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

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3085 was prepared by Technical Committee ISO/TC 102, *Iron ores.*

This second edition cancels and replaces the first edition (ISO 3085-1975), of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Iron ores — Experimental methods for checking the precision of sampling

1 Scope and field of application

This International Standard specifies experimental methods to be applied 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 — 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 specified in ISO 3082 or ISO 3083.

2 References

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

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method.* [1)]

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

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

ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling.* [1)]

ISO 4701, *Iron ores — Determination of size distribution by sieving.*

3 General conditions

3.1 Number of consignments for experiment

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

3.2 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 3081 or ISO 3082. Namely, if the number of increments required for the routine sampling is n_1 and one gross sample is made up of the minimum number of increments, the minimum number of increments required for the experiment shall be $2n_1$ and two gross samples shall be made up.

NOTE — If this is impracticable, the number of increments n_1 may be taken and divided into two parts, each comprising $n_1/2$.

3.3 Sample preparation and testing

The preparation and testing of the sample shall be carried out in accordance with the methods specified in the relevant International Standards.

NOTE — In the case of chemical analysis, such as the determination of the total iron content, it is preferable to carry out a series of determinations on test samples of a consignment on different days.

3.4 Replication of experiment

Even when a series of experiments has been conducted prior to regular sampling operations, the experiments should be carried out occasionally in order to check a possible quality variation in the consignments, and at the same time, to control the methods of sampling, sample division and testing.

Because of the large amount of work involved in this method, it should be carried out as part of routine work of sampling and testing.

4 Method of experiment

4.1 Sampling procedure

The sampling procedure to be followed shall be selected from the three categories of sampling, i.e. periodic systematic

1) At present at the stage of draft.

sampling, stratified sampling and two-stage sampling, depending on the method of taking increments from the consignment in accordance with the relevant clauses of ISO 3081 or ISO 3082.

4.1.1 Periodic systematic sampling

4.1.1.1 The number of increments, n_1, shall be selected from table 4 of ISO 3081 or ISO 3082, depending on the mass of the consignment and the classification category of the iron ore, i.e. "large", "medium", or "small" quality variation.

4.1.1.2 The sampling interval, Δm, in tonnes, shall be calculated by dividing the tonnage, m_1, of the consignment by $2n_1$, i.e. giving intervals equal to one-half of the sampling interval of the routine sampling. The sampling interval thus calculated shall be rounded down to the nearest 10 t.

4.1.1.3 The increments shall be taken at a regular sampling interval (see 4.1.1.2), with a random start from the consignment.

4.1.1.4 The increments shall be placed alternately in two containers, A and B. Thus, two gross samples, A and B, will be made up, each composed of n_1 increments.

Example 1

Suppose that a consignment of 19 000 t of discharged iron ore is transferred by belt conveyors and that the classification category of the ore is "medium" quality variation: the minimum required number of increments, n_1, is 60, as shown in table 4 of ISO 3081 or ISO 3082.

Then the sampling interval, Δm, in tonnes, for taking increments is given by the equation

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

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

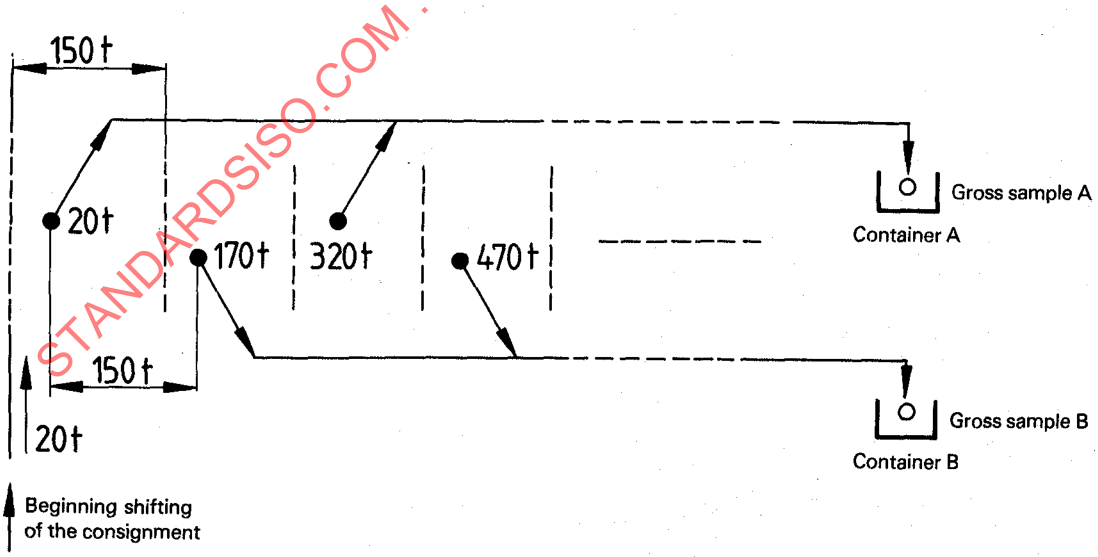
The increments are placed alternately in containers A and B, and two gross samples, A and B, are made up, each composed of 63 increments (see figure 1).

4.1.2 Stratified sampling

4.1.2.1 In the case where the number of wagons or containers,[1] i.e. the number of strata, n_4, forming one consignment, is smaller than the number of increments, n_1, given in table 4 of ISO 3081, the number of increments, n_3, to be taken from each wagon (stratum) shall be obtained by the equation given in 8.2.2 of ISO 3081.

4.1.2.2 $2n_3$ increments shall be taken from each wagon.

4.1.2.3 The $2n_3$ increments taken from each wagon shall be separated at random into two subsamples, each of n_3 increments.



Legend: Dot indicates increment and circle indicates gross sample

Figure 1 — Schematic diagram for example 1

1) Hereinafter referred to simply as "wagons".

4.1.2.4 Each of the two subsamples of all the wagons shall be combined to make up two gross samples, A and B, respectively, each comprising n_1 (= $n_3 n_4$) increments.

NOTE — If the tonnage varies wagon by wagon, the number of increments to be taken from each wagon shall be decided in proportion to the tonnage. This method is called "proportional stratified sampling", for which the procedure is illustrated in example 3.

Example 2

Suppose that a consignment of iron ore is delivered in 11 wagons of capacity 60 t and that the quality variation of the ore within wagons, σ_w, is "medium"; the minimum required number of increments, n_1, for the 660 t consignment is 20, as shown in table 4 of ISO 3081.

Then, the number of increments to be taken from each wagon is given by the equation

$$n_3 = \frac{n_1}{n_4} = \frac{20}{11} \approx 2$$

Four ($2n_3 = 2 \times 2$) increments are taken from each wagon.

The four increments are separated at random into two subsamples, each consisting of two increments.

Each of the two subsamples from the 11 wagons is combined to compose two gross samples, A and B respectively, each comprising 22 ($2n_4 = 2 \times 11$) increments (see figure 2).

Example 3

Suppose that a wagon-borne consignment consists of six wagons of capacity 60 t and eight wagons of capacity 30 t, i.e. $m_1 = (6 \times 60) + (8 \times 30) = 600$ t of iron ore, the classification category of which is "large" quality variation in terms of standard deviation within wagons, σ_w; then the minimum number of increments, n_1, is 40, as shown in table 4 of ISO 3081.

Then the numbers of increments to be collected from the six wagons of capacity 60 t and the eight wagons of capacity 30 t are respectively

$$\frac{n_1 \times 6 \times 60}{m_1} = \frac{40 \times 360}{600} = 24$$

$$\frac{n_1 \times 8 \times 30}{m_1} = \frac{40 \times 240}{600} = 16$$

The numbers of increments to be taken from each wagon of capacity 60 t and from each wagon of capacity 30 t are respectively

$$\frac{24}{6} = 4$$

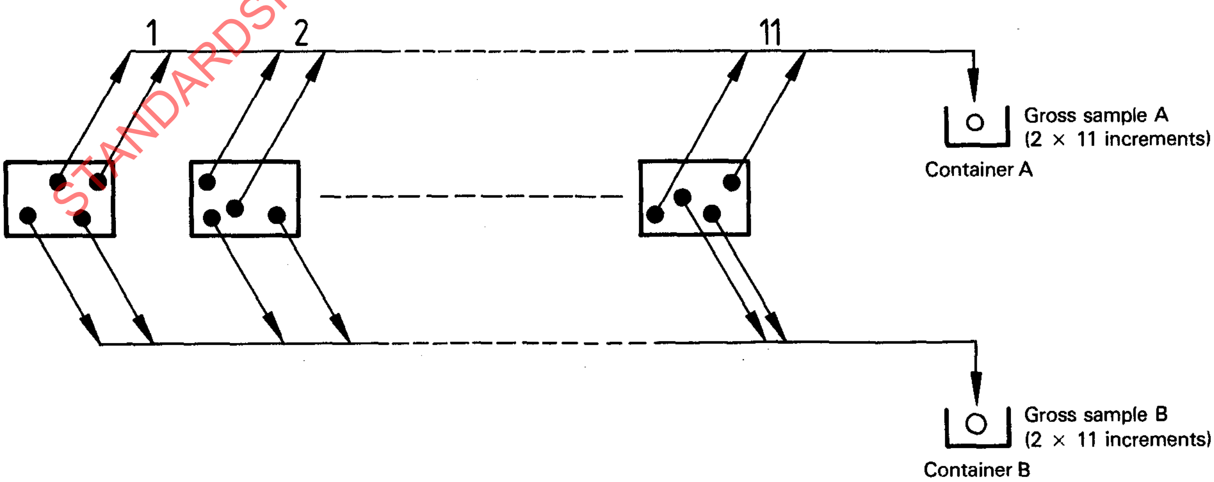
$$\frac{16}{8} = 2$$

For this experiment, eight (= 2×4) increments are taken from each wagon of capacity 60 t, and four (= 2×2) increments from each wagon of capacity 30 t. The increments taken in this way are separated at random into two subsamples.

The two subsamples thus obtained from all of the wagons are combined separately to make up two gross samples, A and B respectively, each comprising 40 increments.

4.1.3 Two-stage sampling

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



Legend: Boxes, dots and circles indicate respectively wagons, increments taken from a wagon, and gross samples.

Figure 2 — Schematic diagram for example 2

4.1.3.2 An additional n_2 wagons shall be selected at random from the the same consignment independently.

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

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

4.1.3.4 All of the increments taken from the wagons selected in accordance with 4.1.3.1 shall be combined to make up gross sample A.

All of the increments taken from the wagons selected in accordance with 4.1.3.2 shall be combined to make up another gross sample B.

Example 4

Suppose that a wagon-borne consignment consists of 80 wagons of capacity 60 t, i.e. $m_1 = 80 \times 60 = 4\,800$ t "medium" quality variation in terms of standard deviation within wagons, σ_w, and "small" quality variation in terms of standard deviation between wagons, σ_b; then the number of wagons to be selected, n_2, is 15, as shown in table 5 of ISO 3081.

From the same consignment, an additional 15 wagons are selected independently of those previously selected.

The number of increments to be taken at random from each of the first 15 wagons selected, n_3, is four, and the total 60 ($n_2 n_3 = 4 \times 15$) increments are combined to make up gross sample A.

An additional four increments are taken at random from each of the second 15 wagons selected, and the total 60 increments are combined to make up gross sample B.

4.2 Sample division and testing

The two gross samples A and B taken in accordance with 4.1 shall be divided separately and subjected to testing by either type 1, type 2 or type 3 as described in 4.2.1, 4.2.2 or 4.2.3.

4.2.1 Division-testing type 1 (see figure 3)

4.2.1.1 The two gross samples A and B shall be divided separately to prepare two test samples.

4.2.1.2 The four test samples, A_1, A_2, and B_1, B_2, shall be tested in duplicate respectively. A total of eight tests shall be run in random order.

NOTE — Type 1 allows the precisions of sampling, division and measurement to be obtained separately.

4.2.2 Division-testing type 2 (see figure 4)

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

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

NOTE — Type 2 also allows the precisions of sampling, division and measurement to be obtained separately. However, the estimates of precisions of division and measurement are inferior to those obtained by type 1.

4.2.3 Division-testing type 3 (see figure 5)

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

4.2.3.2 The two test samples A and B shall be tested individually.

NOTE — By type 3, only the overall precision of sampling, division and measurement is obtained.

5 Analysis of experimental data

The method for analysis of experimental data shall be as specified in this clause depending on the type of division-testing selected, regardless of whether the method of sampling be periodic systematic, stratified, or two stage.

5.1 Division-testing type 1 (see figure 3 and table 2)

The estimated values of approximately 95 % probability precision (hereinafter referred to as simply as precision) of sampling, division and measurement shall be calculated in accordance with the procedure given in 5.1.1 to 5.1.7.

5.1.1 Denote the pair of four measurements (such as % Fe) 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}.

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

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

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

where

i = 1 and 2 stands for A and B;

j = 1 and 2 stands for test samples.

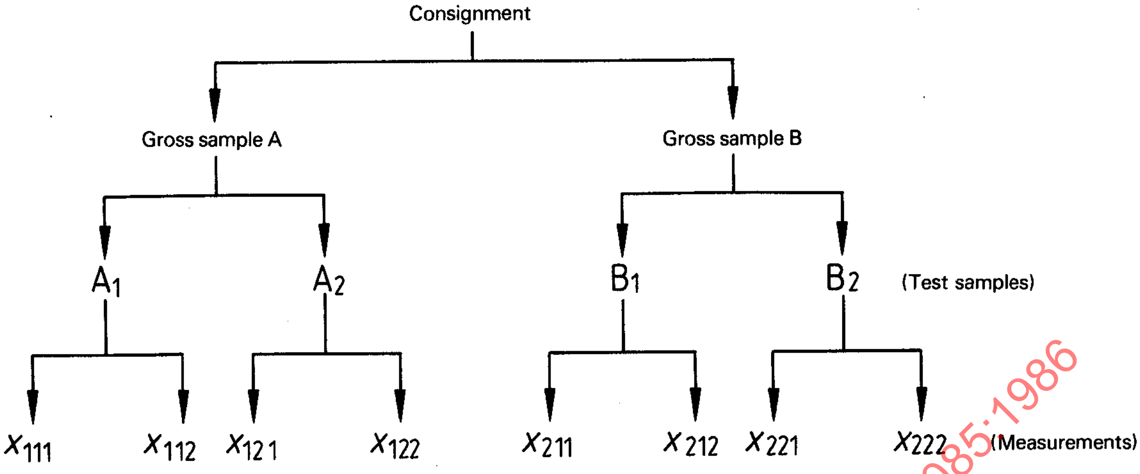


Figure 3 — Flowsheet for division-testing type 1

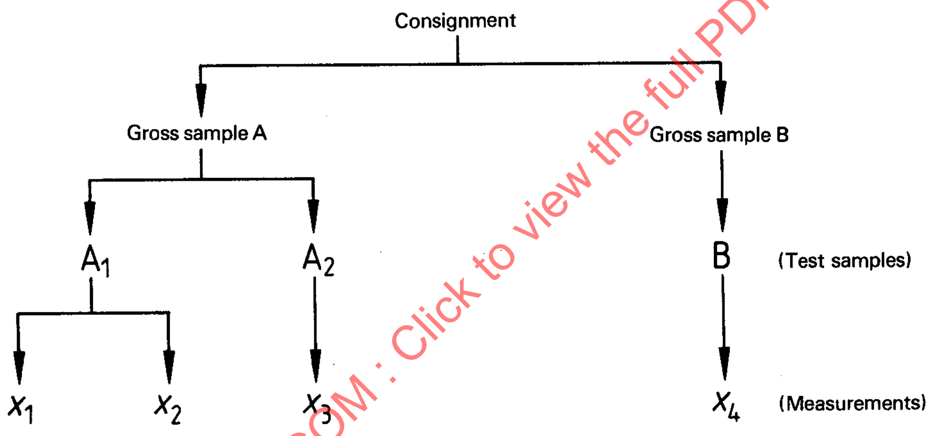


Figure 4 — Flowsheet for division-testing type 2

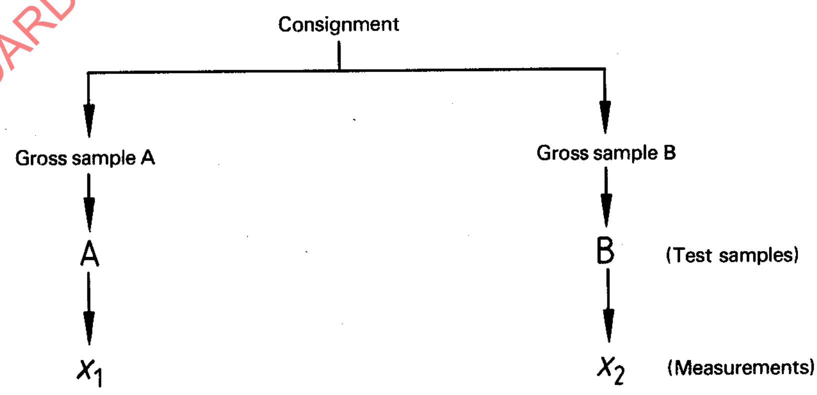


Figure 5 — Flowsheet for division-testing type 3

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

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

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

5.1.4 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.

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

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

5.1.5[1] 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).

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

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

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

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

where n is the number of consignments.

For the preparation of control charts for means and ranges, calculate the control limits using the formulae (11) and (12).

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

$$\bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_1 \qquad \bar{\bar{\bar{\bar{x}}}} \pm A_2\bar{R}_2 \qquad \bar{\bar{\bar{\bar{x}}}} \pm A_3\bar{R}_3 \quad \ldots (11)$$

Upper control limit for R-chart

$$D_4\bar{R}_1 \text{ (for } R_1) \qquad D_4\bar{R}_2 \text{ (for } R_2) \qquad D_4\bar{R}_3 \text{ (for } R_3) \quad \ldots (12)$$

where A_2 = 1,880 and D_4 = 3,267 (for a pair of measurements).

5.1.6 Estimate the values of standard deviation of measurement, $\hat{\sigma}_M$, division, $\hat{\sigma}_D$, and sampling, $\hat{\sigma}_S$, using equations (13) to (15) respectively.

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

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

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

where $1/d_2$ = 0,886 5 (for a pair of measurements).

NOTE — When n_1 increments are taken and divided into two parts in accordance with the note to 3.2, the value of $\hat{\sigma}_S^2$ in equation (15) shall be divided by two to compare with the specified precision, β_S.

The comparison described in 5.1.7 will be made using the value thus obtained.

5.1.7 Calculate the estimated values of precision of measurement, $2\hat{\sigma}_M$, division, $2\hat{\sigma}_D$, and sampling, $2\hat{\sigma}_S$.

Compare the value of $2\hat{\sigma}_S$ thus obtained with the specified precision of sampling, β_S, as given in table 4 of ISO 3081 or ISO 3082.

NOTES

1 See the note to 5.1.6.

2 It is recommended that the values of σ_M and σ_D obtained by this method be compared with the values obtained by another method.

This procedure may also be applied to evaluate the precision of the routine method.

3 The precision of sampling is defined as follows:

Stratified sampling

$$\beta_S = 2\sigma_S = 2\sigma_w/\sqrt{n}$$

Two-stage sampling

$$\beta_S = 2\sigma_S = 2\sqrt{\left(\frac{n_4 - n_2}{n_2 - 1}\right)\frac{\sigma_b^2}{n_2} + \frac{\sigma_w^2}{n_2 n_3}}$$

where n_3 = 4.

5.2 Division-testing type 2 (see figure 4)

The estimated value of precision shall be calculated in accordance with the procedure given in 5.2.1 to 5.2.7.

5.2.1 Denote the four measurements as follows:

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

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

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

5.2.2 Calculate the mean, $\bar{x}$, and range, R_1, for each pair of duplicate measurements using equations (16) and (17).

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

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

1) Sources:
[1] Theoretical background: PEARSON E.S. *The Application of Statistical Methods to Industrial Standardization and Quality Control.* London, British Standards Institution, 1935.
[2] Numerical values: *ASTM Manual on Quality Control of Materials.* Philadelphia, American Society for Testing and Materials, 1951.

5.2.3 Calculate the mean, $\bar{\bar{x}}$, and range, R_2, for each selected pair of measurements, x_1 and x_3, or x_2 and x_3 using equations (18) and (19).

$$\bar{\bar{x}} = \begin{cases} \frac{1}{2}(x_1 + x_3) \\ \text{or} \\ \frac{1}{2}(x_2 + x_3) \end{cases} \quad \text{selected at random} \qquad \ldots (18)$$

$$R_2 = \begin{cases} |x_1 - x_3| \\ \text{or} \\ |x_2 - x_3| \end{cases} \quad \text{selected at random} \qquad \ldots (19)$$

5.2.4 Calculate the mean, $\bar{\bar{x}}$, and range, R_3, for each pair of gross samples, A and B, using equations (20) and (21).

$$\bar{\bar{x}} = \begin{cases} \frac{1}{2}(x_1 + x_4) \\ \text{or} \\ \frac{1}{2}(x_2 + x_4) \\ \text{or} \\ \frac{1}{2}(x_3 + x_4) \end{cases} \quad \text{selected at random} \qquad \ldots (20)$$

$$R_3 = \begin{cases} |x_1 - x_4| \\ \text{or} \\ |x_2 - x_4| \\ \text{or} \\ |x_3 - x_4| \end{cases} \quad \text{selected at random} \qquad \ldots (21)$$

5.2.5 Calculate the overall mean, $\bar{\bar{\bar{x}}}$, and the means of ranges, $\bar{R}_1$, $\bar{R}_2$ and $\bar{R}_3$ using equations (22) to (25) respectively.

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

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

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

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

where n is the number of consignments.

Calculate the control limits to construct the control charts for mean and range using formulae (26) and (27).

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

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

Upper control limits for R-chart

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

where A_2 = 1,880 and D_4 = 3,267 (for a pair of measurements).

5.2.6 Calculate the estimated values of standard deviation of measurement, $\hat{\sigma}_M$, division, $\hat{\sigma}_D$, and sampling, $\hat{\sigma}_S$, using equations (28) to (30) respectively.

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

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

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

where $1/d_2$ = 0,886 5 (for a pair of measurements).

NOTE — See the note to 5.1.6.

5.2.7 Calculate the estimated values of precision of measurements, $2\hat{\sigma}_M$, division, $2\hat{\sigma}_D$, and sampling, $2\hat{\sigma}_S$, respectively.

Compare the value of $2\hat{\sigma}_S$ thus obtained with the specified precision of sampling, β_S, given in table 4 of ISO 3081 or ISO 3082.

5.3 Division-testing type 3 (see figure 5)

In this case the estimated values of precision of sampling, division and measurement are not obtainable separately. What is derived from type 3 is the overall precision, $2\hat{\sigma}_{SDM}$, of these three precisions.

The relationship between these precisions is

$$\hat{\sigma}_{SDM}^2 = \hat{\sigma}_S^2 + \hat{\sigma}_D^2 + \hat{\sigma}_M^2 \qquad \ldots (31)$$

The estimated value of overall precision shall be calculated in accordance with the procedure given in 5.3.1 to 5.3.5.

5.3.1 Calculate the mean, $\bar{x}$, and range, R, for each pair of measurements using equations (32) and (33).

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

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

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

5.3.2 Calculate the overall mean, $\bar{\bar{x}}$, and the mean of range, $\bar{R}$, using equations (34) and (35).

$$\bar{\bar{x}} = \frac{1}{n}\sum\bar{x} \qquad \ldots (34)$$

$$\bar{R} = \frac{1}{n}\sum R \qquad \ldots (35)$$

where n is the number of consignments.

5.3.3 Calculate the control limits to construct control charts for mean and range using formulae (36) and (37).

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

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

Upper control limit for R-chart

$$D_4\bar{R} \qquad \ldots (37)$$

where A_2 = 1,880 and D_4 = 3,267 (for a pair of measurements).

5.3.4 Calculate the estimated value of overall standard deviation, $\hat{\sigma}_{SDM}$, using equation (38).

$$\hat{\sigma}^2_{SDM} = (\bar{R}/d_2)^2 \qquad \ldots (38)$$

where $1/d_2$ = 0,886 5 (for a pair of measurements).

5.3.5 Calculate the estimated value of overall precision, $2\hat{\sigma}_{SDM}$.

6 Interpretation of results and action

6.1 Interpretation

When all of the values of R_3, R_2, and R_1 calculated in accordance with 5.1 and 5.2 are within the upper control limit of the R-chart constructed in accordance with 5.1.5 and 5.2.5, it is an indication that the routine processes of sampling, division and measurement of samples are in a state of control.

When all of the values of R calculated in accordance with 5.3 are within the upper control limit of the R-chart constructed in accordance with 5.3.3, it is an indication that the overall process of sampling, division and measurement is in a state of control.

On the other hand, when several values of R_3, R_2, R_1, from 5.1 and 5.2, and R, from 5.3, fall out of the respective upper control limits, the process (such as sampling, division, or measurement) under investigation is not in a state of control and should be checked in order to detect assignable causes. If there are any values which have assignable causes, the means of ranges should be calculated not including those values.

It is recommended that the control limits obtained by the above experiment should be used for the interpretation of subsequent experimental results.

6.2 Action

When there is an indication that the precision does not attain the specified value in table 4 of ISO 3081 or ISO 3082, the sampling procedure shall be modified according to 6.2.1 to 6.2.3.

6.2.1 Check the changes in quality variations of the iron ore in accordance with the method given in ISO 3084. When it is confirmed that there is a significant change in quality variation of the iron ore in question, the following actions may be taken.

6.2.1.1 Periodic systematic or stratified sampling

Change the number of increments to be taken from a consignment, n_1, by the revised category of quality variation in accordance with table 3 of ISO 3081 or ISO 3082.

6.2.1.2 Two-stage sampling

Change the number of wagons to be selected from a consignment, n_2, in accordance with table 5 of ISO 3081.

6.2.2 In the case of periodic systematic or stratified sampling, a greater number, n'_1, of increments may be collected from a consignment. The contribution of this action to improvement of the precision of sampling is in proportion to $\sqrt{n_1/n'_1}$.

6.2.3 Increase the mass of increment. However, the increase in the mass more than that required will not effect a significant improvement of the precision of sampling.

7 Example of experiment

This example of experiment is based on periodic systematic sampling by division-testing type 1, and conducted by a consumer of iron ores. The experimental results are summarized in tables 1 and 2, and figure 6.

Table 1 shows particulars of the experiment and analysis results of iron determinations. Table 2 shows the records of % Fe and the process of calculation of $\hat{\sigma}_M$, $\hat{\sigma}_D$ and $\hat{\sigma}_S$.

Figure 6 shows the control charts for mean and 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 used in this example.

The numbers of cases where points of data are situated outside the three sigma control limits are recorded in the bottom space of table 2, and the corresponding data are identified by asterisks (see 6.1).

The values of the estimated standard deviation of analysis, division and sampling of this example are

standard deviation of analysis:	$\hat{\sigma}_M$ = 0,077 (% Fe)
standard deviation of division:	$\hat{\sigma}_D$ = 0,11 (% Fe)
standard deviation of sampling:	$\hat{\sigma}_S$ = 0,23 (% Fe)

Of the three, $\hat{\sigma}_S$ is the greatest, and the estimated precision of sampling is $2\hat{\sigma}_S = 2 \times 0,23 = 0,46$ % Fe. This value satisfies the precision of sampling, β_S, shown in table 4 of ISO 3081 or ISO 3082.

Table 1 — Example of recording particulars of experiment

(Name of the Company and Works)

Report on checking the precision of sampling

Period of experiment: ....

Site of experiment: (Location identification)..

Characteristic measured: Iron content (% Fe)

Consignments investigated

Source and type of ore: ....

Loading point: ....

Transportation medium: Ship

Number of consignments: 20

Mass of consignments: Mean 9 920 t; Minimum 7 000 t; Maximum 13 000 t

Particulars of sampling

Maximum particle size of consignments: 110 mm
Type of increment: Unit mass of ore on belt conveyor; for its full cross-section over a certain length of the flow
Nominal mass of increment: 25 kg
Number of increments from a consignment: 2 × 50 = 100
Method of taking increment: Stop belt conveyor at specified tonnage interval of ore discharge, and collect all ore with a shovel on the belt at specified location to obtain a 25 kg increment.

Preparation of samples

Method of making up gross samples: Place alternately individual increments taken successively in containers A and B, and make up 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)

Measurement of % Fe

Statistic	Experimental results	Commercial determination	Manifested at loading point
Mean	61,10	—	—
Minimum	59,90	—	—
Maximum	63,02	—	—

Estimated precision of sampling (% Fe)

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

$\hat{\sigma}_D = 0,11$

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

$\beta_S = 2\hat{\sigma}_S = 0,46$

$\hat{\sigma}_{SDM} = 0,27$

$\beta_{SDM} = 2\hat{\sigma}_{SDM} = 0,54$

Comments and remarks..

....

....

Date:............................ Reported by:..
(Name of supervisor of experiment)

Table 2 — Example of data sheet for checking the precision

(Refer to 5.1 and figure 3)

Source and type of ore: ....

Characteristic measured: iron content   Period of experiment ....   Number of consignments: 20

Date: ....

No. ....

Consignment No.	Date of sampling	Size of consignment t	Number of increments		A_1				A_2				A		B_1				B_2				B			
			A	B	x_{111}	x_{112}	$\bar{x}_{11.}$	R_1	x_{121}	x_{122}	$\bar{x}_{12.}$	R_1	$\bar{\bar{x}}_{1..}$	R_2	x_{211}	x_{212}	$\bar{x}_{21.}$	R_1	x_{221}	x_{222}	$\bar{x}_{22.}$	R_1	$\bar{\bar{x}}_{2..}$	R_2	$\bar{\bar{\bar{x}}}$	R_3
1.		12 100	50	50	60,92	60,99	60,96	0,07	60,98	61,01	61,00	0,03	60,98	0,04	61,40	61,34	61,37*	0,06	61,28	61,35	61,32*	0,07	61,34	0,05	61,16	0,36
2.		7 300	50	50	60,88	60,87	60,88*	0,01	61,02	61,02	61,02	0,00	60,95	0,14	60,27	60,10	60,18*	0,17	60,04	59,93	59,98*	0,11	60,08*	0,20	60,52*	0,87
3.		10 700	50	50	60,82	60,76	60,79*	0,06	60,96	60,88	60,92*	0,08	60,86	0,13	60,70	60,67	60,68*	0,03	60,82	60,60	60,71*	0,22	60,70*	0,03	60,78	0,16
4.		13 000	50	50	61,40	61,30	61,35*	0,10	61,40	61,25	61,32*	0,15	61,34	0,03	61,94	61,97	61,96*	0,03	61,60	61,43	61,52*	0,17	61,74*	0,44	61,54	0,40
5.		11 500	50	50	62,04	62,00	62,02*	0,04	62,27	62,44	62,36*	0,17	62,19*	0,34	61,92	61,77	61,84*	0,15	62,51	62,52	62,52*	0,01	62,18*	0,68*	62,18*	0,01
6.		10 000	50	50	62,70	62,92	62,81*	0,22	62,90	62,72	62,81*	0,18	62,81*	0,00	63,02	62,94	62,98*	0,08	62,98	62,92	62,95*	0,06	62,96*	0,03	62,88*	0,15
7.		11 200	50	50	60,94	60,98	60,96	0,04	60,80	60,85	60,82*	0,05	60,89	0,14	61,14	61,20	61,17	0,06	60,94	61,03	60,98	0,09	61,08	0,19	60,98	0,19
8.		9 700	50	50	60,90	60,87	60,88*	0,03	61,02	61,00	61,01	0,02	60,94	0,13	60,90	60,88	60,89*	0,02	60,70	60,50	60,60*	0,20	60,74	0,29	60,84	0,20
9.		8 600	50	50	61,20	61,00	61,10	0,20	61,08	61,08	61,08	0,00	61,09	0,02	60,88	60,64	60,76*	0,24	60,60	60,55	60,58*	0,05	60,67*	0,18	60,88	0,42
10.		9 300	50	50	60,94	61,07	61,00	0,13	61,00	61,00	61,00	0,00	61,00	0,00	61,00	61,00	61,00	0,00	59,95	59,87	59,91*	0,08	60,46*	1,09*	60,73	0,54
11.		8 300	50	50	59,94	59,90	59,92*	0,04	60,02	60,09	60,06*	0,07	59,99*	0,14	59,96	60,02	59,99*	0,06	59,98	59,90	59,94*	0,08	59,96*	0,05	59,98*	0,03
12.		10 500	50	50	60,08	60,04	60,06*	0,04	60,14	60,26	60,20*	0,12	60,13*	0,14	60,52	60,60	60,56*	0,08	60,46	60,35	60,40*	0,11	60,48*	0,16	60,30*	0,35
13.		8 200	50	50	60,38	60,23	60,30*	0,15	60,30	60,30	60,30*	0,00	60,30*	0,00	60,28	60,18	60,23*	0,10	60,29	60,32	60,30*	0,03	60,26*	0,07	60,28*	0,04
14.		10 600	50	50	61,10	61,00	61,05	0,10	61,00	61,02	61,01	0,02	61,03	0,04	60,84	60,66	60,75*	0,18	61,12	60,96	61,04	0,16	60,90	0,29	60,96	0,13
15.		9 100	50	50	62,00	61,93	61,96*	0,07	62,32	62,27	62,30*	0,05	62,13*	0,34	61,80	61,74	61,77*	0,06	61,74	61,71	61,72*	0,03	61,74*	0,05	61,94*	0,39
16.		10 400	50	50	60,72	60,78	60,75*	0,06	61,14	61,14	61,14	0,00	60,94	0,39	60,82	60,74	60,78*	0,06	60,56	60,38	60,47*	0,18	60,62*	0,31	60,78	0,32
17.		7 900	50	50	61,50	61,42	61,46*	0,08	62,02	62,07	62,04*	0,05	61,75*	0,58	61,06	61,04	61,05	0,02	61,16	61,25	61,20	0,09	61,12	0,15	61,44	0,63
18.		11 200	50	50	61,08	60,94	61,01	0,14	61,04	60,96	61,00	0,08	61,00	0,01	60,78	60,80	60,79*	0,02	60,88	60,89	60,88*	0,01	60,84	0,09	60,92	0,16
19.		11 800	50	50	61,15	61,30	61,22	0,15	61,10	61,08	61,09	0,02	61,16	0,13	62,00	62,05	62,02*	0,05	61,21	61,12	61,16	0,09	61,59*	0,86*	61,38	0,43
20.		7 000	50	50	61,54	61,32	61,43*	0,22	61,50	61,26	61,38*	0,24	61,40	0,05	61,86	61,60	61,73*	0,26	61,66	61,58	61,62*	0,08	61,68*	0,11	61,54	0,28
Sum		198 400	1 000	1 000	1 222,23	1 221,62	1 221,91	1,95	1 224,01	1 223,70	1 223,86	1,33	1 222,88	2,79	1 223,09	1 221,94	1 222,50	1,75	1 220,48	1 219,16	1 219,80	1,92	1 221,14	5,32	1 222,01	6,06
Mean		9 920	50	50	61,11	61,08	61,10	0,10	61,20	61,18	61,19	0,07	61,14	0,14	61,15	61,10	61,12	0,08	61,02	60,96	60,99	0,10	61,06	0,26	61,10	0,30

Calculation

$\hat{\sigma}_M^2 = (0{,}886\,5\,R_1)^2 = 0{,}005\,9$ $(0{,}886\,5\,\bar{R}_2)^2 = 0{,}032\,3$ $(0{,}886\,5\,\bar{R}_3)^2 = 0{,}072\,1$ $\bar{\bar{\bar{x}}} = 61{,}10$ $\bar{R}_1 = 0{,}087$ $3{,}267\,\bar{R}_1 = 0{,}284$

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

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

$\bar{\bar{\bar{x}}} \pm 1{,}880\,\bar{R}_1 = 61{,}10 \pm 0{,}164$ (61,26 and 60,94) $\bar{\bar{\bar{x}}} \pm 1{,}880\,\bar{R}_2 = 61{,}10 \pm 0{,}382$ (61,48 and 60,72) $\bar{\bar{\bar{x}}} \pm 1{,}880\,\bar{R}_3 = 61{,}10 \pm 0{,}570$ (61,67 and 60,53)

Comments and remarks

Recorded by ....

Checked by ....

(Name of supervisor of experiment)

Adjustment for calculated values

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

Number of cases where % Fe 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)

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

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

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

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