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Ģelezove rude ï Eksperimentalne metode za preverjanje natanļ nosti vzorļ enja

Iron ores -- Experimental methods for checking the precision of sampling

Minerais de fer -- Méthodes expérimentales de contrôle de la fidélité de l'échantillonnage

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INTERNATIONAL STANDARD

ISO 3085

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Iron ores — Experimental methods for checking the precision of sampling

Minerais de fer — Méthodes expérimentales de contrôle de la fidélité de l'échantillonnage

Reference number
ISO 3085:1996(E)

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 3085 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 1, *Sampling*.

This third edition cancels and replaces the second edition (ISO 3085:1986), which has been technically revised.

Annexes A to C of this International Standard are for information only.

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Iron ores — Experimental methods for checking the precision of sampling

1 Scope

This International Standard specifies experimental methods for checking the precision of sampling of iron ores being carried out in accordance with the methods specified in ISO 3081 or ISO 3082.

NOTE 1 These methods may also be applied for the purpose of checking the precision of sample preparation and measurement.

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 2597-1:1994, *Iron ores — Determination of total iron content — Part 1: Titrimetric method after tin(II) chloride reduction.*

ISO 3081:1986, *Iron ores — Increment sampling — Manual method.*

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method.*

ISO 3084:1986, *Iron ores — Experimental methods for evaluation of quality variation.*

ISO 3087:1987, *Iron ores — Determination of moisture content of a consignment.*

ISO 4701—[1)], *Iron ores — Determination of size distribution by sieving.*

ISO 9507:1990, *Iron ores — Determination of total iron content — Titanium(III) chloride reduction methods.*

ISO 9508:1990, *Iron ores — Determination of total iron content — Silver reduction titrimetric method.*

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

3 Definitions

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

NOTE 2 The precision of sampling is defined mathematically in ISO 3081:1986, annex A.

4 Principle

From twenty lots or more, preferably taking twice as many increments as specified in ISO 3081 or ISO 3082 and placing the increments alternately into two gross samples. If this is impracticable or the precision testing is carried out in conjunction with routine sampling, the normal number of increments specified in ISO 3081 or ISO 3082 may be used.

Preparation of separate test samples from each gross sample and the determination of relevant quality characteristics.

1) To be published. (Revision of ISO 4701:1985)

Analysis of the experimental data obtained and calculation of the estimated value of precision of sampling for each selected quality characteristic.

Comparison of the estimated precision with that specified in ISO 3081 or ISO 3082, and taking the necessary action if the estimated precision does not attain these specified values.

5 General conditions

5.1 Sampling

The sampling procedure to be followed shall be selected from the three methods of sampling, viz. periodic systematic sampling, stratified sampling or two-stage sampling, depending on the method of taking increments from the lot in accordance with ISO 3081 or ISO 3082.

5.1.1 Number of lots

To reach a reliable conclusion, it is recommended that the experiment be carried out on more than 20 lots of the same type of iron ore. However, if this is impracticable, at least 10 lots should be covered. If the number of lots for the experiment is not sufficient, each lot may be divided into several parts to produce more than 20 parts in total for the experiment, and the experiment should be carried out on each part, considering each part as a separate lot in accordance with ISO 3081 or ISO 3082.

5.1.2 Number of increments and number of gross samples

The number of increments required for the experiment shall preferably be twice the number specified in ISO 3081 or ISO 3082. Hence, if the number of increments required for routine sampling is n_1 and one gross sample is made up from these increments, the number of increments required for the experiment shall be $2n_1$ and two gross samples shall be constituted.

Alternatively, if the experiment is carried out as part of routine sampling, n_1 increments may be taken and two gross samples constituted, each comprising $n_1/2$ increments. In this case, the sampling precision obtained will be for $n_1/2$ increments. The precision thus obtained must be divided by $\sqrt{2}$ to obtain the sampling precision for gross samples comprising n_1 increments (see clause 7).

5.2 Sample preparation and measurement

Sample preparation shall be carried out in accordance with ISO 3082 or ISO 3083. The measurement shall be carried out in accordance with ISO 2597, ISO 9507 or ISO 9508 for total iron content, ISO 3087 for moisture content, and ISO 4701 for size analysis.

NOTE 3 For the determination of the total iron content, it is preferable to carry out a series of determinations on test samples for a lot over a period of several days.

5.3 Replication of experiment

Even when a series of experiments has been conducted prior to regular sampling operations, the experiments should be carried out periodically to check for possible changes in quality variation and, at the same time, to control the precision of sampling, sample preparation and measurement. Because of the large amount of work involved, it should be carried out as part of routine sampling, sample preparation and measurement.

5.4 Record of the experiment

For future reference and to avoid errors and omissions, it is recommended that detailed records of experiments be kept in a standardized format (see clause 8 and annex A).

6 Method of experiment

6.1 Sampling

6.1.1 Periodic systematic sampling

6.1.1.1 The number of increments, n_1, shall be selected from ISO 3081:1986, table 4 or ISO 3082:1987, table 4 depending on the mass of the lot and the classification of quality variation, i.e. "large", "medium", or "small".

6.1.1.2 When $2n_1$ increments are taken, the sampling interval, Δm, in tonnes, shall be calculated by dividing the mass, m_1, of the lot by $2n_1$, i.e. giving intervals equal to one-half of the sampling interval for routine sampling.

$$\Delta m = \frac{m_1}{2n_1}$$

Alternatively, when the experiment is carried out as part of routine sampling and n_1 increments are taken, the sampling interval, Δm, shall be calculated by dividing the mass, m_1, of the lot by n_1.

$$\Delta m = \frac{m_1}{n_1}$$

The sampling intervals thus calculated shall be rounded down to the nearest 10 t.

6.1.1.3 The increments shall be taken at the sampling interval determined in 6.1.1.2, with a random start.

6.1.1.4 The increments shall be placed alternately in two containers. Thus, two gross samples, A and B, will be constituted.

EXAMPLE 1 (see figure 1)

Suppose that a lot of 19 000 t is transferred by belt conveyors and that the ore is classified as "medium" quality variation, then the number of increments required for routine sampling, n_1, is 60, as shown in ISO 3081:1986, table 4 or ISO 3082:1987, table 4.

When $2n_1$ increments are taken, the sampling interval for the experiment, Δm, is given by the equation

$$\Delta m = \frac{m_1}{2n_1} = \frac{19\,000}{60 \times 2} = 158 \rightarrow 150$$

Thus, increments are taken at 150 t intervals. The point for taking the first increment from the first sampling interval of 150 t is determined by a random selection method. If the point for taking the first increment is determined as 20 t from the beginning of handling the lot, subsequent increments should be taken at the point $20 + i\Delta m$, where $i = 1, 2, ..., 2n_1$ (170 t, 320 t and so on). Since the whole lot size is 19 000 t, 126 increments will be taken.

The increments are placed alternately in two containers, and two gross samples, A and B, are constituted, each composed of 63 increments.

6.1.2 Stratified sampling

6.1.2.1 When the number of wagons or containers (hereinafter referred to simply as "wagons"), i.e. the number of strata, n_4, forming one lot, is smaller than the number of increments required, n_1 given in ISO 3081:1986, table 4, the number of increments, n_3, to be taken from each wagon (stratum) shall be calculated from the equation.

$$n_3 = \frac{n_1}{n_4}$$

The number of increments thus calculated shall be rounded up to the next higher whole number if $2n_1$ increments are taken, or to the next higher whole even number if n_1 increments are taken.

6.1.2.2 When $2n_1$ increments are taken, $2n_3$ increments shall be taken from each wagon and shall be separated at random into two partial samples, each of n_3 increments.

Alternatively, when the experiment is carried out as part of routine sampling and n_1 increments are taken, n_3 increments shall be taken from each wagon and shall be separated at random into two partial samples, each of $n_3/2$ increments.

6.1.2.3 The two partial samples from each wagon shall be combined into two gross samples, A and B, respectively.

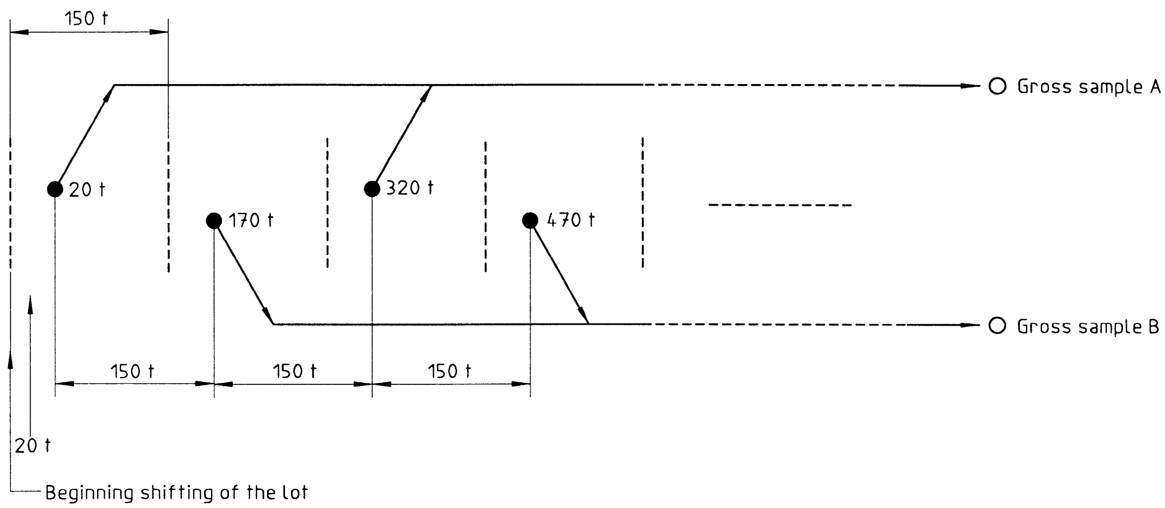
NOTE 4 If the mass varies from wagon to wagon, the number of increments to be taken from each wagon shall be decided in proportion to the mass of ore in each wagon. This method is called "proportional stratified sampling".

EXAMPLE 2 (see figure 2)

Suppose that a lot is delivered in 11 wagons each of 60 t capacity and that the quality variation of the ore within wagons, σ_W, is "medium" (see ISO 3084), then the minimum number of increments required, n_1, for the 660 t lot is 20, as shown in ISO 3081:1986, table 4.

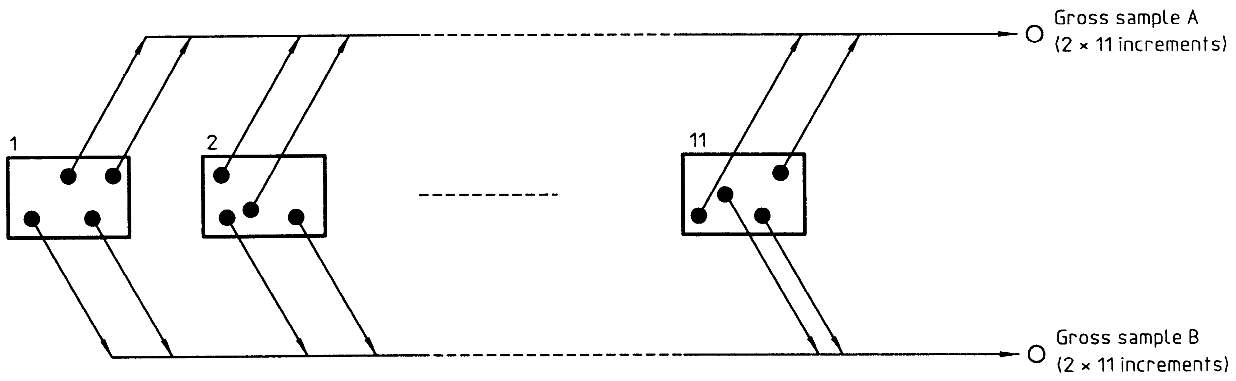
Thus, the number of increments to be taken from each wagon is

$$n_3 = \frac{n_1}{n_4} = \frac{20}{11} = 1{,}8 \rightarrow 2$$



Key
Solid circles and open circles indicate increments and gross samples respectively.

Figure 1 — Schematic diagram for example 1



Key
Boxes, solid circles and open circles indicate wagons, increments taken from a wagon and gross samples respectively.

Figure 2 — Schematic diagram for example 2

When $2n_1$ increments are taken, four ($2n_3 = 2 \times 2$) increments are taken from each wagon, and separated at random into two partial samples, each consisting of two increments.

The two partial samples from each of the 11 wagons are combined into two gross samples, A and B respectively, each comprising 22 ($2n_4 = 2 \times 11$) increments.

6.1.3 Two-stage sampling

6.1.3.1 If the number of wagons, n_4, forming a lot is more than the number of increments required, n_1, from ISO 3081:1986, table 4, or when it is impracticable to take increments from all the wagons, n_2 wagons shall be selected at random from the lot in accordance with ISO 3081:1986, table 5.

6.1.3.2 An additional n_2 wagons shall be selected at random from the same lot independently.

NOTE 5 In the process of random selection, it is possible for the same wagons to be included in each independent selection.

6.1.3.3 The required number of increments shall be taken from each of the n_2 wagons selected in accordance with ISO 3081:1986, 8.2.3.

6.1.3.4 All of the increments taken from the wagons selected in accordance with 6.1.3.1 shall be combined to make up gross sample A.

All of the increments taken from the wagons selected in accordance with 6.1.3.2 shall be combined into gross sample B.

EXAMPLE 3

Suppose that a wagon-borne lot consists of 80 wagons of 60 t capacity, i.e. $m_1 = 80 \times 60 = 4\,800$ t and that the quality variations within wagons, σ_w, and between wagons, σ_b, are "medium" and "small", respectively, then the minimum number of wagons to be selected, n_2, is 15, as shown in ISO 3081:1986, table 5.

From the same lot, an additional 15 wagons are selected at random independently of those previously selected.

The number of increments to be taken at random from each of the first 15 wagons, n_3, is four according to ISO 3081:1986, 8.2.3 and the total 60 ($n_2 n_3 = 15 \times 4$) increments are combined into gross sample A.

Similarly, an additional four increments are taken at random from each of the second 15 wagons, and the total 60 increments are combined into gross sample B.

6.2 Sample preparation and measurement

The two gross samples A and B taken in accordance with 6.1 shall be prepared separately and subjected to testing by either method 1, method 2 or method 3 described below.

6.2.1 Method 1

See figure 3.

The two gross samples A and B shall be divided separately. The resulting four test samples, A_1, A_2, B_1 and B_2, shall be tested in duplicate. The eight tests shall be run in random order.

NOTE 6 Method 1 allows the precisions of sampling, sample preparation and measurement to be obtained separately.

6.2.2 Method 2

See figure 4.

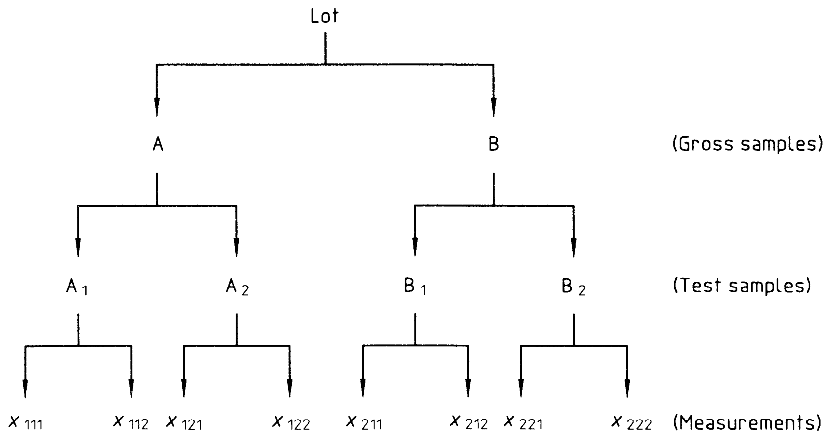


Figure 3 — Flowsheet for method 1

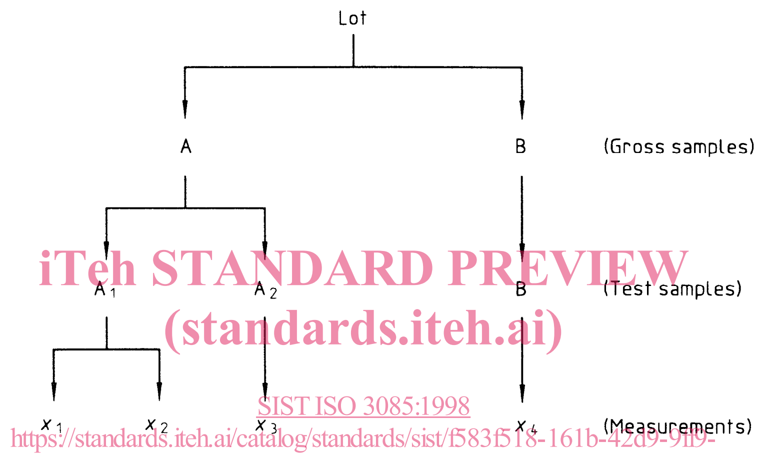


Figure 4 — Flowsheet for method 2

Gross sample A shall be divided to prepare two test samples, A_1 and A_2, and one test sample shall be prepared from gross sample B.

Test sample A_1 shall be tested in duplicate and single tests shall be conducted on test samples A_2 and B.

NOTE 7 Method 2 also allows the precisions of sampling, sample preparation and measurement to be obtained separately. However, the estimates of precision of sample preparation and measurement are less precise than those obtained by method 1.

6.2.3 Method 3

See figure 5.

One test sample shall be prepared from each of the two gross samples A and B, and single tests shall be conducted on each test sample.

NOTE 8 Using method 3, only the overall precision of sampling, sample preparation and measurement is obtained.

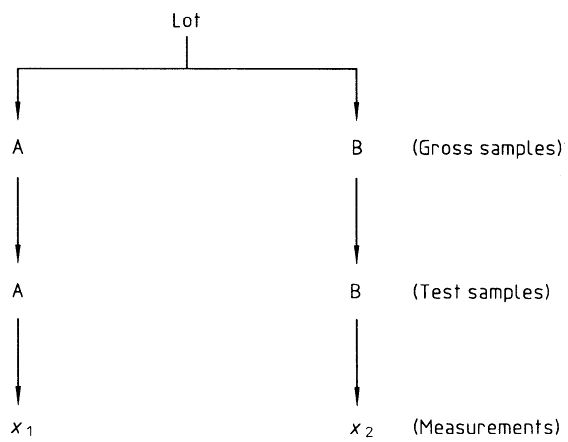


Figure 5 — Flowsheet for method 3

7 Analysis of experimental data

The method for analysis of experimental data shall be as specified below depending on the method of sample preparation and measurement, regardless of whether the method of sampling is periodic systematic, stratified or two-stage.

7.1 Method 1

See figure 3 and annex A.

The estimated values of precision at the 95 % probability level (hereinafter referred to simply as precision) of sampling, sample preparation and measurement shall be calculated as follows.

7.1.1 Denote the four measurements (such as % Fe), for 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}.

7.1.2 Calculate the mean, $\bar{x}_{ij.}$, and range, R_1, for each pair of duplicate measurements using equations (1) and (2) respectively.

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

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

where

i = 1 and 2 and stands for A and B;

j = 1 and 2 and stands for test samples.

7.1.3 Calculate the mean, $\bar{\bar{x}}_{i..}$, and range, R_2, for each pair of duplicate samples, using equations (3) and (4) respectively.

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

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

7.1.4 Calculate the mean, $\bar{\bar{\bar{x}}}$, and range, R_3, for each pair of gross samples, A and B, using equations (5) and (6) respectively.

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

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

7.1.5 Calculate the overall mean, $\bar{\bar{\bar{\bar{x}}}}$, and the means of ranges, $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$, using equations (7) to (10).

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

$$\bar{R}_1 = \frac{1}{4n}\sum R_1 \qquad \text{. . . (8)}$$

$$\bar{R}_2 = \frac{1}{2n}\sum R_2 \qquad \text{. . . (9)}$$

$$\bar{R}_3 = \frac{1}{n}\sum R_3 \qquad \text{. . . (10)}$$

where n is the number of lots.

Calculate the control limits for ranges as follows and construct the charts.

Upper control limit for R chart

$D_4\bar{R}_1$ (for R_1), $D_4\bar{R}_2$ (for R_2), $D_4\bar{R}_3$ (for R_3)

where D_4 = 3,267 (for a pair of measurements).

7.1.6 When all of the values of R_3, R_2 and R_1 are within the upper control limit of the R chart, it is an indication that the processes of sampling, sample preparation and measurement of samples are in a state of statistical control.

On the other hand, when several values of R_3, R_2 and R_1, fall outside the respective upper control limits, the process (such as sampling, sample preparation or measurement) under investigation is not in a state of statistical control and should be checked in order to detect assignable causes. Such values should be excluded and the means of ranges recalculated.

7.1.7 When $2n_1$ increments are taken, calculate the estimated values of the standard deviations of measurement, $\hat{\sigma}_M$, sample preparation, $\hat{\sigma}_P$, and sampling, $\hat{\sigma}_S$, using equations (11) to (13) respectively:

$$\hat{\sigma}_M^2 = \left(\bar{R}_1/d_2\right)^2 \qquad \text{. . . (11)}$$

$$\hat{\sigma}_P^2 = \left(\bar{R}_2/d_2\right)^2 - \frac{1}{2}\hat{\sigma}_M^2 \qquad \text{. . . (12)}$$

$$\hat{\sigma}_S^2 = \left(\bar{R}_3/d_2\right)^2 - \frac{1}{2}\hat{\sigma}_P^2 - \frac{1}{4}\hat{\sigma}_M^2 \qquad \text{. . . (13)}$$

where $1/d_2$ = 0,886 2 (for a pair of measurements).

NOTE 9 As an alternative to using ISO 3084, the quality variation, σ_W, can be determined from the standard deviation of sampling, σ_S, as follows:

$$\sigma_W = \sqrt{n_1}\,\sigma_S .$$

When n_1 increments are taken in accordance with 5.1.2, the estimated value of the standard deviation of sampling, $\hat{\sigma}_S$, from equation (13) shall be divided by $\sqrt{2}$ to obtain the standard deviation of sampling for gross samples comprising n_1 increments. The estimated values of the standard deviations of measurement and sample preparation may be calculated using equations (11) and (12).

7.1.8 Calculate the estimated values of the precisions of sampling $(2\hat{\sigma}_S)$ sample preparation $(2\hat{\sigma}_P)$ and measurement $(2\hat{\sigma}_M)$.

7.2 Method 2

See figure 4.

The estimated values of precision of sampling, sample preparation and measurement shall be calculated as follows.

7.2.1 Denote the four measurements as follows:

x_1, x_2 are the duplicate measurements of test sample A_1 prepared from gross sample A;

x_3 is the single measurement of test sample A_2 prepared from gross sample A;

x_4 is the single measurement of test sample B prepared from gross sample B.

7.2.2 Calculate the mean, $\bar{x}$, and range, R_1, for each pair of duplicate measurements using equations (14) and (15).

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \quad \ldots (14)$$

$$R_1 = |x_1 - x_2| \quad \ldots (15)$$

7.2.3 Calculate the mean, $\bar{\bar{x}}$, and range, R_2, using equations (16) and (17).

$$\bar{\bar{x}} = \frac{1}{2}(\bar{x} + x_3) \quad \ldots (16)$$

$$R_2 = |\bar{x} - x_3| \quad \ldots (17)$$

7.2.4 Calculate the mean, $\bar{\bar{\bar{x}}}$, and range, R_3, for each pair of gross samples, A and B, using equations (18) and (19).

$$\bar{\bar{\bar{x}}} = \frac{1}{2}(\bar{\bar{x}} + x_4) \quad \ldots (18)$$

$$R_3 = |\bar{\bar{x}} - x_4| \quad \ldots (19)$$

7.2.5 Calculate the overall mean, $\bar{\bar{\bar{\bar{x}}}}$, and the means of ranges, $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$ using equations (7), (20), (21) and (10) respectively.

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

$$\bar{R}_1 = \frac{1}{n}\sum R_1 \quad \ldots (20)$$

$$\bar{R}_2 = \frac{1}{n}\sum R_2 \quad \ldots (21)$$

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

where n is the number of lots.

Calculate the control limits for range as in 7.1.5.

7.2.6 When all the values of R_3, R_2 and R_1 are within the upper control limit of the R-chart, it is an indication that the processes of sampling, sample preparation and measurement of samples are in a state of statistical control.

On the other hand, when several values of R_3, R_2 and R_1 fall outside the respective upper control limits, the process (such as sampling, sample preparation, or measurement) under investigation is not in a state of statistical control and should be checked in order to detect assignable causes. Such values should be excluded and the means of ranges recalculated.

7.2.7 When $2n_1$ increments are taken, calculate the estimated values of the standard deviations of measurement, $\hat{\sigma}_M$, sample preparation, $\hat{\sigma}_P$, and sampling, $\hat{\sigma}_S$, using equations (11), (22) and (23) respectively.

$$\hat{\sigma}_M{}^2 = \left(\bar{R}_1/d_2\right)^2 \quad \ldots (11)$$

$$\hat{\sigma}_P{}^2 = \left(\bar{R}_2/d_2\right)^2 - \frac{3}{4}\hat{\sigma}_M{}^2 \quad \ldots (22)$$

$$\hat{\sigma}_S{}^2 = \left(\bar{R}_3/d_2\right)^2 - \frac{3}{4}\hat{\sigma}_P{}^2 - \frac{11}{16}\hat{\sigma}_M{}^2 \quad \ldots (23)$$

where $1/d_2 = 0{,}886\,2$ (for a pair of measurements). See note 9.

When n_1 increments are taken in accordance with 5.1.2, the estimated value of the standard deviation of sampling, $\hat{\sigma}_S$, from equation (23) shall be divided by $\sqrt{2}$ to obtain the standard deviation of sampling for gross samples comprising n_1 increments. The estimated values of the standard deviations of measurement and sample preparation may be calculated using equations (11) and (22).

7.2.8 Calculate the estimated values of the precisions of sampling $(2\hat{\sigma}_S)$, sample preparation $(2\hat{\sigma}_P)$ and measurement $(2\hat{\sigma}_M)$.

7.3 Method 3

See figure 5.

In this case the estimated values of precision of sampling, sample preparation and measurement cannot be separated. Method 3 provides the overall precision, $2\hat{\sigma}_{SPM}$, of sampling, sample preparation and measurement.

The relationship between these precision values is

$$\hat{\sigma}_{SPM}{}^2 = \hat{\sigma}_S{}^2 + \hat{\sigma}_P{}^2 + \hat{\sigma}_M{}^2 \quad \ldots (24)$$

The estimated value of overall precision shall be calculated in accordance with the following procedure.