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**INTERNATIONAL STANDARD**



**3084**

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**Iron ores — Experimental methods for evaluation of quality variation**

*Minerais de fer — Méthodes expérimentales d'évaluation de la variation de qualité*

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## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

International Standard ISO 3084 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	India	Sweden
Austria	Iran	Thailand
Belgium	Italy	Turkey
Bulgaria	Japan	United Kingdom
Canada	Mexico	U.S.A.
Czechoslovakia	Poland	U.S.S.R.
Egypt, Arab Rep. of	Romania	Yugoslavia
France	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

# Iron ores — Experimental methods for evaluation of quality variation

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies experimental methods for the evaluation of quality variation of iron ores for the purpose of defining the sampling procedure by the stratified method, periodic systematic method and the two-stage method as prescribed in ISO 3081 or ISO 3082.

NOTE — The experimental methods may be applied approximately to the time-basis sampling when the variation of the flow of the ores is not so large.

## 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.*<sup>1)</sup>

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

ISO 3085, *Iron ores — Experimental methods for checking the precision of sampling.*

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 Quality variation

The magnitudes of quality variation or degrees of heterogeneity of iron ores shall be determined by standard deviation as described below.

1) *Stratified sampling and periodic systematic sampling:*

Standard deviation within strata or intervals between taking increments (denoted by  $\sigma_w$ ).

2) *Two-stage sampling as applied to sampling from wagons:*

Standard deviation within wagons (denoted by  $\sigma_w$ ) and standard deviation between wagons (denoted by  $\sigma_b$ ).

1) In preparation.

### 3.2 Quality characteristics

The quality characteristic chosen for determining the quality variation is generally the iron content; however, it should be recognized that the moisture content, particle size distribution and other quality characteristics may have to be taken into account.

In this case, the criteria for classification of quality variation (see clause 6) shall be established beforehand.

### 3.3 Iron ores to be classified

Quality variation shall be determined for each type of iron ore specified between the contracting parties.

### 3.4 Sampling, sample preparation and testing

Sampling, sample preparation and testing of the sample for this investigation shall be carried out in accordance with the relevant International Standards.

### 3.5 Execution of investigation

The sampling for this investigation may be conducted in conjunction with routine sampling for the determination of quality of the consignment. In other words, the same sample collected from the consignment may commonly be used for both purposes.

## 4 METHOD OF INVESTIGATION FOR STRATIFIED SAMPLING AND PERIODIC SYSTEMATIC SAMPLING

The procedures for evaluating the standard deviation within strata ( $\sigma_w$ ), applicable to both stratified sampling and periodic systematic sampling, are described below.

### 4.1 Type of investigation

4.1.1 *Type 1* — When large-size consignments are delivered infrequently, the quality variation may be derived from a single consignment.

Create at least ten parts of equal tonnage over the consignment. Then constitute a pair of sub-samples for each part, by combining the increments involved in each part as shown in figure 1 (1) and example 1.

**4.1.2 Type 2** – When small-size consignments are delivered frequently, the quality variation may be derived from several consignments of approximately equal tonnage.

Create at least ten parts of approximately equal tonnage over all the consignments involved. Then constitute a pair of sub-samples for each part, by combining the increments involved in each part as shown in figure 1 (2) and example 2.

**4.1.3 Type 3** – When consignments are delivered frequently and the investigation by type 1 or type 2 is uneconomical, the quality variation may be derived from a larger number of consignments of approximately equal tonnage.

Constitute a pair of sub-samples for each consignment as shown in figure 1 (3) and example 3.

**4.1.4 Type 4** – In the case of sampling from a wagon-borne consignment and when the increments are collected from all of the wagons of the consignment, the sampling scheme may be regarded as the method of stratified sampling.

Constitute a pair of sub-samples for each consignment as shown in figure 1 (4).

## 4.2 Number of increments and composition of sub-samples

### 4.2.1 Number of increments

The number of increments to be collected from one or several consignments for this investigation may be the same as that selected for routine sampling. However, when the routine sampling is based on the classification category of "small" quality variation (see table 4 of ISO 3081 or ISO 3082) and the number of increments is considered to be insufficient to obtain a reliable standard deviation, then the number of increments shall be increased.

1) In the case of investigation type 1, the number of increments shall be selected from table 4 of ISO 3081 or ISO 3082, and the increments shall be divided into at least ten groups in order to constitute a pair of sub-samples (see figure 1 (1)).

2) In the case of investigation type 2, the number of increments to be taken for each consignment shall be in accordance with table 4 of ISO 3081 or ISO 3082. The increments corresponding to each consignment shall then be sub-divided on a stratum basis in order to constitute a pair of sub-samples (see figure 1 (2)).

3) In the case of investigation type 3, the number of increments being collected from each consignment shall be selected from table 4 of ISO 3081 or ISO 3082 (see figure 1 (3)).

4) In the case of investigation type 4, the number of increments being collected from each train of the consignment shall be selected from table 4 of ISO 3081, and the number of increments to be taken from each wagon shall be calculated from sub-clause 6.3.2 (1) of ISO 3081 (see figure 1 (4)). If the number is odd, it shall be increased by one to make it even.

### 4.2.2 Composition of sub-samples

The sub-samples shall be constituted in accordance with the following procedure :

a) allocate a serial number to the increments of one part in consecutive order of collection;

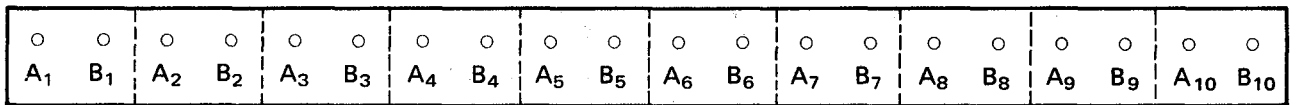
b) constitute a pair of sub-samples from consecutive odd-number increments (denoted by sub-sample A) and consecutive even-number increments (denoted by sub-sample B) in each part (see figure 2);

c) for each investigation, prepare  $k$  sets of paired sub-samples.

NOTE – Each sub-sample shall be constituted from two or more increments.

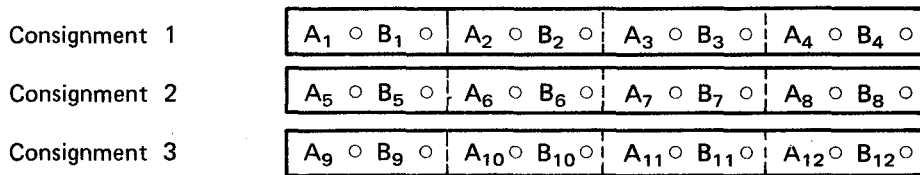
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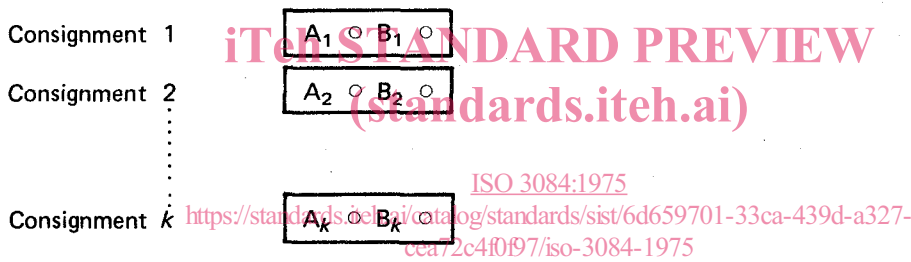


LEGEND : The rectangular box indicates one consignment; each division of the box made by broken lines indicates one part; a pair of circles indicates a pair of sub-samples. (This applies to (1), (2) and (3)).

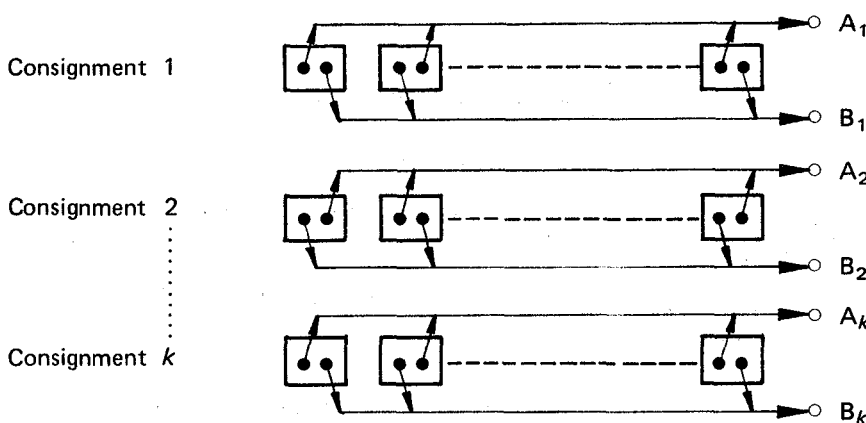
(1) Investigation type 1 – One consignment (Example for ten parts)



(2) Investigation type 2 – Several consignments (Example for three consignments and twelve parts)



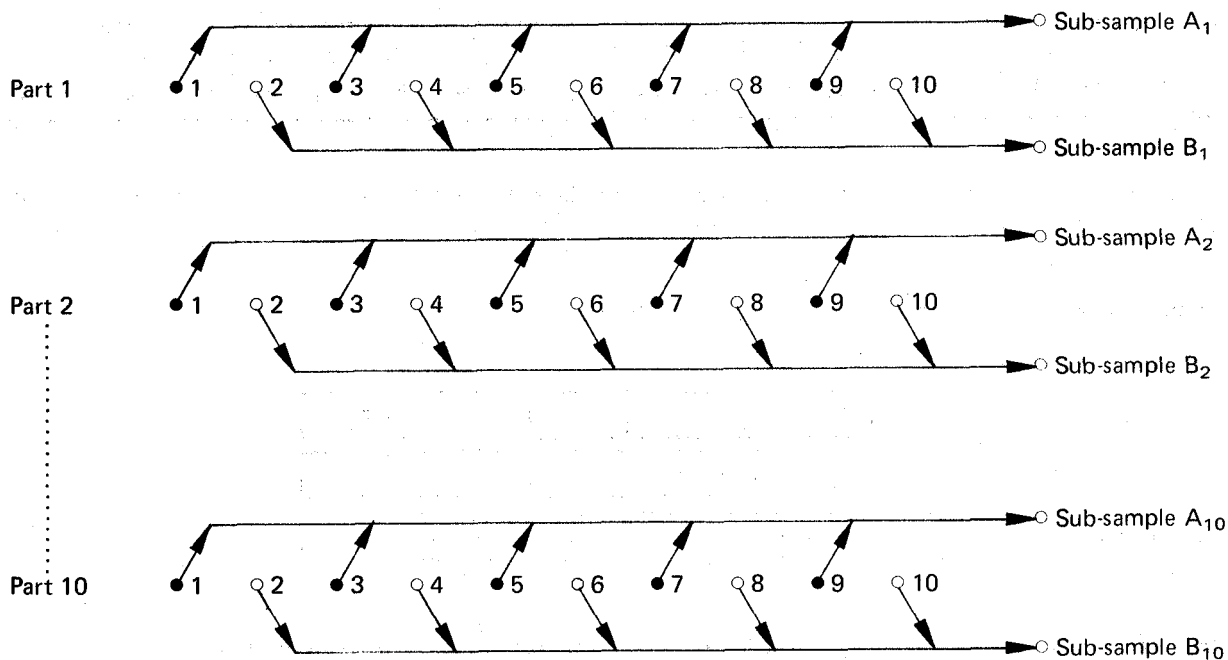
(3) Investigation type 3 – k consignments : one consignment = one part



LEGEND : Each box indicates a wagon; points in box indicate increments; circles indicate sub-samples.

(4) Investigation type 4 – Wagon-borne consignments by stratified sampling

FIGURE 1 – Schematic diagram for one investigation



LEGEND : ● odd-number increment  
○ even-number increment

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NOTE — The above diagram is based on an example for a single consignment of 5 000 to 15 000 tonnes of ore of "large" quality variation. The required minimum number of increments is 100 from table 4 of ISO 3081 or ISO 3082, and ten pairs of sub-samples A and B, each comprising five increments, are prepared.

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FIGURE 2 — Example of schematic diagram for composition of pairs of sub-samples (type 1)

### 4.3 Preparation of final samples and testing

Final samples A (odd number) and B (even number) shall be prepared separately from all the pairs of sub-samples.

The final sample may be an analysis sample, moisture sample, size sample or physical testing sample, as required.

The final samples A and B which have been prepared from respective sub-samples shall be subjected to testing for required quality characteristics separately.

### 4.4 Number of investigations

- a) In the case of investigation types 1 and 2, at least five separate investigations may be conducted.
- b) In the case of investigation types 3 and 4, at least ten separate investigations may be conducted.

NOTE — As  $\sigma_w$  cannot be estimated so precisely with a small number of investigations, about ten separate investigations are recommended.

### 4.5 Calculation of standard deviation within strata

#### 4.5.1 Data sheet

The experimental data of chemical analysis, moisture determination, size determination or physical testing as measured on the individual final samples shall be recorded on a suitable form (see examples 1 to 3).

#### 4.5.2 Calculation

The estimated standard deviation within strata shall be calculated from the formulae given below.

Range of paired measurements :

$$R = |A - B| \quad \dots (1)$$

where

$R$  is the range;

$A$  is the measurement of quality characteristic (such as % Fe) of odd-number final sample A;

$B$  is the measurement of quality characteristic of even-number final sample  $B$  which pairs with  $A$ .

Mean of ranges :

$$\bar{R} = \frac{1}{k} \sum R \quad \dots (2)$$

where

$\bar{R}$  is the mean of ranges;

$k$  is the number of values of  $R$ .

Mean of paired measurements :

$$\bar{x} = \frac{1}{2} (A + B) \quad \dots (3)$$

where  $\bar{x}$  is the mean of paired measurements of each part.

Standard deviation within strata :

$$\hat{\sigma}_w^{*2} = \bar{n}^* \left( \frac{\bar{R}}{d_2} \right)^2 \quad \dots (4)$$

where

$\hat{\sigma}_w^*$  is the estimated standard deviation within strata;

$\bar{n}^*$  is the number of increments comprising each sub-sample  $A$  or  $B$ ;

$d_2$  is the factor to estimate standard deviation from the range; for paired data  $1/d_2 = 0,886 5$ .

NOTES

1 The mean ( $\bar{x}_i$ ) of a consignment may be obtained from formula (5) as the routine determined value of quality characteristic of the consignment.

$$\bar{x}_i = \frac{1}{P_i} \sum \bar{x} \quad \dots (5)$$

where

$\bar{x}_i$  is the mean of  $\bar{x}$  for each consignment;

$P_i$  is the number of parts in each consignment.

2 The square root of  $\hat{\sigma}_w^{*2}$  obtained from formula (4) is the overall standard deviation of sampling, sample division and measurement, which is over-estimating  $\hat{\sigma}_w$ , but the classification in clause 6 may be made with this value (see 4.6). When it is desired to obtain  $\hat{\sigma}_w$ , and the standard deviation of sample division (denoted by  $\hat{\sigma}_D$ ) and the standard deviation of measurement (denoted by  $\hat{\sigma}_M$ ) are known,  $\hat{\sigma}_w$  should be calculated by the formula given below :

$$\hat{\sigma}_w^2 = \bar{n}^* \left[ \left( \frac{\bar{R}}{d_2} \right)^2 - \hat{\sigma}_D^2 - \hat{\sigma}_M^2 \right] \quad \dots (6)$$

3 If the number of increments is determined according to 4.2.1 and those increments are taken, the variation in the number of increments comprising each sub-sample will be small. If the variation is 10 % or less, formulae (4) and (6) can be applied approximately by using the mean value of  $\bar{n}^*$ .

#### 4.6 Expression of results

a) In the case of types 1 and 2, the estimated value of standard deviation within strata ( $\bar{\sigma}_w$ ) of a particular iron ore evaluated from a series of investigations shall be reported by the square root of the mean of all the values of  $\sigma_w^2$ . Symbolically,

$$\bar{\sigma}_w = \sqrt{\frac{1}{h} \sum \hat{\sigma}_w^2} \quad \dots (7)$$

where

$\bar{\sigma}_w$  is the mean of  $\hat{\sigma}_w$ ;

$h$  is the number of individual values of  $\hat{\sigma}_w^2$ .

b) In the case of types 3 and 4, the value of  $\hat{\sigma}_w^*$  obtained by formula (4) or  $\hat{\sigma}_w$  obtained by formula (6) shall be reported as the estimated value of standard deviation within strata of a particular iron ore.

NOTE – In the case of iron content, the value of standard deviation shall be rounded off to the first decimal place.

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#### 5 METHOD OF INVESTIGATION FOR TWO-STAGE SAMPLING

The method described in this clause covers the procedure for evaluating standard deviation within wagons (denoted by  $\sigma_w$ ) and standard deviation between wagons (denoted by  $\sigma_b$ ), being applicable to the method of sampling from wagons.

NOTE – When the number of wagons constituting one train is relatively small, and the sampling scheme requires the collection of increments from all of the wagons in the train, then the method of investigation should be that of the stratified sampling as specified in type 4.

##### 5.1 Number of sample wagons and number of increments

The number of sample wagons to be selected from a train of wagons and the number of increments to be collected from each of the sample wagons selected shall be decided in accordance with the following procedure :

a) the wagon-borne consignment or part of a consignment being delivered as a single train may be regarded as the subject of a single investigation;

b) in the first place, the number of sample wagons (denoted by  $m$ ) shall be decided in accordance with table 6 of ISO 3081. For the sake of convenience in the treatment of subsequent date, it is recommended that  $m$  be an even number;

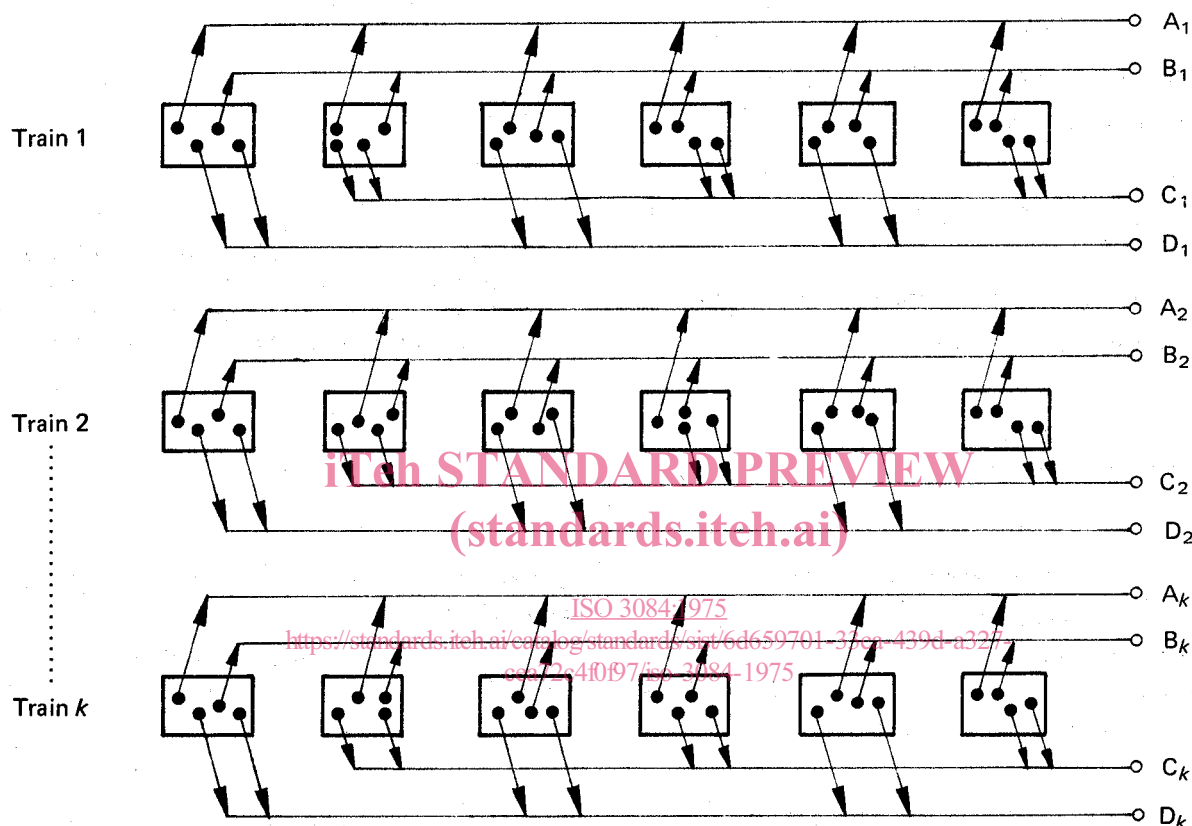
c) in the second place, a fixed number of four increments (denoted by  $\bar{n}$ , i.e.  $\bar{n} = 4$ ) shall be collected from each of the sample wagons.

5.2 Composition of sub-samples

All the increments collected from  $m$  sample wagons in a train shall be combined to constitute two pairs of sub-samples, denoted by A, B, and C, D, in accordance with the procedure indicated in figure 3.

The number of increments to constitute one sub-sample may be calculated as follows :

$$\frac{m \times \bar{n}}{\text{number of sub-samples}} = \frac{m \times 4}{4} = m$$



LEGEND : Each box indicates a sample wagon and a set of four random points indicates the four increments taken from each sample wagon.

NOTE – The above diagram shows those sample wagons which have been selected from each train of wagons of  $k$  trains. It is based on an example for a consignment of 600 to 2 000 tonnes of ore of "small" quality variation for  $\sigma_w$  and "medium" for  $\sigma_b$ .

According to table 6 of ISO 3081, the required minimum number of sample wagons ( $m$ ) is equal to six for one train, and two pairs of sub-samples, A, B, and C, D, each comprising six increments are prepared.

FIGURE 3 – Schematic diagram for one investigation



### 5.3 Preparation of final samples and testing

The two pairs of final samples, A, B, and C, D, shall be prepared from the two pairs of sub-samples from each train of wagons.

The individual final samples prepared separately from respective sub-samples shall be subjected to testing for required quality characteristics. For each investigation, prepare  $k$  sets of two pairs of final samples.

### 5.4 Number of investigations

It is recommended that at least ten separate investigations be carried out.

### 5.5 Calculation of standard deviation within and between wagons

#### 5.5.1 Data sheet

The experimental data of chemical analysis, moisture determination, size determination or physical testing as measured on the individual final samples shall be recorded on a suitable form (see example 4).

#### 5.5.2 Calculation

The estimated standard deviation within wagons and between wagons shall be calculated from the formulae given below.

Range of two paired measurements :

$$R_{AB} = |A - B| \quad \dots (8)$$

$$R_{CD} = |C - D| \quad \dots (9)$$

where

$R_{AB}$ ,  $R_{CD}$  are the ranges of respective paired measurements of final samples;

$A$ ,  $B$ , and  $C$ ,  $D$ , are the measurements of quality characteristic (such as % Fe) of two pairs of final related samples.

Mean of measurements :

$$\bar{x}_i = \frac{1}{4} [(A_i + B_i) + (C_i + D_i)] \quad \dots (10)$$

where

$\bar{x}_i$  is the mean of two pairs of measurements of each train or each investigation;

$i$  is the suffix which designates each train or each investigation.

Standard deviation within wagons and between wagons :

$$\hat{\sigma}_w^{*2} = m \left( \frac{R_{AB}}{d_2} \right)^2 \quad \dots (11)$$

$$\hat{\sigma}_b^2 = m \left( \frac{R_{CD}^2 - R_{AB}^2}{2 d_2^2} \right) \quad \dots (12)$$

where

$\hat{\sigma}_w^*$  is the estimated standard deviation within wagons;

$\hat{\sigma}_b$  is the estimated standard deviation between wagons;

$m$  is the number of sample wagons selected from a train of wagons;

$d_2$  is the factor to estimate standard deviation from the range; for paired data  $1/d_2 = 0,8865$ .

#### NOTES

1 The mean value ( $\bar{x}_i$ ) obtained from formula (10) may be used as the determined value of quality characteristic for a train of wagons.

2 Formulae (11) and (12) are derived from the following simultaneous equations (see figure 3) :

$$\left( \frac{R_{AB}}{d_2} \right)^2 = \frac{\hat{\sigma}_w^2}{m \times \frac{\bar{n}}{4}} \quad \dots (13)$$

$$\left( \frac{R_{CD}}{d_2} \right)^2 = \frac{\hat{\sigma}_b^2}{\frac{m}{2}} + \frac{\hat{\sigma}_w^2}{\frac{m}{2} \times \frac{\bar{n}}{2}} \quad \dots (14)$$

where  $\bar{n} = 4$ .

3 The value of  $\hat{\sigma}_w^*$  obtained from formula (11) is the overall standard deviation of sampling, sample division and measurement of the sample, which is over-estimating  $\hat{\sigma}_w$ . When it is desired to obtain  $\hat{\sigma}_w$ , and the standard deviation of sample division (denoted by  $\hat{\sigma}_D$ ) and the standard deviation of measurement (denoted by  $\hat{\sigma}_M$ ) are known,  $\hat{\sigma}_w$  should be calculated by formula (15) below. However, regardless of  $\hat{\sigma}_D$  and  $\hat{\sigma}_M$ , formula (12) remains unchanged.

$$\hat{\sigma}_w^2 = m \left[ \left( \frac{R_{AB}}{d_2} \right)^2 - \hat{\sigma}_D^2 - \hat{\sigma}_M^2 \right] \quad \dots (15)$$

4 In the case where a series of investigations is carried out on  $k$  trains and the number of sample wagons ( $m$ ) selected from each train is the same, then  $\hat{\sigma}_w^*$  and  $\hat{\sigma}_b$  may be obtained from the following formulae in place of formulae (11) and (12) :

$$\hat{\sigma}_w^{*2} = m \left( \frac{\bar{R}_{AB}}{d_2} \right)^2 \quad \dots (16)$$

$$\hat{\sigma}_b^2 = \frac{m (\bar{R}_{CD}^2 - \bar{R}_{AB}^2)}{2 d_2^2} \quad \dots (17)$$

where  $\bar{R}_{AB}$  and  $\bar{R}_{CD}$  are the means of  $k$  ranges of respective pairs of measurements.

5 When the value obtained from formula (12), (15) or (17) becomes negative, it should be read as zero.