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Iron ores — Experimental methods for evaluation of quality variation

Minerais de fer — Méthodes expérimentales pour l'évaluation de la variation de qualité

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Contents

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3084 was prepared by Technical Committee ISO/TC 102, *Iron ores*, subcommittee SC 1, *Sampling*.

This third edition cancels and replaces the second edition (ISO 3084:1986) which has been technically revised.

Annex A of this International Standard is for information only.

Iron ores — Experimental methods for evaluation of quality variation

1 Scope

This International Standard specifies experimental methods for the evaluation of quality variation of iron ores for each type of iron ore being traded and for each handling plant.

Two distinct approaches are specified. The first is to analyse interleaved samples composed of a number of paired increments taken and combined alternately following stratified sampling or systematic sampling as specified in ISO 3082. The second is to collect and analyse individual increments and then to analyse the data using variographic methods.

Using interleaved samples involves less work, but use of variograms provides a better estimate of quality variation and hence a better estimate of the sampling variance. The variogram method is usually used to fine tune a sampling operation.

NOTE The experimental methods may be applied approximately to time basis sampling when the flowrate is almost uniform.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3082:—[1)], *Iron ores — Sampling and sample preparation procedures*.

ISO 3085:1996, *Iron ores — Experimental methods for checking the precision of sampling*.

ISO 11323:1996, *Iron ores — Vocabulary*.

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 11323 and the following apply.

3.1 interleaved samples
samples constituted by placing consecutive primary increments alternately into two sample containers

[1)] To be published. (Revision of ISO 3081:1986, ISO 3082:1987 and ISO 3083:1986)

4 General conditions

4.1 Quality variation

The quality variation or degree of heterogeneity of iron ore shall be determined in terms of the standard deviation.

The standard deviation of a quality characteristic between increments taken from within strata, denoted by σ_W, shall be determined either by estimating the variance between interleaved samples or by measuring individual increments and determining the slope and intercept of a linear fit to a variogram corrected by subtraction of sample preparation and measurement variances. In both cases, where corrections for sample preparation and measurement variances are made (see 5.6.2, note 2, and 6.1), it is essential that the sample preparation and measurement variances be determined at the same time as the experiments to determine quality variation are carried out.

4.2 Quality characteristics

The quality characteristic chosen for determining the quality variation is generally the total iron content, but silica content, alumina content, moisture content, size distribution and other quality characteristics may also be chosen.

When separate samples are taken for the determination of chemical composition, moisture content, size distribution, etc., the quality variation for the individual characteristics shall be adopted. When the sample is used for the determination of more than one quality characteristic, the largest classification category for quality variation among these characteristics shall be adopted.

4.3 Sampling, sample preparation and measurement

Sampling and sample preparation shall be carried out in accordance with ISO 3082. Measurement of samples shall be carried out in accordance with the relevant International Standards for chemical analysis, moisture content and size analysis of iron ores.

The sampling for evaluation of quality variation may be conducted in conjunction with routine sampling for determination of the quality of the lot. In other words, the samples collected from the lot may be used for both purposes.

5 Evaluation of quality variation using interleaved sampling

5.1 General

The procedures for evaluating the standard deviation within strata, σ_W, applicable to both stratified and systematic sampling, are described in 5.2 to 5.7.

5.2 Type of investigation

5.2.1 Type 1

When lots are frequently delivered, the quality variation may be determined from a large number of lots of almost equal mass as follows:

a) treat each lot separately;

b) make up a pair of interleaved samples for each lot as shown in figure 1a) and example 1.

5.2.2 Type 2

When large lots are infrequently delivered, the quality variation may be determined from a single lot as follows:

a) split the lot into at least 10 parts of almost equal mass;

b) make up a pair of interleaved samples for each part by combining the increments taken from each part as shown in figure 1b) and example 2.

5.2.3 Type 3

When small lots are frequently delivered, the quality variation may be determined from several lots of almost equal mass as follows:

a) split all the lots involved into at least 10 parts of almost equal mass;

b) make up a pair of interleaved samples for each part by combining the increments taken from each part as shown in figure 1c) and example 3.

5.2.4 Type 4

When sampling a wagon-borne lot and when increments are taken from all wagons comprising the lot, the sampling scheme may be regarded as stratified sampling. When lots are frequently delivered, the procedure for determining quality variation is as follows:

a) treat each lot separately;

b) make up a pair of interleaved samples for each lot as shown in figure 1d).

5.3 Number of increments and constitution of interleaved samples

5.3.1 Number of increments

The number of increments to be taken from one or several lots may be the same as that selected for routine sampling. However, when the routine sampling is based on the classification category of "small" quality variation and the number of increments is considered to be insufficient to obtain a reliable standard deviation, then the number of increments shall be increased (see ISO 3082).

5.3.1.1 Type 1 investigation

For type 1 investigations, the number of increments, n_1, to be taken from each lot shall be in accordance with ISO 3082, and a pair of interleaved samples shall be constituted for each lot [see figure 1a)].

5.3.1.2 Type 2 investigation

For type 2 investigations, the number of increments, n_1, shall be determined from ISO 3082, and at least 10 pairs of interleaved samples shall be constituted [see figure 1b)].

5.3.1.3 Type 3 investigation

For type 3 investigations, the number of increments, n_1, to be taken from each lot shall be in accordance with ISO 3082. Each lot shall be divided into a number of strata, and increments from each stratum shall be combined into a pair of interleaved samples [see figure 1c)].

5.3.1.4 Type 4 investigation

For type 4 investigations, the number of increments, n_1, being collected from each lot shall be determined from table 3 of ISO 3082, and the number of increments, n_w, to be taken from each wagon shall be in accordance with

ISO 3082. If the number is odd, it shall be increased by one to make it even. A pair of interleaved samples shall be constituted for each lot [see figure 1d)].

5.3.2 Constitution of interleaved samples

The interleaved samples shall be made up according to the following procedure:

— allocate a serial number to the increments from each lot or part-lot in order of sampling;

— constitute pairs of interleaved samples from consecutive odd-numbered increments (denoted by interleaved sample A_i) and consecutive even-numbered increments (denoted by interleaved sample B_i) for each lot or part-lot (see figure 2);

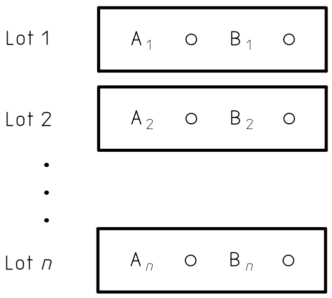
— for each investigation, prepare n sets of paired interleaved samples.

Each interleaved sample shall be made up of two or more increments.

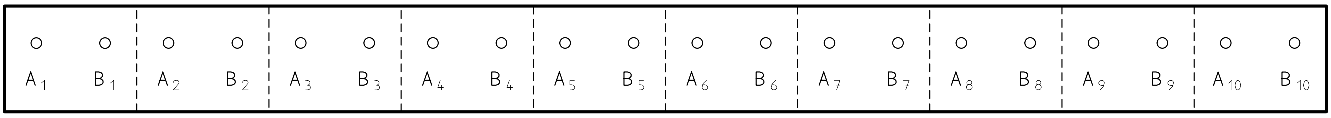
5.4 Preparation of test samples and measurement

Separate test samples shall be prepared from the interleaved samples, A_i and B_i.

Chemical analysis, moisture determination, size determination or physical testing shall be carried out on the test samples as required.

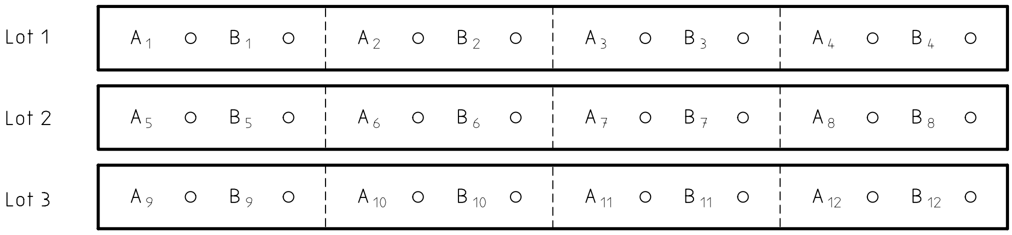


a) Type 1 investigation — *n* lots: one lot = one part

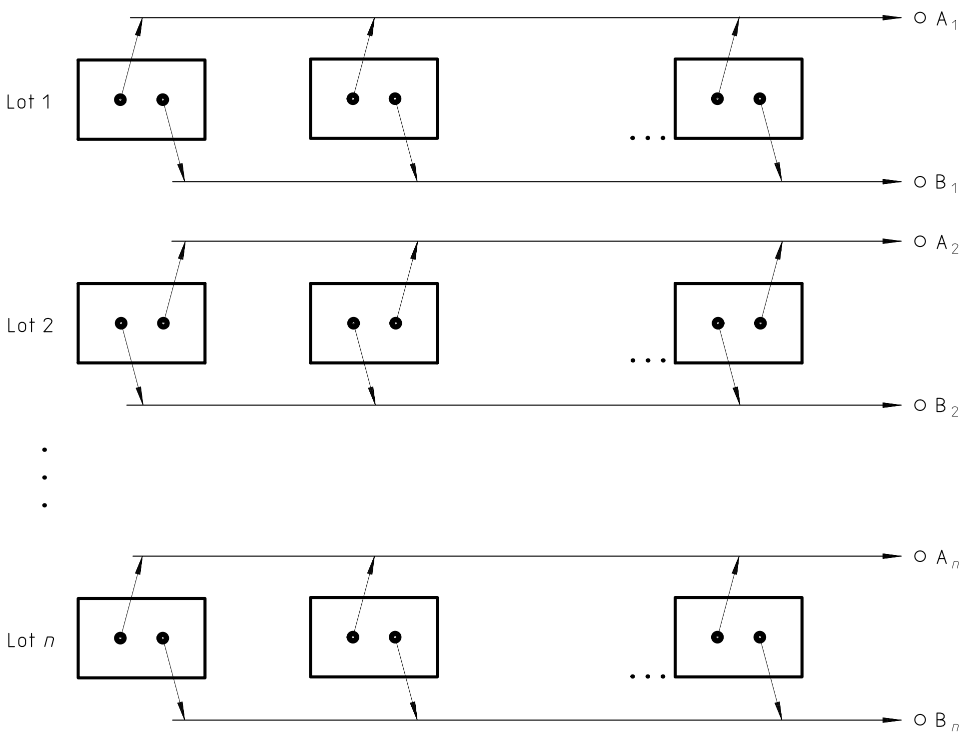


Key: The rectangular box indicates one lot; each division of the box made by broken lines indicates one part; a pair of circles indicates a pair of interleaved samples. This also applies to a) and c).

b) Type 2 investigation — One lot (example for 10 parts)



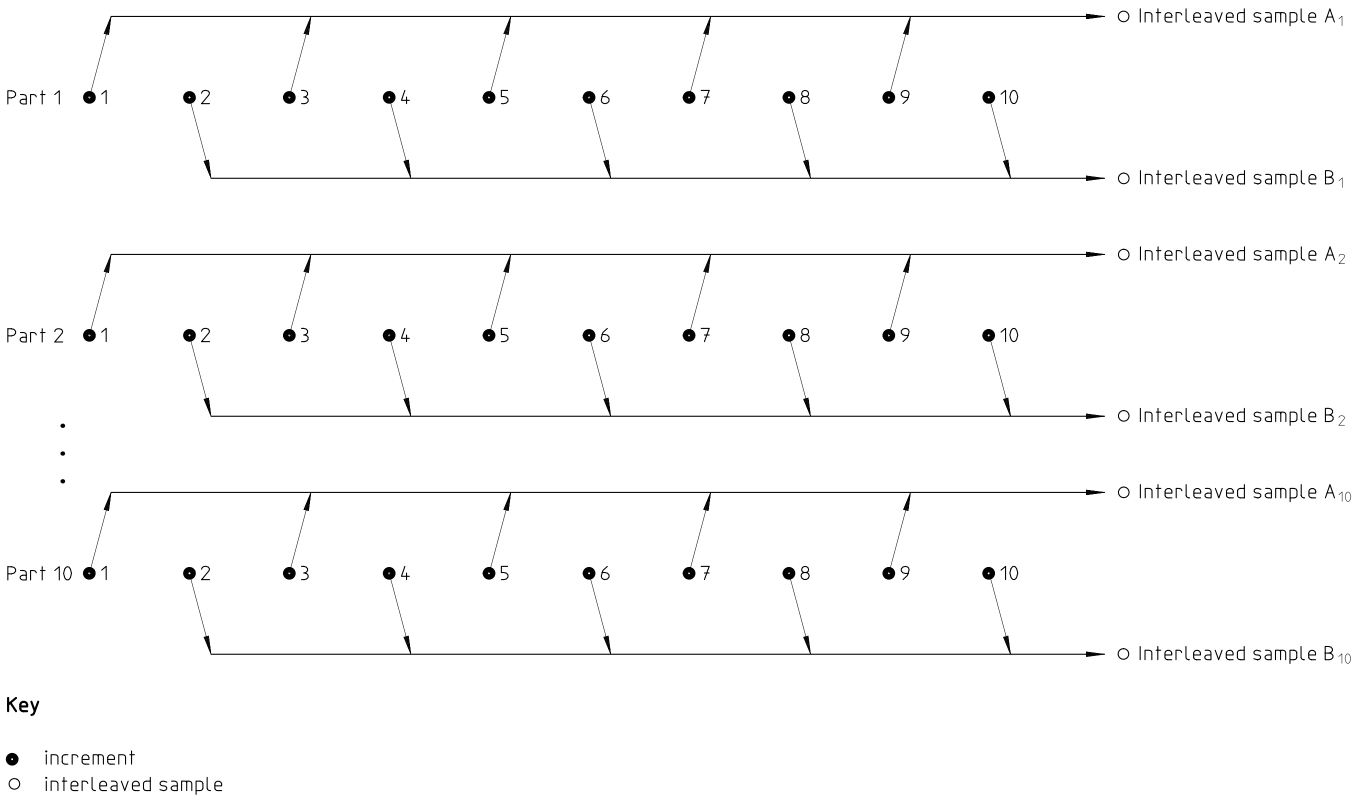
c) Type 3 investigation — Several lots (example for three lots and 12 parts)



Key: Each box indicates a wagon; points in the box indicate increments; circles indicate interleaved samples.

d) Type 4 investigation — Stratified sampling of wagon-borne lots.

Figure 1 — Schematic diagram for one investigation



NOTE This diagram is an example for a single lot of 5 000 t to 15 000 t of ore of "large" quality variation. In accordance with ISO 3082, the required minimum number of increments is 100, and 10 pairs of interleaved samples A_i and B_i (i = 1, 2, ..., 10), each comprising five increments, are prepared.

Figure 2 — Example of schematic diagram for constitution of pairs of interleaved samples (type 2)

5.5 Number of investigations

Because the standard deviation, σ_W, of a quality characteristic within strata cannot be estimated very precisely from a small number of investigations, the following minimum number of investigations is recommended:

a) for type 2 and 3 investigations, at least five separate investigations;

b) for type 1 and 4 investigations, at least 10 separate investigations.

5.6 Calculation of standard deviation within strata

5.6.1 Data sheet

The experimental data generated by chemical analysis, moisture determination, size determination or physical testing of individual test samples shall be recorded on a suitable form (see examples 1 to 3).

5.6.2 Calculation

The estimated standard deviation within strata shall be calculated from equation (4).

The range, R_i, of paired measurements is given by equation (1):

$$R_i = |A_i - B_i| \tag{1}$$

where

A_i is the measured quality characteristic (such as % Fe) of the test sample prepared from interleaved sample A_i;

B_i is the measured quality characteristic of the test sample prepared from interleaved sample B_i, which is from the same part-lot as interleaved sample A_i;

i is a subscript designating each part-lot.

The mean, $\overline{R}$, of ranges R_i is given by equation (2):

$$\overline{R} = \frac{1}{n_4} \sum R_i \qquad (2)$$

where n_4 is the number of ranges, R_i, which is the same as the number of part-lots in the investigation.

The mean, $\overline{x}_i$, of paired measurements for each part is given by equation (3):

$$\overline{x}_i = \frac{1}{2} (A_i + B_i) \qquad (3)$$

The estimated standard deviation within strata, $\hat{\sigma}_W$, is given by equation (4):

$$\hat{\sigma}_w = \sqrt{n_5}\, \frac{\overline{R}}{d_2} \qquad (4)$$

where

n_5 is the number of increments comprising each interleaved sample A_i or B_i;

d_2 is the factor to estimate standard deviation from the range; for paired data $1/d_2$ = 0,886 2.

NOTES

1 For type 3 investigations, the mean value of the quality characteristic for the j-th lot, $\overline{x}_j$, may be obtained from equation (5).

$$\overline{x}_j = \frac{1}{n_6} \sum x_{ji} \qquad (5)$$

where

x_{ji} is the mean of paired measurements for each part in lot j;

n_6 is the number of parts in the lot.

2 The estimated standard deviation within strata, $\hat{\sigma}_W$, obtained from equation (4) is a measure of the combined standard deviation of sampling, sample preparation and measurement. While the standard deviation within strata is overestimated, this value may be used for the classification in clause 7 (see 5.7).

When it is desired to obtain an unbiased estimate of the standard deviation within strata, and when the estimated standard deviation of sample preparation, denoted by $\hat{\sigma}_P$, and the estimated standard deviation of measurement, denoted by $\hat{\sigma}_M$, are known, the estimated standard deviation within strata should be calculated using equation (6):

$$\hat{\sigma}_w = \sqrt{n_5 \left[\left(\frac{\overline{R}}{d_2}\right)^2 - \hat{\sigma}_P^2 - \hat{\sigma}_M^2 \right]} \qquad (6)$$

3 If the number of increments is determined according to 5.3.1 and those increments are taken, the variation among the numbers of increments comprising the various interleaved samples will be small. If the variation is 10 % or less, equations (4) and (6) can be applied approximately by using the mean value of n_5.

5.7 Expression of results

5.7.1 Type 2 and 3 investigations

For type 2 and 3 investigations, the estimated mean value of standard deviation within strata, $\bar{\hat{\sigma}}_{\mathrm{w}}$, for a particular iron ore and handling plant evaluated from a series of investigations shall be reported as the square root of the mean of all measured values of $\hat{\sigma}_{\mathrm{w}}^2$, i.e.:

$$\bar{\hat{\sigma}}_{\mathrm{w}} = \sqrt{\frac{1}{n_7}\sum \hat{\sigma}_{\mathrm{w}}^2} \qquad (7)$$

where n_7 is the number of individual values of $\hat{\sigma}_{\mathrm{w}}$.

5.7.2 Type 1 and 4 investigations

For type 1 and 4 investigations, the value of $\hat{\sigma}_{\mathrm{w}}$ obtained from equation (4) or equation (6) shall be reported as the estimated standard deviation within strata for the particular iron ore and handling plant.

6 Estimation of quality variation from the variogram

6.1 General variogram method

Using this method, a large number (say 20 to 40) of successive increments, n, are extracted, prepared and measured in duplicate. The variogram, which examines the differences between increments at various intervals (called lags) apart, can then be calculated. The value of the variogram, $V_{\mathrm{E}}(t)$, corresponding to a lag of k increments is given by equation (8):

$$V_{\mathrm{E}}(t) = \frac{\sum_{i=1}^{N_k}\left[\bar{x}_{i+k} - \bar{x}_i\right]^2}{2N_k} \qquad (8)$$

where

t is $k\Delta t$, where Δt is the sampling interval in units of time or mass, depending on whether time basis or mass basis sampling is used;

N_k is the number of pairs of increments, $n - k$, at lag k apart;

$\bar{x}_{i+k}$ is the average of the duplicate measurements for increment $i + k$;

$\bar{x}_i$ is the average of the duplicate measurements for increment i.

The resultant variogram, $V_{\mathrm{E}}(t)$, is called the "experimental" variogram, and includes the variance of sample preparation and measurement as well as the sampling variance.

The extracted increments are prepared and measured in duplicate, allowing the sample preparation and measurement variances to be determined from the mean of the range of the measurements in accordance with the procedures described in ISO 3085. Half the sum of the sample preparation and measurement variances so determined, i.e., $\hat{\sigma}^2_{\mathrm{PM}}/2$, is subtracted from the calculated value of $V_{\mathrm{E}}(t)$ at each lag to give the "corrected" variogram, $V_{\mathrm{C}}(t)$, which provides information on the sampling variance only.

The lag is sometimes expressed as an integer, being the multiple of the sampling interval Δt. The variogram $V_{\mathrm{C}}(k\Delta t)$ may then be written as V_k. The sampling interval does not need to be the same for determining the variogram and for using it, so it is important to express the lag in units of time or mass and to express the variogram as a function of the continuous-valued lag if Δt might change.

In most cases, it is found that variograms which occur in practice can be adequately approximated by a straight line over the range from very small values of k to at least twice the spacing between increments (see example 4). Thus it can be assumed that

$$V_{\mathrm{C}}(t) = V_0 + Bt \tag{9}$$

where

V_0 is the random component of variance of the corrected variogram;

B is the slope (or gradient) of the variogram.

The estimated sampling variance $\hat{\sigma}^2_{\mathrm{S}}$ is then given by the following equations:

a) Systematic sampling

$$\hat{\sigma}^2_{\mathrm{S}} = \frac{V_0}{n} + \frac{BT}{6n^2} \tag{10}$$

b) Stratified random sampling

$$\hat{\sigma}^2_{\mathrm{S}} = \frac{V_0}{n} + \frac{BT}{3n^2} \tag{11}$$

c) Random sampling

$$\hat{\sigma}^2_{\mathrm{S}} = \frac{V_0}{n} + \frac{BT}{3n} \tag{12}$$

where T is the total tonnage for the lot (mass basis sampling) or the total time interval (time basis sampling) over which sampling takes place, $n\Delta t$.

The estimated sampling variance depends on the sampling scheme chosen, random sampling being the least precise. When there are no periodic variations in quality, systematic sampling is more precise than stratified random sampling.

6.2 Simplified variogram method

Assuming that systematic sampling is used, a straight line is fitted to the first two points of the variogram (see example 4) to give

$$V_0 = 2V_1 - V_2 \tag{13}$$

$$B = (V_2 - V_1)/\Delta t \tag{14}$$

where

V_1 is $V_{\mathrm{C}}(\Delta t)$ = variogram at lag 1;

V_2 is $V_C(2\Delta t)$ = variogram at lag 2.

The normal expectation is that the straight line through V_1 and V_2 will have a positive or zero slope ($B \geqslant 0$) and hence the value of V_0 obtained from equation (13) should be less than or equal to V_1. However, if the line has negative slope, ($B < 0$) and hence $V_0 > V_1$, then set $V_0 = V_1$ and set $B = 0$.

6.3 Relationship between variogram and quality variation

Replacing T/n by Δt, the sampling interval for subsequent sampling operations following determination of the variogram, equation (10) for systematic sampling becomes

$$\hat{\sigma}_S^2 = \frac{V_0}{n} + \frac{B\Delta t}{6n} \tag{15}$$

The relationship between the estimated sampling variance, $\hat{\sigma}_S^2$, and the estimated quality variation, $\hat{\sigma}_W$, is as follows:

$$\hat{\sigma}_S^2 = \frac{\hat{\sigma}_W^2}{n} \tag{16}$$

Combining equations (15) and (16) gives the variogram estimate of $\hat{\sigma}_W^2$ as follows:

$$\hat{\sigma}_W^2 = V_0 + \frac{B\Delta t}{6} \tag{17}$$

Thus

$$\hat{\sigma}_W = \sqrt{V_0 + \frac{B\Delta t}{6}} \tag{18}$$

Equation (18) enables the estimated quality variation $\hat{\sigma}_W$ to be calculated for any sampling interval Δt. The corresponding sampling precision $\hat{\sigma}_S$ can then be determined using equation (16). Note that, in contrast to the methods in 5, $\hat{\sigma}_W$ is not assumed to be a constant. Its value depends on the size of the sampling strata, i.e., on Δt.

6.4 Number of increments

While the sampling precision can be determined for any proposed sampling interval using the equations in 6.2, calculation of the number of increments required to achieve a desired sampling precision is not straightforward. The number of increments must be determined by iteration as follows:

— calculate $\hat{\sigma}_W$ for the proposed number of increments using equation (18);

— calculate the sampling precision $\hat{\sigma}_S$ using equation (16);

— compare the calculated sampling precision with the desired value;

— increase or decrease the number of increments and repeat the above steps until the calculated sampling precision is equal to the desired value.

7 Classification of quality variation

The quality variation of iron ore shall be classified into one of the three categories specified in table 1 based on the experimentally determined standard deviation obtained from a series of investigations.

Table 1 — Classification of quality variation, σ_W, based on the standard deviations in selected quality characteristics (values as absolute percentages)

Quality characteristics		Classification of quality variation (σ_W)		
		Large	Medium	Small
Total iron content		$\sigma_W \geqslant 2,0$	$2,0 > \sigma_W \geqslant 1,5$	$\sigma_W < 1,5$
Silica content		$\sigma_W \geqslant 2,0$	$2,0 > \sigma_W \geqslant 1,5$	$\sigma_W < 1,5$
Alumina content		$\sigma_W \geqslant 0,6$	$0,6 > \sigma_W \geqslant 0,4$	$\sigma_W < 0,4$
Phosphorus content		$\sigma_W \geqslant 0,015$	$0,015 > \sigma_W \geqslant 0,011$	$\sigma_W < 0,011$
Moisture content		$\sigma_W \geqslant 2,0$	$2,0 > \sigma_W \geqslant 1,5$	$\sigma_W < 1,5$
Size of −200 mm ore	−10 mm fraction mean 20 %	$\sigma_W \geqslant 10$	$10 > \sigma_W \geqslant 7,5$	$\sigma_W < 7,5$
Size of −50 mm ore				
Size of −31,5 + 6,3 mm ore	−6,3 mm fraction mean 10 %	$\sigma_W \geqslant 5$	$5 > \sigma_W \geqslant 3,75$	$\sigma_W < 3,75$
Size of sinter feed	+6,3 mm fraction mean 10 %			
Size of pellet feed	−45 µm fraction mean 70 %	$\sigma_W \geqslant 3$	$3 > \sigma_W \geqslant 2,25$	$\sigma_W < 2,25$
Size of pellets	−6,3 mm fraction mean 5 %			

NOTE It is possible that the quality variation may vary because of changes to factors such as:

a) ore bodies in a mine;

b) the method of mining;

c) the method of ore dressing;

d) the method of stockpiling and reclamation;

e) the method of loading/unloading;

f) the mass of the lot.

Accordingly, the quality variation of any given ore should be checked from time to time to determine the influence of such changes.

Annex A
(informative)

Examples for calculations of standard deviation

Example 1: Stratified sampling for 13 lots: one lot = one part [see figure 1a)]

Particulars of lot

Name of iron ore: ....

Type of iron ore: (e.g. lump ore).................

Classification of quality variation: "Small"

Particulars of sampling

Mass of increment: 25 kg

Lot No.	**Name of lot** (e.g. name of ship)	**Date of delivery**	**Mass of lot** (wet) (t)	**Number of increments** n_1	**Number of increments comprising each interleaved sample** $n_{10} = 20/2 = 10$
1	. . . .	. . . .	3 023	20	10
2	. . . .	. . . .	2 998	20	10
3	. . . .	. . . .	2 895	20	10
4	. . . .	. . . .	2 824	20	10
5	. . . .	. . . .	2 834	20	10
6	. . . .	. . . .	2 825	20	10
7	. . . .	. . . .	2 182	20	10
8	. . . .	. . . .	3 040	20	10
9	. . . .	. . . .	2 925	20	10
10	. . . .	. . . .	3 028	20	10
11	. . . .	. . . .	2 218	20	10
12	. . . .	. . . .	3 056	20	10
13	. . . .	. . . .	3 100	20	10

Lot No.	– 10 mm (undersize) %				Moisture %				Fe %			
	A_i	B_i	$\bar{x}_i$	R_i	A_i	B_i	$\bar{x}_i$	R_i	A_i	B_i	$\bar{x}_i$	R_i
1	30,9	39,2	35,0	8,3	5,99	6,06	6,02	0,07	60,25	60,50	60,38	0,25
2	31,5	35,2	33,4	3,7	5,37	5,81	5,59	0,44	61,80	61,60	61,70	0,20
3	38,1	42,1	40,1	4,0	5,44	5,63	5,54	0,19	61,10	61,20	61,15	0,10
4	37,8	37,2	37,5	0,6	4,70	4,58	4,64	0,12	62,05	62,00	62,02	0,05
5	42,7	40,3	41,5	2,4	4,83	4,96	4,90	0,13	61,58	61,50	61,54	0,08
6	26,0	26,7	26,4	0,7	5,05	5,26	5,16	0,21	62,20	61,65	61,92	0,55
7	36,0	38,3	37,2	2,3	5,61	5,67	5,64	0,06	61,35	61,10	61,22	0,25
8	31,7	38,2	35,0	6,5	5,11	5,53	5,32	0,42	62,00	62,20	62,10	0,20
9	41,0	34,7	37,8	6,3	3,75	3,73	3,74	0,02	64,25	64,40	64,32	0,15
10	15,9	20,1	18,0	4,2	5,46	5,42	5,44	0,04	63,30	63,30	63,30	0,00
11	16,7	19,5	18,1	2,8	5,00	5,16	5,08	0,16	64,05	64,15	64,10	0,10
12	25,0	23,4	24,2	1,6	5,64	5,48	5,56	0,16	62,85	63,05	62,95	0,20
13	30,0	34,8	32,4	4,8	5,75	5,75	5,75	0,00	59,60	59,20	59,40	0,40
$\bar{\bar{x}} = (1/13)\sum \bar{x}_i$			32,0				5,26				62,01	
$\bar{R} = (1/13)\sum R_i$				3,71				0,155				0,195
$\hat{\sigma}_W^2 = 10(\bar{R} \times 0{,}886\ 2)^2$				108,10				0,1887				0,299
$\hat{\sigma}_W$				10,4				0,43				0,55

Example 2: Stratified sampling for one lot [see figure 1b)]

Particulars of lot

Name of iron ore: ....

Type of iron ore: (e.g. lump ore) ....

Classification of quality variation: "Large"

Name of lot: (e.g. name of ship)

Date of delivery:

Mass of lot: 29 874 t (wet)

Particulars of sampling

Mass of increment: 150 kg

Number of increments: 120

Number of parts: $n_4 = 10$

Number of increments comprising each interleaved sample: $n_5 = 120/(10 \times 2) = 6$

Part No.	– 10 mm (undersize) %				Moisture %				Fe %			
	A_i	B_i	$\bar{x}_i$	R_i	A_i	B_i	$\bar{x}_i$	R_i	A_i	B_i	$\bar{x}_i$	R_i
1	30,2	35,5	32,8	5,3	5,75	6,06	5,90	0,31	60,95	61,61	61,28	0,66
2	27,8	34,7	31,2	6,9	6,17	5,90	6,04	0,27	62,29	61,42	61,86	0,87
3	24,7	19,6	22,2	5,1	5,90	6,48	6,19	0,58	61,97	62,90	62,44	0,93
4	22,4	26,3	24,4	3,9	6,10	6,43	6,26	0,33	61,77	62,45	62,11	0,68
5	13,3	7,9	10,6	5,4	5,24	4,60	4,92	0,64	64,62	63,48	64,05	1,14
6	19,7	29,2	24,4	9,5	5,95	6,92	6,44	0,97	63,16	62,13	62,64	1,03
7	28,1	14,3	21,2	13,8	6,26	5,20	5,73	1,06	62,38	63,60	62,99	1,22
8	9,4	14,3	11,8	4,9	4,65	5,38	5,02	0,73	63,98	63,09	63,54	0,89
9	14,0	16,1	15,0	2,1	5,39	5,10	5,24	0,29	63,26	63,80	63,53	0,54
10	17,3	13,1	15,2	4,2	4,95	5,31	5,13	0,36	62,31	63,24	62,78	0,93
$\bar{\bar{x}}\,(1/10)\,\bar{x}_i$			20,9				5,69				62,72	
$\bar{R} = (1/10)\sum R_i$				6,11				0,554				0,889
$\hat{\sigma}_W^2 = 6(\bar{R} \times 0{,}886\,2)^2$				175,91				1,446				3,724
$\hat{\sigma}_W$				13,3				1,20				1,93

Example 3: Stratified sampling for four lots [see figure 1(c)]

Particulars of lots

Name of iron ore: ....

Type of iron ore: (e.g. lump ore) ....

Classification of quality variation: "Medium"

Particulars of sampling

Mass of increment: 5 kg

Number of parts in one investigation: n_4=(3 × 4)= 12

Number of increments comprising each interleaved samples: n_5 = 60/ (3 × 2) = 10

Number of parts in each lot: n_6 = 3

Lot No.	**Name of lot** (e.g. name of ship)	**Date of delivery**	**Mass of lot** (wet) (t)	**Number of increments** n_1
1	. . . .	. . . . . .	21 459	60
2	. . . .	. . . . . .	20 964	60
3	. . . .	. . . . . .	21 400	60
4	. . . .	. . . . . .	20 750	60

Part No.	**Lot No.**	**– 10 mm (undersize)** %				**Moisture** %				**Fe** %			
		A_i	B_i	$\bar{x}_i$	R_i	A_i	B_i	$\bar{x}_i$	R_i	A_i	B_i	$\bar{x}_i$	R_i
1	1	46,8	51,1	49,0	4,3	5,46	6,10	5,78	0,64	62,31	61,44	61,88	0,87
2		44,7	35,4	40,0	9,3	5,69	5,23	5,46	0,46	63,22	61,86	62,54	1,36
3		38,2	35,1	36,6	3,1	5,47	5,16	5,32	0,31	62,15	63,19	62,67	1,04
4	2	51,2	47,2	49,2	4,0	5,44	5,01	5,22	0,43	62,79	62,22	62,50	0,57
5		44,0	41,2	42,6	2,8	5,47	5,12	5,30	0,35	62,08	62,92	62,50	0,84
6		30,9	36,3	33,6	5,4	5,54	4,87	5,20	0,67	63,22	62,57	62,90	0,65
7	3	42,4	38,9	40,6	3,5	5,62	5,42	5,52	0,20	64,42	63,28	63,85	1,14
8		33,7	37,3	35,5	3,6	5,23	5,07	5,15	0,16	63,14	64,01	63,58	0,87
9		25,3	30,7	28,0	5,4	5,01	5,33	5,17	0,32	64,94	63,98	64,46	0,96
10	4	44,5	42,4	43,4	2,1	4,49	4,33	4,41	0,16	64,30	63,56	63,93	0,74
11		37,9	33,2	35,6	4,7	4,34	4,58	4,46	0,24	64,33	65,65	64,99	1,32
12		25,9	32,2	29,0	6,3	4,85	4,38	4,62	0,47	64,12	65,25	64,68	1,13
$\bar{\bar{x}}_1 = (1/3)\sum \bar{x}_{1i}$				41,9				5,52				62,37	
$\bar{\bar{x}}_2 = (1/3)\sum \bar{x}_{2i}$				41,8				5,24				62,63	
$\bar{\bar{x}}_3 = (1/3)\sum \bar{x}_{3i}$				34,7				5,28				63,96	
$\bar{\bar{x}}_4 = (1/3)\sum \bar{x}_{4i}$				36,0				4,50				64,53	
$\bar{R} = (1/12)\sum R_i$					4,54				0,368				0,958
$\hat{\sigma}_W^2 = 10(\bar{R} \times 0{,}886\,2)^2$					161,873				1,064				7,208
$\hat{\sigma}_W$					12,7				1,03				2,68

NOTE — The symbols x_1, x_2,...... correspond to Lot No. 1, 2,...... respectively.

Example 4: Evaluation of quality variation from the simplified variogram

Particulars of lot

Name of iron ore: ....

Type of iron ore: (e.g. lump ore) ....

Classification of quality variation: "Small"

Name of lot: (e.g. name of ship) ....

Date of delivery: ....

Mass of lot: 40 × 2 800 t = 112 000 t (wet) ....

Particulars of sampling

Mass of increment: 500 kg

Number of increments: 40

Systematic mass basis sampling at 2 800 t intervals

Each increment divided into two parts and the duplicate samples A_i and B_i prepared and analysed individually

Characteristic measured: total iron content (% Fe)

Increment	Fe %				Increment	Fe %			
	A_i	B_i	$\overline{x}_i$	R_i		A_i	B_i	$\overline{x}_i$	R_i
1	65,22	64,79	65,01	0,43	21	65,22	65,35	65,29	0,13
2	64,57	64,84	64,71	0,27	22	64,98	64,96	64,97	0,02
3	65,41	65,52	65,47	0,11	23	64,47	64,82	64,65	0,35
4	65,76	65,26	65,51	0,50	24	65,31	65,08	65,20	0,23
5	65,41	65,49	65,45	0,08	25	64,83	64,94	64,89	0,11
6	64,99	65,06	65,03	0,07	26	65,15	65,06	65,11	0,09
7	65,41	65,23	65,32	0,18	27	65,14	65,14	65,14	0,00
8	64,85	64,97	64,91	0,12	28	65,15	64,68	64,92	0,47
9	65,72	65,83	65,78	0,11	29	64,86	64,94	64,90	0,08
10	65,56	65,66	65,61	0,10	30	65,23	65,23	65,23	0,00
11	65,87	65,29	65,58	0,58	31	65,14	65,43	65,29	0,29
12	66,17	65,98	66,08	0,19	32	65,10	65,23	65,17	0,13
13	65,70	65,22	65,46	0,48	33	65,00	65,04	65,02	0,04
14	65,08	64,97	65,03	0,11	34	65,16	65,08	65,12	0,08
15	64,16	64,19	64,18	0,03	35	64,76	64,84	64,80	0,08
16	64,61	64,55	64,58	0,06	36	65,18	65,14	65,16	0,04
17	65,36	65,11	65,24	0,25	37	65,20	65,18	65,19	0,02
18	65,20	64,96	65,08	0,24	38	65,14	65,07	65,11	0,07
19	65,22	65,35	65,29	0,13	39	65,07	64,80	64,94	0,27
20	65,16	65,16	65,16	0,00	40	65,14	65,35	65,25	0,21
$\overline{R} = (1/40)\sum R_i$									0,168 7
$\hat{\sigma}^2_{\mathrm{PM}}/2 = (\overline{R} \times 0{,}886\,2)^2/2$									0,011 2

Equation (8) is used to calculate the experimental variogram for the average values of the duplicate measurements in the example. For example, since there are 39 terms in the sum, corresponding to the 39 pairs of increments separated by 2 800 tonnes, the experimental variogram at lag 1 (Δt = 2 800 tonnes) is:

$$V_E(\Delta t) = \frac{\sum_{i=1}^{39} (\bar{x}_{i+1} - \bar{x}_i)^2}{(2 \times 39)}$$

$$= [(64{,}71 - 65{,}01)^2 + (65{,}47 - 64{,}71)^2 + (65{,}51 - 65{,}47)^2 + ... + (65{,}25 - 64{,}94)^2]/(2 \times 39)$$

$$= 0{,}068\ 6$$

The variance of sample preparation and measurement is determined from the differences between the duplicate analyses A_i and B_i using the method specified in ISO 3085. In this example, half the preparation and measurement variance was found to be 0,011 2. Hence, the corrected variogram at lag 1 is given by:

$$V_1 = V_C(\Delta t) = V_E(\Delta t) - 0{,}011\ 2 = 0{,}068\ 6 - 0{,}011\ 2 = 0{,}057\ 4$$

Similarly, since there are 38 pairs of samples at lag 2 (5 600 tonnes), the experimental variogram for lag 2 is:

$$V_E(2\Delta t) = \frac{\sum_{i=1}^{38} (\bar{x}_{i+2} - \bar{x}_i)^2}{(2 \times 38)}$$

$$= [(65{,}47 - 65{,}01)^2 + (65{,}51 - 64{,}71)^2 + (65{,}45 - 65{,}47)^2 + ... + (65{,}25 - 65{,}11)^2]/(2 \times 38)$$

$$= 0{,}102\ 1$$

The corrected variogram for lag 2 is

$$V_2 = V_C(2\Delta t) = V_E(2\Delta t) - 0{,}011\ 2 = 0{,}102\ 1 - 0{,}011\ 2 = 0{,}090\ 9$$

The corrected variogram for lags 3, 4, ..., n can be calculated similarly. The corrected values for the first 10 lags are given in the following table and are plotted in figure A.1.

Lag	1	2	3	4	5	6	7	8	9	10
V_C	0,057 4	0,090 9	0,122 2	0,130 3	0,120 0	0,113 1	0,087 0	0,091 3	0,101 0	0,121 3

The variogram values can now be used to determine the quality variation in accordance with 6.2.

The intercept V_0 is calculated by extrapolating the straight line through V_2 and V_1 to the axis at zero lag as shown in figure A.1.

Using equation (13):

$$V_0 = 2V_1 - V_2$$

$$= 2\ (0{,}057\ 4) - 0{,}090\ 9 = 0{,}023\ 9$$

The slope, B, is calculated using equation (14) as follows:

$$B = (V_2 - V_1)/\Delta t$$

$$= (0{,}090\ 9 - 0{,}057\ 4)/2\ 800 = 1{,}20 \times 10^{-5}$$

For systematic sampling, the estimated quality variation , $\hat{\sigma}_W$, is calculated using equation (18) as follows:

$$\hat{\sigma}_W^2 = V_0 + B\,\Delta t/6$$

$$= 0{,}023\,9 + 1{,}2 \times 10^{-5} \times 2\,800/6$$

$$= 0{,}029\,5$$

Hence,

$$\hat{\sigma}_W = 0{,}17\%\ \text{Fe}$$

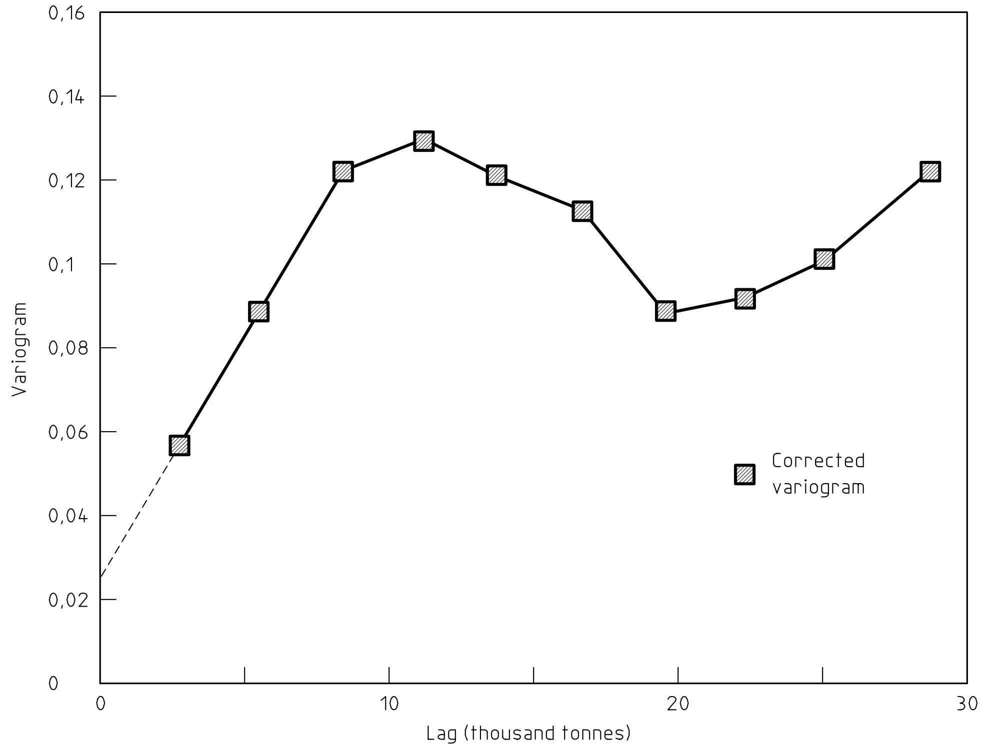


Figure A.1 — Corrected variogram with zero lag extrapolation

ICS 73.060.10

Descriptors: minerals and ores, metalliferous minerals, iron ores, tests, determination, quality.

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