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Aluminium ores — Experimental methods for checking the precision of sampling

Minerais alumineux — Méthodes expérimentales de contrôle de la fidélité d'échantillonnage

Reference number
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Foreword

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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 10277 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*, Subcommittee SC 1, *Sampling*.

Annex A forms an integral part of this International Standard. Annex B is for information only.

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Aluminium ores — Experimental methods for checking the precision of sampling

1 Scope

This International Standard specifies the experimental methods to be applied for checking the precision of sampling of aluminium ores, expressed in terms of the standard deviation, being carried out in accordance with the methods prescribed in ISO 8685.

NOTE 1 These methods may also be applied for the purpose of checking the precision of preparation of samples being carried out in accordance with the methods prescribed in ISO 6140.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10277. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 10277 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 6139:1993, *Aluminium ores — Experimental determination of the heterogeneity of distribution of a lot.*

ISO 6140:1991, *Aluminium ores — Preparation of samples.*

ISO 8685:1992, *Aluminium ores — Sampling procedures.*

3 Symbols

The following symbols are used throughout this International Standard:

d_2 factor to estimate the standard deviation from the range ($d_2 = 1{,}128$ for a pair of determinations)

n number of increments

R_1 absolute difference between determinations on subsample A and subsample B

$\overline{R}_1$ mean absolute difference between determinations on subsamples A and B for n_s sampling units

R_2 absolute difference between determinations on divided subsamples B_1 and B_2

$\overline{R}_2$ mean absolute difference between determinations on divided subsamples B_1 and B_2 for n_s sampling units

R_3 absolute difference between determinations on the same divided subsample B_2

$\overline{R}_3$ mean absolute difference between determinations on the same divided subsample B_2 for n_s sampling units

x subsample values

$\bar{x}$ mean value of a quality characteristic

x_1 determination on subsample A

x_2 determination on subsample B

x_3 determination on divided subsample B_1

x_4 determination on divided subsample B_2

x_i value of non-reference member of ith pair

x_{ri} value of reference member of ith pair

σ_S standard deviation of sampling

$\hat{\sigma}$ estimated value of σ

$\hat{\sigma}_M$ estimated standard deviation of measurement

$\hat{\sigma}_P$ estimated standard deviation of sample preparation

$\hat{\sigma}_{PM}$ estimated standard deviation of sample preparation and measurement

$\hat{\sigma}_S$ estimated standard deviation of sampling

$\hat{\sigma}_{SPM}$ estimated overall standard deviation of sampling, sample preparation and measurement

4 General conditions

4.1 General

The determination of precision of sampling is based on duplicate sampling from lots. If sample preparation and analysis is also carried out in duplicate, it is possible to determine the errors associated with those parameters in addition to the errors due to sampling.

4.2 Number of lots for the experiment

In order to reach a reliable conclusion, it is recommended that the experiment be carried out on more than 20 lots of the same type of aluminium ore. However, if this is impracticable, at least 10 lots should be covered and each lot shall be divided into several parts to produce more than 20 parts for the experiment. The experiment shall be carried out on each part, considering each part as a separate lot in accordance with ISO 8685.

4.3 Number of increments and number of gross samples

The minimum number of increments required for the experiment shall be twice the number specified in ISO 8685. Thus, if the number of increments required for the routine sampling is n and one gross sample is constituted, the minimum number of increments required for the experiments shall be $2n$ and two gross samples shall be constituted.

NOTE 2 If this is impracticable, n increments may be taken and divided into two parts, each comprising $n/2$ increments.

4.4 Sample preparation and testing

The preparation of samples shall be in accordance with ISO 6140 and the testing of samples shall be carried out in accordance with the methods prescribed in the relevant International Standards.

4.5 Replication of experiment

It is recommended that, even after a series of experiments has been conducted, the experiments should be repeated at regular intervals and when there is a change in ore quality. The experiment should also be repeated when there is a change in equipment or of ore supplier.

Because of the large amount of work involved in this method, it is recommended that the procedure should be carried out as a part of routine work of sampling and measurement.

5 Experimental

5.1 Duplicate samples

Each alternate primary increment is set aside in order to form gross samples A and B. The number of divided increments per primary increment is the same as that taken for routine sampling. An example of a sampling plan for gross samples A and B is shown in figure 1.

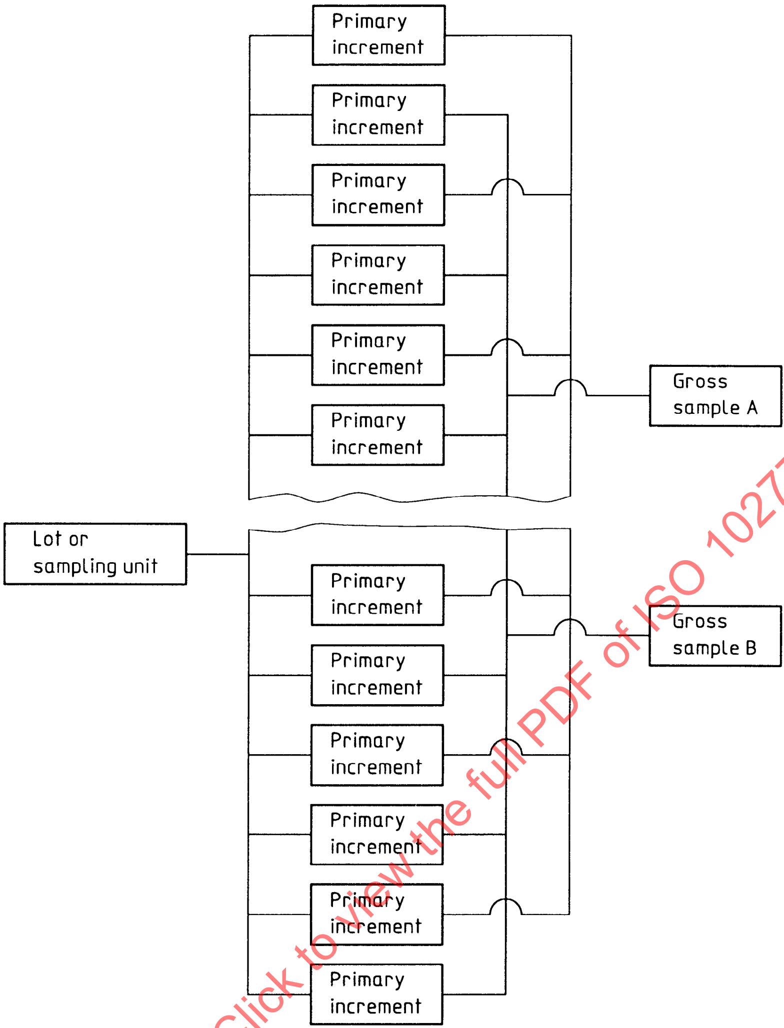


Figure 1 — Example of a plan of duplicate sampling

5.2 Sample division and testing

The two gross samples A and B taken in accordance with 5.1 shall be divided separately and subjected to either type 1, type 2 or type 3 testing as described in 5.2.1, 5.2.2 or 5.2.3 respectively.

5.2.1 Division-testing type 1 (see figure 2)

5.2.1.1 The two gross samples A and B shall be divided separately to prepare two final samples.

5.2.1.2 The four final samples A_1, A_2 and B_1, B_2 shall each be tested in duplicate. A total of eight tests shall be run in random order.

NOTE 3 In type 1 testing, the standard deviations of sampling, preparation and measurement are obtained separately.

5.2.2 Division-testing type 2 (see figure 3)

5.2.2.1 The gross sample A shall be divided to prepare two final samples, A_1 and A_2, and from the gross sample B, one final sample shall be prepared.

5.2.2.2 The final sample A_1 shall be tested in duplicate and the other final samples A_2 and B shall be tested individually.

NOTE 4 In type 2 testing, the standard deviations of sampling, preparation and measurement are obtainable separately. However, the precision for estimating the standard deviations of sampling, preparation and measurement will be lower than that attainable in type 1 testing.

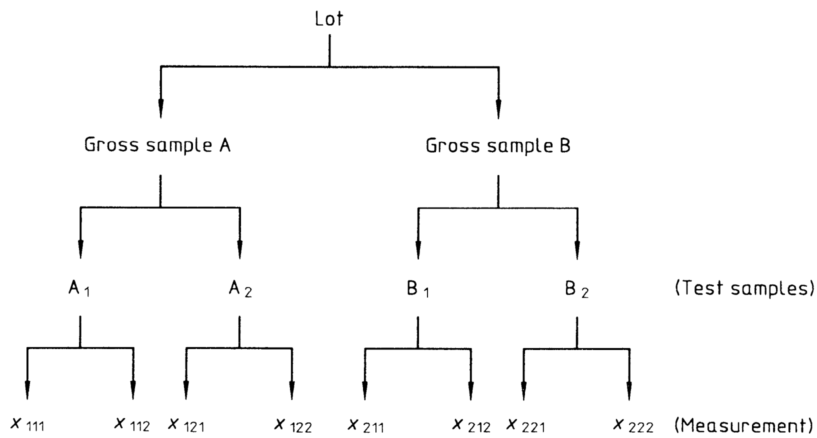


Figure 2 — Flowsheet for division-testing type 1

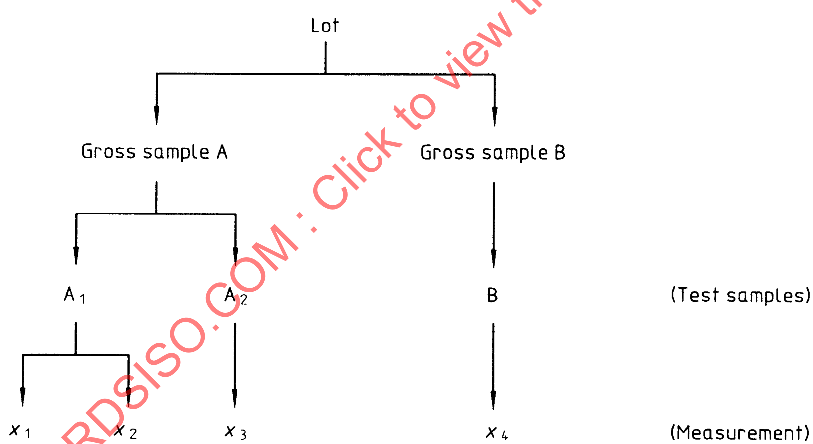


Figure 3 — Flowsheet for division-testing type 2

5.2.3 Division-testing type 3 (see figure 4)

5.2.3.1 From each of the two gross samples A and B, one final sample shall be prepared.

5.2.3.2 The two final samples A and B shall be tested individually.

NOTE 5 In type 3 testing, only the overall standard deviation of sampling, preparation and measurement is obtained.

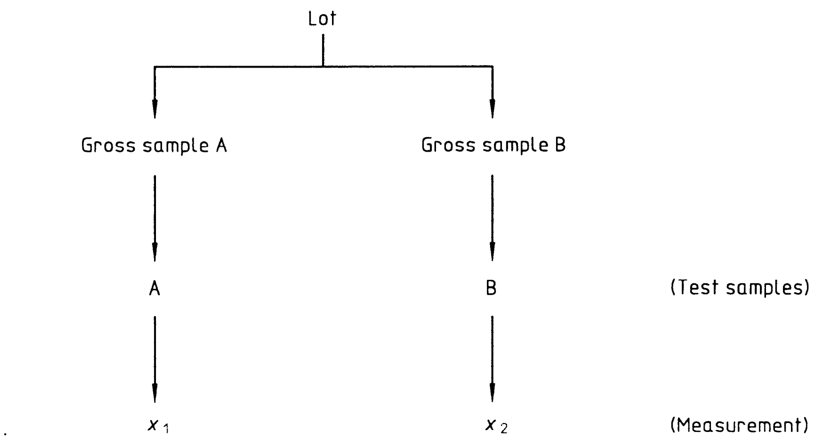


Figure 4 — Flowsheet for division-testing type 3

6 Analysis of experimental data

The procedure for the analysis of experimental data shall be as specified in 6.1 to 6.3 or in annex A for the type of division-testing selected. A procedure for treating data containing rogue results is included in the procedure (see example clause 7). When the data do not contain rogue values, the method in annex A may be used.

6.1 Division-testing type 1 (see figure 2 and sheet 2)

The estimated values of approximately 95 % probability standard deviation (hereinafter referred to simply as standard deviation) of sampling, preparation and measurement shall be calculated in accordance with the procedure given below:

a) Denote the pair of four measurements (such as Al_2O_3 as a percentage by mass) of a pair of two duplicate samples, prepared from the two gross samples A and B, as x_{111}, x_{112}, x_{121}, x_{122} and x_{211}, x_{212}, x_{221}, x_{222}.

b) Calculate the mean and the range for each pair of duplicate measurements:

$$\bar{x}_{ij.} = \frac{1}{2}(x_{ij1.} + x_{ij2}) \quad \ldots (1)$$

$$R_1 = |x_{ij1} - x_{ij2}| \quad \ldots (2)$$

where

$i = 1$ and 2, stands for gross samples A and B respectively;

$j = 1$ and 2, stands for final samples A_1, B_1 and A_2, B_2 respectively.

c) Calculate the mean and the range for each pair of duplicate samples:

$$\bar{\bar{x}}_{i..} = \frac{1}{2}(\bar{x}_{i1.} + \bar{x}_{i2.}) \quad \ldots (3)$$

$$R_2 = |\bar{x}_{i1.} - \bar{x}_{i2.}| \quad \ldots (4)$$

d) Calculate the mean and the range for each pair of gross samples, A and B:

$$\bar{\bar{\bar{x}}} = \frac{1}{2}(\bar{\bar{x}}_{1..} + \bar{\bar{x}}_{2..}) \quad \ldots (5)$$

$$R_3 = |\bar{\bar{x}}_{1..} - \bar{\bar{x}}_{2..}| \quad \ldots (6)$$

e) Calculate the overall mean and the mean of ranges ($\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$):

$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{k}\sum \bar{\bar{\bar{x}}} \quad \ldots (7)$$

$$\bar{R}_1 = \frac{1}{4k}\sum R_1 \quad \ldots (8)$$

$$\bar{R}_2 = \frac{1}{2k}\sum R_2 \quad \ldots (9)$$

$$\bar{R}_3 = \frac{1}{k}\sum R_3 \quad \ldots (10)$$

where k is the number of lots.

Calculate the control limits to construct the control charts for means and ranges.

Control limits for $\bar{x}$-chart:

$$\bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_1,\ \bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_2,\ \bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_3 \quad \ldots (11)$$

Upper control limit for R-chart:

$$D_4\bar{R}_1 \text{ (for } R_1\text{)},\ D_4\bar{R}_2 \text{ (for } R_2\text{)},$$

$$D_4\bar{R}_3 \text{ (for } R_3\text{)} \qquad \ldots (12)$$

where $A_2 = 1{,}880$ and $D_4 = 3{,}267$ (for a pair of measurements). (See clause 8.)

f) Using the ranges of measurement, calculate the estimated values of variance of measurement ($\hat{\sigma}_M^2$), preparation ($\hat{\sigma}_P^2$) and sampling ($\hat{\sigma}_S^2$):

$$\hat{\sigma}_M^2 = (\bar{R}_1/d_2)^2 \qquad \ldots (13)$$

$$\hat{\sigma}_P^2 = (\bar{R}_2/d_2)^2 - \frac{1}{2}(\bar{R}_1/d_2)^2 \qquad \ldots (14)$$

$$\hat{\sigma}_S^2 = (\bar{R}_3/d_2)^2 - \frac{1}{2}(\bar{R}_2/d_2)^2 \qquad \ldots (15)$$

where $1/d_2 = 0{,}886$ (for a pair of measurements). (See clause 8.)

NOTE 6 When n increments are taken and divided into two parts in accordance with note 2 in 4.3, the value of $\hat{\sigma}_S^2$ in equation (15) shall be divided by 2 to compare with the specified variance $(\sigma_S)^2$. The comparison described in step h) below will be made using the value thus obtained.

g) Calculate the estimated values of standard deviation of measurement ($\hat{\sigma}_M$), preparation ($\hat{\sigma}_P$) and sampling ($\hat{\sigma}_S$).

h) Compare the value of $\hat{\sigma}_S$ thus obtained with the desired standard deviation of sampling (σ_S) as given in ISO 8685.

6.2 Division testing type 2 (see figure 3)

The estimated values of standard deviation shall be calculated in accordance with the following procedure:

a) Denote the four measurements as follows:

- x_1, x_2: pair of duplicate measurements of a final sample A$_1$ prepared from gross sample A;
- x_3: single measurement of a final sample A$_2$ prepared from a gross sample A;
- x_4: single measurement of a final sample B prepared from gross sample B.

b) Calculate the mean and the range for each pair of duplicate measurements:

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \qquad \ldots (16)$$

$$R_1 = |x_1 - x_2| \qquad \ldots (17)$$

c) Calculate the mean and the range for each selected pair of measurements, x_1 and x_3, or x_2 and x_3, selected at random:

$$\bar{\bar{x}} = \frac{1}{2}(x_1 + x_3) \text{ or } \frac{1}{2}(x_2 + x_3) \qquad \ldots (18)$$

$$R_2 = |x_1 - x_3| \text{ or } |x_2 - x_3| \qquad \ldots (19)$$

d) Calculate the mean and the range for each pair of gross samples, A and B, selected at random:

$$\bar{\bar{\bar{x}}} = \frac{1}{2}(x_1 + x_4),\ \frac{1}{2}(x_2 + x_4) \text{ or}$$

$$\frac{1}{2}(x_3 + x_4) \qquad \ldots (20)$$

$$R_3 = |x_1 - x_4|,\ |x_2 - x_4| \text{ or } |x_3 - x_4| \qquad \ldots (21)$$

e) Calculate the overall mean and the mean of ranges ($\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$):

$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{k}\sum \bar{\bar{\bar{x}}} \qquad \ldots (22)$$

$$\bar{R}_1 = \frac{1}{k}\sum R_1 \qquad \ldots (23)$$

$$\bar{R}_2 = \frac{1}{k}\sum R_2 \qquad \ldots (24)$$

$$\bar{R}_3 = \frac{1}{k}\sum R_3 \qquad \ldots (25)$$

where k is the number of lots.

Calculate the control limits to construct the control charts for mean and ranges.

Control limits for $\bar{x}$-chart:

$$\bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_1,\ \bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_2,\ \bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_3 \qquad \ldots (26)$$

Upper control limit for R-chart:

$$D_4\bar{R}_1,\ D_4\bar{R}_2,\ D_4\bar{R}_3 \qquad \ldots (27)$$

where $A_2 = 1{,}880$ and $D_4 = 3{,}267$ (for a pair of measurements). (See clause 8.)

f) Using the ranges, calculate the estimated values of variance of measurement ($\hat{\sigma}_M^2$), preparation ($\hat{\sigma}_P^2$) and sampling ($\hat{\sigma}_S^2$):

$$\hat{\sigma}_M^2 = (\bar{R}_1/d_2)^2 \qquad \ldots (28)$$

$$\hat{\sigma}_P^2 = (\bar{R}_2/d_2)^2 - (\bar{R}_1/d_2)^2 \qquad \ldots (29)$$

$$\hat{\sigma}_S^2 = (\overline{R}_3/d_2)^2 - (\overline{R}_2/d_2)^2 \qquad \dots (30)$$

where $1/d_2 = 0{,}886$ (for a pair of measurements). (See clause 8 and note 6 in 6.1.)

g) Calculate the estimated values of standard deviation of measurement ($\hat{\sigma}_M$), preparation ($\hat{\sigma}_P$) and sampling ($\hat{\sigma}_S$).

h) Compare the value of $\hat{\sigma}_S$ thus obtained with the desired standard deviation of sampling (σ_S) as given in ISO 8685.

6.3 Division testing type 3 (see figure 4)

In this case the estimated values of standard deviation of sampling, preparation and measurement are not obtainable separately. Type 3 testing gives the overall standard deviation ($\hat{\sigma}_{SPM}$):

$$\hat{\sigma}_{SPM}^2 = \hat{\sigma}_S^2 + \hat{\sigma}_P^2 + \hat{\sigma}_M^2 \qquad \dots (31)$$

The estimated value of overall standard deviation shall be calculated in accordance with the following procedure:

a) Calculate the mean and the range for each pair of measurements:

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \qquad \dots (32)$$

$$R = |x_1 - x_2| \qquad \dots (33)$$

where x_1, x_2 are the measurements of final samples A and B, respectively.

b) Calculate the overall mean and the mean of the range:

$$\bar{\bar{x}} = \frac{1}{k}\sum \bar{x} \qquad \dots (34)$$

$$\overline{R} = \frac{1}{k}\sum R \qquad \dots (35)$$

where k is the number of lots.

c) Calculate the control limits to construct control charts for mean and range.

Control limit for $\bar{x}$-chart:

$$\bar{\bar{x}} \pm A_2\overline{R} \qquad \dots (36)$$

Upper control limit for R-chart:

$$D_4\overline{R} \qquad \dots (37)$$

where $A_2 = 1{,}880$ and $D_4 = 3{,}267$ (for a pair of measurements). (See clause 8.)

d) Calculate the estimated values of overall variance ($\hat{\sigma}_{SPM}^2$):

$$\hat{\sigma}_{SPM}^2 = (\overline{R}/d_2)^2 \qquad \dots (38)$$

e) Calculate the estimated value of overall standard deviation ($\hat{\sigma}_{SPM}$).

7 Interpretation of results and action

7.1 Interpretation

7.1.1 Data containing no rogue results

When all of the values of R_3, R_2 and R_1 calculated in accordance with 6.1 and 6.2 are within the upper control limit of the R-chart constructed in accordance with 6.1 e) and 6.2 e), it is an indication that the routine processes of sampling, division and measurement of samples are under control.

When all of the values of R calculated in accordance with 6.3 are within the upper control limit of the R-chart constructed in accordance with 6.3 c) , it is an indication that the overall process of sampling, division and measurement is under control.

On the other hand, when any of the values of R_3, R_2, R_1, calculated in accordance with 6.1 and 6.2 and R, calculated in accordance with 6.3, fall outside the respective upper control limit, the process (such as sampling, preparation, or measurement) under investigation is not under control, and should be checked in order to detect assignable causes.

7.1.2 Data containing rogue results

When a greater number of the values of $\bar{x}_{ij.}$ or $\bar{\bar{x}}_{i..}$ calculated in accordance with 6.1, $\bar{x}$ or $\bar{\bar{x}}$ calculated in accordance with 6.2 or $\bar{x}$ calculated in accordance with 6.3, is outside the control limits of the corresponding x-chart, it is an indication that the standard deviation of measurement or standard deviation of preparation is reasonably sufficient.

When most of the values of $\bar{\bar{x}}$ calculated in accordance with 6.1 and 6.2 or $\bar{x}$ calculated in accordance with 6.3 are within the control limits of the corresponding $\bar{x}$-chart, the standard deviation of sampling is insufficient, and the variation in quality characteristics of the lots under experiment could not be detected. Under such circumstances, the methods of sampling, preparation and measurement shall be reviewed for modification (see 7.2).

NOTE 7 These tests are necessary to ensure that the standard deviation of measurement or standard deviation of preparation are sufficient to enable the other components of error to be identified.

7.2 Action

When there is an indication that the standard deviation does not attain the desired value, the sampling procedure may be modified as follows:

a) Check the changes in heterogeneity of distribution of the aluminium ore in accordance with the method given in ISO 6139. If it is confirmed that there is a significant change in heterogeneity of distribution of the aluminium ore in question, the number of increments taken from a lot shall be revised accordingly.

   In the case of systematic or stratified random sampling when a greater number (denoted by n_1) of increments is collected from a lot, the standard deviation of sampling is improved in proportion to $\sqrt{n/n_1}$.

b) Increase the mass of increment. There is, however, a limit above which increasing the sample mass will not effect a significant improvement of the standard deviation of sampling.

8 Experimental example

The following experimental example is based on periodic, systematic sampling by division-testing type 1, and conducted by a consumer of aluminium ores. The experimental results are summarized in sheets 1 and 2, and in figure 5.

Sheet 1 shows details of the experiment and analysis results of alumina (Al_2O_3) determinations.

Sheet 2 shows the Al_2O_3 content and the process of calculation of $\hat{\sigma}_M$, $\hat{\sigma}_P$ and $\hat{\sigma}_S$.

Figure 5 shows the control charts for the mean and the range for $\bar{x}$, $\bar{\bar{x}}$, $\bar{\bar{\bar{x}}}$ and R_1, R_2, R_3.

In order to avoid errors and omissions, and for future reference, it may be convenient to keep detailed records of experiments in a standardized form such as that used in the example shown.

The number of cases where points of data are situated outside the 3-sigma control limits are recorded in the bottom space of sheet 2, and the corresponding data in the body of the sheet are identified by asterisks (see 7.1).

The values of estimated standard deviation of measurement, preparation and sampling of this example are as follows:

Standard deviation of measurement:

$$\hat{\sigma}_M = 0{,}077 \text{ [\% } (m/m) \text{ of } Al_2O_3]$$

Standard deviation of preparation:

$$\hat{\sigma}_P = 0{,}17 \text{ [\% } (m/m) \text{ of } Al_2O_3]$$

Standard deviation of sampling:

$$\hat{\sigma}_S = 0{,}23 \text{ [\% } (m/m) \text{ of } Al_2O_3]$$

Of the three, $\hat{\sigma}_S$ is the greatest.

Sheet 1 — Example of recording experimental details

[Name of Company and Works]

Report of checking the precision of sampling

Date of experiment: ....

Site of experiment: ....

Characteristic measured: Alumina content as a percentage by mass

Lots investigated

Source and type of ore: ....

Loading point: ....

Means of transportation: Ship

Number of lots: 20

Mass of lots: Mean 9 920 t; minimum 7 000 t; maximum 13 000 t

Sampling details

Maximum particle size of lots: 110 mm

Type of increment: Unit mass of ore on belt conveyor; for its full cross-section over a certain length of flow

Nominal mass of increment: 25 kg

Number of increments: Stop belt conveyor at specified tonnage intervals of ore discharge, and collect all ore with a shovel on the belt at the specified location to obtain a 25 kg increment

Preparation of samples

Method of constituting gross samples: Place alternately individual increments taken successively in containers A and B, and constitute gross samples A and B, each comprising 50 increments.

Mass of gross samples: Mean 1 250 kg; minimum 1 220 kg; maximum 1 285 kg

Type of dividing of gross samples: Division-testing type 1 (duplicate samples)

Mesurements of Al_2O_3 [% (m/m)]

Statistic	Experimental results	Commercial determination	Found at loading point
Mean	51,10	—	—
Minimum	49,90	—	—
Maximum	53,02	—	—

Estimated precision of sampling [% (m/m) of Al_2O_3]

$\hat{\sigma}_M = 0{,}077$ $\hat{\sigma}_{SPM} = 0{,}29$

$\hat{\sigma}_P = 0{,}17$

$\hat{\sigma}_S = 0{,}23$

Comments and remarks: ....

....

Date: .... Reported by: ....

[Name of supervisor of experiment]

Sheet 2 — Example of recorded values

(See 6.1 and figure 5)

Source and type of ore: ....

Date: ....

Characteristic measured: alumina content

Number of lots: 20

Date of experiment: ....

Lot No.		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	**Sum**	**Mean**
Date of sampling																							
Mass of lot, t		12 100	7300	10 700	13 000	11 500	10 000	11 200	9700	8600	9300	8300	10 500	8200	10 600	9100	10 400	7900	11 200	11 800	7000	198 400	9920
Number of increments	A	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	1 000	50
	B	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	50	1 000	50
A_1	x_{111}	50,92	50,88	50,82	51,40	52,04	52,70	50,94	50,90	51,20	50,94	49,94	50,08	50,38	51,10	52,00	50,72	51,50	51,08	51,15	51,54	1 022,23	51,11
	x_{112}	50,99	50,87	50,76	51,30	52,00	52,92	50,98	50,87	51,00	51,07	49,90	50,04	50,23	51,00	51,93	50,78	51,42	50,94	51,30	51,32	1 021,62	51,08
	$\bar{x}_{11.}$	50,96	50,88*	50,79*	51,35*	52,02*	52,81*	50,96	50,88*	51,10	51,00	49,92*	50,06*	50,30*	51,05	51,96*	50,75*	51,46*	51,01	51,22	51,43*	1 021,91	51,10
	R_1	0,07	0,01	0,06	0,10	0,04	0,22	0,04	0,03	0,20	0,13	0,04	0,04	0,15	0,10	0,07	0,06	0,08	0,14	0,15	0,22	1,95	0,10
A_2	x_{121}	50,98	51,02	50,96	51,40	52,27	52,90	50,80	51,02	51,08	51,00	50,02	50,14	50,30	51,00	52,32	51,14	52,02	51,04	51,10	51,50	1 024,01	51,20
	x_{122}	51,01	51,02	50,88	51,25	52,44	52,72	50,85	51,00	51,08	51,00	50,09	50,26	50,30	51,02	52,27	51,14	52,07	50,96	51,08	51,26	1 023,70	51,18
	$\bar{x}_{12.}$	51,00	51,02	50,92*	51,32*	52,36*	52,81*	50,82*	51,01	51,08	51,00	50,06*	50,20*	50,30*	51,01	52,30*	51,14	52,04*	51,00	51,09	51,38*	1 023,86	51,19
	R_1	0,03	0,00	0,08	0,15	0,17	0,18	0,05	0,02	0,00	0,00	0,07	0,12	0,00	0,02	0,05	0,00	0,05	0,08	0,02	0,24	1,33	0,07
A	$\bar{\bar{x}}_{1..}$	50,98	50,95	50,86	51,34	52,19*	52,81*	50,89	50,94	51,09	51,00	49,99*	50,13*	50,30*	51,03	52,13*	50,94*	51,75*	51,00	51,16	51,40	1 022,88	51,14
	R_2	0,04	0,14	0,13	0,03	0,34	0,00	0,14	0,13	0,02	0,00	0,14	0,14	0,00	0,04	0,34	0,39	0,58	0,01	0,13	0,05	2,79	0,14
B_1	x_{211}	51,40	50,27	50,70	51,94	51,92	53,02	51,14	50,90	50,88	51,00	49,96	50,52	50,28	50,84	51,80	50,82	51,06	50,78	52,00	51,86	1 023,09	51,15
	x_{212}	51,34	50,10	50,67	51,97	51,77	52,94	51,20	50,88	50,64	51,00	50,02	50,60	50,18	50,66	51,74	50,74	51,04	50,80	52,05	51,60	1 021,94	51,10
	$\bar{x}_{21.}$	51,37*	50,18*	50,68*	51,96*	51,84*	52,98*	51,17	50,89*	50,76*	51,00	49,99*	50,56*	50,23*	50,75*	51,77*	50,78*	51,05	50,79*	52,02*	51,73*	1 022,50	51,12
	R_1	0,06	0,17	0,03	0,03	0,15	0,08	0,06	0,02	0,24	0,00	0,06	0,08	0,10	0,18	0,06	0,06	0,02	0,02	0,05	0,26	1,75	0,08
B_2	x_{221}	51,28	50,04	50,82	51,60	52,51	52,98	50,94	50,70	50,60	49,95	49,98	50,46	50,29	51,12	51,74	50,56	51,16	50,88	51,21	51,66	1 020,48	51,02
	x_{222}	51,35	49,93	50,60	51,43	52,52	52,92	51,03	50,50	50,55	49,87	49,90	50,35	50,32	50,96	51,71	50,38	51,25	50,89	51,12	51,58	1 019,16	50,96
	$\bar{x}_{22.}$	51,32*	49,98*	50,71*	51,52*	52,52*	52,95*	50,98	50,60*	50,58*	49,91*	49,94*	50,40*	50,30*	51,04	51,72*	50,47*	51,20	50,88*	51,16	51,62*	1 019,80	50,99
	R_1	0,07	0,11	0,22	0,17	0,01	0,06	0,09	0,20	0,05	0,08	0,08	0,11	0,03	0,16	0,03	0,18	0,09	0,01	0,09	0,08	1,92	0,10
B	$\bar{\bar{x}}_{2..}$	51,34	50,08*	50,70*	51,74*	52,18*	52,96*	51,08	50,74	50,67*	50,46*	49,96*	50,48*	50,26*	50,90	51,74*	50,62*	51,12	50,84	51,59*	51,68*	1 021,14	51,06
	R_2	0,05	0,20	0,03	0,44	0,68*	0,03	0,19	0,29	0,18	1,09*	0,05	0,16	0,07	0,29	0,05	0,31	0,15	0,09	0,86*	0,11	5,32	0,26

$\bar{\bar{\bar{x}}}$	51,16	50,52*	50,78	51,54	52,18*	52,86*	50,98	50,84	50,86	50,73	49,98*	50,30*	50,28*	50,96	51,94*	50,78	51,44	50,92	51,38	51,54	1 022,01	51,10
R_3	0,36	0,67	0,16	0,40	0,01	0,15	0,19	0,20	0,42	0,54	0,03	0,35	0,04	0,13	0,39	0,32	0,63	0,16	0,43	0,28	6,06	0,30

Calculation

$\hat{\sigma}_M^2 = (0{,}886\ 5\ R_1)^2 = 0{,}005\ 9$ $\quad (0{,}886\ 5\ \bar{R}_2)^2 = 0{,}032\ 3$ $\quad (0{,}886\ 5\ \bar{R}_3)^2 = 0{,}072\ 1$ $\quad \bar{\bar{\bar{x}}} = 51{,}10$ $\quad \bar{R}_1 = 0{,}087$ $\quad 3{,}267\ \bar{R}_1 = 0{,}204$

$\hat{\sigma}_M = 0{,}077$ $\quad \hat{\sigma}_P^2 = 0{,}032\ 3 - \dfrac{0{,}005\ 9}{2} = 0{,}029\ 4$ $\quad \hat{\sigma}_S^2 = 0{,}072\ 1 - \dfrac{0{,}032\ 3}{2} = 0{,}056\ 0$ $\quad \bar{R}_2 = 0{,}203$ $\quad 3{,}267\ \bar{R}_2 = 0{,}664$

$\hat{\sigma}_P = 0{,}171$ $\quad \hat{\sigma}_S = 0{,}237$ $\quad \bar{R}_3 = 0{,}303$ $\quad 3{,}267\ \bar{R}_3 = 0{,}991$

$\bar{\bar{\bar{x}}} \pm 1{,}880\ \bar{R}_1 = 51{,}10 \pm 0{,}164$ (51,26 and 50,94); $\bar{\bar{\bar{x}}} \pm 1{,}880\ \bar{R}_2 = 51{,}10 \pm 0{,}382$ (51,48 and 50,72); $\bar{\bar{\bar{x}}} \pm 1{,}880\ \bar{R}_3 = 51{,}10 \pm 0{,}570$ (51,67 and 50,53)

Adjustment for calculated values

Individual % Al_2O_3 identified by asterisk (*) are outside the 3-sigma control limits.

Number of cases where % Al_2O_3 fell outside the limits are

R_1: 0 out of 80 data (Simplify as 0/80), R_2: 3/40, R_3: 0/20, $\bar{x}$: 57/80, $\bar{\bar{x}}$: 21/40, $\bar{\bar{\bar{x}}}$: 7/20

$\hat{\sigma}_M^2 = 0{,}005\ 9$

$\hat{\sigma}_M = 0{,}077$

First adjustment for R_2 :

$\bar{R}'_2 = 0{,}148$

$3{,}267\ \bar{R}'_2 = 0{,}484$ (One point outside the UCL)

Second adjustment for R_2 :

$\bar{R}''_2 = 0{,}136$

$3{,}267\ \bar{R}''_2 = 0{,}445$ (No point outside the UCL)

$(0{,}886\ 5\ \bar{R}''_2)^2 = 0{,}014\ 5$

$\hat{\sigma}_P = 0{,}107\ 5$

$(0{,}886\ 5\ \bar{R}'_3)^2 = 0{,}060\ 7$

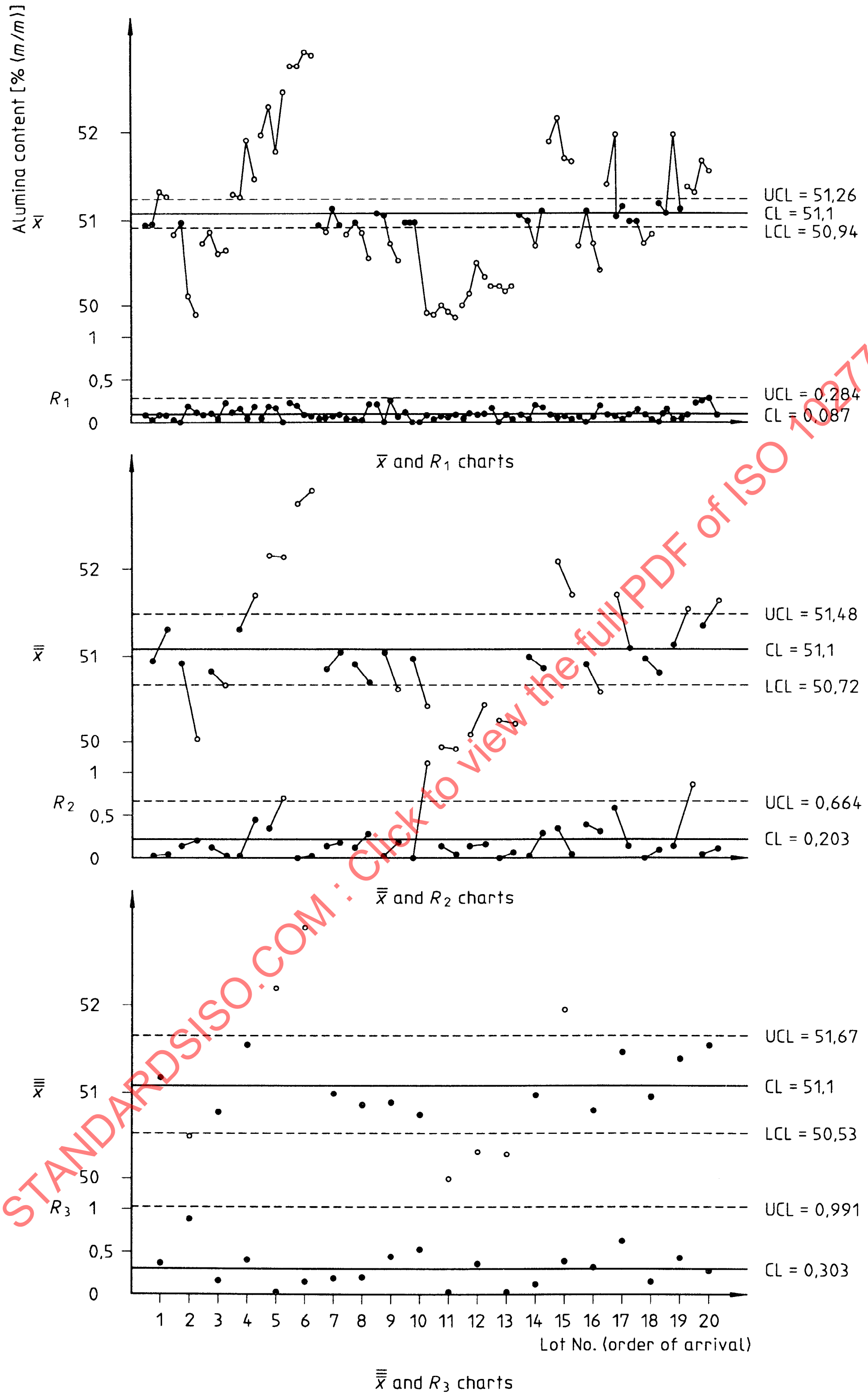
$\hat{\sigma}_S = 0{,}231\ 2$

Comments and remarks:

Recorded by: ....

Checked by: ....

[Name of supervisor of experiment]



Key

°	Data outside the control limits	CL	Central line
UCL	Upper control limit	LCL	Lower control limit

Figure 5 — Example of control charts for mean and range (Graphical presentation of data in sheet 2)

Annex A
(normative)

Alternative method for analysis of experimental data

When the data do not contain rogue values (see 7.1.1) this alternative method may be used for analysing the experimental data in place of the method specified in clause 6.

A.1 Division-testing type 1

The estimated values of approximately 95 % probability standard deviation (hereinafter referred to simply as standard deviation) of sampling, division and measurement should be calculated in accordance with the procedure given below:

a) Denote the pair of four measurements (such as Al_2O_3 as a percentage by mass) of a pair of two duplicate samples, prepared from the two gross samples A and B, as x_{111}, x_{112}, x_{121}, x_{122} and x_{211}, x_{212}, x_{221}, x_{222}.

b) Calculate the mean and the range for each pair of duplicate measurements:

$$\bar{x}_{ij.} = \frac{1}{2}(x_{ij1} + x_{ij2}) \qquad \ldots \text{(A.1)}$$

$$R_1 = |x_{ij1} - x_{ij2}| \qquad \ldots \text{(A.2)}$$

where

$i = 1$ and 2, stands for A and B respectively;

$j = 1$ and 2, stands for final samples, A_1, B_1 and A_2, B_2 respectively.

c) Calculate the mean and the range for each pair of duplicate samples:

$$\bar{\bar{x}}_{i..} = \frac{1}{2}(\bar{x}_{i1.} + \bar{x}_{i2.}) \qquad \ldots \text{(A.3)}$$

$$R_2 = |\bar{x}_{i1.} - \bar{x}_{i2.}| \qquad \ldots \text{(A.4)}$$

d) Calculate the mean and the range for each pair of gross samples, A and B:

$$\bar{\bar{\bar{x}}} = \frac{1}{2}(\bar{\bar{x}}_{1..} + \bar{\bar{x}}_{2..}) \qquad \ldots \text{(A.5)}$$

$$R_3 = |\bar{\bar{x}}_{1..} - \bar{\bar{x}}_{2..}| \qquad \ldots \text{(A.6)}$$

e) Calculate the overall mean and the variance in accordance with the sum of squares of the ranges:

$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{k}\sum \bar{\bar{\bar{x}}} \qquad \ldots \text{(A.7)}$$

$$\sum R_1^2 = |x_{ij1} - x_{ij2}|^2 \qquad \ldots \text{(A.8)}$$

$$\sum R_2^2 = |\bar{x}_{i1} - \bar{x}_{i2}|^2 \qquad \ldots \text{(A.9)}$$

$$\sum R_3^2 = |\bar{\bar{x}}_1 - \bar{\bar{x}}_2|^2 \qquad \ldots \text{(A.10)}$$

$$\hat{\sigma}_1^2 = \frac{1}{8k}\sum R_1^2 \qquad \ldots \text{(A.11)}$$

$$\hat{\sigma}_2^2 = \frac{1}{4k}\sum R_2^2 \qquad \ldots \text{(A.12)}$$

$$\hat{\sigma}_3^2 = \frac{1}{2k}\sum R_3^2 \qquad \ldots \text{(A.13)}$$

where k is the number of lots.

f) Calculate the estimated values of the variance of measurement ($\hat{\sigma}_M^2$), preparation ($\hat{\sigma}_P^2$) and sampling ($\hat{\sigma}_S^2$):

$$\hat{\sigma}_M^2 = \hat{\sigma}_1^2 \qquad \ldots \text{(A.14)}$$

$$\hat{\sigma}_P^2 = \hat{\sigma}_2^2 - \frac{1}{2}\hat{\sigma}_M^2 \qquad \ldots \text{(A.15)}$$

$$\hat{\sigma}_S^2 = \hat{\sigma}_3^2 - \frac{1}{2}\hat{\sigma}_2^2 \qquad \ldots \text{(A.16)}$$

g) Calculate the estimated values of standard deviation of measurement ($\hat{\sigma}_M$), preparation ($\hat{\sigma}_P$) and sampling ($\hat{\sigma}_S$).

h) Compare the value of $\hat{\sigma}_S$ thus obtained with the desired standard deviation of sampling (σ_S) as given in ISO 8685.

A.2 Division-testing type 2

The estimated values of standard deviation should be calculated in accordance with the procedure given below:

a) Denote the four measurements as follows:

x_1, x_2: pair of duplicate measurements of a final sample A_1 prepared from gross sample A;

x_3: single measurement of a final sample A_2 prepared from gross sample A;

x_4: single measurement of a final sample B prepared from gross sample B.

b) Calculate the mean and the range for each pair of duplicate measurements:

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \qquad \ldots \text{(A.17)}$$

$$R_1 = |x_1 - x_2| \qquad \ldots \text{(A.18)}$$

c) Calculate the mean and the range for each selected pair of measurements, x_1 and x_3 or x_2 and x_3 selected at random:

$$\bar{\bar{x}} = \frac{1}{2}(x_1 + x_3) \text{ or } \frac{1}{2}(x_2 + x_3) \qquad \ldots \text{(A.19)}$$

$$R_2 = |x_1 - x_3| \text{ or } |x_2 - x_3| \qquad \ldots \text{(A.20)}$$

d) Calculate the mean and the range for each pair of gross samples, A and B selected at random:

$$\bar{\bar{\bar{x}}} = \frac{1}{2}(x_1 + x_4), \frac{1}{2}(x_2 + x_4) \text{ or } \frac{1}{2}(x_3 + x_4) \qquad \ldots \text{(A.21)}$$

$$R_3 = |x_1 - x_4|, \ |x_2 - x_4| \text{ or } |x_3 - x_4| \qquad \ldots \text{(A.22)}$$

e) Calculate the overall mean and the variances in accordance with the sum of squares of the ranges:

$$\bar{\bar{\bar{\bar{x}}}} = \frac{1}{k}\sum \bar{\bar{\bar{x}}} \qquad \ldots \text{(A.23)}$$

$$\hat{\sigma}_1^2 = \frac{1}{2k}\sum R_1^2 \qquad \ldots \text{(A.24)}$$

$$\hat{\sigma}_2^2 = \frac{1}{2k}\sum R_2^2 \qquad \ldots \text{(A.25)}$$

$$\hat{\sigma}_3^2 = \frac{1}{2k}\sum R_3^2 \qquad \ldots \text{(A.26)}$$

where k is the number of lots.

f) Calculate the estimated values of the variance of measurement ($\hat{\sigma}_M^2$), preparation ($\hat{\sigma}_P^2$) and sampling ($\hat{\sigma}_S^2$):

$$\hat{\sigma}_M^2 = \hat{\sigma}_1^2 \qquad \ldots \text{(A.27)}$$

$$\hat{\sigma}_P^2 = \hat{\sigma}_2^2 - \hat{\sigma}_M^2 \qquad \ldots \text{(A.28)}$$

$$\hat{\sigma}_S^2 = \hat{\sigma}_3^2 - \hat{\sigma}_2^2 \qquad \ldots \text{(A.29)}$$

g) Calculate the estimated values of standard deviation of measurement ($\hat{\sigma}_M$), preparation ($\hat{\sigma}_P$) and sampling ($\hat{\sigma}_S$).

h) Compare the value of $\hat{\sigma}_S$ thus obtained with the desired standard deviation of sampling (σ_S) as given in ISO 8685.

A.3 Division-testing type 3

In this case, the estimated values of standard deviation of sampling, preparation and measurement are not obtainable separately. Type 3 testing gives the overall standard deviation ($\hat{\sigma}_{SPM}$):

$$\hat{\sigma}_{SPM}^2 = \hat{\sigma}_S^2 + \hat{\sigma}_P^2 + \hat{\sigma}_M^2 \qquad \ldots \text{(A.30)}$$

The estimated value of overall standard deviation shall be calculated in accordance with the procedure given below:

a) Calculate the mean and the range for each pair of measurements:

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \qquad \ldots \text{(A.31)}$$

$$R = |x_1 - x_2| \qquad \ldots \text{(A.32)}$$

where x_1, x_2 are the measurements of final samples A and B, respectively.

b) Calculate the overall mean and the estimated value of overall variance in accordance with the sum of squares of the ranges:

$$\bar{\bar{x}} = \frac{1}{k}\sum \bar{x} \qquad \ldots \text{(A.33)}$$

$$\hat{\sigma}_{SPM}^2 = \frac{1}{k}\sum R^2 \qquad \ldots \text{(A.34)}$$

where k is the number of lots.

c) Calculate the estimated value of overall standard deviation ($\hat{\sigma}_{SPM}$).