

Australian Standard™

Guide to the sampling of particulate materials

Part 5: Sampling of slurries



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Guide to the sampling of particulate materials

Part 5: Sampling of slurries

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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 mineral slurries from moving streams and stationary situations.

This Guide is Part 5 of the AS 4433 series for the sampling of particulate materials, to be used as a basis for the preparation of sampling standards for a number of mineral commodities. Other Parts in the series are as follows:

- Part 1: Sampling procedures
- Part 2: Preparation of samples
- Part 3: Estimating sampling precision
- Part 4: Checking for bias
- Part 6: Inspection of mechanical sampling systems

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STANDARDS AUSTRALIA

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Guide to the sampling of particulate materials

Part 5: Sampling of slurries

1 SCOPE

This Guide sets out basic methods for sampling particulate material that is mixed with a liquid, usually water, to form a slurry. In industry and in the mining and mineral processing literature, slurry is also referred to as pulp, but this term is not used in this Guide. At very high ratios of fine particulate solids to liquids where material assumes a soft plastic form, the mixture is correctly termed a paste. Sampling of pastes is not covered in this Guide.

If the liquid contains chemical compounds in solution rather than as particulate material, the liquid is more correctly termed a liquor. While the principles of sampling slurries generally apply to liquors as well as slurries, this Guide does not cover all aspects of liquor sampling.

The procedures described in this Guide apply to sampling of particulate materials that are transported in moving streams as slurries. These streams may fall freely or be confined in pipes, launders, flumes, sluices, spirals or similar channels.

Sampling of slurries in stationary situations, such as a settled or even a well-stirred slurry in a holding vessel or dam, is not recommended and is not covered in this Guide.

The procedures described in this Guide are designed to provide samples representative of the slurry solids and particle size distribution of the slurry under examination. After filtering the slurry sample and measuring the fluid volume, damp samples of the contained particulate material in the slurry are available for drying (if required) and measurement of one or more characteristics in an unbiased manner and with a known degree of precision. The characteristics are measured by chemical analysis, physical testing or both.

The sampling methods described are applicable to slurries that require inspection to verify compliance with product specifications, determination of the value of a characteristic as a basis for settlement between trading partners or estimation of a set of average characteristics and variances that describes a system or procedure. The sampling methods are also applicable to acceptance sampling.

Provided that flow rates are not too high, the reference method against which other sampling procedures are compared is one where the entire stream is diverted into a vessel for a specified time or volume interval. This method corresponds to the stopped-belt method described in AS 4433.1.

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
4433.6	Part 6: Inspection of mechanical sampling systems

ISO

3085 Iron ores—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 PRINCIPLES OF SAMPLING SLURRIES

5.1 General

For the purposes of this Guide, a slurry is defined in Appendix A as ‘any fluid mixture of a solid with a liquid (usually water) used as a convenient form to handle solids in bulk’. Slurry flows are found in many mineral processing plants, with the water and entrained solids mixture being transported through the plant circuits by means of pumps and pipelines and under gravity in sluices, flumes and launders. Water is the preferred medium for carrying out many mineral concentration, grinding and classification processes. In a number of operations, ore is transported to the mill in slurry form, and in others concentrates are transported long distances in slurry pipelines. Tailings from wet plants are also discharged as slurries through pipelines to the tailings dam or other tailings storage facility. In many of these operations, collection of increments at selected sample points is required for evaluation of the particulate material in the slurry.

A sample is constituted from a set of unbiased primary increments from a lot or a sub-lot. If the percentage solids by mass is to be determined, the sample container is weighed immediately after collection and combination of increments to avoid water loss by evaporation or spillage. Weighing is necessary to determine the percentage of solids by mass in the slurry sample. The sample may then be filtered, dried and weighed. Alternatively, the sample may be sealed in plastic bags after filtering for transport and drying at a later stage. The liquid removed during filtration should be retained if it needs to be analysed.

Test samples are prepared from samples after filtering and drying. Test portions may then be taken from the test sample and analysed using an appropriate and properly calibrated analytical method or test procedure under prescribed conditions.

The objective of the measurement chain is to determine the characteristic of interest in an unbiased manner with an acceptable and affordable degree of precision. The general sampling theory, which is based on the additive property of variances, can be used to determine how the variances of sampling, sample preparation and chemical analysis or physical testing propagate and hence determine the overall variance for the measurement chain. This sampling theory can also be used to optimize mechanical sampling systems and manual sampling methods.

If a sampling scheme is to provide representative samples, all parts of the slurry in the lot must have an equal opportunity of being selected and appearing in the lot sample for testing. Hence, slurries are to be sampled in such a manner that all possible primary increments in the set into which the slurry can be divided have the same probability of being selected. Any deviation from this basic requirement can result in an unacceptable loss of accuracy. A sampling scheme having incorrect selection techniques, i.e. with non-uniform selection probabilities, cannot be relied upon to provide representative samples.

Sampling of slurries should preferably be carried out by systematic sampling on a time basis (see Clause 10). If the slurry flow rate and the solids concentration vary with time, the slurry volume and the dry solids mass for each increment will vary accordingly. If there are any regular patterns in the quality or flow rate of material, then it needs to be shown that no systematic error (bias) is introduced by having the proposed sampling interval approximately equal to a multiple of the period of variation in quantity or quality. Otherwise, stratified random sampling should be used (see Clause 11).

Best practice for sampling slurries is to cut freely falling streams mechanically (see Clause 12), with a complete cross-section of the stream being taken during the traverse of the cutter. Access to freely falling streams can sometimes be engineered at the end of pipes. Alternatively, a full-stream sample by-line can be added that diverts the slurry into a holding tank, or weirs can be incorporated in launders, sluices and flumes. If samples are not collected by taking a complete cross-section, non-uniform concentration of solids in the slurry due to segregation and stratification of the solids may lead to bias in the sample that is collected. Slurry in pipes can be homogeneous across the flow, with very fine particles such as clays dispersed uniformly in turbulent suspension across the diameter of the pipe. However, more commonly, the slurry in a pipe will have significant particle concentration gradients across the pipe. This is called heterogeneous flow. Examples of such flow are full pipe flow of a heterogeneous suspension or partial pipe flow of a fine particle suspension above a slower moving or even stationary bed of coarser particles.

For heterogeneous flow, bias is likely to occur where a tapping is made into the slurry pipe to locate either a flush fitting sample take-off pipe or a sample tube projecting into the slurry stream for extraction of samples. The bias is caused by non-uniform concentration profiles in the pipe and the different trajectories followed by particles of different masses due to their inertia, resulting in larger or denser particles being preferentially rejected from or included in the sample.

In slurry channels such as launders, flow is almost always heterogeneous, and the non-uniformity in particle concentration is not completely eliminated at the discharge over a weir or step. However, weirs and steps are useful sampling points because they allow complete access to the full width and breadth of the stream, thereby enabling all parts of the slurry stream to be collected with equal probability.

5.2 Sampling errors

5.2.1 General

The processes of sampling, sample preparation and measurement contribute variation to the final results. Where the average of these variations is close to zero, they are called random errors. Systematic errors and departures from prescribed procedures leading to the average of these contributions being biased away from zero, are not random errors, and statistical procedures are not applicable.

The characteristics of the solids component of a slurry can be determined by extracting samples from the slurry stream, preparing test samples and measuring the required quality characteristics. The total sampling error TE can be expressed as the sum of a number of independent components (Gy, 1982; Pitard, 1993). Such a simple additive combination would not be possible if the components were correlated. The sampling error, expressed as a sum of its components, is given by:

$$TE = QE_1 + QE_2 + QE_3 + WE + DE + EE + PE \quad \dots 5.2(1)$$

where

QE_1 = short-range quality fluctuation error associated with short-range variations in quality of the solids component of the slurry

QE_2 = long-range quality fluctuation error associated with long-range variations in quality of the solids component of the slurry

- QE_3 = periodic quality fluctuation error associated with periodic variations in quality of the solids component of the slurry
- WE = weighting error associated with variations in slurry flow rate
- DE = increment delimitation error introduced by incorrect increment delimitation
- EE = increment extraction error introduced by incorrect increment extraction from the slurry
- PE = preparation error (also known as accessory error) introduced by departures (usually unintentional) from correct practices, e.g. during constitution of the lot sample, draining and filtering away the water, and transportation and drying of the sample

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

$$QE_1 = FE + GE \quad \dots 5.2(2)$$

where

- FE = fundamental error due to variation in quality between particles
- GE = segregation and grouping error.

The fundamental error results from the composition heterogeneity of the lot, i.e. the heterogeneity that is inherent to the variation in the composition of the particles making up the solids component of the lot. The greater the differences in the compositions of particles, the greater the composition heterogeneity and the higher the fundamental error variance. The fundamental error can never be completely eliminated. It is an inherent error resulting from the variation in composition of the particles in the slurry being sampled.

The segregation and grouping error results from the distribution heterogeneity of the sampled material (Pitard, 1993). The distribution heterogeneity of a lot is the heterogeneity arising from the manner in which particles are distributed in the slurry. It can be reduced by blending before sampling or by taking a greater number of smaller increments, but it can never be completely eliminated.

A number of the components of the total sampling error, namely DE , EE and PE , can be minimized, or reduced to an acceptable level, by correct design of the sampling procedure.

5.2.2 Preparation error, PE

In this context, the preparation error includes errors associated with non-selective sample preparation operations that should not change mass, such as sample transfer, draining and filtering, drying, crushing, grinding or mixing. It does not include errors associated with sample division. Preparation errors, also known as accessory 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 made negligible by correct design of the sample plant and by staff training. For example, cross-stream slurry cutters should have caps to prevent entry of splashes when the cutter is in the parked position and care needs to be taken during filtering to avoid loss of fines that are still suspended in the water to be discarded.

5.2.3 Delimitation and extraction errors, *DE* and *EE*

Delimitation and extraction errors arise from incorrect sample cutter design and operation. The increment delimitation error, *DE*, results from an incorrect shape of the volume delimiting the slurry increment, and this can be due to both design and operation faults. Because of the incorrect shape of the slurry increment volume, sampling with non-uniform selection probabilities results. The average of *DE* is often non-zero, which makes it a source of sampling bias. The delimitation error can be made negligible if all parts of the stream cross-section are diverted by the sample cutter for the same length of time.

Sampling from moving slurry streams usually involves methods that fall into three broad operational categories as follows (Pitard, 1993):

- (a) Taking the whole stream part of the time with a cross-stream cutter as shown in Figure 1(a) (after Pitard, 1993), usually where the slurry falls from a pipe or over a weir or step. Cuts 1 and 2 show correct sampling with the cutter diverting all parts of the stream for the same length of time. Cuts 3 and 4 show incorrect sampling where the cutter diverts different parts of the stream for different lengths of time.
- (b) Taking part of the stream all of the time as shown in Figure 1(b) (after Pitard, 1993) with an on-stream point sampler or probe within a pipe or channel, which is always incorrect.
- (c) Taking part of the stream part of the time as shown in Figure 1(c) (after Pitard, 1993), also with an on-stream point sampler or probe within a pipe or channel, which is always incorrect.

The increment extraction error, *EE*, results from incorrect extraction of the slurry increment. The extraction is said to be correct if, and only if, all particles in the slurry that have their centre of gravity inside the boundaries of the correctly delimited increment are extracted. The average of *EE* is often non-zero, which makes it a source of sampling bias. The extraction error can be made negligible by ensuring that the slurry increment is completely extracted from the stream without any particulate material being lost from the cutter in splashes or slops. The depth and capacity of the cutter has to be sufficient to avoid slurry reflux from the cutter aperture, resulting in loss of part of the extracted slurry increment.

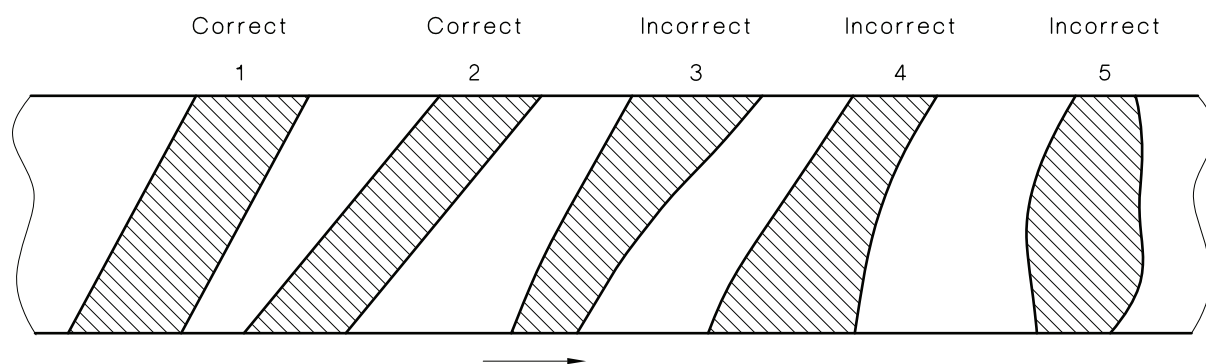
5.2.4 Weighting error, *WE*

The weighting error is an error component arising from the selection model underlying Equation 5.2(1). In the model, the time-dependent flow rate of the solids in the slurry stream is a weighting function applied to the corresponding time-dependent quality characteristic over time, which gives the weighted average quality characteristic of the solids component of the lot. The weighting error results from the application of incorrect weights to the quality characteristics. It should be negligible for time-basis sampling where the sizes of sample increments are proportional to the average of flowrate for each stratum; otherwise, the best solution to reducing the weighting error is to stabilize the flow rate. As a general rule, the weighting error is negligible for variations in flow rate up to 10%, and acceptable for variations in flow rate up to 20%.

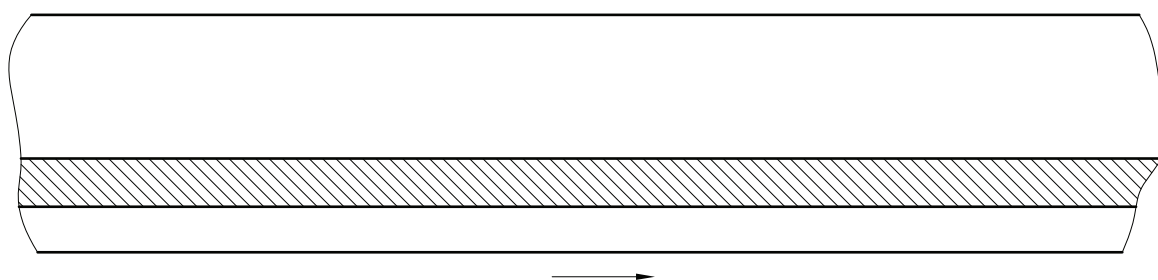
5.2.5 Periodic quality fluctuation error, *QE₃*

Periodic quality fluctuation errors result from periodic variations in quality generated by some equipment used for slurry processing and transportation, e.g. grinding and screening circuits, splitters and pumps. The presence of periodic variations can be detected by determining the variogram (see Appendix C). While in most cases variogram values can be fitted with a simple linear or quadratic function, if periodic behaviour (characterized by regularly spaced maxima and minima) is observed, the fitting function can include a sine wave term with a period and amplitude to be determined as parameters of the fit (Gy, 1982). In such cases, stratified random sampling should be carried out as discussed in Clause 11.

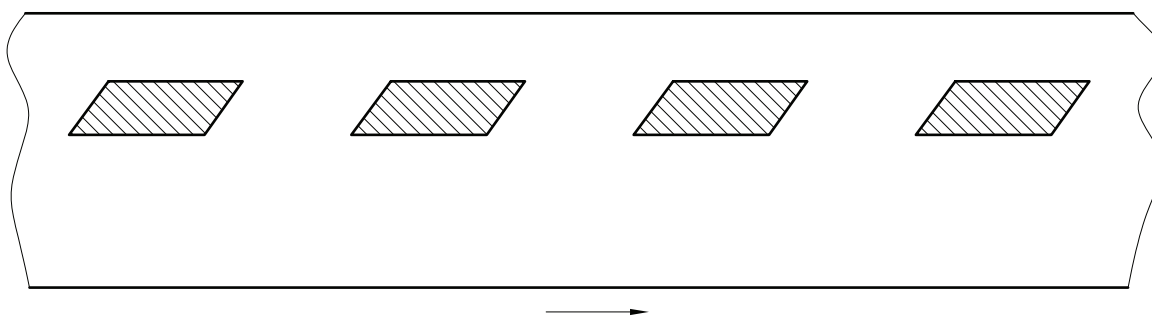
The alternative is to reduce the source of periodic variations in quality significantly, which may require plant redesign.



(a) Taking all of the stream part of the time



(b) Taking part of the stream all of the time (always incorrect)



(c) Taking part of the stream part of the time (always incorrect)

FIGURE 1 PLAN VIEW OF SLURRY VOLUMES DIVERTED BY SAMPLE CUTTERS

5.3 Sampling variance

5.3.1 General

Assume that the weighting (*WE*), increment delimitation (*DE*), increment extraction (*EE*) and preparation errors (*PE*) described in Clause 5.2 have been eliminated or reduced to insignificant values by careful design and sampling practice. In addition, assume that periodic variations in quality have been eliminated and that the flow rate has been regulated. The sampling error in Equation 5.2(1) then reduces to the form:

$$TE = QE_1 + QE_2 \quad \dots 5.3(1)$$

Hence, the sampling variance (σ_s^2) is given by:

$$\sigma_s^2 = \sigma_{QE_1}^2 + \sigma_{QE_2}^2 \quad \dots 5.3(2)$$

The short-range quality fluctuation variance, $\sigma_{QE_1}^2$ 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 solids in the slurry.

The long-range quality fluctuation variance, $\sigma_{QE_2}^2$, arises from the continuous trends in quality that occur while sampling a slurry and is usually space and time dependent. This component is often the combination of a number of trends generated by diverse causes.

The sampling variance can be estimated by using the increment variance method or, alternatively, the variogram method (see Clauses 5.3.2 and 5.3.3).

5.3.2 Increment variance method

The increment variance σ_i^2 is defined as the variance of the selected quality characteristic for increments taken from the lot, i.e.

$$\sigma_i^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2 \quad \dots 5.3(3)$$

where

- n = number of increments
- x_i = value of quality characteristic for increment i
- $\bar{x}$ = mean quality characteristic for all increments

The sample preparation and measurement variance σ_{PM}^2 subtracted from the increment variance to obtain the sampling variance component of the variation between increments, namely $\sigma_i^2 - \sigma_{PM}^2$. The estimated sampling variance for the average of n increments is then given by:

$$\sigma_s^2 = \frac{\sigma_i^2 - \sigma_{PM}^2}{n} \quad \dots 5.3(4)$$

NOTE: Equation 5.3(4) provides a conservative estimate of the sampling variance if the slope of the variogram is large (see Clause 5.3.3).

5.3.3 Variogram method

The variogram method provides a less conservative estimate of the sampling variance that takes into account the correlation between adjacent increments, but it is more complex to apply. The method is described in detail in Appendix C.

5.4 Overall variance and precision

The overall variance, denoted by σ_{SPM}^2 , comprises three components, namely the variance of sampling, the variance of sample preparation and the variance of measurement, as follows:

$$\sigma_{\text{SPM}}^2 = \sigma_{\text{S}}^2 + \sigma_{\text{P}}^2 + \sigma_{\text{M}}^2 \quad \dots 5.4(1)$$

where

σ_{S}^2 = sampling variance

σ_{P}^2 = sample preparation variance

σ_{M}^2 = measurement variance

NOTE: The distinction between ‘sampling’ and ‘sample preparation’ is not always clear. For the purposes of this Guide, ‘sampling’ stages denote those stages of sampling and sample division that take place within the sampling plant where slurry increments are extracted and where drainage of clear water is carried out after the contained solids of the sample settle. On the other hand, ‘sample preparation’ stages denote those stages that take place away from the sampling plant, typically in the plant laboratory. Sample preparation stages may include additional drainage, filtering and drying of samples before size reduction, sample division and preparation of test samples for measurement. The principles of sampling given in Clause 5.2 apply to sample preparation stages as well as to the sampling stages.

The overall precision, β_{SPM} , is a measure of the combined precisions of sampling, sample preparation, and measurement. For a symmetrical two-sided confidence level of 95% and where the number of independent comparisons (degrees of freedom) that can be made among the set of measurements is large,

$$\beta_{\text{SPM}} = 1.96\sigma_{\text{SPM}}$$

In practice, the approximation

$$\beta_{\text{SPM}} = 2\sigma_{\text{SPM}} = 2\sqrt{\sigma_{\text{S}}^2 + \sigma_{\text{P}}^2 + \sigma_{\text{M}}^2} \quad \dots 5.4(2)$$

is often used in sampling standards for bulk materials, irrespective of whether the materials are dry or in slurry form.

Where a very precise result is required and the sampling variance has been minimized, consideration has to be given to increasing the number of sample preparations and measurements to reduce these components of the overall variance. This is achieved by—

- (a) carrying out multiple determinations on the contained solids in the lot sample;
- (b) analysing the contained solids in individual increments; or
- (c) dividing the slurry lot into a number of sub-lots or part-lots and analysing the contained solids in a sample from each sub-lot.

The overall variance in each case is then given by one of the following equations:

- (i) Where a single lot sample is constituted from a lot and r replicate determinations on the contained solids are carried out on the lot sample,

$$\sigma_{\text{SPM}}^2 = \sigma_{\text{S}}^2 + \frac{\sigma_{\text{M}}^2}{r} \quad \dots 5.4(3)$$

- (ii) Where k sub-lot samples are prepared, each constituted from the contained solids of an equal number of increments, and r replicate determinations are carried out on each sub-lot sample,

$$\sigma_{\text{SPM}}^2 = \sigma_{\text{S}}^2 + \frac{\sigma_{\text{P}}^2 + \frac{\sigma_{\text{M}}^2}{r}}{k} \quad \dots 5.4(4)$$

- (iii) Where all n increments are prepared and a single determination is carried out on the contained solids of each increment,

$$\sigma_{\text{SPM}}^2 = \sigma_{\text{S}}^2 + \frac{\sigma_{\text{P}}^2}{n} + \frac{\sigma_{\text{M}}^2}{n} \quad \dots 5.4(5)$$

6 SAMPLING SCHEMES

Most sampling operations are routine and are carried out to determine the average quality characteristics of a lot as well as variations in quality characteristics between sub-lots and lots for monitoring and controlling quality. In establishing a sampling scheme for routine sampling so that the required precision for a lot can be obtained, it is necessary to carry out the following sequence of steps. This sequence includes experimental procedures, such as Step (g) below, that are non-routine and carried out infrequently, e.g. determining quality variation, particularly if a significant change has occurred to the slurry source or to the sampling equipment. The procedure is as follows:

- (a) Define the purpose for which the samples are being taken. Sampling for commercial transactions is usually the main purpose of sampling standards. However, the procedures described in this standard are equally applicable to monitoring plant performance, process control and metallurgical accounting.
- (b) Define the lot by specifying the duration of slurry flow, e.g. one day of operation.
- (c) Identify the quality characteristics to be measured and specify the overall precision (combined precision of sampling, sample preparation and measurement) required for each quality characteristic. If the required precision results in impractical numbers of increments and sub-lots, it may be necessary to adopt a poorer precision.
- (d) Ascertain the nominal top size and particle density of the solids in the slurry for determining the minimum mass of solids in the lot sample (see Clause 9).
- (e) Check that the procedures and equipment for taking slurry increments avoid bias (see Clause 7).
- (f) Determine the increment variance of the quality characteristics under consideration and the variance of preparation and measurement (see Clause 5).
- (g) Determine the number of increments to be taken from the lot and the number of sub-lots required to achieve the desired precision (see Clause 8).
- (h) Determine the sampling interval in minutes for time-basis systematic sampling (see Clause 10) or stratified random sampling within fixed time intervals (see Clause 11).
- (i) Take slurry increments at the intervals determined in Step (h) during the whole period of handling the lot.

During sampling operations, sub-lot samples may be combined to constitute a single lot sample for analysis (see Figure 2). To improve the overall precision of the measured quality characteristics of the lot, increments taken from sub-lots may be used to constitute sub-lot samples for analysis (see Figure 3). Other reasons for separate preparation and analysis of sub-lot samples are—

- (i) for convenience of materials handling;
- (ii) to provide progressive information on the quality of the lot; or
- (iii) to provide reference or reserve samples after division.

Each increment may also be analysed separately (see Figure 4) to assess the variability of the quality characteristics of the lot. In addition, it is recommended that the precision achieved in practice be checked on an ongoing basis by duplicate sampling where alternate increments are diverted to lot samples A and B (see Figure 5) from which two test samples are prepared and analysed. A substantial number (greater than 15) of data pairs are required to obtain a reliable estimate of precision.

In most situations, the solids in the slurry increment will not need to be crushed or pulverized to allow further division, since most slurries contain only fine particulate material. However, if the particles are coarse and particle size reduction is required to allow further division, it is necessary to recalculate the minimum sample mass for the lot using the new nominal top size of the crushed solids (see Clause 9).

The initial design of a sampling scheme for a new plant or a slurry having unfamiliar characteristics should, wherever possible, be based on experience with similar handling plants and material types. Alternatively, a substantial number of increments, e.g. 100, can be taken and used to determine the quality variation of the contained solids, but the precision of sampling cannot be determined *a priori*.

Sampling of slurries in stationary situations, such as storage tanks, mixer tanks and tailings dams, is not recommended and is not covered in this Guide.

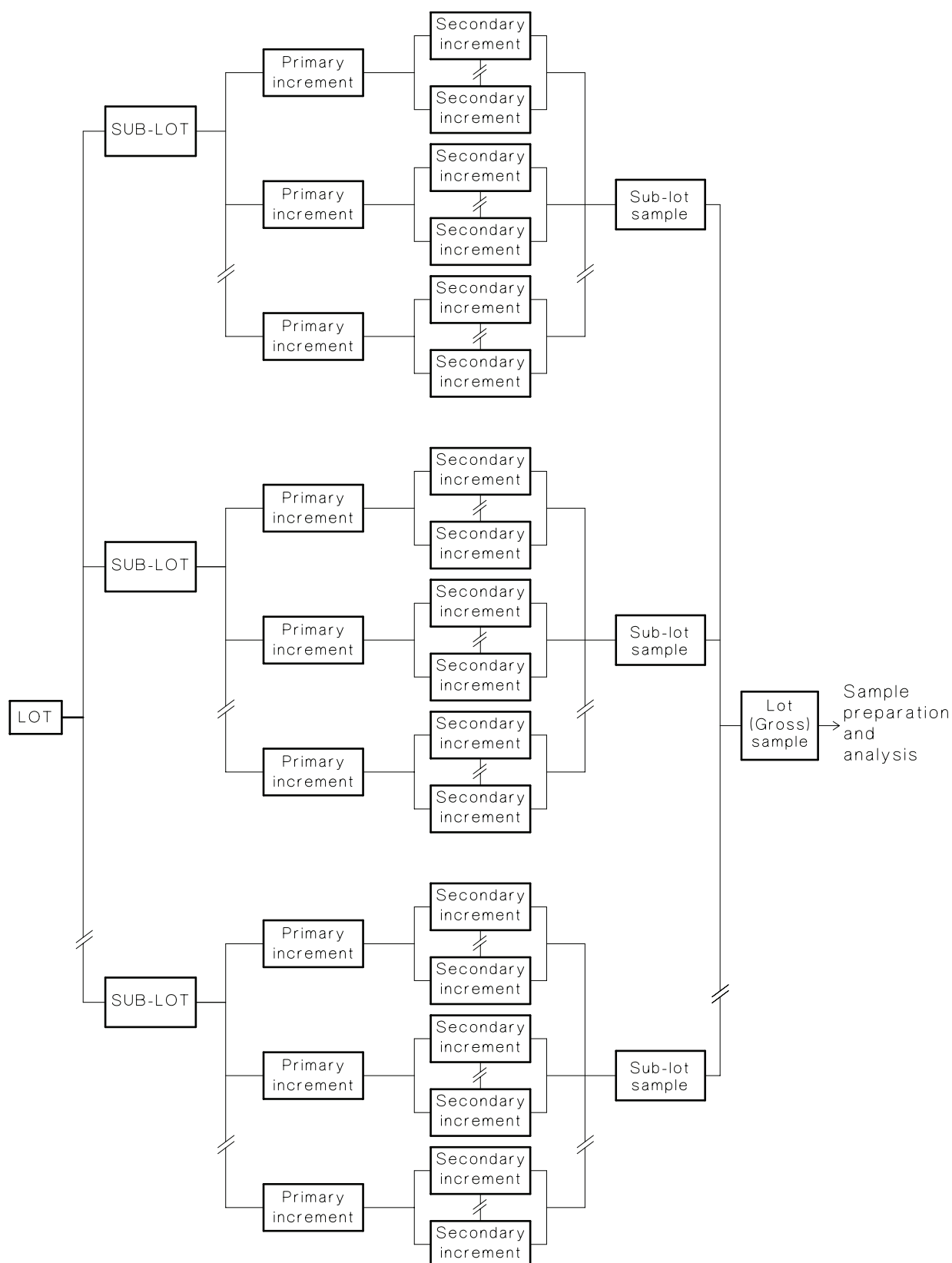


FIGURE 2 EXAMPLE OF A SAMPLING PLAN WHERE A SINGLE LOT SAMPLE IS CONSTITUTED FOR ANALYSIS