

Australian Standard™

Guide to the sampling of particulate materials

Part 3: Estimating sampling precision



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Guide to the sampling of particulate materials

Part 3: Estimating sampling precision

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PREFACE

This Guide was prepared by the Standards Australia Committee MN-010, Sampling of Minerals as a basis for preparing Standards for the sampling of a range of minerals from moving streams and stationary situations.

This Guide is Part 3 of the AS 4433 series for the sampling of particulate materials, to be used for the preparation of sampling Standards for a number of mineral commodities. Other Standards in the series are as follows:

- Part 1: Sampling procedures
- Part 2: Preparation of samples
- Part 4: Checking for bias
- Part 5: Sampling of slurries
- Part 6: Inspection of mechanical sampling systems

An investigation of sampling theory has been carried out to justify the principles adopted.

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STANDARDS AUSTRALIA

Australian Standard

Guide to the sampling of particulate materials

Part 3: Estimating sampling precision

1 SCOPE

This Guide sets out methods for checking and adjusting the precision of sampling of particulate materials, and checking the precision of subsequent sample preparation and measurement.

The procedures described in this Guide apply to sampling of particulate materials from both moving streams and stationary situations, and to the subsequent sample preparation and measurement.

Experiments to check for precision and bias should be carried out for all sampling programs, whether they use manual effort or employ mechanical devices. The experiments should be implemented on a regular basis. Procedures should be repeated whenever significant change has occurred in the character of materials being sampled, especially in the nominal top size or particle size distribution. Check experiments for precision and bias for the installation as a whole should always be conducted when a mechanical installation is commissioned or when principal parts are modified.

Methods for checking for bias are described separately in AS 4433.4.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Guide:

AS

1152 Specification for test sieves

4433 Guide to the sampling of particulate materials

4433.1 Part 1: Sampling procedures

4433.2 Part 2: Preparation of samples

4433.4 Part 4: Checking for bias

ISO

12744 Copper, lead and zinc sulfide concentrates—Experimental methods for checking the precision of sampling

3 DEFINITIONS

For the purpose of this Guide, the definitions given in Appendix A apply.

4 NOTATION

A list of symbols used in this Guide is presented in Appendix B.

5 ACCURACY AND PRECISION OF SAMPLING**5.1 General**

An experimental measurement can usefully be regarded as the result of the processes of sampling, sample preparation and laboratory testing. This result can always be regarded as being equal to the true (and generally unknown) reference value plus an experimental error.

which varies randomly from experiment to experiment. Terms like accuracy, precision and bias describe various aspects of the distribution of this error.

In general terms, bias is the difference between the reference value and the average of a number of experimental values. This difference is equal to the average of the error distribution, and accuracy is the degree to which bias is absent from the experimental results.

However, a totally unbiased experimental procedure, if repeated many times, may yield results which, while having the true value as their average, are spread out over a relatively wide range. In this case, there could be no confidence that any single result from this experiment is close to the true value. The precision of such an experiment is said to be relatively poor (i.e. the measure of precision is relatively high).

Precision is an expression of how closely repeated experimental values agree among themselves. A good experimental procedure is therefore one whose accuracy is high; i.e. the bias is small, and whose precision is good; i.e. the results of repeated experiments do not vary widely and the measure of precision is relatively small.

Methods for assessing the precision of sampling, sample preparation and measurement procedures are given in Clauses 6, 7 and 8. The influences of the number of increments in a gross sample, and the number of gross samples on the precision of a sampling result is discussed below.

5.2 Precision

Where a set of replicate samples are taken from a single lot or sub-lot of particulate material and are prepared and measured separately, the standard deviation of the set can be estimated from the average range of the measurements (see Clause 7). A measure of the precision of any single result is provided by the width of an interval centred on the result within which the true mean lies with a given probability or level of confidence. This measure of precision is called the index of precision, which is referred to as precision in this Standard and defined by the following equation:

$$\beta_{\text{SPM}} = \pm \sigma_{\text{SPM}} \quad \dots (1)$$

where

β_{SPM} = the overall precision of sampling

σ_{SPM} = the overall sampling, sample preparation and measurement standard deviation

t = the Student t -value corresponding to the same confidence level and to the number of replicate samples

For this Standard, the 68% confidence level has been chosen. Table C1 in Appendix C gives Student t -values for the 68% confidence level and for various numbers of replicate results. In addition, Table C2 in Appendix C gives the corresponding set of Student t -values for the 95% confidence level which is used in many Standards.

For the 68% confidence interval and when the number of replicate results is higher than 8, it is an acceptable approximation to assume $t = 1$ in Equation (1) (see Table C1). This approximation is adopted in this Guide, permitting a direct equivalence between precision and estimated standard deviation as indicated by Equation (1). Methods are given in the following Clauses for estimating from measurements on replicate samples the standard deviations of sampling, sample preparation and measurement, thereby giving directly the corresponding precisions at the 68% confidence level.

The overall standard deviation of sampling, sample preparation and measurement, σ_{SPM} in Equation (1), is a function of the variability of the particulate material, the number and, to a lesser extent, the mass of the increments, and the random errors introduced in sample preparation and measurement.

This relationship can be expressed as:

$$\sigma_{\text{SPM}}^2 = \frac{\sigma_{\text{I}}^2}{n} + \sigma_{\text{PM}}^2 \quad \dots (2)$$

where

σ_{I} = the primary increment standard deviation (see Clause 5.3.3 of AS 4433.1)

σ_{PM} = the preparation and measurement standard deviation

n = the total number of primary increments in the lot sample

The primary increment standard deviation, σ_{I} , depends upon the type and nominal top size of the particulate material, the degree of pretreatment and segregation, and the absolute value of the quality characteristic to be determined. The primary increment standard deviation may be affected also by the mass of the primary increments.

The preparation and measurement standard deviation, σ_{PM} , is related to σ_{I} and to the method of testing, but is independent of the number of increments.

5.3 Number of increments

As stated in Clause 5.2, the precision is determined by the variability of the particulate material, the number of increments and the preparation and measurement standard deviation. By combining Equations (1) and (2), it can be shown that, for a desired precision, σ_{SPM} , the required number of primary increments, n , in a single lot sample can be estimated from the following equation:

$$n = \frac{\sigma_{\text{I}}^2}{\sigma_{\text{SPM}}^2 - \sigma_{\text{PM}}^2} \quad \dots (3)$$

The use of Equation (3) to assist in the design of sampling schemes is illustrated by several examples given in Table 2 of the initial numbers of primary increments required for coals in different variability classes for moisture and ash at various levels of precision, selected merely to give realistic examples. The values of σ_{PM} have been set at levels found by experience to be achievable using off-line sample preparation.

TABLE 1
EXAMPLES OF NUMBER OF PRIMARY INCREMENTS
FOR ASH IN COAL

Primary increment standard deviation	$\sigma_{\text{I}} = 1.5$	$\sigma_{\text{I}} = 2$	$\sigma_{\text{I}} = 3$
Preparation and measurement standard deviation, σ_{PM}	0.1	0.2	0.15
Overall standard deviation, σ_{SPM}	0.2	0.5	0.2
No. of primary increments, n	75	19	514

Where samples are being taken to determine more than one quality characteristic, as for example the taking of coal samples to determine both moisture and ash, the number of increments is calculated from the desired precision, increment standard deviation and preparation and measurement standard deviation separately for each quality characteristic, and then the highest of these evaluated numbers of increments is used for the sampling plan.

Where a better precision is desired (i.e. a smaller σ_{SPM} is desired in Equation (3)), it may be achieved by collecting a larger number of primary increments and thereby reducing the effects of random sampling errors. However, there is an upper limit to the number of primary increments that can usefully be taken unless the errors of sample preparation and measurement are also reduced. This limit is reached, as can be seen from Equation (3), when

$$\sigma_{\text{SPM}} = \sigma_{\text{PM}}$$

Even before this limit is reached, the number of increments required may reach unmanageable proportions.

Example 1 (Quality characteristic is alumina in ilmenite)

Increment standard deviation for alumina, $\sigma_I = 0.56$

Desired overall precision = overall standard deviation $\sigma_{\text{SPM}} = 0.1$

Preparation and measurement standard deviation, $\sigma_{\text{PM}} = 0.095$

Therefore from Equation (3) the number of increments is given by—

$$n = \frac{0.56^2}{0.1^2 - 0.095^2} = 322$$

Where it is difficult or impossible to achieve the desired precision by testing a single gross sample from the lot, it is necessary to divide the lot into two or more, say n_s , sub-lots. Then n primary increments are taken from each of these n_s sub-lots to constitute n_s lot samples, each of which is then prepared and tested. The n_s results are then averaged to give a result for the lot. The total variance of the average of the n_s results is given by an equation similar to Equation (2), viz.

$$\sigma_{\text{SPM}}^2 = \frac{\frac{\sigma_I^2}{n} + \sigma_{\text{PM}}^2}{n_s} \quad \dots (4)$$

Equation (4) can be rearranged so that, for a desired precision, σ_{SPM} , the number of primary increments n required from each of the n_s sub-lots can be estimated from the following equation:

$$n = \frac{\sigma_I^2}{n_s \sigma_{\text{SPM}}^2 - \sigma_{\text{PM}}^2} \quad \dots (5)$$

The total number of primary increments for the whole lot in this case becomes $n_s \times n$.

Example 2 (Quality characteristic is alumina in ilmenite)

Requirements as for Example 1, but with the lot divided into 4 sub-lots.

$$n = \frac{0.56^2}{4(0.1)^2 - 0.095^2} = 10$$

Thus 10 primary increments are required per sub-lot, i.e. a total of

$4 \times 10 = 40$ primary increments from the whole lot.

Example 3 (Quality characteristic is moisture (% by wt) in coal)

Given the following parameters:

Primary increment standard deviation for moisture $\sigma_1 = 0.57$

Desired overall precision = overall standard deviation, $\sigma_{SPM} = 0.075$

Preparation and measurement standard deviation, $\sigma_{PM} = 0.1$

Coal is received each day. The overall desired standard deviation is that for a week's supply.

Therefore, the number of primary increments required per day is given by

$$n = \frac{0.57^2}{4(0.075)^2 - 0.1^2} = 11$$

Therefore, 11 primary increments per sub-lot (day) are required, or 77 for the whole lot (week).

If, in Example 3, an attempt had been made to treat the total week's delivery as a lot not broken into sub-lots, formal calculation of the number of primary increments would have yielded.

$$n = \frac{0.57^2}{0.075^2 - 0.1^2} = -74$$

The interpretation of this negative number is that it is not possible to obtain the desired overall standard deviation, because of the size of the preparation and measurement standard deviation relative to this overall standard deviation.

It is often possible, within the practical limits of the system, to achieve a desired overall standard deviation using different combinations of n and n_s , so that the optimum scheme can be designed.

5.4 Number of lots

It is recommended that pairs of interleaved samples are collected from more than 20 lots of the same type of particulate material to reach a reliable conclusion. The lot size shall be chosen to ensure this requirement is met.

5.5 Number of increments and number of samples

The minimum number of increments for checking precision should preferably be twice the number determined in accordance with AS 4433.1. Hence, if the number of increments required for routine sampling is n and one lot sample is constituted, the minimum number of increments should be $2n$, and two interleaved samples shall be constituted.

Alternatively, if the precision is being checked as part of routine sampling, n increments may be taken and two interleaved samples constituted, each comprising $n/2$ increments. The sampling variance thus obtained must be divided by 2 to obtain the sampling variance for lot samples comprising n increments.

5.6 Sample processing and analysis

Sample processing shall be carried out in accordance with AS 4433.2. The analysis of samples shall be carried out according to the methods prescribed in the relevant International or Australian Standards for the particulate material under consideration.

5.7 Frequency of precision checks

It is recommended that, even after a precision check has been conducted, further checks should be carried out at regular intervals. Precision checks should also be carried out when there is a change in equipment.

Because of the large amount of work involved in checking precision, it is recommended that checks should be carried out as a part of routine sampling and analysis.

6 PRECISION OF SAMPLE PREPARATION AND MEASUREMENT

6.1 General

As shown in Clause 5.2, the overall precision (at the 68% confidence level) of sampling, sample preparation and measurement σ_{SPM} , is determined by the primary increment standard deviation, σ_i , the total number of primary increments in the lot sample, n , and the sample preparation and measurement standard deviation, σ_{PM} .

Accordingly, the estimates of σ_{PM} should be determined periodically, and particularly whenever particulate material from a new source is being handled, or when up-stream process conditions have altered substantially.

Where the number of replicate results used to estimate σ_{PM} is higher than 8, it is a good approximation to assume that

$$\text{precision of sample preparation and measurement} = \pm \sigma_{PM} \quad \dots (6)$$

for the 68% confidence interval.

6.2 Estimating the standard deviation of sample preparation and measurement

6.2.1 General

Where the sample preparation and measurement standard deviation, σ_{PM} is unknown, or where it must be estimated again because of alterations to process operating conditions such as equipment changes, a procedure involving duplicate measurements, or a procedure involving higher order sets of replicate measurements, as described generally in Appendix D, can be conducted to determine an estimate for σ_{PM} .

The normal procedure is to make duplicate measurements. These measurements are made on test samples prepared from subsamples obtained by one of several recommended sampling procedures. For example, subsamples can be obtained as successive increments taken from a lot (Clause 6.2.2), as replicate samples constituted from successive increments taken from a lot (Clause 6.2.3), or as lot or sub-lot samples taken from a number of lots or sub-lots (Clause 6.2.4).

6.2.2 Standard deviation of sample preparation and measurement from increment samples

For a new material or when operating conditions change, an experimental sampling program should be carried out on a single lot or sub-lot in accordance with Clause 8.2.3.2 of AS 4433.1 to determine estimates for both the primary increment standard deviation, σ_i and the standard deviation of sample preparation and measurement, σ_{PM} .

The experimental sampling program to be followed is shown diagrammatically in Figure 1.

In such a program, n successive increments are taken from the lot or sub-lot and each of these increments is equally divided. From each divided portion a test sample is prepared and measured. The number of increments taken must be at least 30.

For the i th increment, the duplicate measurement values are denoted as $x_1(i)$ and $x_2(i)$.

Using the general statistical procedure in Appendix D (see Equations (D2) and (D7)), the estimate s_{PM} for the sample preparation and measurement standard deviation is given by the equation

$$s_{PM} = \frac{\sqrt{\pi} \bar{R}_{PM}}{2} \quad \dots (7)$$

The average in Equation (7) is over $R_{PM}(i)$ ($i = 1, \dots, n$) where

$$R_{PM}(i) = |x_1(i) - x_2(i)| \quad \dots (8)$$

The procedure for obtaining an estimate for the primary increment variance, s_I^2 , from the variance of the average of the duplicate measurements,

$$\bar{x}(i) = \frac{x_1(i) + x_2(i)}{2} \quad \dots (9)$$

is discussed in detail in Clause 8.2.3 of AS 4433.1.

6.2.3 Standard deviation of sample preparation and measurement from lot or sub-lot samples

In routine sampling, it is inconvenient to separately hold individual sequential increments for sample preparation and measurement as described in Clause 6.2.2, or to constitute replicate samples for preparation and measurement as described in Clause 6.2.4.

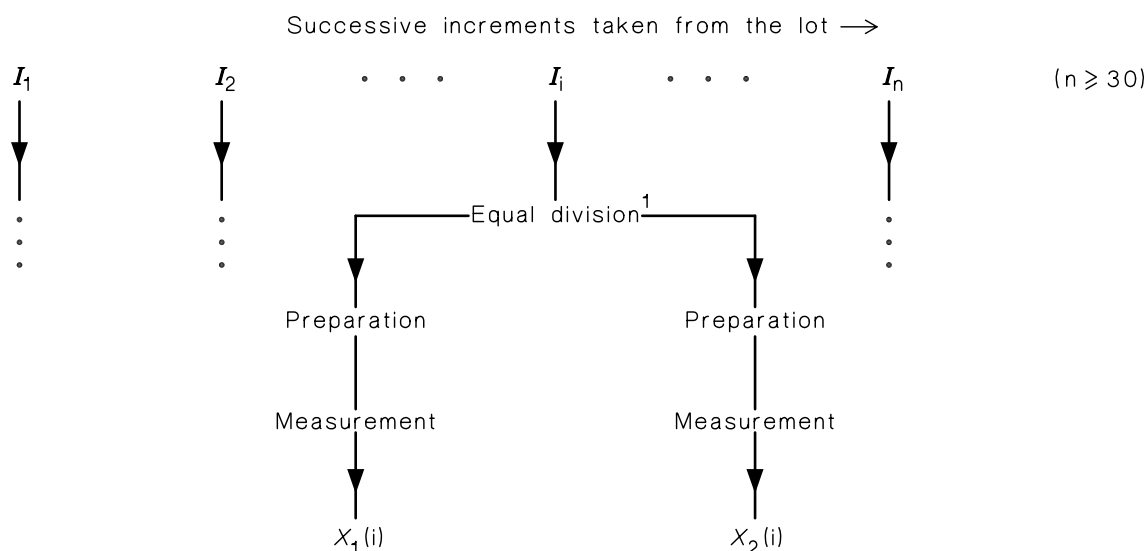
However, in routine sampling, two subsamples can be taken by equal division of a lot sample or a sub-lot sample, as shown in Figure 2, and each subsample can be prepared and measured. This procedure can be repeated on a series of sub-lot or lot samples as shown in Figure 2 until u pairs of measurements are obtained. The number of measured pairs, u , should be 10 or greater.

Following the same statistical procedure on these paired data as in Clause 6.2.2, the standard deviation of sample preparation is estimated to be

$$s_{PM} = \frac{\sqrt{\pi} \bar{R}_{PM}}{2} \quad \dots (10)$$

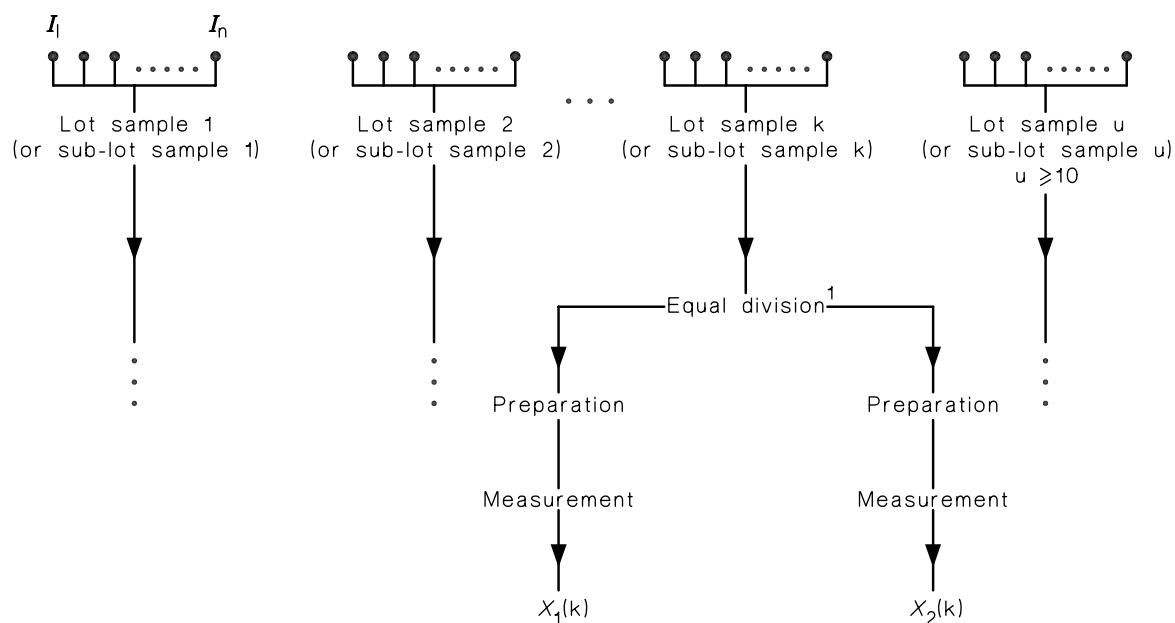
The average in Equation (10) is over $R_{PM}(k)$ ($k = 1, \dots, u$) where

$$R_{PM}(k) = |x_1(k) - x_2(k)| \quad \dots (11)$$



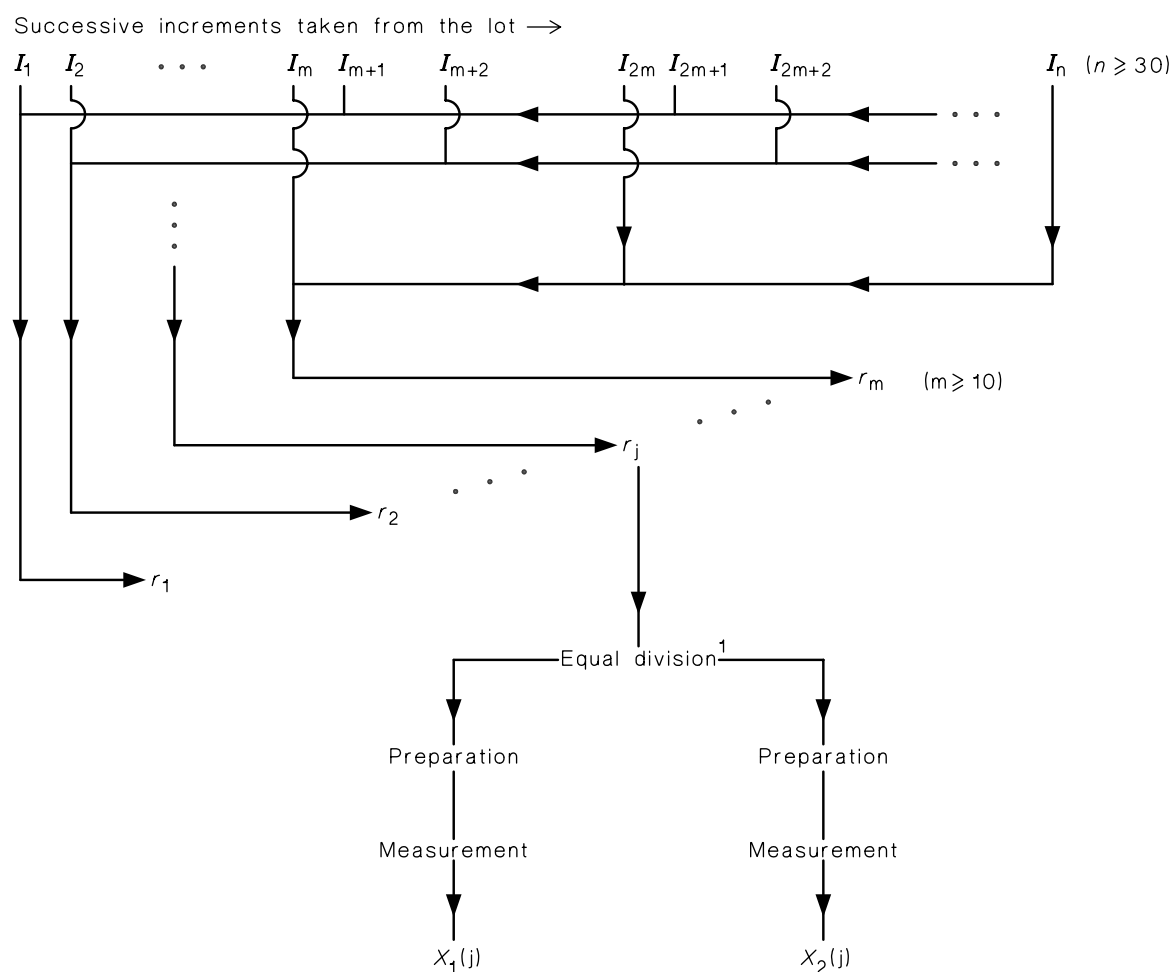
NOTE: Equal division is carried out by a normally used procedure such as riffing or rotary sample division.

FIGURE 1 SAMPLING SCHEME TO DETERMINE BOTH s_{PM} AND s_I



NOTE: Equal division is carried out by a normally used procedure such as riffing or rotary sample division.

FIGURE 2 SAMPLING SCHEME TO DETERMINE s_{PM} FROM LOT OR SUB-LOT SAMPLES



NOTE: Equal division is carried out by a normally used procedure such as riffing or rotary sample division.

FIGURE 3 SAMPLING SCHEME TO DETERMINE s_{PM} BY REPLICATE SAMPLING