The reported mass balance error of -0,47% has a range of -0,3 to -0,6 %, i.e. consistently negative but not large enough to significantly affect the statements of PGE grade and recovery. At this stage of the investigation it is concluded that the loss is probably a result of residual moisture in the 2,5 kg sample of "dry" mill feed at weighing prior to the flotation tests. A test to quantify this was performed, taking 200,0 g samples of such "dry" mill feed and drying them in a sample drying oven at 106 degrees Centigrade. The result was a moisture loss of 0,15%, so not all of the mass loss is due to moisture; some 0,32% must be loss of material during the filtering and drying process. Had the mass loss been anything significant, there would have been an unaccounted loss of PGE. This was not observed in the 30 replicate flotation tests.

Flotation Test Products

Concentrates

High-grade concentrates have been shown to incur a significant grade estimation error due to the bimodal nature of the PGE/PGM distribution when PGM are present in large quantities. This was shown to be in the order of 2%. Normalisation of this error to mill head grade through the recovery reduces the figure to approximately 1,8%. Unless the high-grade sample was pulverised in clean silica prior to analysis this error will show as part of the unaccounted gain, and overstate the recovery by as much. This group of concentrates are provisionally categorised as richer than 20 g/t residual PGM.

Concentrates of lower tenor did not show the same bias, therefore provided the flotation tests do not reach the abovementioned grade this error does not appear to be applicable within the scope of this study, and in particular the 30 replicate flotation tests. This effect is probably due to the lower grade being accompanied by larger quantities of free gangue in the concentrate that disperse the PGM more evenly in a manner similar to the silica used for the high grade material.

Cleaner flotation tests that simulate high-grade cleaning, however, would benefit from this knowledge. In such cases it would be advisable to superpan trial samples of such concentrates to determine the residual PGM prior to submitting the test concentrates for analysis. The above argument, however, does not rule out the possibility of conventional grades of concentrate such as in the 30 flotation tests having a mill feed sample with an unusual quantity of metallics present. These would float to the first concentrate, as discrete PGM are positive floaters, and nugget the sample. This would lead to an outlier effect in the metal balance.

Flotation Tailings

Although the rougher tailings studied found very significant grade estimation effects in the order of 15% by incorrect grind presentation at analysis, the replicate tests in this instance were conducted at the ideal grind, so most of the underestimation of tails grade does not apply here, rather it is worth noting should the investigator require rougher flotation tests at coarser grinds. In such a case it will be necessary to either grind or pulverise the tailings to the optimum grind prior to analysis. The deliberate recalculation of tailings grades for the replicate test floats showed that for this type of final tailing, the bimodality does not appear to affect the grade estimation in this instance.

The potential error in this case from tailings is therefore not measurable.

Analysis

The t-distribution has been instrumental in modelling the one-tailed error in mean estimation of concentrate and tailings. The 5-6 prill requirement for concentrates incurs an error of 2,28 and 1,75% for high and conventional concentrate grade error respectively. In the replicate floats the concentrate grade was conventional, so the evaluation error is ± 1,43 % in the built-up head grade.

The same applies for the middling, or second concentrate. Here the error is ± 1,23 % of the built-up head grade.

In tailings the t-distribution has to be recalculated as the earlier exercise on rougher tails was for a much higher grade of sample. From t = 2 the sextuplicate fire assays used produce a one-tailed error of 11,6 %.

In mill feed terms this amounts to 0,61 % of built-up head grade.

The individual errors in the flotation test products are summarised in Table 4.8.

These errors amount to a one-tailed error of ± 3,27% in the built-up head, but may compensate for each other as the error is signed and additive.

Table 4.8
Comparative Errors
Flotation Test Products

Test Product	One-Tailed Error %	Error in BUH g/t PGE
Conc	1,75	1,43
Midd	14,16	1,23
Tails	11,60	0,61
Total		3,27

In comparing the errors of inputs and outputs at their optimised evaluation conditions, a best case error comparison using the t-distribution for mill feed and built-up head is shown in Table 4.9.

Inspection of this table shows that the mill feed sample incurs a higher evaluation error than built-up head. It is reasoned that this result was to be expected because of the heterogeneous nature of mineral deportment in the mill feed sample, whereas the built-up head is calculated from more specialised, or homogeneous, samples that are slightly easier to evaluate.

Table 4.9
Comparison of Total Errors
Mill Feed and Built-Up Head
Best Case Data

Total Error	Mill Feed	Built-Up Head
% of Head Grade	± 4,80	± 3,27

4.4 STRUCTURE OF THE QUALITY CONTROL MODEL

The foregoing experimentation has drawn a series of conclusions that form the foundation of a quality control model that may now be used as a basis for other flotation testwork on Merensky Ore.

The main sections of the model, described in section 4.4.2. (i) to (vi), are shown in ISO format [Halton,1994] in Figure 4.5, Sheets 1-6. A glossary of terms and icons precedes these.

4.4.1 Glossary of Terms

Refer to Figure 4.4.

Operation Symbol

The rectangular symbol describes a single work operation. It should contain a brief description of this operation inside the rectangle. The first word should be a verb, e.g. CALIBRATE SCALE, WEIGH SAMPLE, etc.

Initiator Description

This symbol begins a flowchart. The description of the initiating operation must be included in the symbol.

Terminator Description

This symbol ends a flowchart. The description of the terminating operation must be included in the symbol.

Decision Symbol

This symbol indicates that a decision must be made. The symbol must include a description of the decision to be made. The arrows leaving the symbol must indicate the YES and NO alternatives. Ideally these should lead to further operations.

Connector Symbol

When flowcharts from one operation to another must be linked, the connector symbol is used. In the circle, the linked flow number is used. These symbols must be linked by arrows with the arrow heads placed at the beginning or the end of a line joining a symbol.

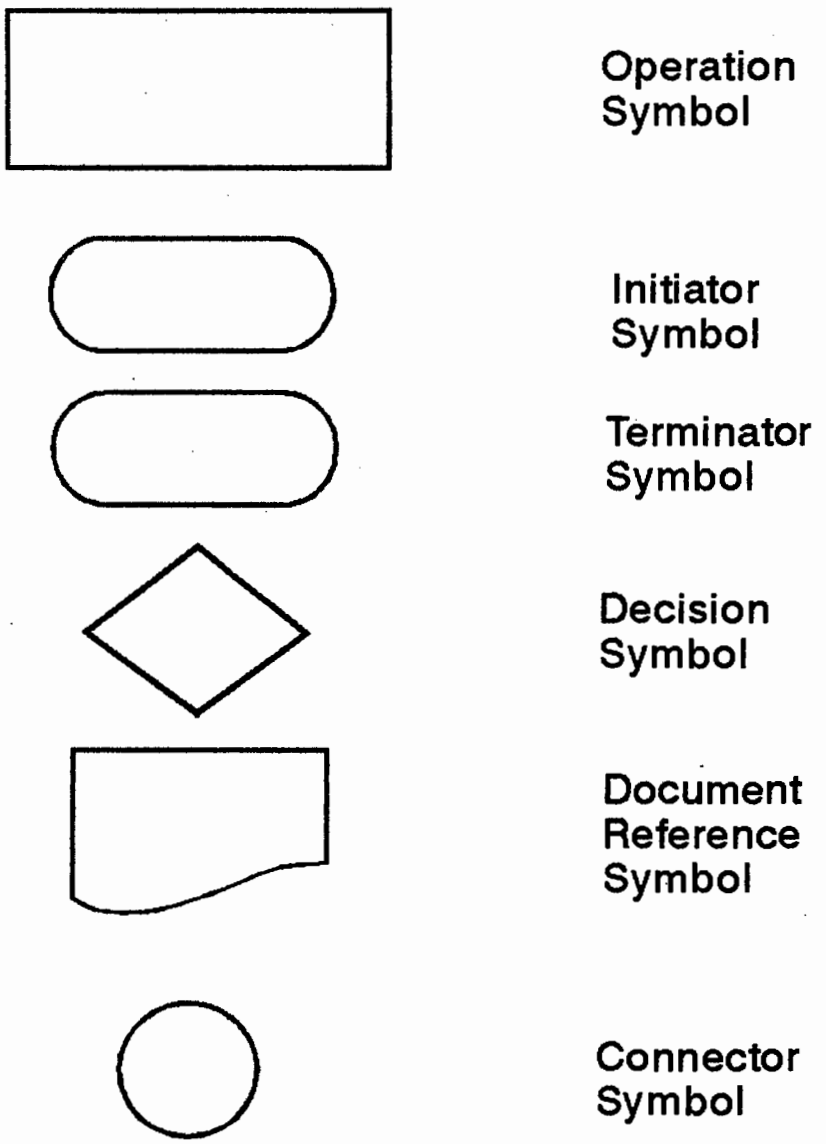


Figure 4.4

ISO Flowsheet Symbols

4.4.2 Model Structure

Six main sections are identified for this model:

(i) Mill Feed Sampling
(ii) Mill Feed Reference Distribution
(iii) Grinding Preparation
(iv) Flotation Testing
(v) Sample Preparation
(vi) Reconciliation of Metal Balance.

(i) Mill Feed Sampling (Figure 4.5 Sheet 1)

The mill feed stream to be sampled must be first identified. If an M_s model exists for this stream it may be used *a priori* and adjusted for the current topsize in the production stream (§ 3.1.5). If no M_s model exists for this stream the M_s experiment (§ 2.4.1) should be conducted and the results tabulated as shown in § 3.1.5. M_s should be found for a fundamental variance of less than 3,4%.

The mill feed sample should be taken using Gy's rules of probable inclusion (§ 1.2.3) to provide a bulk sample of mass at least as large as M_s. This sample is dried at 106° C, and is blended using the spinning riffler. The odds and evens blending method is used here (§ 2.1).

The blended sample is then crushed to - 3mm (§ 2.4.1) and blended again using the odds and evens method. The crushed and blended bulk sample is then subsampled using the spinning riffler (§ 2.4.1) to produce replicate subsamples of assay heads. These weigh 2,5 kg each. Here a total subsample mass of 10 kg is necessary if the grind/assay function is known otherwise sufficient study material for the exercise (§ 2.4.1) is necessary, amounting to 50 kg.

The grind/assay function is a test of the sensitivity of lead collection by fire assay for PGE to the state of grind of the ore sample. Refer § 2.4.1.

(ii) Mill Feed Reference Distribution (Figure 4.5 Sheet 2)

Refer to laboratory records and confirm whether the grinding requirements of this mill feed have been determined for optimal assaying. If known, proceed to mill the reference mill feed samples from Figure 4.5 Sheet 1 to the optimum grind. Blend the milled products using the spinning riffler (§ 2.1) and submit 20 x 500 g replicate samples for analysis. If the grinding requirements of this mill feed are not known for assay purposes, first conduct the necessary experiment (§ 2.4.1) then return to the standard preparation of replicate assay heads at known optimum grind as above.

Submit the prepared replicate mill feed samples for analysis and construct the reference distribution per § 2.8, except that all these results are off one grind and are pooled. Report the mean and 95% confidence limits.

(iii) Grinding Preparation (Refer Figure 4.5. Sheet 3)

The bulk of the remaining mill feed sample is now subsampled using the spinning riffler (§ 2.1). Test grinds are conducted on some of these to calibrate the laboratory ball mill grinding curve for the range of grinds to be studied (§ 2.4.1). A subsample of the natural reference tailings sample is included in the grading tests with the unknown test grinds. The database for the latter is developed by testwork described in § 2.1.

The result on the check sample for this database is first compared to the database control limits. If the check grading is outside these limits, the results of the test grinds are disqualified and the test sieves inspected and washed. The test grind gradings are then repeated with another check sample of reference tailing material until the results fall within the control limits. At this stage the test grinds are declared valid.

The natural standard reference grading sample has 95% limits of ± 1,84% passing 75 microns, so the test grinds are allowed to vary within these limits about the target grinds. Any grind outside these limits must be recalculated for grind time.

General experience shows that the finer grinds 70-80% passing 75 microns are the most vulnerable to this estimation.

Accepted test grinds are then reported.

(iv) Flotation Testing (Refer Figure 4.5 Sheet 4)

The 2,5 kg mill feed replicate subsample is ground to the desired grind (§ 2.6). The milled ore is transferred as a slurry to the laboratory flotation cell. The flotation test is conducted in terms of the particular test conditions desired and these are noted on a logsheet. The flotation test products, viz. concentrate, middling and tailing, are separately filtered and dried using procedures outlined in § 2.6. Five replicates are performed.

The mean and standard deviation of the concentrate masses are calculated (§Appendix 1). If the RSD of the concentrate masses exceeds 5%, the individual replicate tests are examined at 1 standard deviation about the mean as rejection limit. The ith replicate so rejected is then repeated as above to replace the flotation test products, and the calculation repeated.

When the 5 concentrate masses have an RSD of ≤ 5%, the tests are all accepted and the respective flotation test products composited. The total mass balance is recorded.

(v) Sample Preparation (Refer Figure 4.5. Sheet 5)

The composited mass balances per quintuplicate float are checked for error. If the mass balance incurred a loss greater than 0,5%, the test is repeated from sheet 4. Otherwise the balance is acceptable and sample preparation may proceed.

Three grades of sample are handled. These are high, medium and low. The high grade, being concentrate richer than 150 g/t PGE, is pulverised in an equal mass of silica prior to analysis (§ 2.5) and the assay result multiplied by 2.

The medium grade, being middlings, has no significant sample preparation problems and is submitted as is for assay.

The tailings, being low grade, require special preparation. These must be ground to finer than 60% passing 75 microns prior to analysis. This is done either in the laboratory mill or in the ring pulveriser (§ 2.4.3).

The analyses required for acceptable accuracy in the mean grades of concentrate, middling and tailings are 6, 6 and 10 respectively. It is possible that the tailings replicates may increase with lower grade to achieve similar confidence to that in the rougher tailings. The acceptance limits for the assay replicates are concentrate: 3,00; middlings: 2,00; and tailings: 11,1 % RSD respectively.

Mean grades and built-up heads are now calculated (§ 2.6).

(vi) Reconciliation of Head Grades (Refer Figure 4.5 Sheet 6)

The standard deviation and RSD of the composite built-up heads are calculated (§ 3.2.4). If the RSD is more than 5%, the sets of built-up head grades are examined with Grubbs' t outlier test (§ 4.2). The rejected replicate is replaced with a repeat test per sheet 4, and this recycles via sheets 4, 5 and 6.

(The 5% RSD is calculated from the exercise on 30 replicate flotation tests with the outlier removed and justifies rejection on this basis).

The call factor i.e. percentage ratio of built-up to assayed head, is now calculated for the mean built-up head and the reference assayed head (§ 2.8). If this call factor is in error by more than ± 3,3 % the entire test must be repeated from sheet 1. The 3,3% error has been found in § 4.3 Comparative Errors. Otherwise the data are accepted and the metallurgical performances are calculated for each test condition and reported.

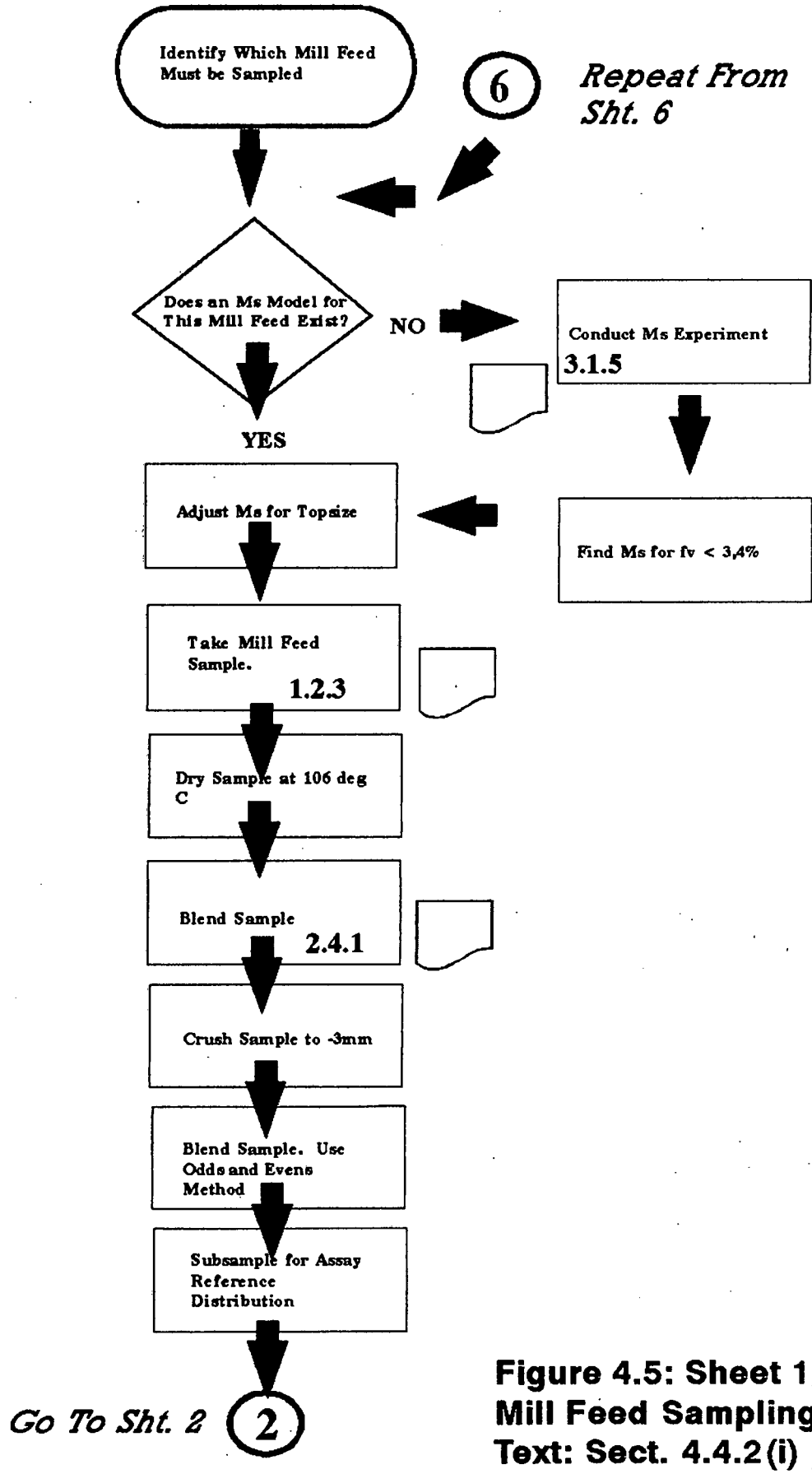


Figure 4.5: Sheet 1/6
Mill Feed Sampling
Text: Sect. 4.4.2(i)

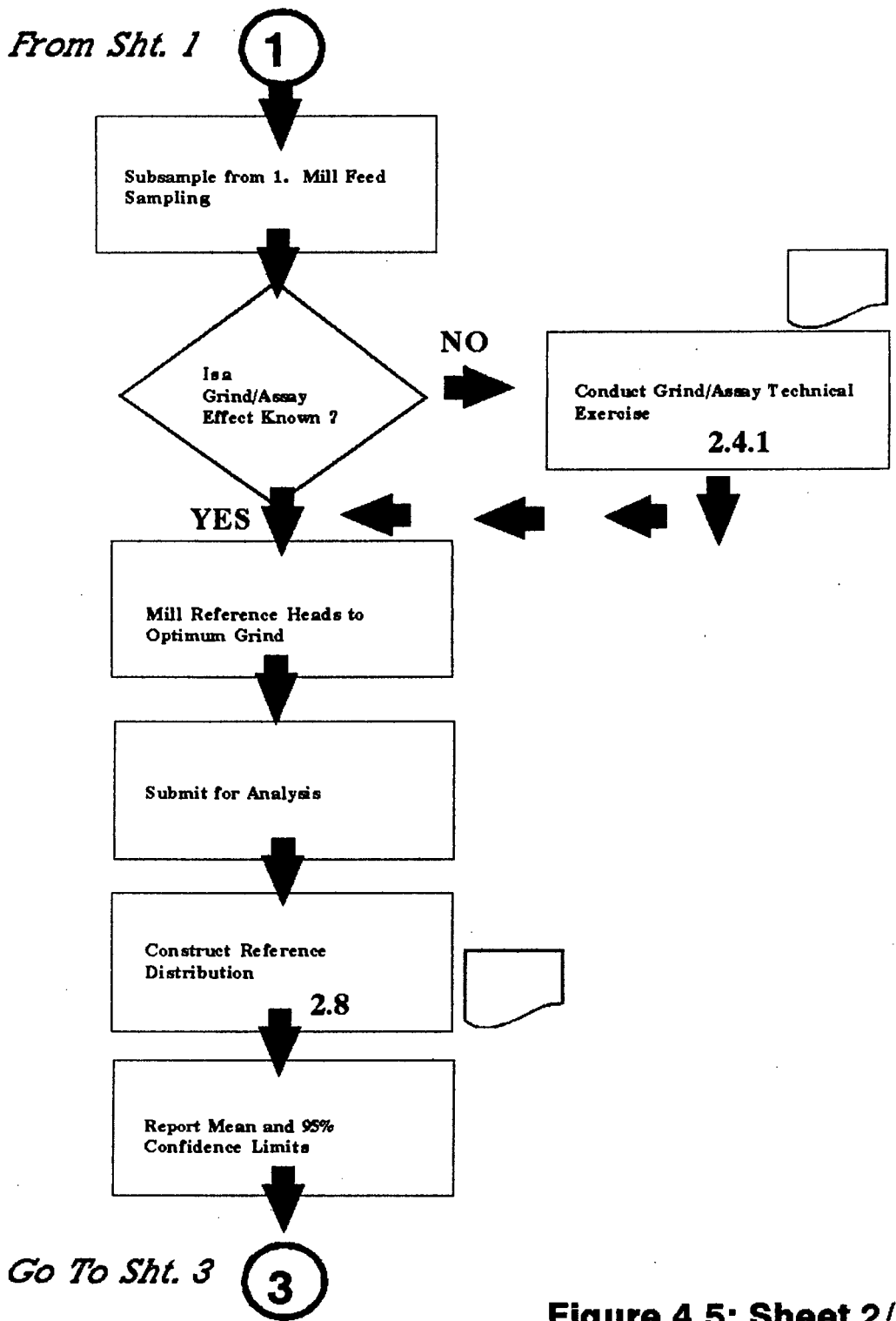


Figure 4.5: Sheet 2/6
Mill Feed Reference Distribution
Text: Sect. 4.4.2(ii)

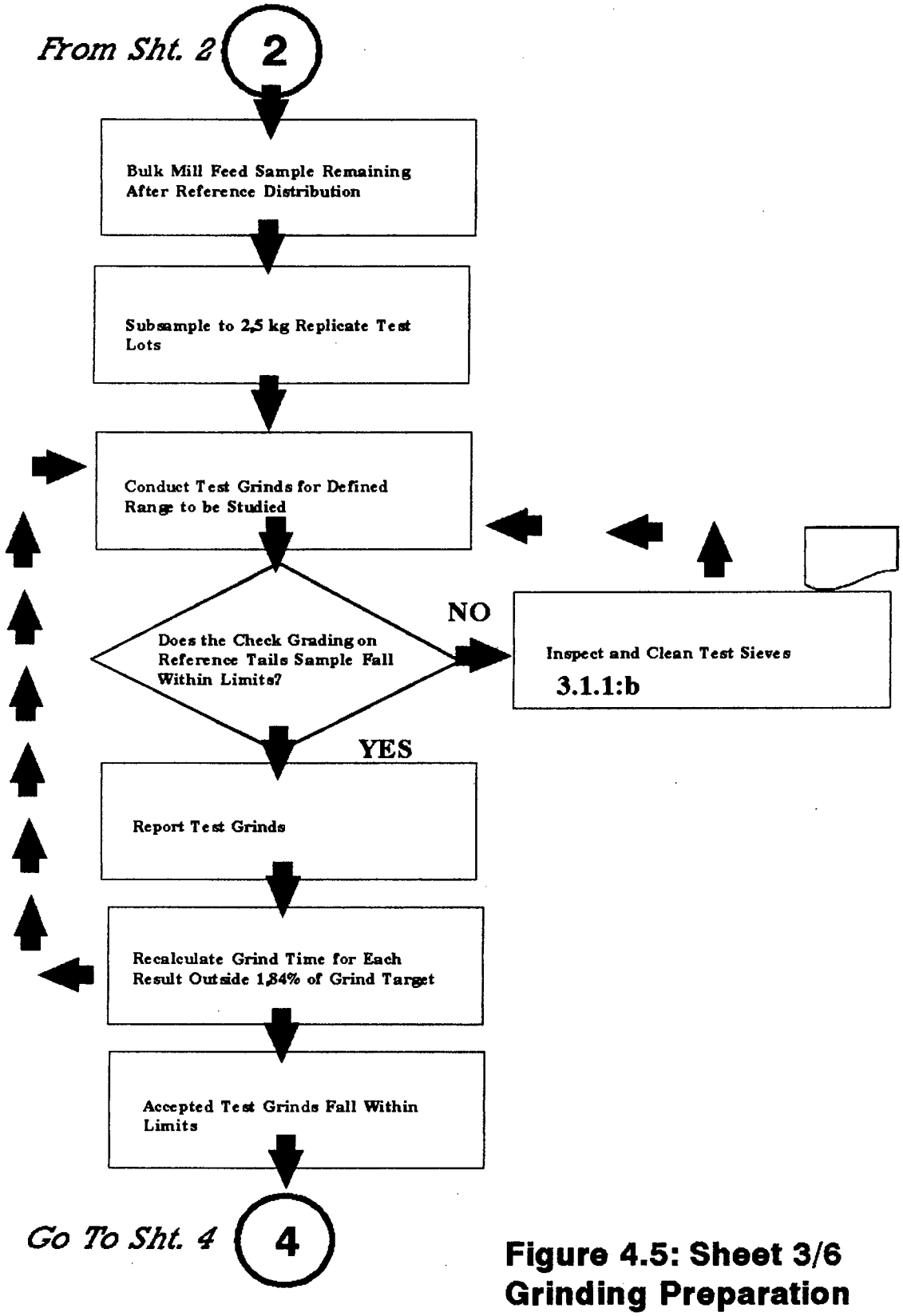


Figure 4.5: Sheet 3/6 Grinding Preparation Text: Sect. 4.4.2(iii)

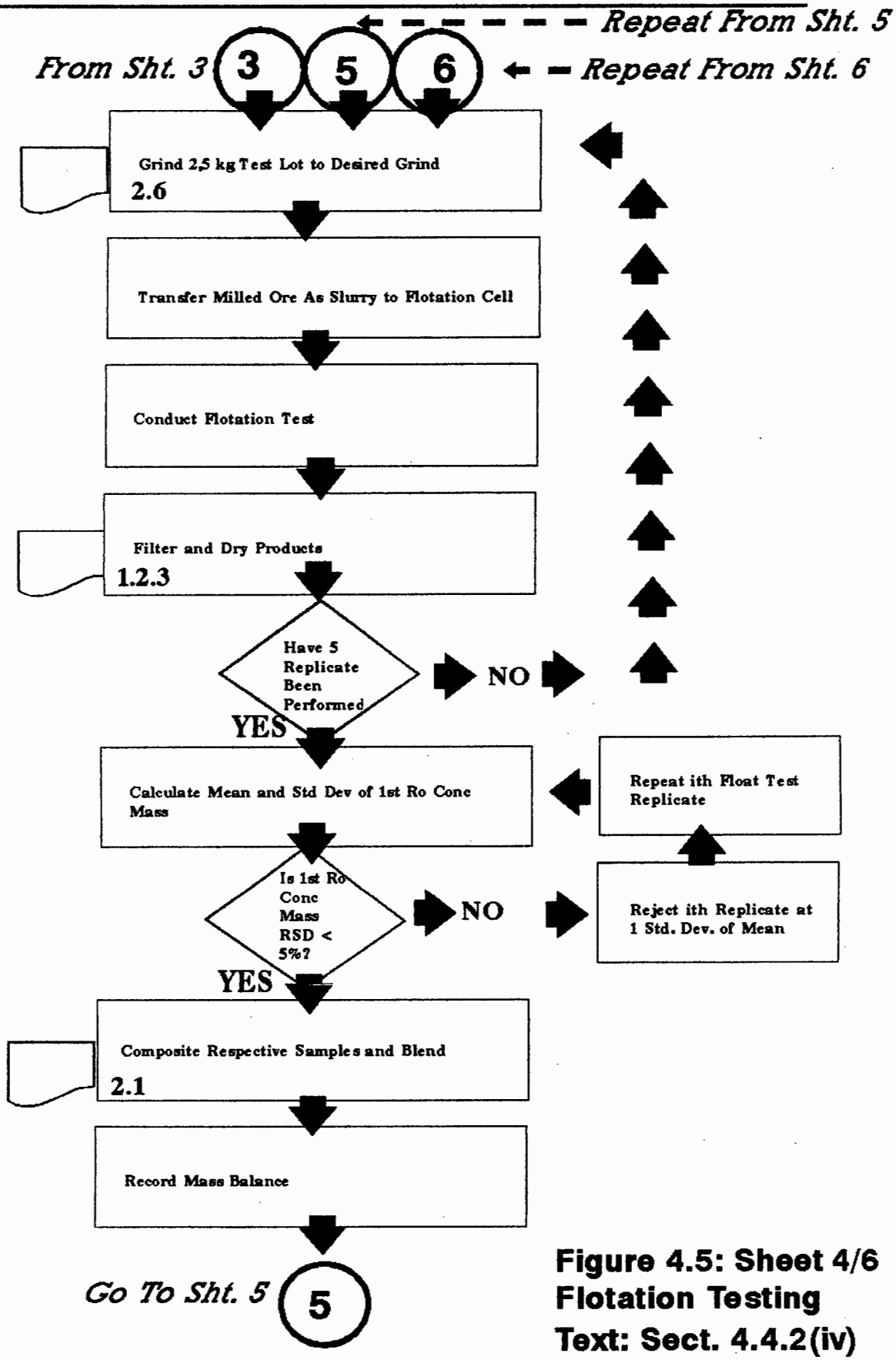


Figure 4.5: Sheet 4/6
Flotation Testing
Text: Sect. 4.4.2(iv)

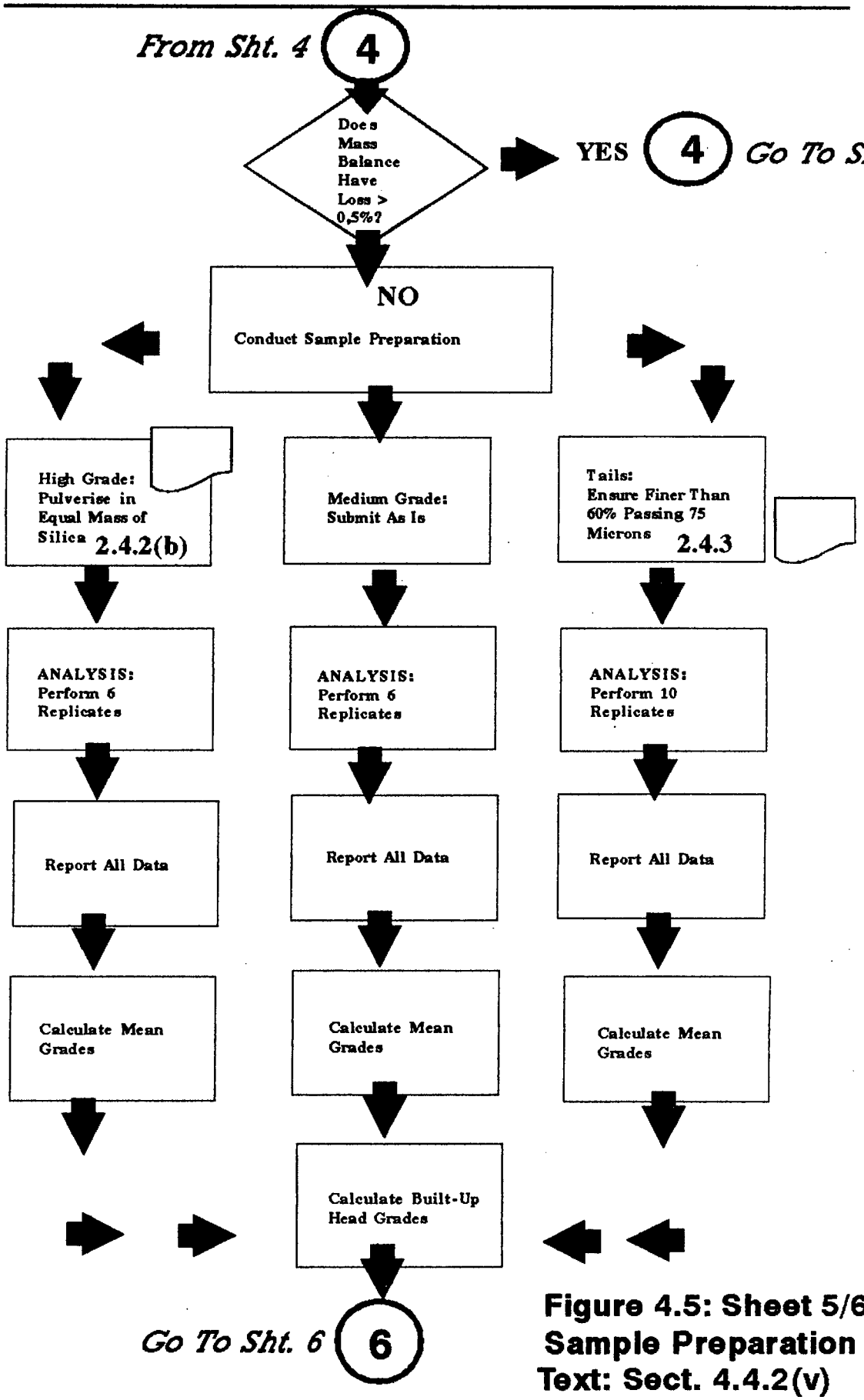


Figure 4.5: Sheet 5/6
Sample Preparation
Text: Sect. 4.4.2(v)

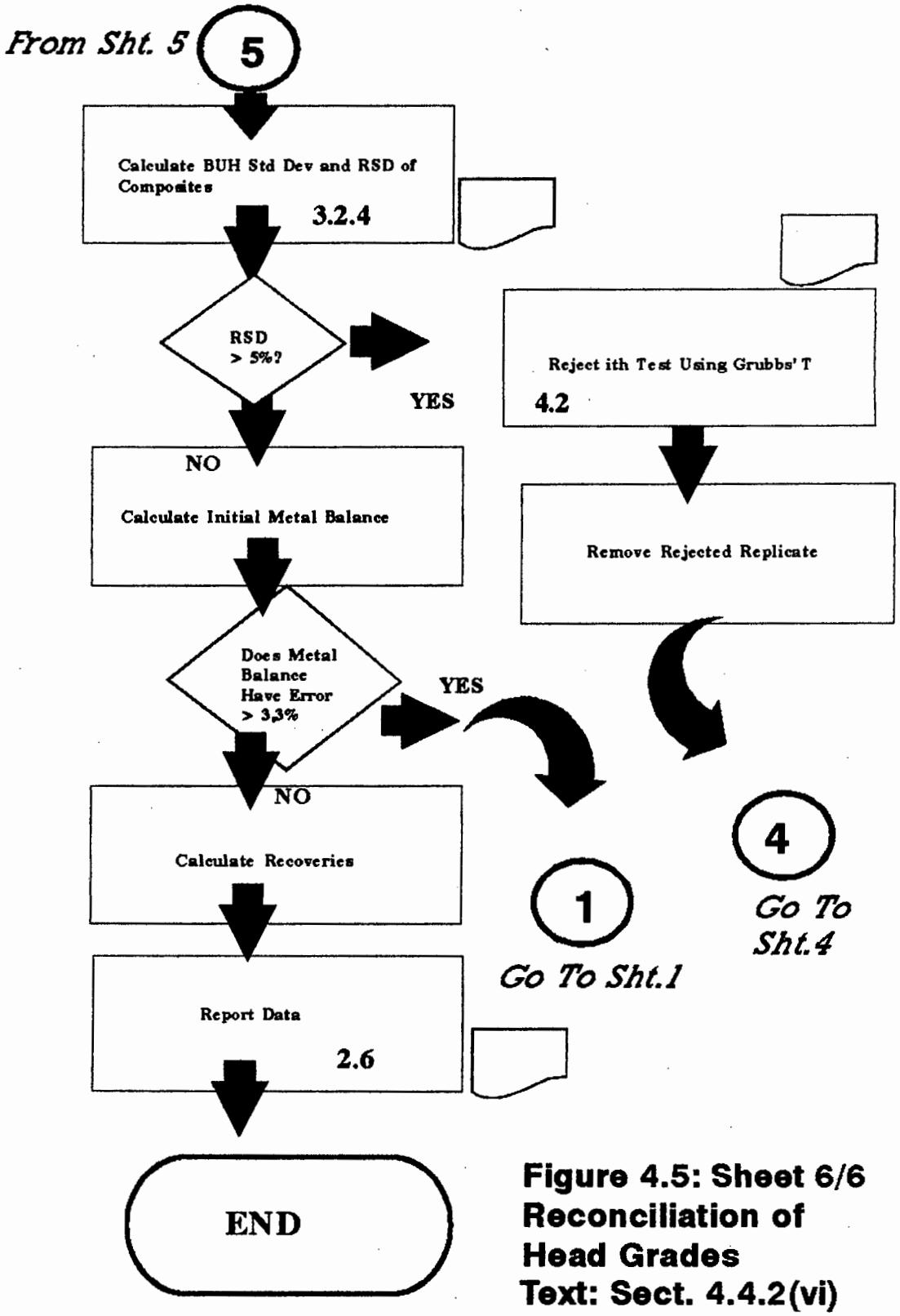


Figure 4.5: Sheet 6/6 Reconciliation of Head Grades Text: Sect. 4.4.2(vi)

5. CONCLUSIONS AND FUTURE WORK

These studies have attempted to bridge the approaches of several disciplines on the specific subject of Merensky evaluation at a small scale. A set of conclusions is now evident, also a list of further work is identified.

5.1. CONCLUSIONS

5.1.1. Geology, Mineralogy and Geostatistics

A review of the geology and mineralogy of the Merensky ore shows evidence of several different PGE host minerals. Sampling models available indicated that certain basic exploratory tests would be necessary to define sample extraction and minimum sample mass. A study of the relevant geostatistical models showed that the value frequency distributions and their probability plots could tell of the lognormality of a distribution and whether correction would be necessary prior to the calculation of mean grade.

Other work in a similar format was identified for the diagnosis of composite distributions. In this regard decomposition of the Merensky distributions has been attempted, and a prototype interpretation made of the probability plot to quantify the amount of discrete PGM, or metallics, present in a sample. The errors in this graphical estimation method are presently 2-15%, but will improve once certain regression methods are used.

This section of work concluded that composite lognormal distributions are characteristic of Merensky and its mineral process products, and that the major feature was the step change in grade from solid solution PGE to discrete PGM, where the probability plot shows a distinct change in gradient.

5.1.2. Mill Feed Sampling

The obtaining of a representative sample of mill feed is fundamental to achievement of representative metallurgical responses in testwork. For a conventional ball mill feed of d80 size 5 mm, Gy's minimum sample mass (M_s) model underestimates M_s by some 176% at the 10% variance level.

A more reliable estimate of M_s is obtained from the Bartlett and Hawkins model which adds a corrective term for size-by-size variance of PGE values in the mill feed sample. The following range of fundamental variance by sample mass shows that a 500 kg sample of mill feed is suitable for flotation testing purposes:

Effect of M_s on Fundamental Variance
Conventional Ball Mill Feed
Bartlett and Hawkins Model

M_s kg	Fundamental Variance %
50	34,1
100	17,0
200	8,5
300	5,7
400	4,3
500	3,4

The 500 kg sample achieves a fundamental variance well below 5%, and provides adequate study material for an internal reference distribution as well as for testwork.

The preparation of the mill feed sample for analysis was found to have a critical effect on the resolution of the replicate analyses. A specialised blending procedure for the bulk sample using the spinning riffler was used to homogenise the sample prior to subsampling. The sample must also be ground to 60% passing 75 microns to minimise this scatter. The controls established for the measurement and use of particle size distribution, especially supporting the activities whereby mill feed samples are ground to a desired state of grind, have proved useful.

Under these conditions the replicates agreed to within an 8% relative standard deviation (RSD). A total of > 13 replicates is necessary to obtain a reliable estimate of the grade of this ore. The t-distribution for this data was used for this calculation. The use of a semivariogram agreed with this finding.

5.1.3 Flotation Test Products

Flotation Concentrate

An initial distinction is made between high-grade and conventional grade concentrates. These differ not only in overall PGE grade but especially in their discrete PGM (metallics) content. The discrete PGM are present as nuggets in the high-grade sample and skew the value frequency distribution. This type of concentrate required pulverisation in clean silica prior to analysis; a 50/50 mass ratio of sample to silica was used. Under these circumstances the estimated mean grade was some 2% lower than the untreated sample. The probability plots for these also showed that the pulverised sample had correct lognormality.

The significance of the difference in mean grade estimation was successfully tested at the 95% level, and implies that a 2% unaccounted gain in the metal balance would accrue across a flotation test working at this grade unless appropriate sample preparation is seen to.

Conventional grade concentrate showed no benefit from this treatment, confirming the theory that the residual metallics are responsible for such overstatement of grade. In both cases, however, a minimum of 5-6 prills was necessary to obtain a reliable mean estimate.

Flotation Tailings

Rougher tailings were tested in terms of the state of grind prior to analysis. Data within one sample showed that the grade is significantly underestimated if the sample is not ground to a suitable fineness. Within the range of grinds studied, any grind coarser than 60% passing 75 microns appears to cause understatement of the grade of the tailings sample. This is believed to be due to incomplete fusion in the fire assay lead collection process. The order of this understatement is ca. 15% for a grind of 40% passing 75 microns. Such an oversight would cause an unaccounted loss in the metal balance of approximately 2%.

A probability plot of the results at the 70% grind shows that the distribution of values within the composite sample is bimodal. If the subdistribution in the left of the composite distribution is ignored on grounds of incomplete fusion, real data are discarded and the overall grade of tailing overestimated.

A total of 10 replicate fusions per determination of tailings grade at this grind is necessary to estimate the grade with an RSD of < 7%. A lower rejection limit of 0,16 g/t PGE is suggested for control purposes.

5.1.4 Reconciliation of Head Grades

Built-Up Head Grade

Thirty replicate flotation tests were arranged with a standard flotation method. Sufficient mill feed material was kept spare for accounting purposes. This was treated in accordance with the optimised method described for mill feed evaluation. The flotation tests were performed at a grind of 60% passing 75 microns, and were conducted under the same test conditions. The results of this exercise show that the mass balance has an error of -0,47%, which is regarded as insignificant in terms of other potential errors aforementioned.

The resolution of PGE metal balance on a raw data basis shows an unaccounted gain of +1,9%, which is acceptable for initial analysis. One outlier is detected at 2 robust standard deviations from the mean. Construction of the minimum observations model finds 5 replicates necessary to obtain reliable estimation of the built-up head grade. Averaging the 30 tests into simulated quintuplicate composites produced 6 flotation test results whose built-up head grades were compared to the robust assay head data. This averaging included the outlier and the various outlier rejection models tested.

The heuristic model relying on the presence of an outlier to increase the estimated standard deviation, with one crude standard deviation as the rejection limit, produced an unaccounted gain of +0,2%. Trial of the Sichel t-estimator for similar purposes only succeeded in identifying that the assayed head and the built-up head were not within the same 95% confidence limits. Use of the Grubbs outlier test, a form of the t-distribution, arrived at exactly the same conclusion as did the heuristic model. Here the rejection limit was 1,82 crude standard deviations. Analysis of comparative errors in the assayed and built-up head data showed that the visible errors in head grade were ± 4,80 and ± 3,27% respectively. This confirms the earlier experience that the built-up head grade is easier to obtain accurately than the assayed head grade. This comparison is made for optimised conditions such as have been determined by the experimental procedure in these studies. Accordingly non-optimised conditions are likely to enlarge these errors.

5.1.5. Quality Control Model

The quality control model has been developed from first principles, and a flow schematic described for the sampling of mill feed, its evaluation, and treatment of flotation test products. Accurate reconciliation of input and output grades has been shown. Use of the Grubbs test has been instrumental in fundamental proof of the outlier rejection system. Tailored sample preparation methods have been helpful in minimising evaluation errors in this model.

5.2. FURTHER WORK

It is logical to pursue further work in the following areas:

5.2.1 UG2 Ore

A repeat of the examinations made in this thesis for Merensky is likely to reveal equally useful evaluation controls for UG2.

5.2.2 Solid Solution Tenor of PGE in Base Metal Sulphides of Merensky; and that of Silicates

Clear indications are seen in the data reported as to amenability of the PGE/PGM composite distributions to decomposition. If the Fortran programme ROKE is used in this regard a more accurate decomposition is likely, and if performed for both PGE and base metal sulphides the solid solution tenors of PGE in pentlandite and pyrrhotite may be possible. Equally it is worthwhile pursuing the estimation of PGE in solid solution with silicates and oxides.

5.2.3. Production Plant Process Survey

If the principles of quality control shown for laboratory scale work could be extended to the more complex application of production plant process surveying, a useful tool might be developed. Here more use of the reference distribution may be viable; possibly the controls slightly different; however worthwhile investigating.

5.2.4. Flotation Tests for Borehole Core

When supporting an exploration programme the flotation laboratory will have to take account of the smaller sample mass. Here a tailored approach may be possible with the Sichel t estimator as the logvariance will be larger.

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Appendix 1
Reference Section of Useful Statistics

1 Basic Statistics

Mean, Variance, Standard Deviation and Relative Standard Deviation

Terms:

Mean	x
Variance	s^2
Standard Deviation	s
Number of Observations	n
Relative Standard Deviation	RSD

These apply to the sample data.

The estimated sample mean value of a set of n data x_i is given by x, (read as " x bar "), where

$$x = (1/n)\sum_{i=1}^{n} x_i$$

The sample variance of these n data is calculated as s^2, where

$$s^2 = (1/(n-1))\Sigma\, x_i^{\,2} - nx^2$$

The standard deviation of these sample data is then calculated as the square root of the sample variance, viz.

$$s = \sqrt{s^2}$$

The relative standard deviation of these data is calculated as the standard deviation expressed as a percentage of the mean, ie

$$RSD = 100s/x$$

This term is also called the Coefficient of Variance, or the Percent Standard Deviation.

The skewness and kurtosis of the distribution are estimated using the third and fourth moments, viz:

$$\text{Skewness} = m3/(s^3)$$
$$\text{Kurtosis} = m4/(s^4)$$

For the normal distribution the skewness =0 and the kurtosis = 3. The moments are calculated as

$$m3 = (1/n)\sum_{i=1}^{n} (x_i - \bar{x})^3$$

$$m4 = (1/n)\sum_{i=1}^{n} (x_i - \bar{x})^4$$

Bessel Correction

The Bessel Correction is used in small data sets (generally $n < 10$) to correct for overestimation of the standard deviation in small samples. This takes the form

$$s_c = s\sqrt{(n/(n-1)}$$

and naturally s_c, the corrected standard deviation, will converge to s as n becomes large enough for n-1 to converge on n.

2 Krige's Lognormal Distribution Law

The widely applicable lognormal distribution law by Krige transforms the normal distribution law by a naperian logarithm transform of the raw grade values, viz.

z = raw grade of each sample

x = ln (z)

The lognormal distribution law takes two forms. These are two-parameter and three-parameter. The lognormal distribution of values is given by

$$\Phi(x) = [\sigma.(2\pi)^{0,5}]^{-1}\exp[-(1/2\sigma^2)(x-\alpha)^2]$$

where

x = ln(z+a)

α = mean of ln(z+a)

a = additive constant

σ^2 = variance of ln(z+a).

For the two-parameter model, a= 0; for the three-parameter, a > 0, the value of which is calculated as

$$a = (m^2 - f_1 f_2)/(f_1+f_2-2m)$$

where

m = the sample value corresponding to the cumulative frequency of 50%, i.e. the median,

f_1= the sample value corresponding to p frequency in the distribution,

f_2= the sample value corresponding to 1-p frequency in the distribution.

The above parameters are estimated graphically on a lognormal distribution plot of values.

3 **The Sichel t Estimator**
(Using Two-Parameter Lognormal Form)
(Refer Also Fig. A1 : Value Frequency Distribution)

The model is written in natural logarithm base, having used the transform

$x = \ln(z)$ z = arithmetic grade

t = Estimated mean grade for n observations

$\theta_{L,\alpha}$ = lower confidence limit

$\theta_{U,\alpha}$ = upper confidence limit

with

$$\overline{x} = (1/n) \sum_{i=1}^{n} (x_i)$$ $\overline{x}$ = geometric mean

and

$$V = (1/n) \sum_{i=1}^{n} (x_i - x)^2$$ V = logvariance

The value of t is calculated as

$$t = e^{\overline{x}} . \tau_n(V)$$ t = estimated mean grade

where

$$\tau_n V = 1+\sum_{r=1}^{\infty}((n-1)^r . V^r)/(2^r . r! . (n-1)(n+1)....(n+2r-3))$$

Confidence limits $\theta_{L,\alpha}$ and $\theta_{U,\alpha}$ are calculated as

$$\theta_{L,\alpha} = t . \Phi_p(V,n)$$

and

$$\Phi_{U,\alpha} = t.\ \Phi_{1-p}(V,n)$$

The multipliers $\Phi_p(V,n)$ and $\Phi_{1-p}(V,n)$ are given by

$$\Phi_p(V,n) = \exp((\sigma t^2(V)/2) - T_p \sigma t(V))$$

and

$$\Phi_{1-p}(V,n) = \exp((\sigma t^2(V)/2) - (T_{1-p} \sigma t(V)).$$

Thus the mean of a skew VFD can be estimated at an assigned confidence level, and upper and lower limits for the estimate formulated.

4 Value Frequency Distribution

(Refer Figure A1)

The Value Frequency Distribution, or VFD, is a histogram, or frequency plot, of the percent frequencies observed per group of ore grade, for a collection of samples in the spatial or time dimensions.

5 The Semi-Variogram

Refer Figure A2

The semi-variogram is used to determine the minimum spacing, in distance or time, between sample values, at which there is no significant interelationship between the values. The spacing less than this value is termed the area of influence, where predictions may be made.

The spherical semi-variogram is modelled as

$$\tau(1) = (1/2N) * \sum_{i=1}^{N} (g_i - g_j)^2$$

where

i and j are data separated by 1 distance unit.

g_i = the grade at location i

N = the number of data.

Equally $\tau(2)$ is calculated from the same formula but using 2 distance units between i and j. The successive results of $\tau(h)$ are plotted on the y - axis and the corresponding h value, on the x - axis. A typical format of this version has the following construction:

$$\gamma(h) = 0 \qquad h = 0;$$

$$\gamma(h) = C[(1,5*h/a) - (0,5*(h^3/a^3)] + C_o \qquad 0 < h \leq a;$$

$$\gamma(h) = C + C_o \qquad h > a.$$

The overall sample data variance should be equivalent to the sill value $C+C_o$. The value of C_o is equivalent to the nugget effect, or the semivariance of replicate determinations within a given sample.

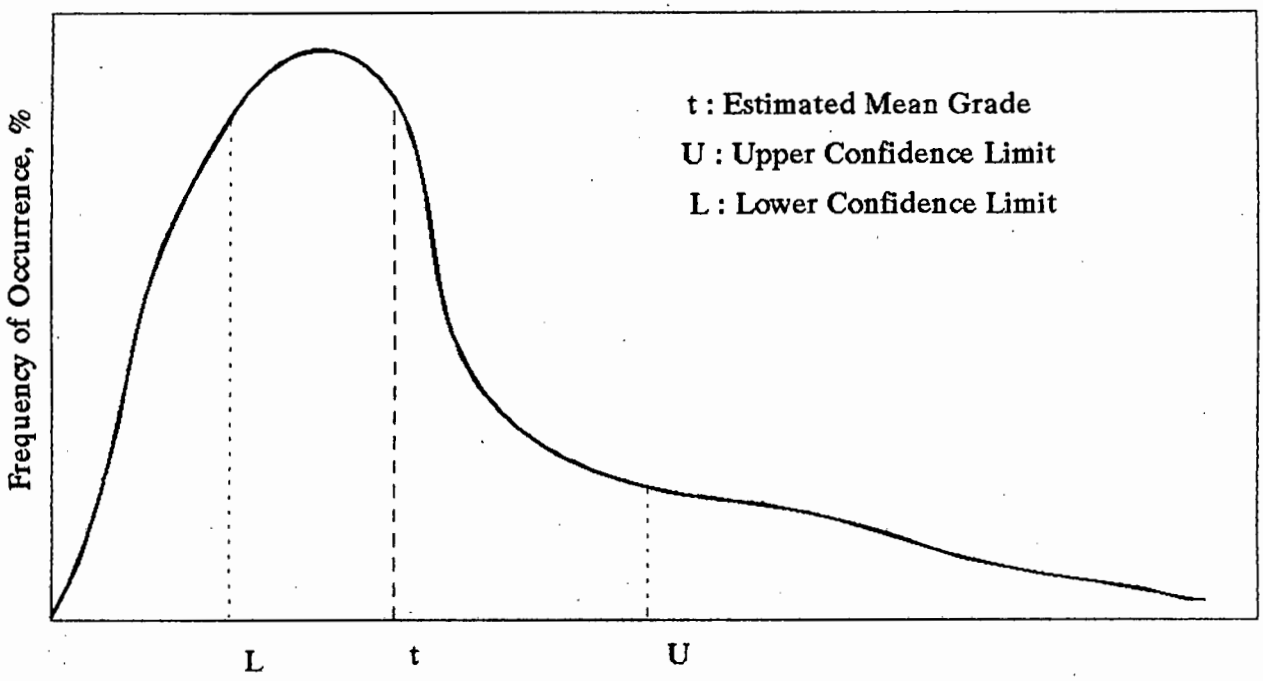

Figure A1 Value Frequency Distribution

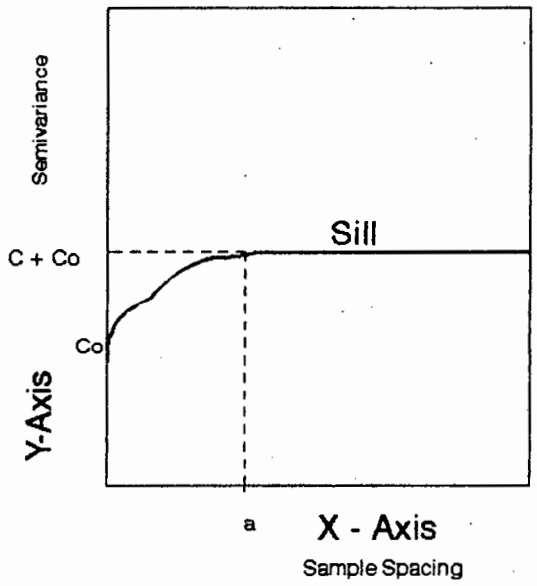

Figure A2
The Semi-Variogram

APPENDIX 2

REFERENCE GRADING SAMPLE DATABASE: WATERVAL FINAL TAILS

DATUM NUMBER	SERIES 1 DML	SERIES 2 PFR	DATUM NUMBER	SERIES 1 DML
	% – 75 mic.	% – 75 mic.		% – 75 mic.
1	66.7	66.4	46	65.9
2	66.7	66.8	47	62.2
3	66.3	65.9	48	66.4
4	66.1	65.6	49	65.9
5	65.6	65	50	65.6
6	66.2	68	51	64.4
7	66.8	67.9	52	66
8	66.8	67.6	53	66.1
9	66.5	65.6	54	65.9
10	66.9	66.6	55	66.1
11	67	66.1	56	66
12	66.8	64.3	57	66
13	66.5	65.5	58	68
14	66.9	65.1	59	66.3
15	67.1	67.4	60	65.4
16	66	65.1	61	66.2
17	67	65.2	62	66.5
18	66.1	65.4	63	66.4
19	66.9	64	64	67
20	66	66.5	65	66.5
21	66.7	66.3	66	65.8
22	65.9	66.1	67	65.1
23	66.7	66	68	65.9
24	65.7	67.2	69	65.2
25	66.5	67.6	70	65
26	65.7	67.1	71	66.2
27	69.3	66.5	72	65.9
28	66.5	64.8	73	66.1
29	66.1	66.2	74	64.6
30	68	65.6	75	66.1
31	66.6	64.9	76	65
32	64.8	65.9	77	65
33	65.4	66.3	78	65.1
34	65	65.7	79	65
35	65.3	64.3	80	65.1
36	66	65.9	81	64.8
37	66.8	67.1	82	65.4
38	65.8	65.5	83	65.4
39	66.2	64.5	84	65.6
40	66.4	65.6	85	64.8
41	66.1		86	65.3
42	66.6		87	64.5
43	66.1		88	
44	65.8		89	
45	65.6		90	