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Manganese and chromium ores — Experimental methods for checking the bias of sampling and sample preparation

Minerais de manganèse et de chrome — Méthodes expérimentales de contrôle de l'erreur systématique d'échantillonnage et de préparation des échantillons

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Foreword

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International Standard ISO 8541 was prepared by Technical Committee ISO/TC 65, *Manganese and chromium ores*.

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.

Manganese and chromium ores — Experimental methods for checking the bias of sampling and sample preparation

1 Scope and field of application

This International Standard specifies experimental methods for checking the bias of sampling and preparation of samples of manganese and chromium ores, whether natural or processed, when the sampling is carried out in accordance with the methods specified in the relevant International Standards.

2 References

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

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

3 General conditions

3.1 The sampling and preparation of samples shall be carried out in accordance with the methods specified in the relevant International Standards.

3.2 In the experimental method, the results obtained from the method to be checked (referred to as method B) shall be compared with the results of a specified reference method (referred to as method A) which is assumed to produce practically unbiased results from technical and empirical viewpoints. The comparison shall be made by use of a statistical method of test for significance of difference between two mean values of test results at the 5 % level of significance.

3.3 The quality characteristics to be selected for the purposes of experiment shall be the major chemical elements, particle size distribution or moisture content of the ore, as the case may be.

3.4 The experiment shall be conducted on at least 10 consignments or 10 parts of the consignments.

3.5 When individual increments of a sample taken by method A and that by method B are correctly paired, the method of data analysis for paired data shall be applied. In order to apply the method of paired data analysis, it is indispensable to design and to perform the field experiment so that the pairing of samples, one by method A and the other by method B, is assured technically. Such pairing is generally assured if the two samples taken by method A and method B are from identical material and undergo identical processing and analysis almost simultaneously under similar conditions.

3.6 In cases where the pairing of individual increments of a sample taken by method A and that by method B is considered to be not sufficient, the method of data analysis for unpaired data shall be applied.

The method of unpaired data analysis used in this International Standard is based on the condition that the numbers of measurements of method A and method B are the same. Care shall be taken to obtain the same number of increments of a sample for both methods.

4 Experimental methods

4.1 Experiments for bias of sampling

4.1.1 Methods to be checked

Examples of checking the bias of methods of sampling are given in 4.1.1.1 to 4.1.1.5.

4.1.1.1 Sampling from conveyors

Method A: Stopped-belt sampling shall be the reference method in which each increment is taken from a stopped-belt conveyor at a specified place over the full width and thickness of the ore stream with the required length.

Method B: Each increment is taken from a point selected at random each time within the ore stream.

4.1.1.2 Sampling from wagons

Method A: Stopped-belt sampling (see 4.1.1.1) shall be the reference method. It is required to provide a belt conveyor system for the experiment.

Method B: A sample consisting of the required mass and number of increments is taken at random from the surface of the ore exposed during the loading or discharging of wagons or trucks.

4.1.1.3 Sampling from stockpiles

Method A: The method of sampling on conveyors as specified in the relevant International Standards shall be the reference method. Where practicable, method A given in 4.1.1.1 is recommended.

Method B: The method of sampling from stockpiles as specified in the relevant International Standards is the method to be checked.

4.1.1.4 Sampling from containers

Method A: Each increment shall be the ore contained in a container in its entirety, which has been selected at random in the sampling.

Method B: Each increment is taken at random from the ore in a container which has been selected at random in the sampling.

4.1.1.5 Mechanical sampling

Method A: The increments taken by method A given in 4.1.1.1 shall be the reference sample.

Method B: Each increment is taken by the installed mechanical primary sampler.

4.1.2 Experimental sampling

Two gross samples shall be taken from the same consignment under study, or the same part of the consignment, one of the two shall be taken by method A and the other by method B. The gross sample taken by method A is designated gross sample A and that taken by method B gross sample B.

4.1.3 Preparation of experimental samples

These two gross samples shall be processed separately for the preparation of the respective final sample by the same method, and shall be tested for the selected quality characteristics by the same method.

4.1.4 Pairing the increments

The gross samples A and B shall be taken from the portion of the ore as being regarded to be practically the same, on an individual increment basis. The selected quality characteristics shall be determined and the measurements recorded on a data sheet such as that given in table 3.

4.1.5 Unpaired increments

In the event that the pairing of increments is not achieved, the measurements shall be recorded on a data sheet such as that given in table 4.

4.2 Experiment for bias of sample preparation

4.2.1 Methods to be checked

Examples of checking the bias of methods of sample preparation are as follows:

Method A: The residue of sample in its entirety obtained after having been taken from the divided sample by a dividing apparatus shall be the reference sample.

Method B: The divided sample obtained by the dividing apparatus is a controlled sample.

The dividing apparatus to be used for method A and method B shall be the same.

Other examples may be given in terms of stages of division, different dividing apparatus, etc. For instance, if method A is the reference method, method B may be the one which uses a different dividing apparatus or involves fewer stages of division.

4.2.2 Preparation of experimental samples

A pair of final samples shall be prepared from the same sample under study, one by method A, designated sample A, and the other by method B, designated sample B.

4.2.3 Determination and recording

The quality characteristics shall be determined and the measurements recorded on a data sheet such as that given in table 4.

5 Data analysis

The test for significance of difference between two experimental results, in other words deviation of the results of method B from the results of the reference method A, shall be carried out by the t-test. For unpaired data, the F-test for equality of variances shall be carried out prior to the t-test.

5.1 Paired data

5.1.1 Calculate the difference between paired measurements

$$d_i = x_{Bi} - x_{Ai} \qquad i = 1, 2, \ldots, k \qquad \ldots (1)$$

where

x_{Ai}, x_{Bi} are respectively the ith measurements of sample A obtained by the reference method A and the ith measurement of sample B obtained by the method B to be checked;

d_i is the ith difference of deviation of x_{Bi} from x_{Ai};

k is the number of pairs of measurements of sample A or of sample B.

5.1.2 Calculate the mean of differences to one decimal place further than that used in the measurements themselves

$$\bar{d} = \frac{1}{k}\sum d_i \qquad \ldots (2)$$

where $\bar{d}$ is the mean value of k differences.

5.1.3 Calculate the unbiased estimates of variance of the differences

$$V_d = \frac{1}{\phi}\left\{\sum d_i^2 - \left(\sum d_i\right)^2 / k\right\} \qquad \ldots (3)$$

where

V_d is the unbiased estimate of variance of differences;

ϕ is the number of degrees of freedom, $\phi = k - 1$.

5.1.4 Calculate the observed value of t, denoted by t_o, by rounding off to the third decimal place

$$t_o = \frac{\bar{d}}{\sqrt{V_d / k}} \qquad \ldots (4)$$

5.1.5 Obtain the t-statistic at the 5 % level of significance for ϕ degrees of freedom, denoted $t(\phi, 0{,}05)$, from table 1.

5.1.6 Compare the absolute value of t_o obtained by experiment with the $t(\phi, 0{,}05)$ point obtained from the table.

When

$|t_o| < t(\phi, 0{,}05)$, then $\bar{d}$ is insignificant

When

$|t_o| \geqslant t(\phi, 0{,}05)$, then $\bar{d}$ is significant $\qquad \ldots (5)$

NOTE — To arrive at a conclusion see clause 6.

5.2 Unpaired data

5.2.1 *F*-test for equality of two variances

Prior to conducting the t-test for significance of difference between two mean values, the variance of the results of method A and that of method B are required to test for equality in a statistical sense by the F-test, also referred to as the variance ratio test.

5.2.1.1 For the sake of simplicity, transform the crude measurements to integers

$$X_{Ai} = (x_{Ai} - c_1)h \qquad i = 1, 2, \ldots, n$$
$$X_{Bi} = (x_{Bi} - c_2)h \qquad i = 1, 2, \ldots, n \qquad \ldots (6)$$

where

x_{Ai}, x_{Bi} are respectively the ith crude measurements of samples A and B;

X_{Ai}, X_{Bi} are respectively the ith transformed measurements of samples A and B;

c_1, c_2 are respective subtracting constants, arbitrarily selected separately, for the transformation;

h is a multiplying constant arbitrarily selected for the transformation;

n is the number of measurements of sample A or of sample B.

5.2.1.2 Calculate the mean values of measurements of samples A and B, denoted $\bar{x}_A$ and $\bar{x}_B$ respectively, and express them by inverting the transformation

$$\bar{x}_A = c_1 + \frac{\bar{X}_A}{h}$$
$$\bar{x}_B = c_2 + \frac{\bar{X}_B}{h} \qquad \ldots (7)$$

where

$$\bar{X}_A = \frac{1}{n}\sum X_{Ai}$$

and $\qquad i = 1, 2, \ldots, n$

$$\bar{X}_B = \frac{1}{n}\sum X_{Bi}$$

5.2.1.3 Calculate the sums of squares, denoted S_A and S_B respectively

$$S_A = \sum X_{Ai}^2 - \frac{1}{n}\left(\sum X_{Ai}\right)^2$$
$$S_B = \sum X_{Bi}^2 - \frac{1}{n}\left(\sum X_{Bi}\right)^2 \qquad i = 1, 2, \ldots, n \qquad \ldots (8)$$

Table 1 — $t(\phi, 0{,}05)$ [1)]

ϕ	9	10	11	12	13	14	15	16	17	18	19	20
t	2,262	2,228	2,201	2,179	2,160	2,145	2,131	2,120	2,110	2,101	2,093	2,086

1) Source: FISHER, R.A. and YATES, F., *Statistical Tables for Biological, Agricultural and Medical Research*, 4th edition, 1953.

5.2.1.4 Calculate the unbiased estimates of variance, denoted V_A and V_B respectively

$$V_A = \frac{S_A}{\phi_A}, \qquad V_B = \frac{S_B}{\phi_B}, \qquad V_A, V_B \geqslant 0 \qquad \ldots (9)$$

where ϕ_A, ϕ_B are the respective numbers of degrees of freedom of samples A and B, $\phi_A = n_A - 1$, $\phi_B = n_B - 1$; in this method $n_A = n_B = n$.

5.2.1.5 Calculate the ratio of V_B of method B to V_A of the reference method A by equation (10) and round off to the second decimal place.

In this method it is assumed that the variance of the reference method A would be smaller than that of method B ($V_A < V_B$), however, if it turns out that $V_A > V_B$, change the denominator for numerator so that the ratio is greater than unity, and use equation (10a).

$$F_o = V_B / V_A \qquad F_o \geqslant 1 \qquad \ldots (10)$$

$$F_o = V_A / V_B \qquad F_o \geqslant 1 \qquad \ldots (10a)$$

5.2.1.6 Obtain the F-statistic at the 5 % level of significance for ϕ_B and ϕ_A degrees of freedom ($\phi_B = \phi_A = \phi$), denoted by $F(\phi, \phi; 0{,}05)$. (See table 2.)

5.2.1.7 Compare the observed F_o value obtained by experiment with the $F(\phi, \phi; 0{,}05)$ point obtained from table 2.

When

$$F_o < F(\phi, \phi; 0{,}05), \text{ then the test is passed} \qquad \ldots (11)$$

When

$$F_o \geqslant F(\phi, \phi; 0{,}05), \text{ then the test is failed} \qquad \ldots (12)$$

5.2.1.8 When the F-test is passed, conduct the t-test as specified in 5.2.2. When the F-test is failed, reject the experimental results, improve the technique and, if necessary, carry out a further experiment.

5.2.2 t-test for significance of difference

5.2.2.1 Calculate the observed value of the t-statistic, denoted t_o, by rounding off to the third decimal place

$$t_o = \frac{\bar{x}_B - \bar{x}_A}{\sqrt{\dfrac{S_A + S_B}{\phi_A + \phi_B}\left(\dfrac{1}{n_A} + \dfrac{1}{n_B}\right)}} \qquad \ldots (13)$$

hence

$$t_o = \frac{\bar{x}_B - \bar{x}_A}{\sqrt{\dfrac{S_A + S_B}{\phi n}}} \qquad \ldots (14)$$

where

$\phi_A = n_A - 1$

$\phi_B = n_B - 1$

$\phi_A = \phi_B = \phi$

$n_A = n_B = n$

5.2.2.2 Compare the absolute value of t_o obtained by experiment with the $t(2\phi, 0{,}05)$ point obtained from table 1.

When

$|t_o| < t(2\phi, 0{,}05)$, then $\bar{d}$ is insignificant

When

$|t_o| \geqslant t(2\phi, 0{,}05)$, then $\bar{d}$ is significant ... (15)

NOTE — See clause 6 for the conclusions that may be drawn from the outcome of these tests.

6 Review of experimental results

If the difference between the results obtained from method B and method A is insignificant by the t-test, then method B may be adopted as routine method, provided that agreement is achieved between the parties concerned, where such is required. Also, the conditions specified in 6.1 to 6.3 shall be observed.

6.1 The unbiasedness means that the value obtained from routine operations does not depart from the true value, or reference value in this method, with a statistically significant difference at the 5 % level of significance.

6.2 If, even though the difference is statistically significant, it is regarded as being negligibly small from a practical or economic standpoint, method B may be adopted as a routine method, provided that agreement is achieved between the parties concerned, where such is required.

6.3 When the difference is statistically insignificant but is regarded as being so large as not to be negligible from a practical or economic standpoint upon investigation of the parties concerned, it is required that a further experiment be carried out.

Table 2 — $F(\phi, \phi; 0{,}05)$ 1)

ϕ	9	10	11	12	13	14	15	16	17	18	19	20
F	3,18	2,98	2,82	2,69	2,58	2,48	2,40	2,33	2,27	2,22	2,17	2,12

1) Source: Merrington, M. and Thompson, C.M., Tables of percentage points of the inverted beta distribution, *Biometrika* **33** 1943.

Table 3 — Data sheet for *t*-test of paired data (example)

Designation of experiment: Type of ore: (for example manganese ore) Dates of experiment:					
Consignment No.	**Name of ore**	**Quality characteristic** (for example % Mn)			
		x_{Bi}	x_{Ai}	$d_i = x_{Bi} - x_{Ai}$	d_i^2
1. 2. . . . *k*.					
			Sum		

***t*-test**

$$\bar{d} = \frac{1}{k} \sum d_i \begin{cases} > 0 \\ < 0 \end{cases}$$

$$V_d = \frac{1}{\phi} \left\{ \sum d_i^2 - \left(\sum d_i \right)^2 / k \right\} = \ldots\ldots\ldots\ldots$$

$$t_o = \frac{\bar{d}}{\sqrt{V_d / k}} = \ldots\ldots\ldots\ldots$$

$t(\phi, 0{,}05) = \ldots\ldots\ldots\ldots$

$|t_o| \lesseqqgtr t(\phi, 0{,}05)$

Statement of the result of *t*-test: ....

Table 4 — Data sheet for *t*-test of unpaired data (example)

Designation of experiment: Type of ore: (for example manganese ore) Dates of experiment:							
Consignment No.	**Name of ore**	**Quality characteristic** (for example % Mn)					
		Gross sample B			**Gross sample A**		
		x_{Bi}	X_{Bi}	X^2_{Bi}	x_{Ai}	X_{Ai}	X^2_{Ai}
1. 2. . . . *n*.							
Sum		$\sum x_{Bi}$	$\sum X_{Bi}$	$\sum X^2_{Bi}$	$\sum x_{Ai}$	$\sum X_{Ai}$	$\sum X^2_{Ai}$

***t*-test**

$\bar{x}_B$ = ... $\bar{x}_A$ = ...

S_B = ... S_A = ...

t = ...

$t(2\phi, 0{,}05)$ = ...

$|t_o| \lesseqgtr t(2\phi, 0{,}05)$

Statement of the result of *t*-test: ....