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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

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

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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 $i = 1, 2, \dots, 2n_1$ (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).

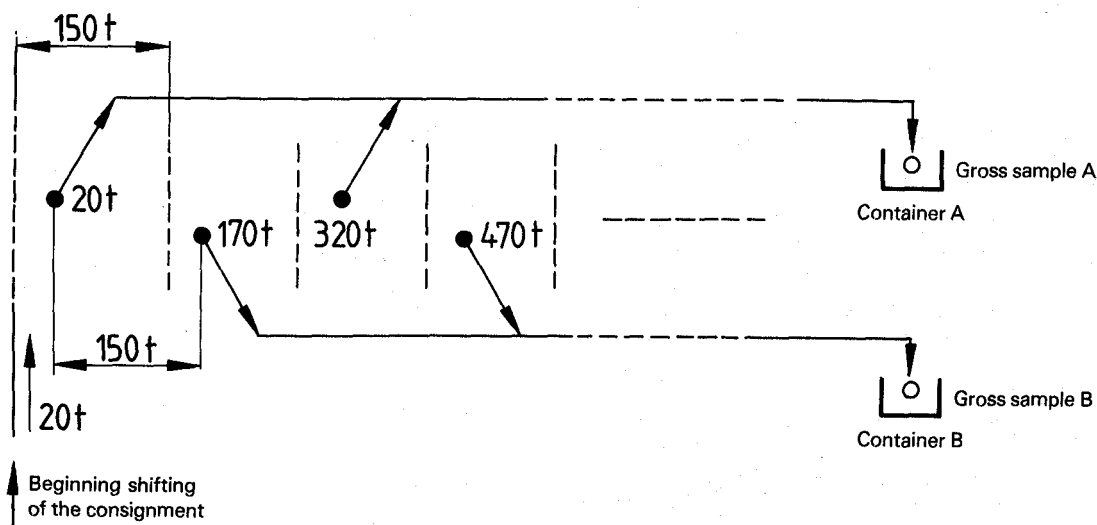
4.1.2 Stratified sampling

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.

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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 ($= n_3n_4$) 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

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

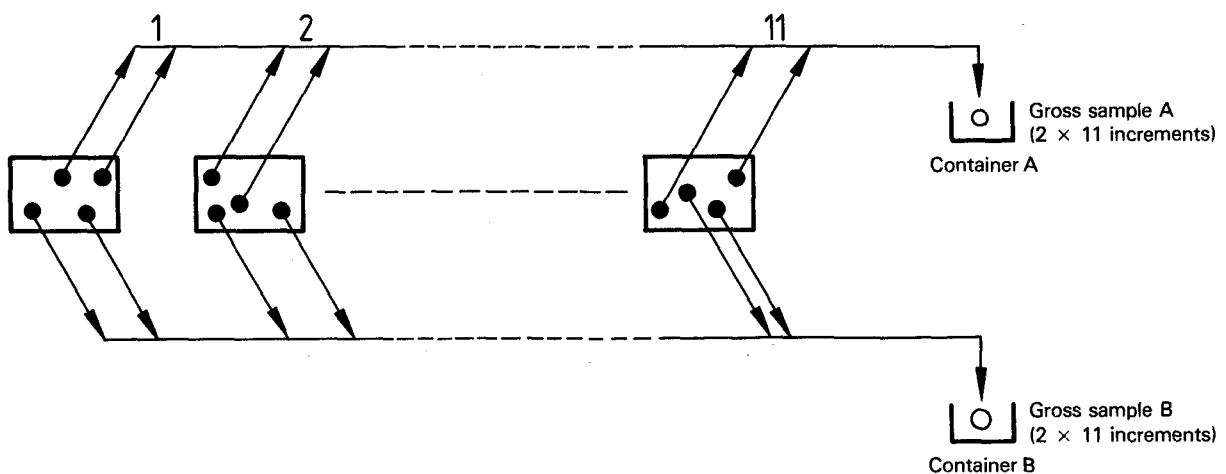
$$\frac{16}{8} = 2$$

For this experiment, eight ($= 2 \times 4$) increments are taken from each wagon of capacity 60 t, and four ($= 2 \times 2$) 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 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.

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

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

where

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

$j = 1$ and 2 stands for test samples.

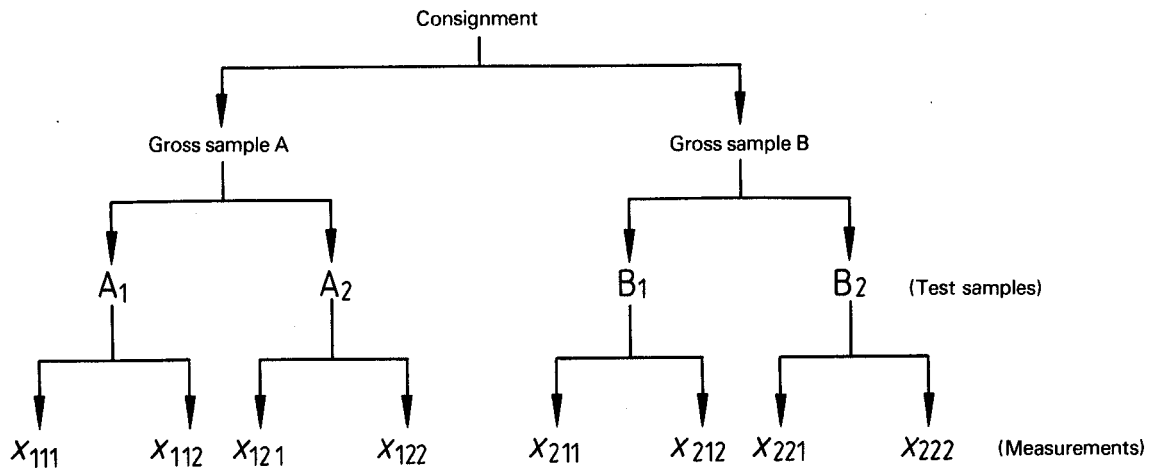


Figure 3 — Flowsheet for division-testing type 1

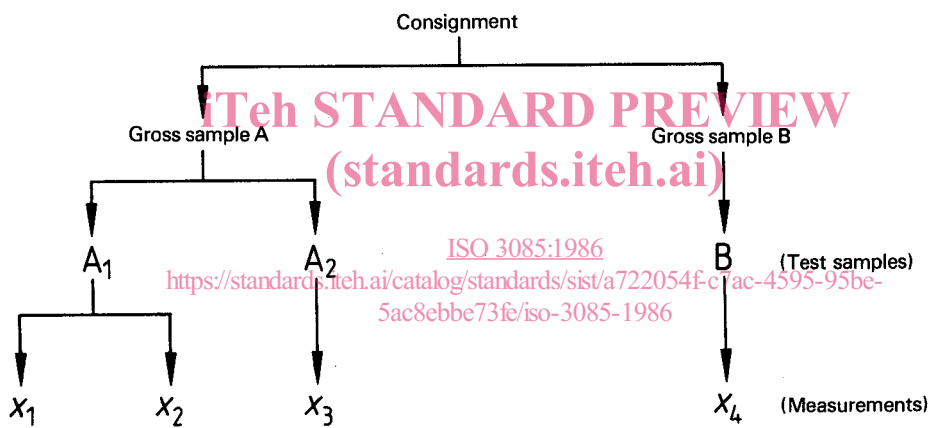


Figure 4 — Flowsheet for division-testing type 2

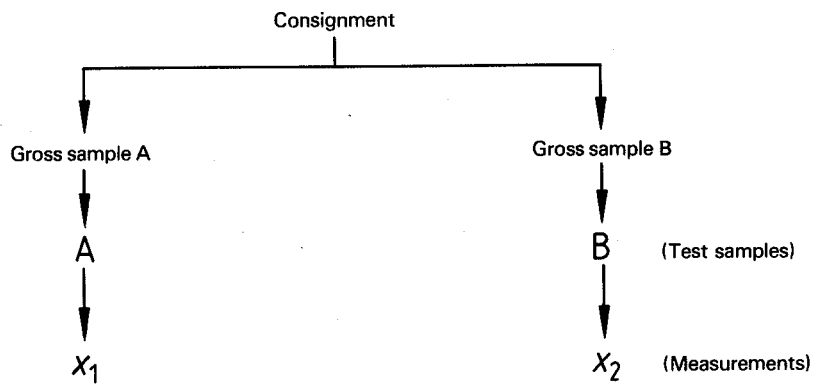


Figure 5 — Flowsheet for division-testing type 3

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

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

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

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

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

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

5.1.5¹⁾ 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 (7) to (10).

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

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

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

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

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

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

Upper control limit for R -chart

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

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.

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

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

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

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).

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

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

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{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).

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

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

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

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

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

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

$$\bar{\bar{x}} = \frac{1}{n} \sum \bar{x} \quad \dots (22)$$

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

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

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

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

$$\bar{\bar{x}} \pm A_2 \bar{R}_1 \quad \bar{\bar{x}} \pm A_2 \bar{R}_2 \quad \bar{\bar{x}} \pm A_2 \bar{R}_3 \quad \dots (26)$$

Upper control limits for R -chart

$$D_4 \bar{R}_1 \quad D_4 \bar{R}_2 \quad D_4 \bar{R}_3 \quad \dots (27)$$

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.

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

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

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

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.

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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

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

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).

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

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

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).

$$\bar{\bar{x}} = \frac{1}{n} \sum \bar{x} \quad \dots (34)$$

$$\bar{R} = \frac{1}{n} \sum R \quad \dots (35)$$

where n is the number of consignments.