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Manganese and chromium ores — Experimental methods for evaluation of quality variation and methods for checking the precision of sampling

Minerais de manganèse et de chrome — Méthodes expérimentales d'évaluation de la variation de qualité et méthodes de contrôle de la fidélité de l'échantillonnage

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Foreword

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Manganese and chromium ores — Experimental methods for evaluation of quality variation and methods for checking the precision of sampling

1 Scope and field of application

This International Standard specifies experimental methods for the evaluation of quality variation of manganese and chromium ores, whether natural or processed, for the purpose of defining sampling procedure by the mass-basis systematic method and the two-stage method as specified in the relevant International Standards. It also specifies methods for checking the precision of sampling by the mass-basis systematic method and two-stage method.

NOTE — The evaluation of quality variation for the sampling procedure by the time-basis systematic method shall be derived on the basis of the mass-basis systematic method.

2 References

ISO 4296/1, *Manganese ores — Sampling — Part 1: Increment sampling.*

ISO 4296/2, *Manganese ores — Sampling — Part 2: Preparation of samples.*

3 Experimental methods for evaluation of quality variation — General conditions

3.1 Quality variation

The quality variation is a measure of the heterogeneity of the ore and is expressed in terms of standard deviation, denoted by σ. It shall be standard deviation within strata or intervals between taking increments, denoted by σ_w, in case of mass-basis systematic sampling; and shall be standard deviation between wagons, lorries, containers (hereinafter — wagons), denoted by σ_b, or standard deviation within wagons, denoted by σ_w, in case of two-stage sampling.

3.2 Quality characteristic

The quality characteristic chosen for determining the quality variation shall be manganese content for manganese ores and chromium oxide content for chromium ores.

NOTE — It should be recognized that other quality characteristics, such as moisture content, particle size distribution, etc., may have to be taken into account.

3.3 Determination of quality variation

The quality variation shall be determined for each type of an ore specified between the parties concerned.

3.4 Consignment for experiment

It is recommended that consignments to be selected for the experimental purposes be 3 000 t or over in mass and 50 or more increments constitute a gross sample.

3.5 Methods for sampling and testing

The sampling, sample preparation, chemical analysis and related testing for experimental purposes shall be carried out in accordance with the relevant International Standards.

3.6 Scale of experiment

The experiment shall be conducted on a consignment basis and shall be repeated five times. For systematic sampling, an experiment shall cover either a whole consignment or part of the consignment. For two-stage sampling, it shall cover 10 wagons out of M wagons of a consignment.

3.7 Frequency of experiment

It is recommended that the experiment for the evaluation of quality variation of each type of an ore of regular delivery be conducted at regular intervals, for example biennially, or as necessary.

4 Experimental methods

4.1 Method for systematic sampling

4.1.1 This method is applicable for the estimation of standard deviation within strata, σ_w, in the case of the mass-basis systematic method of sampling being applied to a consignment constituted at ports of loading or transferred at ports of discharge.

4.1.2 For the purpose of experiment, a whole consignment or part of the consignment shall be divided into five parts ($k = 5$) of equal mass.

4.1.3 Ten increments shall be collected from each part, an experimental sample being constituted from 50 increments. Each increment shall be taken at equal mass intervals after a random start within the first interval.

4.1.4 The five consecutive odd-number increments from the start shall constitute an odd-number subsample which is designated subsample A; and the five consecutive even-number increments from the start shall constitute an even-number subsample which is designated subsample B. See figure 1.

4.1.5 The samples for chemical analysis shall be prepared from subsamples A and B: a single final sample is prepared by discarding one of the binary divided portions for subsample A; and paired final samples are prepared from the binary divided portions for subsample B. See figure 2.

4.1.6 A single chemical determination shall be carried out on all of the final samples. The sequence of the 15 chemical determinations of the experimental samples shall be randomized, or otherwise both experimental and routine determinations shall be combined and the determinations shall be in a random order. The individual measurements shall be identified by the designations as shown in figure 2.

4.1.7 It is recommended that the experimental data be recorded on a data sheet such as that given in table 1.

4.2 Method for two-stage sampling

4.2.1 This method is applicable to the estimation of standard deviation between wagons, σ_b, and standard deviation within wagons, σ_w, in the case of the two-stage method of sampling being applied to a consignment loaded on to or discharged from wagons.

4.2.2 In the first stage of two-stage sampling, when sampling from a wagon-borne consignment, a total of 10 (m = 10) wagons shall be selected at equal mass intervals with a random start.

4.2.3 In the second stage of two-stage sampling, each of four ($\bar{n}$ = 4) increments shall be taken at random from the ore within the selected wagons to obtain a total of 40 increments.

4.2.4 The two different paired subsamples, denoted (C_1, C_2), (D_1, D_2), each composed of 10 increments, shall be constituted as follows: C_1 and C_2 each comprise one increment of the 10 selected wagons; D_1 comprises two increments of the five even-number wagons; D_2 comprises two increments of the five odd-number wagons. (See figure 3.)

4.2.5 The samples for chemical analysis shall be prepared from subsamples C_1, C_2; D_1, D_2: a single final sample from each of the two different pairs of subsamples.

4.2.6 Duplicate chemical determinations shall be made on all the final samples. The sequence of chemical determinations of the experimental samples shall be randomized, or otherwise both experimental and routine samples are combined and the determinations shall be in random order. The individual measurements shall be identified by designations as shown in figure 3.

4.2.7 It is recommended that the experimental data be recorded on a data sheet such as that given in table 2.

5 Methods for analysis of experimental data

5.1 Method for systematic sampling

This method is applicable to the experimental data obtained in accordance with the method specified in 4.1. The procedure to evaluate the quality variation shall be as given in 5.1.1 to 5.1.3.

5.1.1 Calculate the estimated value of combined precision of division and measurement, in terms of variance, of the subsample

$$\bar{R}_1 = \frac{1}{5}(|b_{11} - b_{12}| + |b_{21} - b_{22}| + \ldots + |b_{51} - b_{52}|) \quad \ldots (1)^{1)}$$

$$\hat{\sigma}^2_{DM} = (\bar{R}_1/d_2)^2 \quad \ldots (2)^{1)}$$

where

b_{i1}, b_{i2} are the ith measurements of each of the paired samples;

$\bar{R}_1$ is the mean of ranges of the five paired samples;

$\hat{\sigma}^2_{DM}$ is the estimated value of combined precision of division and measurement, in terms of variance;

d_2 is a factor for obtaining the standard deviation from range (d_2 = 1,128 for paired measurements).

When the estimated value of precision of measurement ($\hat{\sigma}^2_M$) is available, the estimated precision of division ($\hat{\sigma}^2_D$) can be calculated by the equation

$$\hat{\sigma}^2_D = \hat{\sigma}^2_{DM} - \hat{\sigma}^2_M \quad \ldots (3)$$

5.1.2 Calculate the estimated value of quality variation within strata in which precision of division and measurement is included

$$\bar{R}_2 = \frac{1}{5}(|a_1 - b_{11}| + |a_2 - b_{21}| + \ldots + |a_5 - b_{51}|) \quad \ldots (4)$$

1) Source: ASTM. *ASTM Manual on Quality Control of Materials.* Philadelphia, PA, American Society for Testing and Materials, 1951.

or

$$\bar{R}_2 = \frac{1}{5}(|a_1 - b_{12}| + |a_2 - b_{22}| + \cdots + |a_5 - b_{52}|) \quad \ldots (4')$$

$$(\hat{\sigma}'_w)^2 = \bar{n}(\bar{R}_2/d_2)^2 \quad \ldots (5)$$

hence

$$(\hat{\sigma}'_w)^2 = 5(\bar{R}_2/1{,}128)^2 \quad \ldots (6)$$

where

a_1, b_{i1}, b_{i2} are the ith measurements of a single final sample, and those of each of the final samples;

$\bar{R}_2$ is the mean of ranges of the five paired samples, either of (a and b_1) or (a and b_2);

$\bar{n}$ is the number of increments constituting a subsample (in this method $\bar{n} = 5$);

$(\hat{\sigma}'_w)^2$ is the estimated quality variation within strata by including σ^2_{DM}.

When b_1 has been chosen by random selection, equation (4) is applied; when b_2 is chosen, equation (4′) is used.

5.1.3 Calculate the estimated quality variation within strata, $\hat{\sigma}^2_w$, excluding σ^2_{DM}, by equation (7), which is derived from equations (2) and (5)

$$\hat{\sigma}^2_w = (\hat{\sigma}'_w)^2 - \hat{\sigma}^2_{DM} \quad \ldots (7)$$

5.2 Method for two-stage sampling

This method applies to experimental data obtained in accordance with the method specified in 4.2. The procedure for evaluation of the quality variation shall be as given in 5.2.1 and 5.2.2.

5.2.1 Calculate the estimated value of combined precision of division and measurement, in terms of variance, of the subsamples

$$\bar{R}_1 = \frac{1}{4}(|C_{11} - C_{12}| + |C_{21} - C_{22}| + |D_{11} - D_{12}| + |D_{21} - D_{22}|) \quad \ldots (8)$$

$$\hat{\sigma}^2_{DM} = (\bar{R}/d_2)^2 \quad \ldots (9)$$

where

C_{1i}, C_{2i}; D_{1i}, D_{2i} are the ith measurements of the two different paired samples;

$\bar{R}$ is the mean of ranges of the two different paired samples;

$\hat{\sigma}^2_{DM}$ and d_2 are as defined in 5.1.1.

To calculate $\hat{\sigma}^2_D$, see equation (3).

5.2.2[1] Calculate the estimated value of quality variation between wagons and that of quality variation within wagons by means of equations (10) to (14)

$$\left.\begin{aligned} \bar{x}_{C1} &= \frac{1}{2}(C_{11} + C_{12}) \qquad \bar{x}_{C2} = \frac{1}{2}(C_{21} + C_{22}) \\ \bar{x}_{D1} &= \frac{1}{2}(D_{11} + D_{12}) \qquad \bar{x}_{D2} = \frac{1}{2}(D_{21} + D_{22}) \end{aligned}\right\} \quad \ldots (10)$$

$$R_C = |\bar{x}_{C1} - \bar{x}_{C2}| \qquad R_D = |\bar{x}_{D1} - \bar{x}_{D2}| \quad \ldots (11)$$

Hence

$$\hat{\sigma}^2_b = m[(R_D/d_2)^2 - (R_C/d_2)^2]/2 \quad \ldots (12)$$

$$(\hat{\sigma}'_w)^2 = m[(R_C/d_2)^2 - (\hat{\sigma}_{DM})^2/2] \quad \ldots (13)$$

or

$$\hat{\sigma}^2_w = m\{(R_C/d_2)^2 - [\hat{\sigma}^2_D + \hat{\sigma}^2_M/2]\} \quad \ldots (14)$$

where

$\bar{x}_{C1}$, $\bar{x}_{C2}$, $\bar{x}_{D1}$, $\bar{x}_{D2}$ are the arithmetic means of each of the duplicate measurements of the two different paired samples;

R_C, R_D are the ranges of each of the arithmetic means of the two different paired samples;

m is the number of selected wagons in the first stage of two-stage sampling, in this method $m = 10$;

$\hat{\sigma}^2_b$ is the estimated quality variation between wagons in terms of variance;

$\hat{\sigma}^2_w$ is the estimated quality variation within wagons in terms of variance.

When the estimated value of combined precision of division and measurement, $\hat{\sigma}^2_{DM}$, of a specific type of an ore is made available, either from the experimentation according to 4.1 and 5.1 (systematic sampling) or from a different experiment, that estimated value may be used and part of this experiment can be simplified. In this case, each of the single chemical determinations shall be made on the two different paired samples to obtain measurements C_1, C_2; D_1, D_2 (see figure 3), then $(\hat{\sigma}_{DM})^2/2$ in equation (13) can be replaced with $\hat{\sigma}^2_{DM}$, and $\hat{\sigma}^2_M/2$ in equation (14) can be replaced with $\hat{\sigma}^2_M$.

1) Source of theoretical background: Pearson, E.S. *The Application of Statistical Methods to Industrial Standardization and Quality Control*, No 600-1935. London, UK, British Standards Institution, 1935.

5.3 Observation

During the process of data analysis, if the calculated value of variance turns out to be negative, it should be assumed that $\hat{\sigma}^2 = 0$, provided that this is not attributable to defects in the experimental operations.

6 Expression of results

The evaluated values of quality variation derived from five experiments shall be summarized by the procedure specified in 6.1 and 6.2.

6.1 Systematic sampling

The arithmetic mean of the estimated values of standard deviation within strata, $\bar{\sigma}_w$, shall be obtained by equation (15).

$$\bar{\sigma}_w = \sqrt{\frac{1}{h} \sum \hat{\sigma}_w^2} \qquad \dots (15)$$

where

$\hat{\sigma}_w^2$ is the value obtained from equation (7);

h is the number of values of $\hat{\sigma}_w^2$ (normally $h = 5$).

The quality variation is overestimated when the estimated value of σ_w^2 in which σ_{DM}^2 is included, $(\hat{\sigma}'_w)^2$, is used in equation (15).

6.2 Two-stage sampling

The arithmetic mean of the estimated values of standard deviation between wagons, $\bar{\sigma}_b$, and that of standard deviation within wagons, $\bar{\sigma}_w$, shall be obtained by equations (16) and (17) respectively.

$$\bar{\sigma}_b = \sqrt{\frac{1}{h} \sum \hat{\sigma}_b^2} \qquad \dots (16)$$

$$\bar{\sigma}_w = \sqrt{\frac{1}{h} \sum \hat{\sigma}_w^2} \qquad \dots (17)$$

where

$\hat{\sigma}_b^2$ is the value obtained from equation (12);

$\hat{\sigma}_w^2$ is the value obtained from equation (14);

h is the number of values of $\hat{\sigma}_b^2$ or $\hat{\sigma}_w^2$ (normally $h = 5$).

The quality variation is overestimated when the estimated value of σ_w^2 in which σ_{DM}^2 is included, $(\hat{\sigma}'_w)^2$, is used in equations (16) and (17).

7 Classification of quality variation

7.1 The evaluated values of quality variation by experimentation are those of "estimated" values of the sampling parameters. Therefore, factors concerning the situation at mines of the ore of each type shall be taken into consideration by the authorized parties responsible for sampling, for the determination of values of standard deviation as "known" values.

NOTE — The parameters of sampling utilized for the classification of magnitude of quality variation in the relevant International Standards are given as the "known" standard deviation.

7.2 When the value or values of standard deviation of the ore of a given type are determined for the purposes of sampling, the ore shall be classified into either one of the two categories ("large" and "small") or the three categories ("large", "medium" and "small") of quality variation, as the case may be.

7.3 It should be noted that the quality variation can change as a result of modification of factors such as

a) ore bodies in a mine;

b) methods of mining;

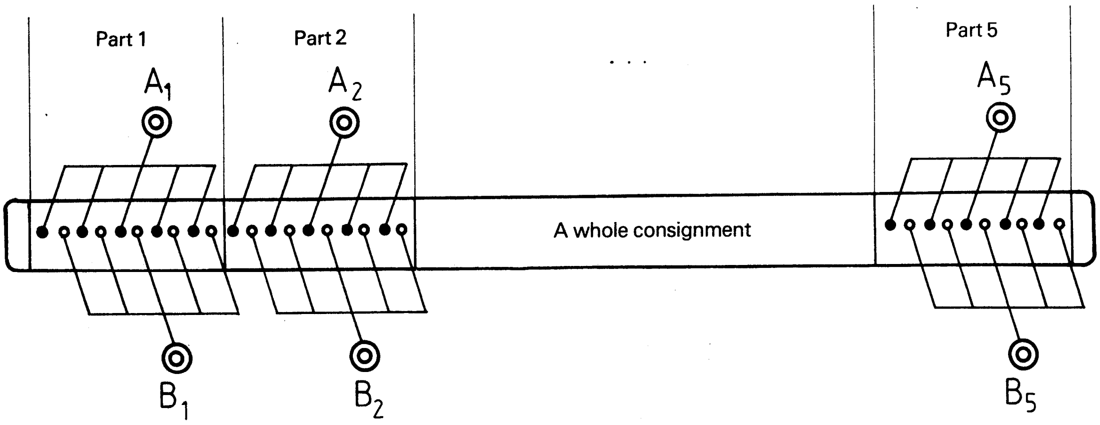
c) methods of ore dressing;

d) methods of stockpiling and reclamation;

e) methods of loading and discharging;

f) mass of consignment.

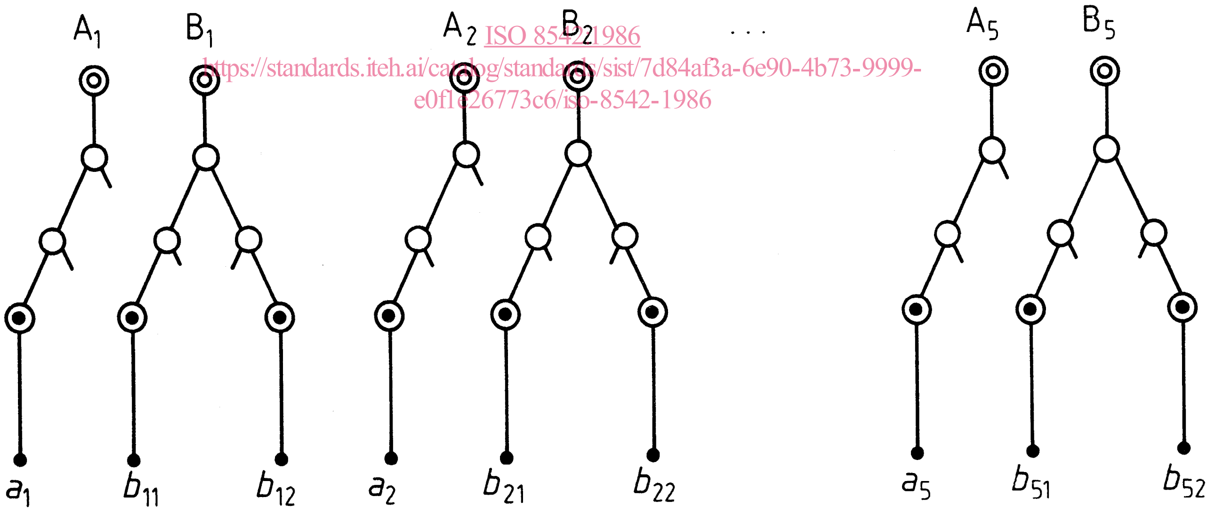
See 3.7.



Legend :

- ● Odd-number increment
- o Even-number increment
- ◎ Subsample

Figure 1 — Flow diagram for constitution of subsample (example)



Legend :

- ◎ Subsample
- ○ Division
- ⊙ Final sample for chemical analysis
- ● Measurement of test portion

Figure 2 — Flow diagram for preparation of final samples (example)

(Wagons selected from a single consignment)

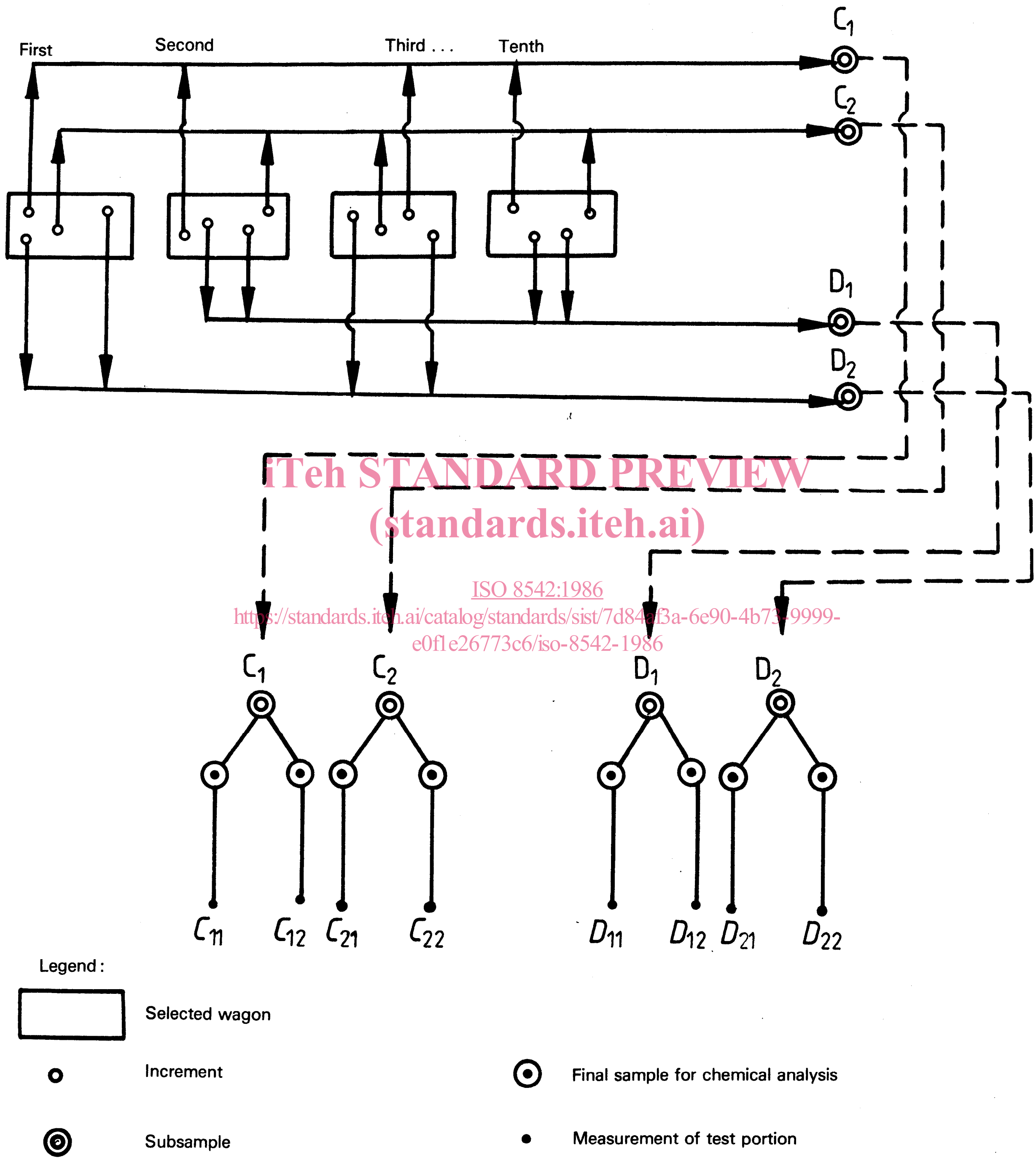


Figure 3 — Flow diagram for constitution of subsamples and preparation of final samples (example)

Table 1 — Data sheet for experiment on systematic sampling (example)

Designation of experiment: Type of ore: (for example manganese ore "A") Identification and mass of consignment: Mass of increment (kg): No. of increments: Dates of experiment:					
Part No.	**Quality characteristic** (for example % Mn)				
	a_i	b_{i1}	b_{i2}	$\lvert b_{i1} - b_{i2} \rvert$	$\lvert a_i - b_{i1} \rvert$ or $\lvert a_i - b_{i2} \rvert$
1. 2. 3. 4. 5.					
			Mean range	$\bar{R}_1$	$\bar{R}_2$

Calculation

$\hat{\sigma}^2_{DM} = (\bar{R}_1/1{,}128)^2 =$ ....

$(\hat{\sigma}'_w)^2 = 5(\bar{R}_2/1{,}128)^2 =$ ....

$\hat{\sigma}^2_w = (\hat{\sigma}'_w)^2 - \hat{\sigma}^2{}_{DM} =$ ....

Table 2 — Data sheet for experiment on two-stage sampling (example)

Designation of experiment: Type of ore: (for example manganese ore "A") Identification and mass of consignment: Mass of increment (kg): No. of increments: Dates of experiment:				
Subsample designation	**Quality characteristic** (for example % Mn)			
	Analysis 1	Analysis 2	R	$\bar{x}$
C_1 C_2 D_1 D_2				$\bar{x}_{C1}$ $\bar{x}_{C2}$ $\bar{x}_{D1}$ $\bar{x}_{D2}$
		Mean range	$\bar{R}$	

Calculation

$\hat{\sigma}^2_{DM} = (\bar{R}/1{,}128)^2 =$ ....

$R_C = \lvert \bar{x}_{C1} - \bar{x}_{C2} \rvert =$ ....

$R_D = \lvert \bar{x}_{D1} - \bar{x}_{D2} \rvert =$ ....

$\hat{\sigma}^2_b = 5[(R_D/1{,}128)^2 - (R_C/1{,}128)^2] =$ ....

$\sigma^2_w = 10[(R_C/1{,}128)^2 - (\hat{\sigma}_{DM})^2/2] =$ ....