

Australian Standard[®]

Coal and coke—Sampling

Part 1: Coal—Sampling procedures



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 - Australian Building Codes Board
 - Australian Coal Association
 - Australian Coal Preparation Society
 - Coalfield Geology Council of NSW
 - CSIRO, Energy Technology
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Coal and coke—Sampling

Part 1: Coal—Sampling procedures

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PREFACE

This Standard was prepared by the Standards Australia Committee MN-001, Coal and Coke, as a revision of AS 4264.1—1995, *Coal and coke—Sampling, Part 1: Higher rank coal—Sampling procedures* and AS 4264.3—1996, *Coal and coke—Sampling, Part 3: Lower rank coal—Sampling procedures*.

This Standard incorporates Amendment No. 1 (September 2011). The changes required by the Amendment are indicated in the text by a marginal bar and amendment number against the clause, note, table, figure or part thereof affected.

Other Standards in this series are as follows:

Part 2: Coke—Sampling procedures

Part 4: Determination of precision and bias

Part 5: Guide to the inspection of mechanical sampling systems

This edition revises and amalgamates the content of AS 4264.1—1995 and AS 4264.3—1996 in order to provide a single standard on sampling procedures for all ranks of coal.

In preparing this revision, reference was made to ISO 13909, Parts 1 to 4 on the mechanical sampling of hard coal and includes tables and information obtained from those standards.

The objective of this Standard is to provide those responsible for sampling coal with a set of rules that will ensure that the subsequent testing of the coal is representative of the full quantity of coal being sampled.

The terms ‘normative’ and ‘informative’ have been used in this Standard to define the application of the Appendices to which they apply. A normative appendix is an integral part of a Standard, whereas an informative appendix is for information and guidance only.

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STANDARDS AUSTRALIA

Australian Standard
Coal and coke—Sampling

Part 1: Coal—Sampling procedures

SECTION 1 SCOPE AND GENERAL

1.1 SCOPE

This Standard sets out methods for the sampling of coal from moving streams and stationary situations, including stopped-belt sampling, for both routine and special purposes, to provide samples for general analysis and for the determination of total moisture.

The provisions of this Standard specifically include stopped-belt sampling. Although this Standard is intended to cover all coal sampling from moving streams, the procedures recommended may not be applicable in cases of extreme segregation of material, as found with very wet coal owing to moisture run-off, or with very dry coal owing to generation of dust. In such cases, it may be necessary to revert to stopped-belt sampling.

Stopped-belt sampling is the reference method against which other sampling procedures are compared.

Methods for the preparation of samples are also included in this Standard, and it is recognized that some on-line sampling installations perform part of the sample preparation.

Sampling from coal seams is covered in AS 2617, and is excluded from this Standard.

1.2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

1038	Coal and coke—Analysis and testing
1038.1	Part 1: Higher rank coal—Total moisture
1038.10.0	Part 10.0 Determination of trace elements—Guide to the determination of trace elements
1038.12.3	Part 12.3: Higher rank coal—Caking and coking properties—Dilatation
1038.12.4.1	Part 12.4.1: Higher rank coal—Caking and coking properties—Plasticity—Continuous-torque Gieseler method
1038.19	Part 19: Higher rank coal—Abrasion index
1038.20	Part 20: Higher rank coal—Hardgrove grindability index
1152	Specification for test sieves
1681	Safety requirements for electrically heated Type 1 ovens in which flammable volatiles occur
2267	Guide to the conduct of pilot coke oven tests
2418	Coal and coke—Glossary of terms
2434	Methods for the analysis and testing of lower rank coal and its chars
2434.1	Part 1: Determination of the total moisture content of lower rank coal
2434.3	Part 3: Lower rank coal—Determination of the moisture holding capacity

AS	
2617	Sampling from coal seams
2856	Coal petrography
2856.1	Part 1: Preparation of coal samples for incident light microscopy
3881	Higher rank coal—Size analysis
4156	Coal preparation
4156.1	Part 1: Higher rank coal—Float and sink testing
4264	Coal and coke—Sampling
4264.4	Part 4: Determination of precision and bias
4264.5	Part 5: Guide to the inspection of mechanical sampling systems

1.3 DEFINITIONS

For the purpose of this Standard, the definitions given in AS 2418 and those below apply.

1.3.1 Bias

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

1.3.2 Coefficient of variation (*cv*)

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

1.3.3 Common sample

A gross sample on which total moisture and other tests are conducted.

1.3.4 Constant-mass division

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

1.3.5 Cut

A single traverse of the sample cutter through the coal stream.

1.3.6 Divided increment

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

1.3.7 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.

1.3.8 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 sub-lots.

1.3.9 Error

The differences between the experimental results and the true figures; errors can be systematic (bias), random, or due to either loss or contamination of sample.

1.3.10 Fixed-rate division

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

1.3.11 General analysis sample

The sample crushed to pass a sieve aperture of 212 μm ; used for the determination of some physical and most chemical characteristics of the coal.

1.3.12 Gross sample

A sample formed when all of the primary increments or sub-lot samples for the lot, 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.

1.3.13 Increment

The quantity of coal taken in one operation.

1.3.14 Laboratory sample

The sample delivered to the laboratory for analysis or testing.

1.3.15 Lot

A quantity of coal delivered at one time; it may comprise one or more sub-lots.

1.3.16 Manual sampling

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

1.3.17 Mass-basis sampling

The taking of increments at or within uniform mass intervals throughout the sub-lot or lot.

1.3.18 Mechanical sampling

The operation of sampling whereby the increments forming sub-lot samples and gross samples are taken by mechanical means.

1.3.19 Nominal top size

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

1.3.20 Pass

The passage of retained material once through a sample divider.

1.3.21 Precision

A measure of the extent to which the observations within a set agree with each other; expressed as twice the standard deviation (95% confidence level).

1.3.22 Random error

Errors that arise from the experimental procedure and lead to results that are sometimes higher and sometimes lower than the true values; in the absence of bias, the average of a number of experimental results subject to only random errors will approach the true value as the number of results increases.

1.3.23 Range of influence

The separation in mass, time or space between increments above which they cease to be correlated.

1.3.24 Reduction (in particle size)

The process of decreasing the dimension of the pieces constituting the sample, without modification of the mass of the sample or of its composition.

1.3.25 Replicate sampling

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

1.3.26 Sample preparation

The process of preparing the sample for analysis or testing; it covers air-drying, mixing, particle size reduction, and division, and may be performed in several stages.

1.3.27 Size analysis sample

A sample taken specifically for the determination of size analysis.

1.3.28 Stage

A sequence of operations comprising reduction in particle size, mixing, and culminating in sample division. The number of stages in sample preparation is equal to the number of divisions made.

1.3.29 Standard deviation

A measure of the dispersion of a statistical sample, based on the square root of the mean squared deviation from the arithmetic mean. For a series of n observations $x_1, x_2 \dots, x_n$

having mean $\sigma = \frac{1}{n-1} \sum x_p$ an estimate of the standard deviation (σ) is given by—

$$\sigma = \left[\frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2 \right]^{0.5}$$

NOTE: Although the standard deviations used in this Standard are sample estimates of the corresponding population parameters, they are denoted by the symbol σ and not s .

1.3.30 Strata

Approximately equal parts of a lot or sub-lot based on intervals of time, mass or space.

1.3.31 Stratified random sampling

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

1.3.32 Sub-lot

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

1.3.33 Sub-lot sample

A sample representative of the whole sub-lot and constituted to prepare laboratory samples.

1.3.34 Systematic sampling

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

1.3.35 Time-basis sampling

The taking of increments at or within uniform time intervals throughout the lot or sub-lot.

1.3.36 Total moisture sample

A sample used exclusively for the determination of total moisture.

1.3.37 Top size

The size of aperture of the finest sieve (complying with AS 1152) through which a minimum of 99% of the mass of material passes.

1.3.38 Variance

The square of the standard deviation.

SECTION 2 SAMPLING PROCEDURES

2.1 SCOPE OF SECTION

This Section sets out procedures for the sampling of coal from stationary situations and moving streams. Stopped-belt sampling is included.

2.2 ESTABLISHING A SAMPLING SCHEME

2.2.1 General

Sampling should be carried out by systematic sampling, either on a time basis (see Clause 2.6) or on a mass basis (see Clause 2.7), but only if it can be shown that no systematic error could be introduced by periodic variation in quality or quantity having a period approximately equal to any multiples of figures within the proposed range of sampling intervals.

As an example, a primary cutter may be cutting a stream of coal that is being reclaimed from a stockpile by a bucket-wheel reclaimer. At both limits of the bucket-wheel traverse across the coal face on the stockpile, the coal may have different properties from those of the stockpile average (e.g. due to 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 coincides with coal being delivered from the limit of a traverse of the bucket-wheel reclaimer. A systematic error could arise.

The same recommendation applies to secondary and subsequent stages of division, where it is felt that, because of the manner in which the coal is handled and presented to subsequent division apparatus, a systematic error could arise.

In such cases, it is strongly recommended that sampling be carried out by the method of stratified random sampling within fixed time or mass intervals (see Clause 2.8).

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

The general procedure for sampling is as follows:

- (a) Decide for what purpose the samples are being taken, e.g. monitoring plant performance, process control, use in commercial transactions.
- (b) Decide on the quality characteristics that are to be measured.
- (c) Define the lot.
- (d) Specify the overall precision (sampling, sample preparation and measurement) required for each quality characteristic. It may be found that the required precision gives impractical numbers of primary increments and sub-lots. In such cases, it may be necessary to accept a poorer precision.
- (e) Ascertain the nominal top size of the coal, to determine the minimum gross sample mass and the minimum cutter opening where a mechanical sampler is used, or the size of the ladle where manual sampling is used.
- (f) Determine the masses of the increments at the primary, secondary and subsequent stages of sampling, to minimize the likelihood of bias (see Clause 2.3).

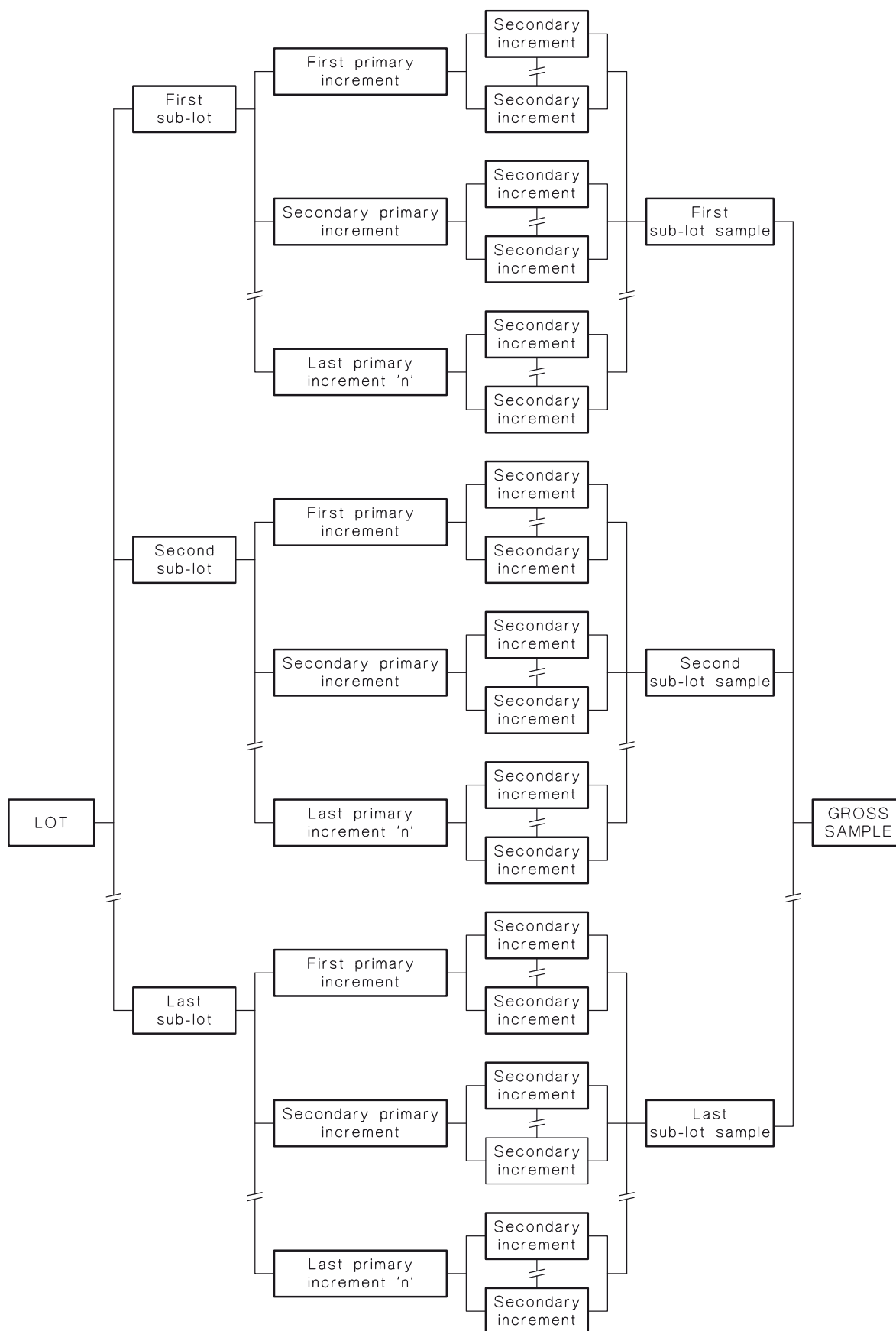
- (g) Determine the number of primary increments and the number of sub-lots required to achieve the desired precision. Sub-lots should be of equal mass or equal time within a lot. If the initial data required for determination of the numbers of primary increments and sub-lots are not known, preliminary testing will have to be carried out (see Clause 2.4.2.2(b)).
- (h) Determine the sampling interval, in tonnes for mass-basis sampling and stratified random sampling within fixed mass intervals, or in minutes for time-basis sampling and stratified random sampling within fixed time intervals.
- (i) Take primary increments at the intervals determined in Step (h), by either mass-basis sampling or time-basis sampling, during the whole period of handling the entire lot.
- (j) Determine the method of combining the increments (primary, secondary or tertiary) and then constitute a gross sample or sub-lot samples, if required. (An example is given in Figure 2.1)

2.2.2 Total moisture determination

Normally, the total moisture content of a lot is determined on the gross sample taken from the lot. However, where a lot requiring a total moisture determination is large (e.g. where increments are taken over a period of more than 2 h), it is recommended that the tests be carried out on a number of sub-lots, and the mean of the test results, weighted according to the sizes of the sub-lots, reported. Sub-lot samples may be formed for periods exceeding 2 h, provided that it can be demonstrated that no significant loss of moisture would occur in the sub-lot sample. This should reduce any bias in the test result caused by moisture loss (or gain) due to climatic conditions, and will also result in better precision (see Clause 2.4.3).

In general, the amount of moisture loss (or gain) for an increment, when exposed to the atmosphere between the time it is extracted from the lot and the time at which laboratory testing commences, will be a function of—

- (a) climatic conditions (e.g. higher temperature can lead to greater evaporation of surface moisture); and
- (b) degree of particle size reduction (e.g. greater size reduction gives an increased surface area per unit mass, which can result in greater evaporation of surface moisture).



NOTE: Mixing, reduction and division steps have been omitted for simplicity.

FIGURE 2.1 EXAMPLE OF A PLAN OF SAMPLING

2.3 MASS OF INCREMENT

2.3.1 General

To minimize bias, it is essential that increments be extracted from the lot in such a manner that all parts of the coal have an equal probability of being selected and becoming part of the final sample for testing, irrespective of the size, mass or density of individual particles.

The minimum mass of an increment needs to be determined for the following three circumstances:

- (a) Sampling from a falling stream.
- (b) Sampling from a moving conveyor belt.
- (c) Sampling manually from stationary situations.

In the first two cases, the minimum mass of an increment is dependent upon, and will be determined by, the flow rate of the coal on the belt, the minimum cutter aperture, and either the maximum cutter speed for falling-stream samplers or the conveyor belt speed for cross-belt samplers. In the third case, the increment mass is determined by the nominal top size and bulk density of the coal; it is not dependent on any other variable factor.

To maintain the integrity of the increment as it passes through the sampling system, the minimum mass of an increment should never be less than 100 g, except where it can be shown that there is no loss of integrity of the individual increment, and the increment obtained reports directly to the sample container and not via chutes or other conveying devices. In such cases, increments having smaller masses can be tolerated. However, the minimum increment mass shall never be less than 35 g.

2.3.2 Minimum mass of increment for falling-stream samplers

At any sampling stage, the minimum mass of increment, taken by a cutter-type sampler (mechanical or manual) from the coal stream at the discharge end of a moving stream, is determined by the minimum cutter aperture and the maximum cutter speed. It may be calculated using the following equation:

$$m_i = \frac{Ca}{3.6 v_c} \quad \dots 2.1$$

where

- m_i = minimum mass of increment, in kilograms
- C = flow rate of coal stream, in tonnes per hour
- a = minimum cutting aperture of the sampler, in metres (see Clause 2.10.3.2)
- v_c = maximum cutter speed of the sampler, in metres per second (see Clause 2.10.3.4)

2.3.3 Minimum mass of increment for cross-belt samplers

The minimum mass of increment taken by a cross-belt sampler from a moving stream is determined by the minimum cutter aperture and the speed of the conveyor belt. The cutter speed shall be at least 1.5 times the belt speed. The minimum increment mass may then be calculated using the following equation:

$$m_i = \frac{Ca}{3.6 v_b} \quad \dots 2.2$$

where

- m_i = minimum mass of increment, in kilograms
- C = flow rate of coal stream, in tonnes per hour

- a = minimum cutting aperture of the sampler, in metres (see Clause 2.10.3.2)
- v_b = speed of the conveyor belt, in metres per second

2.3.4 Minimum mass of increment for manual sampling

The minimum mass of increment for manual sampling (see Clause 2.9.4) is dependent upon the size of the sampling implement. These masses are specified in Appendix A.

2.4 NUMBER OF PRIMARY INCREMENTS AND SUB-LOTS

2.4.1 General

The number of primary increments to be taken from a lot, to attain a certain precision, is a function of the variability of the lot. This variability depends upon the size of the lot, the variation in coal properties as the stream passes the sampling point, the range of particle sizes in the stream, the particle-to-particle variability of the quality characteristics, the local segregation of the coal, and the manner in which it is fed onto the belt or laid down.

The variability (total variance) consists of three components, namely the sampling variance, the sample preparation variance and the measurement variance. Therefore, where a very precise result is required, e.g. for commercial transactions, consideration needs first to be given to increasing the number of primary increments. Further improvement in precision may then be achieved by carrying out a number of sample preparations and measurements to reduce these components of the overall variance. This is achieved by dividing the lot into a number of sub-lots and preparing and testing a sample from each sub-lot.

NOTE: Certain parameters, most notably total moisture, require special consideration, because it is extremely difficult, to retain the integrity of the sample over extended periods of sample collection.

There are three methods for determining the sampling variance, and hence the number of primary increments and sub-lots.

The first and simplest method is to determine the increment standard deviation σ_i from analysis of successive increments taken from a lot or sub-lot (see Clause 2.4.2). This method assumes that the coal quality varies in a random manner throughout the mass being sampled and hence tends to overestimate the sampling variance if there is significant serial correlation between increments (see Clause 2.4.4). Consequently, the calculated number of primary increments and sub-lots required to achieve the desired sampling variance will be larger than really necessary. However, the method is easier to apply than the alternatives and it errs on the safe side.

The second method is to determine the standard deviation within strata σ_w , by conducting duplicate sampling (see Clause 2.4.3), which takes some account of serial correlation between increments. However, the standard deviation obtained may depend on the size of the strata used for data collection, so this method should preferably be used where the resultant sampling interval is not greatly different from that used to collect the data. A significant advantage of the method is that it can be applied during routine sampling operations.

The third method is to determine the variogram from the analysis of successive increments taken from the coal stream. This method provides the best estimate of the sampling variance, and hence the number of primary increments and sub-lots. Extrapolation to different sampling intervals is possible, because full account is taken of serial correlation between increments which frequently occurs, i.e. even though the variation in quality is essentially random, the random variations are such that increments taken from the stream within a certain time (or mass) range will tend to be similar in analysis, with increments becoming more similar as the time (or mass) interval between their acquisition becomes smaller. The variogram method uses exactly the same data as the increment standard deviation method, although the calculations are more complex.

2.4.2 Calculation of number of primary increments and sub-lots from the increment standard deviation

2.4.2.1 Preliminary considerations

There are a number of items to be considered in determining the number of primary increments and sub-lots. These are as follows:

- (a) The coal to be sampled shall be well defined. As the variability will depend upon such factors as seams mined, processing and blending practice, it is important to establish that all of these factors are to remain virtually unchanged over the period in which sampling is to be performed, to ensure that the overall precision of sampling, sample preparation and measurement remains constant.
- (b) The size of the lot or sub-lot on which the average quality is to be measured shall be defined. The size of the lot or sub-lot may change, for example, with export shipments, owing to variations in vessel sizes and client requirements. In such cases, the sampling scheme should be designed to give the most acceptable precision for the smallest lots or sub-lots handled.
- (c) Samples to be taken for determining total moisture or free moisture should receive special consideration, to reduce bias in the test result caused by moisture loss or gain.
- (d) The precision required, expressed as twice the standard deviation (95% confidence level), has to be established.

NOTE: For commercial sampling of coal, the precision required will normally be about 0.2% absolute up to 10% ash and about 2% relative above 10% ash (e.g. at 12% ash, the precision required will be about 0.24%). In the case of mine and coal preparation plant sampling for plant control, such high precisions are not normally required, as it is possible to average the results of several shifts or a week's production to achieve results of high precision if needed. For mine and preparation plant applications therefore, the precision required per lot will normally be of the order of about 5% relative in ash. It is recommended that a sampling, preparation and analysis precision check for ash be carried out on each coal type handled by a sampling plant involved in commercial sampling of coal. This can be done by forming duplicate samples as per AS 4264.4 from a lot or sub-lot.

2.4.2.2 Calculation of number of primary increments

The following methods apply to calculation of the number of primary increments:

- (a) *Calculation of number of primary increments where the overall standard deviation, increment standard deviation and preparation and measurement standard deviation are known or specified* The number of primary increments, n , required to achieve a desired overall standard deviation, σ_{SPM} , can be calculated from the following equation:

$$n = \frac{\sigma_I^2}{\sigma_{\text{SPM}}^2 - \sigma_{\text{PM}}^2} \quad \dots 2.3$$

where

n = number of primary increments

σ_I = primary increment standard deviation, normally of ash, although other critical parameters such as sulfur or calorific value may be used

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

σ_{PM} = standard deviation of sample preparation and measurement

As an example of the use of Equation 2.3, it can be seen that if the required overall standard deviation in ash, σ_{SPM} , is 0.5 and σ_I and σ_{PM} are 3 and 0.2 respectively, the number of increments n is 43, as shown.

$$\sigma_I = 3$$

$$\sigma_{\text{SPM}} = 0.5$$

$$\sigma_{\text{PM}} = 0.2$$

$$n = \frac{(3)^2}{(0.5)^2 - (0.2)^2} = 42.857 = 43 \text{ primary increments}$$

Equation 2.3 is not applicable in all cases. If, for instance, the overall standard deviation in the above example is changed to 0.1, $\sigma_{\text{SPM}}^2 - \sigma_{\text{PM}}^2$ becomes negative and n cannot be calculated. If the introduction of sub-lots, as outlined in Clause 2.4.3, fails to produce a practical number of sub-lots and increments, a new overall standard deviation will have to be chosen. Such a standard deviation will necessarily be worse than 0.1.

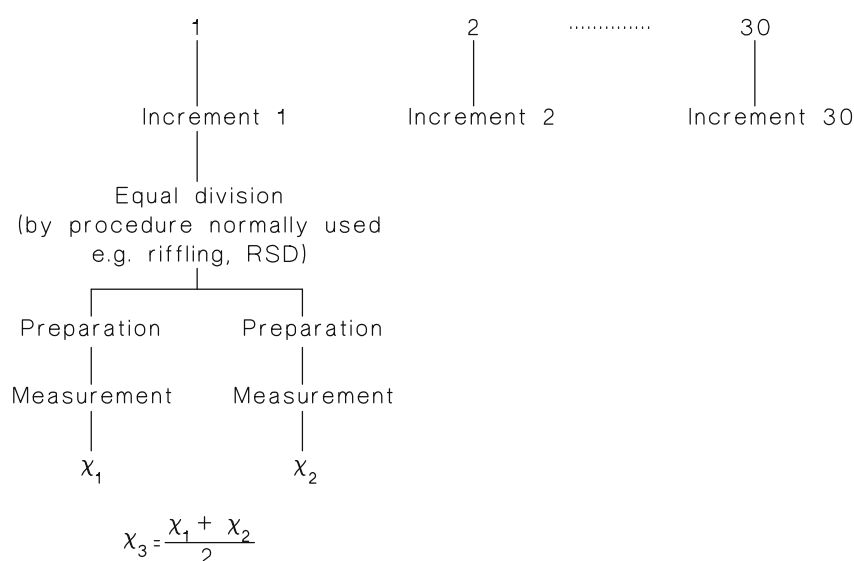
- (b) *Calculation of number of primary increments where the overall standard deviation is specified, but the increment standard deviation and preparation and measurement standard deviation are not known* Where the primary increment standard deviation (σ_I) and the preparation and measurement standard deviation (σ_{PM}) are unknown, or where the sampling characteristics of the coal have not been established previously, a preliminary sampling program has to be conducted in accordance with Figure 2.2 to enable the determination of σ_I and σ_{PM} . A minimum of 30 increments are taken from a lot or sub-lot and subsequently prepared and analyzed in duplicate.

Because the sampling characteristics of the coal are unknown at the commencement of the sampling program, no prior statement can be made about the overall standard deviation.

NOTE: If an existing sampling system is being checked, the sampling interval should correspond to that normally used.

When σ_I and σ_{PM} have been calculated (see Figure 2.2), these values, together with the desired overall standard deviation, are substituted in Equation 2.3 to enable calculation of the number of primary increments.

An example of the number of primary increments calculated according to the above is shown in Table 2.1.



NOTE: For $n_s = 10$ sub-lots and $n = 5$ increments per sub-lot

- (a) Collect at least 30 successive increments from a sub-lot and prepare and analyse each increment in duplicate.
- (b) Calculate the preparation and measurement standard deviation σ_{PM} as follows:

$$\sigma_{PM} = \frac{\sum_{i=1}^n R_{PMi}}{n} \times 0.886$$

where

R_{PMi} = absolute difference between analyses for each increment, i.e.
 $|x_1 - x_2|$

0.886 = a statistical factor relating range to standard deviation for a normal distribution

$$= (\pi/4)^{0.5}$$

n = number of increments prepared and analysed

- (c) Calculate the variance V of the mean analyses $\bar{x}_i$ as follows:

$$V = \frac{\sum_{i=1}^n (\bar{x}_{3i} - \bar{\bar{x}})^2}{n-1}$$

where

$\bar{x}_{3i}$ = mean analysis for increment i

$\bar{\bar{x}}$ = mean analysis for all increments

n = number of increments

- (d) Calculate the increment standard deviation σ_I as follows:

$$\sigma_I = \left(V - \frac{\sigma_{PM}^2}{2} \right)^{0.5}$$

FIGURE 2.2 SAMPLING SCHEME TO DETERMINE σ_I AND σ_{PM}

TABLE 2.1
EXAMPLE OF HOW TO CALCULATE THE INCREMENT
STANDARD DEVIATION

(where the overall standard deviation is specified, but the increment standard deviation and preparation and measurement standard deviation are not known)

Increment No.	Ash % (d) x_1	Ash % (d) x_2	$x_3 = (x_1+x_2)/2$	$R_{PM} = \text{abs}(x_1-x_2)$	$x_3 - \bar{x}$	$(x_3 - \bar{x})^2$
1	13.9	14.1	14.00	0.2	1.14	1.31
2	11.8	11.5	11.65	0.3	-1.21	1.46
3	9.7	9.5	9.60	0.2	-3.26	10.61
4	14.0	13.8	13.90	0.2	1.04	1.09
5	11.4	11.7	11.55	0.3	-1.31	1.71
6	15.1	15.3	15.20	0.2	2.34	5.49
7	8.3	8.0	8.15	0.3	-4.71	22.15
8	10.5	10.7	10.60	0.2	-2.26	5.09
9	11.1	10.8	10.95	0.3	-1.91	3.64
10	12.6	12.4	12.50	0.2	-0.36	0.13
11	14.8	14.8	14.80	0.0	1.94	3.78
12	11.3	11.2	11.25	0.1	-1.61	2.58
13	10.7	10.6	10.65	0.1	-2.21	4.87
14	12.6	12.4	12.50	0.2	-0.36	0.13
15	13.8	13.7	13.75	0.1	0.89	0.80
16	14.3	14.3	14.30	0.0	1.44	2.08
17	10.7	10.6	10.65	0.1	-2.21	4.87
18	15.6	15.6	15.60	0.0	2.74	7.53
19	16.4	16.6	16.50	0.2	3.64	13.27
20	9.2	9.2	9.20	0.0	-3.66	13.37
21	13.3	13.2	13.25	0.1	0.39	0.15
22	14.3	14.5	14.40	0.2	1.54	2.38
23	12.0	11.6	11.80	0.4	-1.06	1.12
24	15.2	15.5	15.35	0.3	2.49	6.22
25	10.7	10.5	10.60	0.2	-2.26	5.09
26	16.8	17.0	16.90	0.2	4.04	16.35
27	17.0	17.0	17.00	0.0	4.14	17.17
28	15.8	15.6	15.70	0.2	2.84	8.08
29	11.1	11.0	11.05	0.1	-1.81	3.26
30	12.5	12.2	12.35	0.3	-0.51	0.26

Using the Table above and the equations in Figure 2.2:

$$\begin{aligned}
 n &= 30 \\
 \sum(R_{PM}) &= 5.2 \\
 \sigma_{PM} &= 0.154 \\
 \bar{x} &= 12.86 \\
 V &= 5.72 \\
 \sigma_I &= 2.39
 \end{aligned}$$

2.4.2.3 Examples of calculation of numbers of primary increments

To assist with the design of sampling schemes, examples are given in Table 2.2 of the initial numbers of primary increments required for coals in different variability classes for ash at an arbitrary level of precision, selected merely to give realistic examples. The values of σ_{PM} have been set at levels found to be achievable using off-line sample preparation.

The number of primary increments used for ash is assumed to be adequate for total moisture.

From the number of primary increments shown in Table 2.2, it can be seen that if the coal is treated as a single lot the number of primary increments required to be taken may be quite large. It may then be necessary to divide the lot into a number of sub-lots.

NOTE: Where a single lot of coal of unknown sampling characteristics is to be sampled, and where it is not possible to carry out a preliminary measurement program to determine the primary increment standard deviation and the preparation and measurement standard deviation, the minimum number of primary increments to be taken should be decided. Taking the number of increments below provides no guarantee that the desired overall standard deviation will be achieved. The following numbers may be taken as a guide:

Mass of lot, t	Number of primary increments
1 000	60
5 000	135
10 000	180
25 000	300
50 000	420
100 000	600

2.4.3 Calculation of number of primary increments and sub-lots from the standard deviation within strata

The within-strata standard deviation method may be implemented by replacing σ_I with σ_w in the equations in Clause 2.4.2, although different methods are used to determine σ_I and σ_w . Where the standard deviation within strata (σ_w) and the preparation and measurement standard deviation (σ_{PM}) are unknown, or where the sampling characteristics of the coal have not been established previously, a preliminary duplicate sampling program has to be conducted in accordance with Figure 2.3 to enable the determination of σ_w and σ_{PM} .

If no previous information is available the sampling scheme shown in Figure 2.3. can be implemented. A total of 100 primary increments are taken and divided into 10 sub-lots, two sets of samples (comprising $n = 5$ increments) being taken from each sub-lot.

NOTE: Alternatively, if an existing sampling system is being checked, the number of increments should be twice the number normally taken for routine sampling.

Because the sampling characteristics of the coal are unknown at the commencement of the sampling program, no prior statement can be made about the overall standard deviation. However, the sampling scheme shown in Figure 2.3 gives, as additional information, the overall standard deviation actually obtained.

TABLE 2.2

AN EXAMPLE OF HOW TO CALCULATE THE NUMBER OF PRIMARY INCREMENTS PER LOT AND SUB-LOT
(For overall precisions (β_{SPM}) ranging from 0.2 to 0.4% ash for preparation and testing standard deviations (σ_{PM}) of 0.1, 0.15 and 0.2% ash and primary increment standard deviations (σ_I) of 1.5, 2 and 3% ash)

Lot and sub-lot details			$\beta_{SPM} = 0.2\% \text{ ash}$																	
Mass of lot, t	Mass of sub-lot, t	Number of sub-lots	$\sigma_{PM} = 0.1\% \text{ ash}$						$\sigma_{PM} = 0.15\% \text{ ash}$						$\sigma_{PM} = 0.2\% \text{ ash}$					
			Number of primary increments						Number of primary increments						Number of primary increments					
			$\sigma_I = 1.5\% \text{ ash}$		$\sigma_I = 2\% \text{ ash}$		$\sigma_I = 3\% \text{ ash}$		$\sigma_I = 1.5\% \text{ ash}$		$\sigma_I = 2\% \text{ ash}$		$\sigma_I = 3\% \text{ ash}$		$\sigma_I = 1.5\% \text{ ash}$		$\sigma_I = 2\% \text{ ash}$		$\sigma_I = 3\% \text{ ash}$	
			Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot
150 000	10 000	15	16	240	29	435	64	960	18	270	31	465	71	1065	20	300	36	540	82	1230
100 000	10 000	10	25	250	44	440	100	1000	29	290	52	520	116	1160	38	380	67	670	150	1500
50 000	5000	10	25	250	44	440	100	1000	29	290	52	520	116	1160	38	380	67	670	150	1500
40 000	4000	10	25	250	44	440	100	1000	29	290	52	520	116	1160	38	380	67	670	150	1500
30 000	3000	10	25	250	44	440	100	1000	29	290	52	520	116	1160	38	380	67	670	150	1500
20 000	2000	10	25	250	44	440	100	1000	29	290	52	520	116	1160	38	380	67	670	150	1500
10 000	1000	10	25	250	44	440	100	1000	29	290	52	520	116	1160	38	380	67	670	150	1500

(continued)

TABLE 2.2 (continued)

Lot and sub-lot details			$\beta_{\text{SPM}} = 0.3\% \text{ ash}$																	
Mass of lot, t	Mass of sub-lot, t	Number of sub-lots	$\sigma_{\text{PM}} = 0.1\% \text{ ash}$						$\sigma_{\text{PM}} = 0.15\% \text{ ash}$						$\sigma_{\text{PM}} = 0.2\% \text{ ash}$					
			Number of primary increments						Number of primary increments						Number of primary increments					
			$\sigma_{\text{I}} = 1.5\% \text{ ash}$		$\sigma_{\text{I}} = 2\% \text{ ash}$		$\sigma_{\text{I}} = 3\% \text{ ash}$		$\sigma_{\text{I}} = 1.5\% \text{ ash}$		$\sigma_{\text{I}} = 2\% \text{ ash}$		$\sigma_{\text{I}} = 3\% \text{ ash}$		$\sigma_{\text{I}} = 1.5\% \text{ ash}$		$\sigma_{\text{I}} = 2\% \text{ ash}$		$\sigma_{\text{I}} = 3\% \text{ ash}$	
			Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot
150 000	10 000	15	7	105	12	180	27	405	7	105	13	195	29	435	8	120	13	195	30	450
100 000	10 000	10	10	100	19	190	42	420	11	110	20	200	44	440	12	120	22	220	49	490
50 000	5000	10	10	100	19	190	42	420	11	110	20	200	44	440	12	120	22	220	49	490
40 000	4000	10	10	100	19	190	42	420	11	110	20	200	44	440	12	120	22	220	49	490
30 000	3000	10	10	100	19	190	42	420	11	110	20	200	44	440	12	120	22	220	49	490
20 000	2000	10	10	100	19	190	42	420	11	110	20	200	44	440	12	120	22	220	49	490
10 000	1000	10	10	100	19	190	42	420	11	110	20	200	44	440	12	120	22	220	49	490

Lot and sub-lot details			$\beta_{\text{SPM}} = 0.4\% \text{ ash}$																	
Mass of lot, t	Mass of sub-lot, t	Number of sub-lots	$\sigma_{\text{PM}} = 0.1\% \text{ ash}$						$\sigma_{\text{PM}} = 0.15\% \text{ ash}$						$\sigma_{\text{PM}} = 0.2\% \text{ ash}$					
			Number of primary increments						Number of primary increments						Number of primary increments					
			$\sigma_{\text{I}} = 1.5\% \text{ ash}$		$\sigma_{\text{I}} = 2\% \text{ ash}$		$\sigma_{\text{I}} = 3\% \text{ ash}$		$\sigma_{\text{I}} = 1.5\% \text{ ash}$		$\sigma_{\text{I}} = 2\% \text{ ash}$		$\sigma_{\text{I}} = 3\% \text{ ash}$		$\sigma_{\text{I}} = 1.5\% \text{ ash}$		$\sigma_{\text{I}} = 2\% \text{ ash}$		$\sigma_{\text{I}} = 3\% \text{ ash}$	
			Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot
150 000	10 000	15	4	60	7	105	15	225	4	60	7	105	16	240	4	60	7	105	16	240
100 000	10 000	10	6	60	10	100	23	230	6	60	11	110	24	240	6	60	11	110	25	250
50 000	5000	10	6	60	10	100	23	230	6	60	11	110	24	240	6	60	11	110	25	250
40 000	4000	10	6	60	10	100	23	230	6	60	11	110	24	240	6	60	11	110	25	250
30 000	3000	10	6	60	10	100	23	230	6	60	11	110	24	240	6	60	11	110	25	250
20 000	2000	10	6	60	10	100	23	230	6	60	11	110	24	240	6	60	11	110	25	250
10 000	1000	10	6	60	10	100	23	230	6	60	11	110	24	240	6	60	11	110	25	250

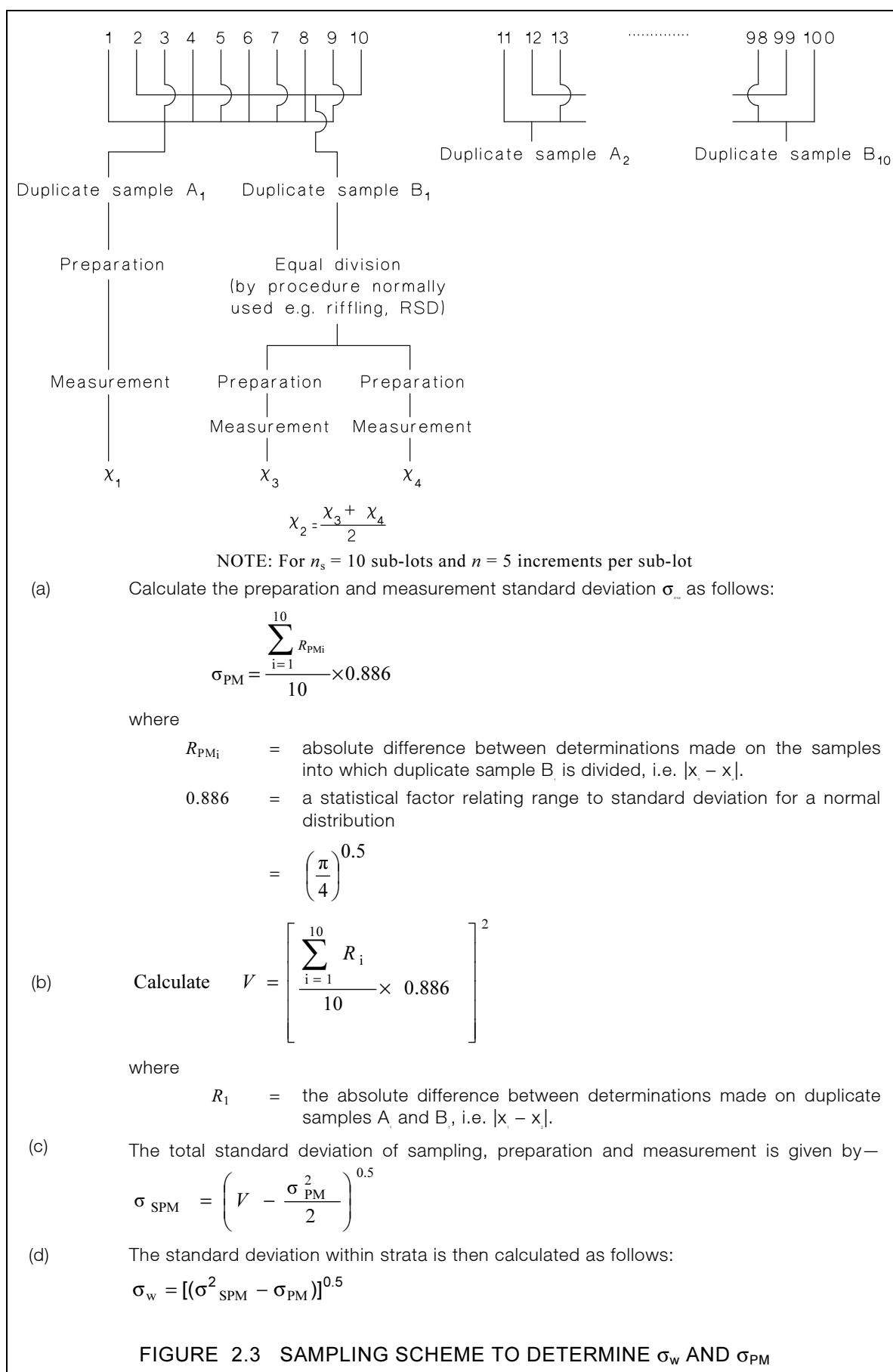


TABLE 2.3
EXAMPLES OF CALCULATIONS OF σ_w

	x_1	x_3	x_4	$x_2=(x_3+x_4)/2$	$R_{PM}=\text{abs}(x_3-x_4)$	$R_i=\text{abs}(x_1-x_2)$
1	12.8	12.5	13.0	12.75	0.5	0.05
2	7.9	7.3	7.5	7.40	0.2	0.50
3	11.4	12.7	12.4	12.55	0.3	1.15
4	8.7	9.4	9.2	9.30	0.2	0.60
5	13.2	14.5	14.8	14.65	0.3	1.45
6	11.5	10.3	10.1	10.20	0.2	1.30
7	11.0	9.1	9.5	9.30	0.4	1.70
8	13.8	16.9	16.6	16.75	0.3	2.95
9	13.3	11.0	11.0	11.00	0.0	2.30
10	10.5	10.3	10.5	10.40	0.2	0.10

From the equation in Figure 2.3(d) the standard deviation within strata is:

$$\begin{aligned}
 n &= 10 \\
 \Sigma(R_{PM}) &= 2.6 \\
 \sigma_{PM} &= 0.230 \\
 \Sigma(R_i) &= 12.10 \\
 V &= 1.15 \\
 \sigma_{SPM} &= 1.08 \\
 \sigma_w &= 2.36
 \end{aligned}$$

2.4.4 Calculation of number of primary increments using variogram method

The variogram method is based on a mathematical model for sampling*, which accounts for serial correlation between increments as well as the interval between increments. Consequently, extrapolation to different sampling intervals is possible. The method can also assist in the detection of any periodic variations in quality.

The variogram is a plot of the mean square difference between the quality of two increments as a function of the interval (called the lag) between them. The data are obtained by collecting at least 30 successive increments (or divided increments) from a lot or sub-lot and analyzing each increment separately. The increments should be collected at about half the sampling interval estimated by the method given in Clause 2.4.2.2(a). This should be carried out for each type of coal and each sampling situation.

The value of the variogram $V_e(t)$ at lag k can be calculated from the following equation:

$$V_e(t) = \frac{\sum_{i=1}^{N_k} [x_{i+k} - x_i]^2}{2N_k} \quad \dots 2.4$$

where

- x_{i+k} = the analysis for increment $i + k$
- x_i = the analysis for increment i
- N_k = the number of pairs of increments at lag k apart

It is recommended that $V_e(t)$ be calculated for the first 10 lags. The principal features of a variogram are shown in Figure 2.4. The intercept A on the vertical axis arises from the random particle-to-particle variations in quality. The values of $V_e(t)$ for $t > 0$ reflect the variations in quality with time, which increase with lag and may reach a maximum value at the 'range of influence'.

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

The 'experimental' variogram obtained using Equation 2.4 includes the sample preparation and measurement variance σ_{PM}^2 (which is constant) as well as the sampling variance σ_{S}^2 . This affects the variogram intercept A , and consequently a correction must be made to obtain the sampling standard deviation.

Experience shows that variograms that occur in practice can be adequately approximated by a straight line over the range $k = 1$ to $k = 5$ or 10 , i.e.

$$V_{\text{e}}(t) = A_{\text{e}} + Bt \quad \dots 2.5$$

where

A_{e} = the random component (the intercept) of the experimental variogram

B = the gradient (or slope) of the variogram

t = $k\Delta m$ for mass-basis sampling

where

Δm = the sampling interval used for data collection

t = $k\Delta t$ for time-basis sampling

where

Δt = the sampling interval used for data collection

The values of A_{e} and B in Equation 2.5 are estimated by fitting a straight line to the first N points of the experimental variogram using linear regression. The number of points N is chosen to cover the linear portion of the variogram and is typically between 5 and 10. The procedure for mass-basis sampling is as follows:

let

$$y = V_{\text{e}}(t), z = k\Delta m$$

then

$$A_{\text{e}} = \frac{\sum y - B \sum z}{N} \quad \dots 2.6$$

$$B = \frac{N \sum z y - \sum z \sum y}{N \sum z^2 - (\sum z)^2} \quad \dots 2.7$$

Subtracting the sample preparation and measurement variance σ_{PM}^2 from the value of A_{e} obtained using Equation 2.6 gives the intercept A_{c} for the 'corrected' variogram, which provides information on sampling errors only, i.e.

$$A_{\text{c}} = A_{\text{e}} - \sigma_{\text{PM}}^2 \quad \dots 2.8$$

After A_{c} and B have been determined, the sampling variance σ_{S}^2 can be calculated as follows:

(a) For mass-basis systematic sampling

$$\sigma_{\text{S}}^2 = \frac{A_{\text{c}}}{n} + \frac{Bm_{\text{L}}}{6n^2} \quad \dots 2.9$$

(b) For mass-basis stratified random sampling

$$\sigma_{\text{S}}^2 = \frac{A_{\text{c}}}{n} + \frac{Bm_{\text{L}}}{3n^2} \quad \dots 2.10$$

where

m_{L} = the total mass of the sub-lot

n = the number of increments taken from the sub-lot

NOTE: The above equations apply to mass-basis sampling. For time-basis sampling, m_L is replaced by t_L , the total sampling time, and Δm is replaced by Δt , the time interval between successive increments.

The number of increments required to achieve a desired sampling standard deviation σ_s can be obtained by transposing Equations 2.9 and 2.10 and solving for n . This gives the following equations for systematic and stratified random sampling:

(i) For mass-basis systematic sampling

$$n = \frac{A_c \left(A_c^2 + \frac{2}{3} B m_L 2\sigma_s^2 \right)^{0.5}}{2\sigma_s^2} \quad \dots 2.11$$

(ii) For mass-basis stratified random sampling

$$n = \frac{A_c \left(A_c^2 + \frac{4}{3} B m_L \sigma_s^2 \right)^{0.5}}{2\sigma_s^2} \quad \dots 2.12$$

If all the increments are combined into a single gross sample for the sub-lot, the total variance σ_{SPM}^2 for sampling, preparation and measurement can be calculated as in Equations 2.13 and 2.14:

(c) For mass-basis systematic sampling

$$\sigma_{SPM}^2 = \frac{A_c}{n} + \frac{B m_L}{6n^2} + \sigma_{PM}^2 \quad \dots 2.13$$

(d) For mass-basis stratified random sampling

$$\sigma_{SPM}^2 = \frac{A_c}{n} + \frac{B m_L}{3n^2} + \sigma_{PM}^2 \quad \dots 2.14$$

Example 2.1—Periodic variogram

As mentioned above, the variogram assists in the detection of periodic variations in quality. An example for total moisture content of coal is shown in Figure 2.5, the periodic behaviour of the variogram indicating the presence of cyclic variations in quality. If the cause of these cyclic variations cannot be located and eliminated, stratified random sampling should be used in future.

Example 2.2—Calculation of sampling standard deviation

The ash data in Table 2.4 were obtained by collecting and analyzing to a dry basis individual increments taken at 0.25 min intervals. The preparation and measurement standard deviation σ_{PM} was determined separately as 0.1.

Having collected the data, the experimental variogram points can be calculated using Equation 2.4. For lag 1 ($k = 1$), the variogram value is as follows:

$$\begin{aligned} V_e(0.25) &= \frac{(13.8-14.6)^2 + (14.7-13.8)^2 + (16.1-14.7)^2 + \dots + (14.9-15.2)^2}{2 \times 29} \\ &= \frac{9.0306}{58} \\ &= 0.1557 \end{aligned}$$

For lag 2 ($k = 2$), the variogram value is given by—

$$\begin{aligned}
 V_e(0.25) &= \frac{(14.7-14.6)^2 + (16.1-13.8)^2 + (15.6-14.7)^2 + \dots + (14.9-14.7)^2}{2 \times 28} \\
 &= \frac{10.3096}{56} \\
 &= 0.1841
 \end{aligned}$$

The first 10 variogram points are tabulated in Table 2.5 and plotted in Figure 2.6. Fitting a straight line to the first five points of the variogram gives—

$$\begin{aligned}
 \Sigma y &= 1.0774 \\
 \Sigma z &= 3.7500 \\
 \Sigma zy &= 0.87443 \\
 \Sigma z^2 &= 3.4375
 \end{aligned}$$

Thus, Equation 2.7 gives—

$$\begin{aligned}
 B &= \frac{5 \times 0.87443 - 3.75 \times 1.0774}{5 \times 3.4375 - (3.75)^2} \\
 &= 0.1062
 \end{aligned}$$

Hence, Equation 2.6 gives

$$\begin{aligned}
 A_e &= \frac{1.0774 - 0.1062 \times 3.75}{5} \\
 &= 0.1358
 \end{aligned}$$

Finally, Equation 2.8 gives—

$$\begin{aligned}
 A_c &= 0.1358 - 0.1^2 \\
 &= 0.1258
 \end{aligned}$$

With the above values for A_c and B , the sampling standard deviation can now be calculated.

Assuming that systematic sampling is to be used, and that 30 increments are to be taken at 2 min intervals, i.e. $t_L = 60$, the sampling variance is as follows:

$$\begin{aligned}
 \sigma_s^2 &= \frac{0.1258}{30} + \frac{0.1062 \times 60}{6 \times 30^2} \\
 &= 4.19 \times 10^{-3} + 1.18 \times 10^{-3} \\
 &= 5.37 \times 10^{-3}
 \end{aligned}$$

Thus $\sigma_s = 0.073\%$ ash

TABLE 2.4
ASH OF INDIVIDUAL INCREMENTS TAKEN
AT 0.25 min INTERVALS

Increment number	Ash %	Increment number	Ash %
1	14.6	16	15.5
2	13.8	17	14.6
3	14.7	18	14.9
4	16.1	19	15.3
5	15.6	20	15.4
6	15.6	21	15.4
7	15.8	22	15.4
8	15.3	23	16.0
9	15.8	24	15.5
10	15.4	25	15.5
11	15.9	26	15.4
12	16.1	27	15.4
13	15.3	28	14.7
14	16.0	29	15.2
15	15.2	30	14.9

TABLE 2.5
EXPERIMENTAL VARIOGRAM POINTS
FOR THE DATA IN TABLE 2.4

Lag <i>(k)</i>	Lag in minutes <i>(z)</i>	Experimental variogram $V_e(t)$ <i>(y)</i>
1	0.25	0.1557
2	0.50	0.1841
3	0.75	0.2346
4	1.00	0.2450
5	1.25	0.2580
6	1.50	0.2965
7	1.75	0.2470
8	2.00	0.2611
9	2.25	0.2798
10	2.50	0.2973