INTERNATIONAL STANDARD ISO 3085

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Iron ores — Experimental methods for checking the precision of sampling

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

First edition — 1975-11-01

UDC 553.31.001.4

Ref. No. ISO 3085-1975 (E)

Descriptors : iron ores, sampling, tests, chemical analysis.

ISO 3085-1975 (E)

Price based on 13 pages

FOREWORD

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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.

International Standard ISO 3085 was drawn up by Technical Committee ISO/TC 102, *Iron ores*, and circulated to the Member Bodies in May 1974.

It has been approved by the Member Bodies of the following countries :

Australia	Iran	South Africa, Rep. of
Austria	Italy	Sweden
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Egypt, Arab Rep. of	Poland	U.S.S.R.
France	Portugal	Yugoslavia
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No Member Body expressed disapproval of the document.

Printed in Switzerland

Iron ores — Experimental methods for checking the precision of sampling

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the experimental methods to be applied for checking the precision of sampling of iron ores being carried out in accordance with the methods prescribed 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 prescribed in ISO 3083.

2 REFERENCES

This document should be read in conjunction with the following International Standards :

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

ISO 3082, *Iron ores — Increment sampling — Mechanical method.*[1]

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

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

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

ISO 3087, *Iron ores — Determination of moisture content.*

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 and one gross sample is constituted, the minimum number of increments required for the experiment shall be $2n$ and 2 gross samples shall be constituted.

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

3.3 Sample preparation and testing

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

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

3.4 Replication of experiment

It is recommended that, even after a series of experiments has been conducted, the experiments should be carried out occasionally in order to check a possible quality variation in the consignments, as well as to control the methods for sampling, sample division and testing.

Because of the large amount of work involved in this method, it is recommended that it be carried out as a 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 following three categories, depending on the method of taking increments from the consignment in

1) In preparation.

accordance with the relevant clause of ISO 3081 or ISO 3082.

a) *Periodic systematic sampling :*

ISO 3081

Sub-clause 6.2	Sampling on conveyors
Sub-clause 6.4	Sampling from loading bunker discharge
Annex, B.1	Sampling from ships
Annex, B.2	Sampling from stockpiles

ISO 3082

Sub-clause 6.8	Methods of taking increments

b) *Stratified sampling :*

ISO 3081

Sub-clause 6.2	Sampling on conveyors
Sub-clause 6.3.3(1)	Sampling from wagons
Sub-clause 6.4	Sampling from loading bunker discharge
Annex, B.1	Sampling from ships
Annex, B.2	Sampling from stockpiles

c) *Two-stage sampling :*

ISO 3081

Sub-clause 6.3.3(2)	Sampling from wagons

4.1.1 *Periodic systematic sampling*

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

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

3) The increments shall be taken at a regular sampling interval, obtained by 2) above, with a random start from the consignment.

4) The increments shall be placed alternately in 2 containers, A and B. Thus, 2 gross samples, A and B, will be constituted, each comprising n increments.

EXAMPLE 1

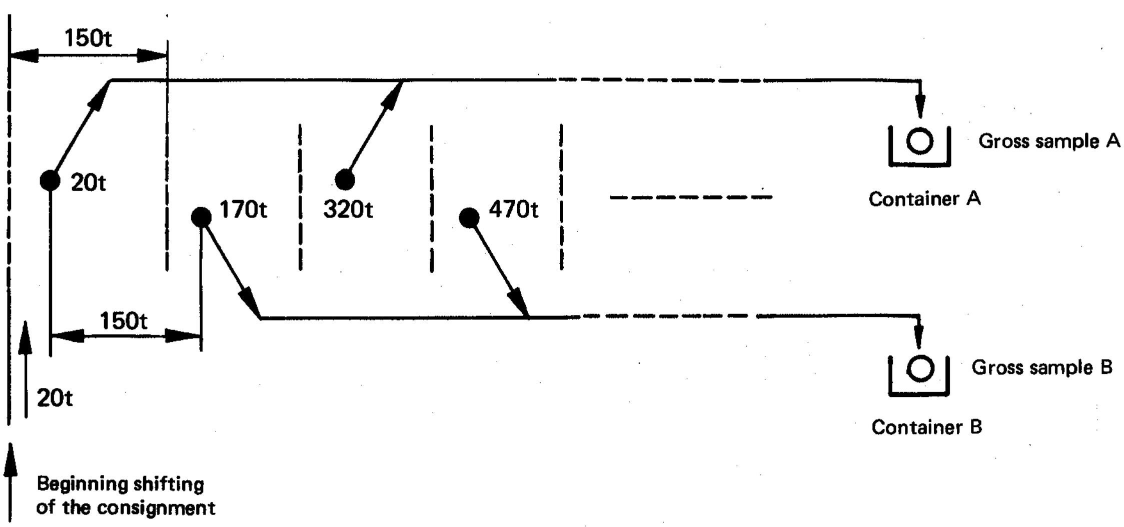
1) Suppose that a consignment of 19 000 tonnes of iron ore discharged is transferred by belt conveyors and that the classification category of the ore is "medium" quality variation : the minimum required number of increments (n) is 60, as shown in table 4 of ISO 3081 or ISO 3082.

2) The sampling interval for taking increments is determined as follows :

$$\frac{19\,000}{60 \times 2} \approx 158 \to 150 \text{ (tonnes)}$$

3) Thus, increments are taken at 150-tonne intervals. The point for taking the first increment from the first sampling interval of 150 tonnes should be determined by a random selection method. If the point for taking the first increment is determined as 20 tonnes from beginning shifting of the consignment, subsequent increments should be taken at the points of 170 tonnes (= 20 + 150), 320 tonnes (= 20 + 150 × 2), . . . Since the whole consignment amounts to 19 000 tonnes, 126 increments will be collected.

4) The increments are placed alternately in containers A and B, and 2 gross samples, A and B, are constituted, each comprising 63 increments (see figure 1).



LEGEND : Dot indicates increment and circle indicates gross sample.

FIGURE 1 — **Schematic diagram for example 1**

4.1.2 *Stratified sampling*

1) In the case where the number of wagons, i.e. the number of strata (k), forming one consignment is smaller than the number of increments (n) given in table 4 of ISO 3081, the number of increments ($\bar{n}$) to be taken from each wagon (stratum) shall be obtained by the formula given in 6.3.3(1) of ISO 3081.

2) Two times the $\bar{n}$ increments ($2\bar{n}$) shall be taken from each wagon.

3) The $2\bar{n}$ increments taken from each wagon shall be separated at random into 2 sub-samples, a and b, each of $\bar{n}$ increments.

4) Each of the 2 sub-samples a and b of all the wagons shall be combined to constitute 2 gross samples, A and B respectively, each comprising n ($= k\bar{n}$) increments.

NOTE — If the tonnage varies wagon by wagon, the number of increments (n_i) 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

1) Suppose that a consignment of iron ore is delivered by eleven 60-tonne wagons and that the quality variation of the ore within wagons (σ_W) is "medium"; the minimum required number of increments for the 660 tonne consignment is 20, as shown in table 4 of ISO 3081.

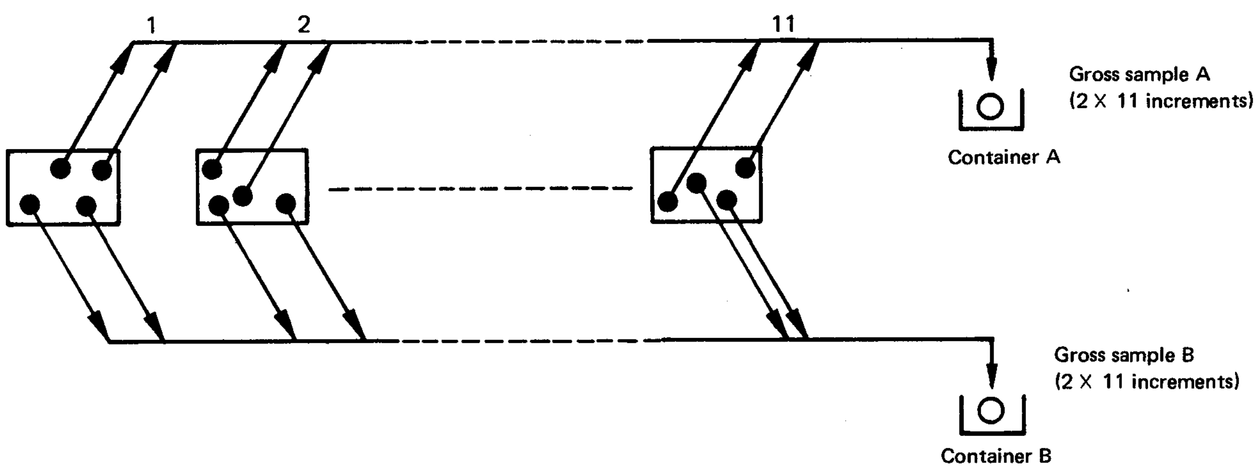
Then, the number of increments to be taken from each wagon is :

$$\frac{20}{11} \approx 2$$

2) The 4 (= 2 × 2) increments are taken from each wagon.

3) The 4 increments are separated at random into 2 sub-samples, a and b, each consisting of 2 increments.

4) Each of the 2 sub-samples a and b from the 11 wagons is combined to constitute 2 gross samples, A and B respectively, each comprising 22 (= 2 × 11) increments (see figure 2).



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

FIGURE 2 — **Schematic diagram for example 2**

EXAMPLE 3

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

Then the numbers of increments to be collected respectively from six 60-tonne wagons and eight 30-tonne wagons are :

$$\frac{40 \times 360}{600} = 24$$

$$\text{and} \quad \frac{40 \times 240}{600} = 16$$

The numbers of increments to be taken respectively from each 60-tonne wagon and from each 30-tonne wagon are :

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

$$\text{and} \quad \frac{16}{8} = 2$$

2) For this experiment, 8 (= 2 × 4) increments are taken from each of the 60-tonne wagons, and 4 (= 2 × 2) increments from each of the 30-tonne wagons.

3) The increments taken by 2) above are separated at random into 2 sub-samples, a and b.

4) The 2 sub-samples a and b thus obtained from all of the wagons are combined separately to constitute 2 gross samples, A and B respectively, each comprising 40 increments.

4.1.3 *Two-stage sampling*

1) If the number of wagons (k) forming one consignment is more than the number of increments (n) required from table 4 of ISO 3081, or when it is impracticable to take increments from all of the wagons, m wagons shall be selected at random from the consignment in accordance with table 6 of ISO 3081.

2) An additional m wagons shall be selected at random from 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.

3) The required number of increments shall be taken from each of the wagons selected in accordance with 6.3.3(2) (b) of ISO 3081.

4) All of the increments taken from the wagons selected in accordance with 1) above shall be combined to constitute gross sample A.

All of the increments taken from the wagons selected in accordance with 2) above shall be combined to constitute another gross sample B.

EXAMPLE 4

1) Suppose that a wagon-borne consignment consists of eighty 60-tonne wagons, i.e. $80 \times 60 = 4\,800$ (tonnes) of iron ore, the classification category of which is "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 is 15, as shown in table 6 of ISO 3081.

2) From the same consignment, an additional 15 wagons are selected independently of those selected in 1) above.

3) 4 increments are taken at random from each of the 15 wagons selected in 1) above, and the total 60 (= 4 × 15) increments are combined to constitute gross sample A.

Another 4 increments are taken at random from each of the 15 wagons selected in 2) above, and this further total 60 (= 4 × 15) increments are combined to constitute gross sample B.

4.2 Sample division and testing

The 2 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)

1) The 2 gross samples A and B shall be divided separately to prepare 2 final samples.

2) The 4 final samples, A_1, A_2, and B_1, B_2, shall be tested in duplicate, respectively. A total of 8 tests shall be run in random order.

NOTE — By type 1, each of the precisions of sampling, division and measurement is obtainable separately.

4.2.2 *Division-testing type 2* (see figure 4)

1) The gross sample A shall be divided to prepare 2 final samples, A_1 and A_2, and from the gross sample B, one final sample shall be prepared.

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 — By type 2 also, each of the precisions of sampling, division and measurement is obtainable separately. However, the respective precisions for estimating the precisions of sampling, division and measurement will be lower than those to be attained by the above type-1 experiment.

4.2.3 *Division-testing type 3* (see figure 5)

1) From each of the 2 gross samples A and B, one final sample shall be prepared.

2) The 2 final samples A and B shall be tested individually.

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

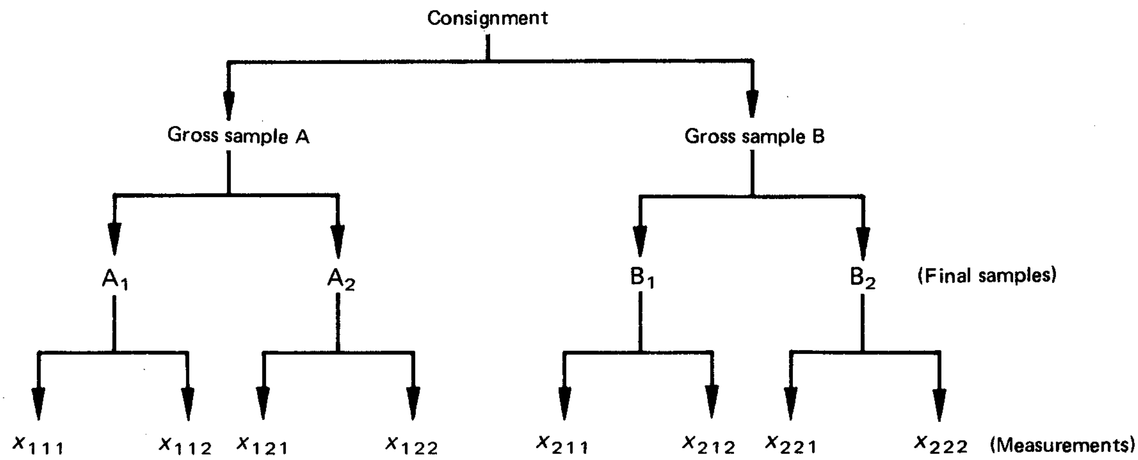


FIGURE 3 — **Flowsheet for division-testing type 1**

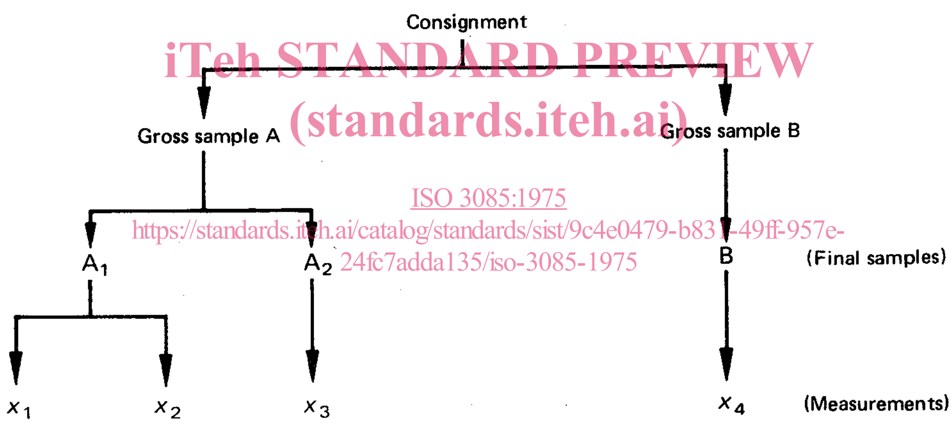


FIGURE 4 — **Flowsheet for division-testing type 2**

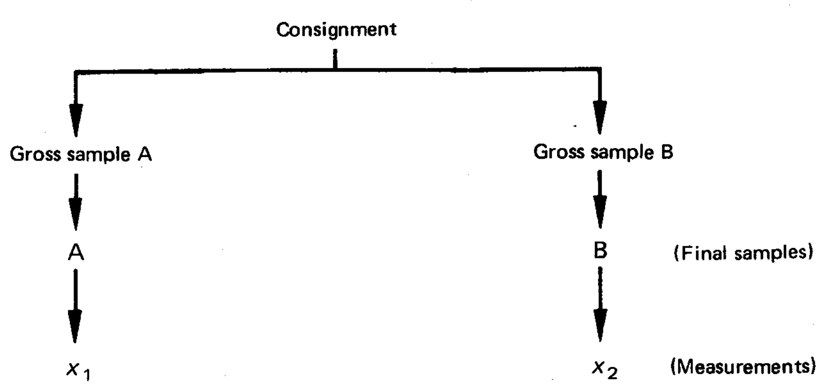


FIGURE 5 — **Flowsheet for division-testing type 3**

5 ANALYSIS OF EXPERIMENTAL DATA

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

5.1 Division-testing type 1 (see figure 3 and exhibit 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 below.

1) Denote the pair of 4 measurements (such as % Fe) of a pair of 2 duplicate samples, prepared from the 2 gross samples A and B, as x_{111}, x_{112}; x_{121}, x_{122} and x_{211}, x_{212}, x_{221}, x_{222}.

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

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

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

where

i = 1 and 2 stands for A and B;

j = 1 and 2 stands for final samples.

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

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

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

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

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

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

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

$$\bar{\bar{\bar{x}}} = \frac{1}{k}\Sigma\,\bar{\bar{x}} \qquad \ldots (7)$$

$$\bar{R}_1 = \frac{1}{4k}\Sigma\,R_1 \qquad \ldots (8)$$

$$\bar{R}_2 = \frac{1}{2k}\Sigma\,R_2 \qquad \ldots (9)$$

$$\bar{R}_3 = \frac{1}{k}\Sigma\,R_3 \qquad \ldots (10)$$

where k is the number of consignments.

For the preparation of control charts for means and ranges, calculate the control limits as follows :

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

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

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).

SOURCE

— *Theoretical background :* E. S. Pearson : The application of Statistical Methods to Industrial Standardization and Quality Control. British Standards Institution (1935).

— *Numerical values :* ASTM Manual on Quality Control of Materials. American Society for Testing and Materials (1951).

6) Calculate the estimated values of standard deviation of measurement ($\hat{\sigma}_M$), division ($\hat{\sigma}_D$) and sampling ($\hat{\sigma}_S$) which are estimated by the range :

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

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

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

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

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

The comparison described in 7) below will be made using the value thus obtained.

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 note in 6) above.

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 as defined below.

Stratified sampling :

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

Two-stage sampling :

$$\beta_S = 2\,\sigma_S = 2\sqrt{\frac{M-m}{M-1}\cdot\frac{\sigma_b{}^2}{m} + \frac{\sigma_w{}^2}{m\bar{n}}}$$

where $\bar{n} = 4$

5.2 Division-testing type 2 (see figure 4)

The estimated value of precision shall be calculated in accordance with the procedure given below.

1) Denote the 4 measurements as follows :

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

where k is the number of consignments.

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

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

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

Upper control limits for R-chart :

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

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

6) Calculate the estimated values of standard deviation of measurement ($\hat{\sigma}_M$), division ($\hat{\sigma}_D$) and sampling ($\hat{\sigma}_S$) :

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

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

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

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

NOTE — See note in 5.1(6).

7) Calculate the estimated values of precision of measurement ($2\,\hat{\sigma}_M$), 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) as 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 below.

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

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

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

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

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

$$\bar{\bar{x}} = \frac{1}{k}\Sigma \bar{x} \qquad \ldots (34)$$

$$\bar{R} = \frac{1}{k}\Sigma R \qquad \ldots (35)$$

where k is the number of consignments.

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

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

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