

Australian Standard™

## **Heavy mineral sand concentrates— Sampling**

### **Part 2: Sampling from stationary situations**



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The following are represented on Committee MN-004:

Australian Institute of Mining and Metallurgy

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Producers of heavy mineral sand concentrates

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Australian Standard™

## Heavy mineral sand concentrates— Sampling

### Part 2: Sampling from stationary situations

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## PREFACE

This Standard was prepared by the Standards Australia Committee MN-004, Heavy Mineral Sands to supersede AS 2884.2—1993, *Heavy mineral sand concentrates—Sampling, Part 2: Sampling from stationary situations*.

The objective of this Standard is to provide sampler and superintending companies with a method for safely obtaining samples of heavy mineral sands for analytical purposes when the preferred method of sampling from a moving stream is not possible.

This revision confirms the method of sampling from stationary situations. Editorial changes have been made to bring the Standard into line with current style.

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is only for information and guidance.

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## STANDARDS AUSTRALIA

## Australian Standard

## Heavy mineral sand concentrates—Sampling

## Part 2: Sampling from stationary situations

**1 SCOPE**

This Standard sets out methods for the sampling of heavy mineral sands from stationary situations to provide samples for chemical analysis, physical testing and determination of moisture. Stopped-belt sampling is specifically included.

The methods of sampling outlined in this Standard should be considered only where the preferred method of sampling from a moving stream is not possible.

The preparation of samples taken by methods outlined in this Standard is covered in AS 2884.3.

Clauses 4 to 8 provide general sampling theory that may not be relevant to sampling from stationary situations.

**2 REFERENCED DOCUMENTS**

The following documents are referred to in this Standard:

AS

|        |                                             |
|--------|---------------------------------------------|
| 1152   | Specification for test sieves               |
| 2884   | Heavy mineral sand concentrates—Sampling    |
| 2884.3 | Part 3: Preparation of samples              |
| 2884.4 | Part 4: Determination of precision and bias |

**3 DEFINITIONS**

For the purpose of this Standard, the definitions below apply.

**3.1 Bias**

The tendency to obtain a value that is either persistently higher or persistently lower than the reference value. In practice, it is the difference between the reference value and the average result obtained from a large number of determinations using a biased method.

**3.2 Coefficient of variation**

The ratio of the standard deviation to the mean value, expressed as a percentage.

**3.3 Constant-mass division**

The method of sample division in which the retained portion is of uniform mass.

**3.4 Divided increment**

The quantity of concentrate obtained by division of the increment to decrease its mass.

**3.5 Division**

The process of decreasing the sample mass (without modification of the particle size of the constituent pieces) whereby one or more representative parts of the sample are retained.

### 3.6 Duplicate sampling

A particular case of replicate sampling (involving only two replicate samples), for the purpose of estimating the average precision of sampling from a number of lots or sampling units.

### 3.7 Fixed-rate division

The method of sample division in which the retained portion from individual increments is a constant proportion of the original mass.

### 3.8 Gross sample

A sample formed when all of the primary increments or partial samples, either as taken or after having been prepared individually to a particular stage of sample preparation, are combined in the correct proportions for preparation of a laboratory sample.

### 3.9 Increment

The quantity of concentrate taken in one operation.

### 3.10 Lot

A quantity of concentrate delivered at one time; it may comprise one or more sampling units.

### 3.11 Manual sampling

The operation of sampling whereby the increments forming partial samples and gross samples are taken using a hand-held implement.

### 3.12 Mass-basis sampling

The taking of increments at or within uniform mass intervals throughout the lot or sampling unit.

### 3.13 Mechanical sampling

The operation of sampling whereby the increments forming partial samples and gross samples are taken by a sampling machine.

### 3.14 Nominal top size

The size of aperture of the finest sieve (complying with AS 1152) through which a minimum of 95 percent of the mass of the material passes.

### 3.15 Partial sample

A sample representative of a part of the whole sampling unit and constituted to prepare laboratory samples or test samples; it may be obtained by combining all increments from a sampling unit into two or more sets, each set comprising consecutive increments, the number of which need not be the same in all sets.

### 3.16 Quality variation

The standard deviation of the quality characteristic of interest.

### 3.17 Replicate sampling

The taking of increments from the lot or sampling unit at equal intervals of time, mass or space, whereby the increments are placed in rotation into different containers to give several replicate samples of approximately equal mass.

### 3.18 Sampling unit

The discrete units (trains, sections of belt, daily production) that comprise a lot.

### 3.19 Standard deviation

The positive square root of the variance.

### 3.20 Strata

Approximately equal parts of a lot or sampling unit based on intervals of time, mass or space.

### 3.21 Stratified random sampling

The taking of increments whereby the concentrate being sampled is divided into strata, one increment being taken at random within each stratum.

### 3.22 Systematic sampling

The taking of increments whereby the concentrate being sampled is divided into strata, one increment being taken at the same location within each stratum.

### 3.23 Time-basis sampling

The taking of increments at or within uniform time intervals throughout the lot or sampling unit.

### 3.24 Variance

A measure of dispersion based on the mean squared deviation from the arithmetic mean.

For a series of  $n$  observations  $X_1, X_2, \dots, X_n$  with mean  $\bar{X} = \frac{1}{n} \sum X_i$  an estimate of the variance is given by—

$$\frac{1}{n-1} \sum (X_i - \bar{X})^2$$

## 4 PRINCIPLES OF SAMPLING

### 4.1 General

The basic rule for a correct sampling scheme is that all parts of concentrate in the stream have an equal opportunity of being selected and appearing in the final gross sample for testing. Any deviation from this basic requirement can result in an unacceptable loss of accuracy and precision. No incorrect sampling scheme can be relied on to provide representative samples.

Sampling should preferably be carried out by systematic sampling. However, it needs to be shown that no systematic error could be introduced by periodic variation in quality or quantity having a period approximately equal to any multiple of the proposed sampling interval.

As an example, a primary cutter may be cutting a stream of concentrate that is being reclaimed from a stockpile by a bucket-wheel reclaimer. At both limits of the bucket-wheel traverse across the face on the stockpile, the concentrate may have different properties from those of the stockpile average (e.g. due to segregation, surface drying, oxidation, addition of dust-suppressing chemicals, or water sprays). In this case, it is quite possible that every time the primary cutter makes a cut, the cut will coincide with concentrate being delivered from the limit of a traverse of the bucket-wheel reclaimer. A systematic error could arise.

Also, where it is felt that, owing to the manner in which the concentrate is handled and presented to subsequent division apparatus, a systematic error could arise in secondary and subsequent stages of division, it is strongly recommended that stratified random sampling within fixed time or mass intervals be carried out.

The methods for sampling and sample preparation depend on the final choice of sampling scheme and on the steps necessary to minimize possible systematic errors arising during subsequent division steps.

## 4.2 Sampling errors

### 4.2.1 General

The total sampling error (TE) can be broken up into a number of independent components, as follows:\*

$$TE = QE_1 + QE_2 + QE_3 + WE + DE + EE + PE \quad \dots 4(1)$$

where

$QE_1$  = short-range quality fluctuation error associated with short-range variations in quality

$QE_2$  = long-range quality fluctuation error associated with long-range variations in quality

$QE_3$  = periodic quality fluctuation error associated with periodic variations in quality

$WE$  = weighting error associated with variations in increment mass

$DE$  = increment delimitation error introduced by incorrect increment delimitation

$EE$  = increment extraction error introduced by incorrect increment extraction

$PE$  = accessory error introduced by careless handling of the sample

The short-range quality fluctuation error consists of two components, as shown by the following equation:

$$QE_1 = FE + GE \quad \dots 4(2)$$

where

$FE$  = fundamental error due to variation between particles

$GE$  = segregation and grouping error

A number of these components, namely  $DE$ ,  $EE$  and  $PE$ , can be eliminated by using correct sampling practices. The remainder can be minimized, or reduced to an acceptable level, by correct design of the sampling procedure.

### 4.2.2 Accessory error

Accessory errors, also referred to as preparation errors, include sample contamination, loss of sample material, alteration of the chemical or physical composition of the sample, operator mistakes, fraud or sabotage. These errors can be eliminated by correct design of the sampling implement and by staff training.

### 4.2.3 Delimitation and extraction errors

Delimitation and extraction errors arise from incorrect sample cutter design and operation. The delimitation error can be eliminated if all parts of the stream cross-section are diverted by the sample cutter for the same length of time. The extraction error can also be eliminated by ensuring that the increment is completely extracted from the stream without any material rebounding or being lost from the cutter.

\* GY, P. *Sampling of particulate materials---Theory and practice*. Amsterdam: Elsevier, 1982.

#### 4.2.4 *Weighting error*

Weighting errors result from fluctuations in the increment mass. The weighting error can be estimated from a variographic experiment on flow rate, but it is better to regulate the increment mass thereby making the weighting error insignificant. As a general rule, the weighting error is negligible for variations in increment mass up to 10%, and acceptable for variations in increment mass up to 20%.

#### 4.2.5 *Periodic quality fluctuation error*

Periodic quality fluctuation errors result from periodic variations in quality generated by some equipment used for concentrate handling, e.g. screening circuits and bucket-wheel reclaimers. The presence of periodic variations can be detected by determining the variogram (see Clause 4.3). In such cases, stratified random sampling should be carried out as discussed in Clause 4.1. The alternative is to eliminate the source of periodic variations in quality.

### 4.3 **Sampling variance**

#### 4.3.1 *General*

Assuming that periodic variations in quality have been eliminated and that the flow rate has been regulated, the sampling error in Equation 4(1) reduces to—

$$TE = QE_1 + QE_2 \quad \dots 4(3)$$

Hence, the sampling variance ( $\sigma_s^2$ ) is given by—

$$\sigma_s^2 = \sigma_{QE_1}^2 + \sigma_{QE_2}^2 \quad \dots 4(4)$$

The short-range quality fluctuation variance ( $\sigma_{QE}^2$ ) arises from the different internal composition of increments taken at the shortest possible interval apart. This is a local or random variance due to the particulate nature of the concentrate.

The long-range quality fluctuation variance ( $\sigma_{QE}^2$ ) arises from the continuous trends in quality that occur while sampling a concentrate. This component is time-dependent, because increments become less and less alike as the intervals between them increase.

#### 4.3.2 *Estimation of sampling variance from the variogram*

The short-range and long-range quality fluctuation variances are determined from a variographic experiment in which the quality characteristics of individual increments taken from a lot are determined and processed in accordance with standard methods. The differences between increments at increasing intervals (called lags) are calculated, giving a picture of how variability changes with lag. The resulting plot is called a variogram.

The value of the variogram  $V_e(t)$  at lag  $t$  is given by—

$$V_e(t) = \sum_i (x_{i+t} - x_i)^2 / 2 N_t \quad \dots 4(5)$$

where

$x_{i+t}$  = the value of the quality characteristic for increment  $i + t$

$x_i$  = the value of the quality characteristic for increment  $i$

$N_t$  = the number of pairs of increments at lag  $t$  apart

The resultant variogram is called the ‘experimental’ variogram  $V_e(t)$  and includes the variance of sample preparation and analysis as well as the sampling variance. Subtraction of the sample preparation and analysis variance gives the ‘corrected’ variogram  $V_c(t)$ , which provides information on sampling errors only.