
International Standard



8530

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

Manganese and chromium ores — Experimental methods for checking the precision of sample division

Minerais de manganèse et de chrome — Méthode expérimentale de contrôle de la fidélité de la division des échantillons

First edition — 1986-10-15

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UDC 553.32 + 553.461 : 620.113

Ref. No. ISO 8530-1986 (E)

Descriptors: minerals and ores, manganese ores, chromate minerals, quality audit, statistical quality control, sampling, rules of calculation.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8560 was prepared by Technical Committee ISO/TC 10, *Technical drawings*.

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 precision of sample division

1 Scope and field of application

This International Standard specifies experimental methods for checking the precision of sample division of manganese and chromium ores, whether natural or processed, being carried out in accordance with the methods specified in the relevant International Standards.

2 Reference

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

3 General conditions

3.1 The division of experimental samples shall be carried out in accordance with the methods specified in the relevant International Standards.

3.2 The quality characteristics to be selected for the purposes of experiment shall be manganese content (% Mn) for manganese ores and chromium oxide content (% Cr₂O₃) for chromium ores.

NOTE — It should be recognized that other chemical elements under question and particle size distribution may have to be taken into account.

3.3 It should be recognized that the precision of division is liable to worsen in the following circumstances:

- a) when a sample having a large particle size is divided excessively at one time into a divided sample of small mass;
- b) when the division is carried out in a large number of stages;
- c) when apparatus for sample division with insufficiently checked precision is employed;
- d) when the established operational instructions for sample preparation are not implemented adequately;
- e) when a gross sample of a very large mass is divided.

3.4 The experiment shall be carried out at least 10 times for each type of an ore either on gross samples or subsamples.

3.5 The samples taken for routine determinations may be used for obtaining the samples for chemical analysis, by splitting the samples.

4 Experimental methods

The experiment shall be conducted by one of the following two methods:

- a) method to obtain the final sample for chemical analysis in three or more stages of crushing and division;
- b) method in one or two stages of crushing and division.

4.1 Method of division in three or more stages

4.1.1 This method is normally applied to coarse ores.

4.1.2 The mass of divided sample in relation to the whole-through sieve size at each stage of division shall be in accordance with the rules for sample division specified in the relevant International Standards.

4.1.3 A flow diagram for preparation of final samples for chemical analysis is given in figure 1, as an example.

The number of stages in the crushing and division of the gross sample (or subsample) shall be the same in order to obtain paired final samples.

4.1.4 A final sample shall be prepared from each of the paired samples.

4.1.5 A single chemical determination shall be made on one of the final samples and duplicate determinations on the other.

The duplicate determinations shall be carried out on two test portions taken from a final sample.

4.1.6 The sequence of chemical determinations of the experimental samples shall be randomized, or experimental samples and routine samples may be kept in one sequence and the determinations may be carried out in a random order.

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

4.2 Method of division in one or two stages

4.2.1 This method is applicable to coarse ores when it is possible to employ a high performance apparatus for crushing, as well as to fine ores.

When the experiment is intended to be conducted on an increment basis, the method of subsamples shall be applied.

4.2.2 The mass of divided sample in relation to the whole-through sieve size at each stage of division shall be in accordance with the rules for sample division specified in the relevant International Standards.

4.2.3 A flow diagram for preparation of final samples for chemical analysis is given in figure 2, as an example.

See the second paragraph of 4.1.3.

4.2.4 The procedures specified in 4.1.4 to 4.1.7 shall also be applied.

5 Data analysis

The analysis of experimental data shall be carried out for each type of ore separately. The methods of data analysis for the experiments specified in 4.1 and 4.2 are the same and it shall be carried out according to the following procedure.

5.1 Precision of measurement¹⁾

Calculate the estimated value of precision of measurement of chemical analysis by the equations

$$\bar{R}_1 = \frac{1}{r} \sum |x_{i21} - x_{i22}| \quad \dots (1)$$

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

where

x_{i21} and x_{i22} are the first and second measurements of the duplicate determination of the i th final sample x_{i2} ;

r is the number of experiments;

\bar{R}_1 is the mean of the ranges of the duplicate measurements;

$\hat{\sigma}_M$ is the estimated value of precision of measurement in terms of standard deviation;

d_2 is the factor to obtain standard deviation from range ($d_2 = 1,128$ for duplicate measurements).

5.2 Precision of division²⁾

Calculate the estimated value of precision of division by the equations

$$\bar{R}_2 = \frac{1}{r} \sum |x_{i1} - x_{i21}| \quad \dots (3)$$

or

$$\bar{R}_2 = \frac{1}{r} \sum |x_{i1} - x_{i22}| \quad \dots (3')$$

NOTE — When x_{i21} has been chosen by a random selection, equation (3) is applied; when x_{i22} is chosen, equation (3') is used.

$$\hat{\sigma}_D = \sqrt{(\bar{R}_2/d_2)^2 - \hat{\sigma}_M^2}$$

where

\bar{R}_2 is the mean of the ranges as defined in equations (3) and (3');

$\hat{\sigma}_D$ is the estimated value of precision of division in terms of standard deviation.

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5.3 Observation

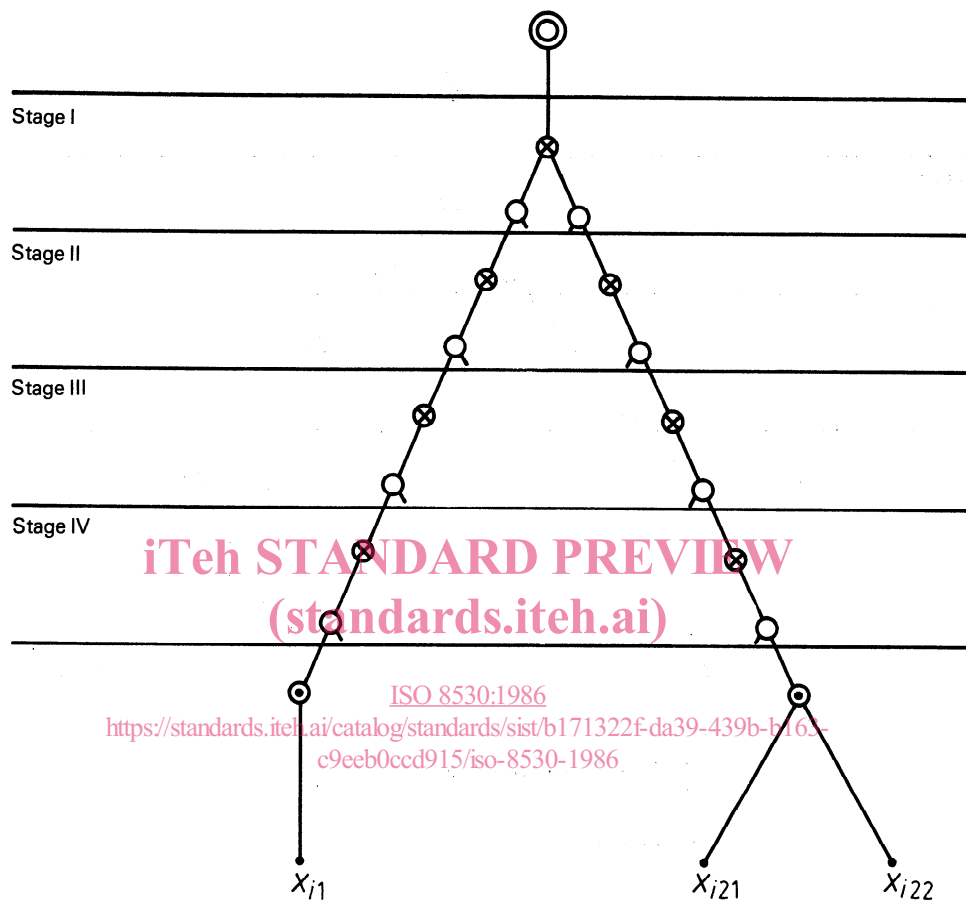
During the process of data analysis, if the calculated value within the square root turns out to be negative, the standard deviation should be regarded as being zero ($\hat{\sigma} = 0$), provided that this is not attributable to defects in the experimental operations.

6 Review of experimental results

In the event that there is an indication that the precision of division of sample and/or precision of measurement in chemical analysis do not attain the specified value or values given in the relevant International Standards, necessary action shall be taken to improve the procedure of sample preparation and/or chemical analysis by the respective sections in charge of the organization. Refer to 3.3 to identify the deficiency or deficiencies and then take remedial action.

1) Source: PEARSON, E.S. and HARTLEY, H.O. *Biometrika Tables for Statisticians*, Vol. 1. Cambridge, UK, Cambridge University Press for Biometrika Trustees.

2) 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.



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Legend :

- ⊙ Gross sample or subsample
- ⊗ Crushing
- Division
- ⊙ Final sample for chemical analysis
- Measurement of test portion

Figure 1 — Flow diagram for sample division in four stages (example)

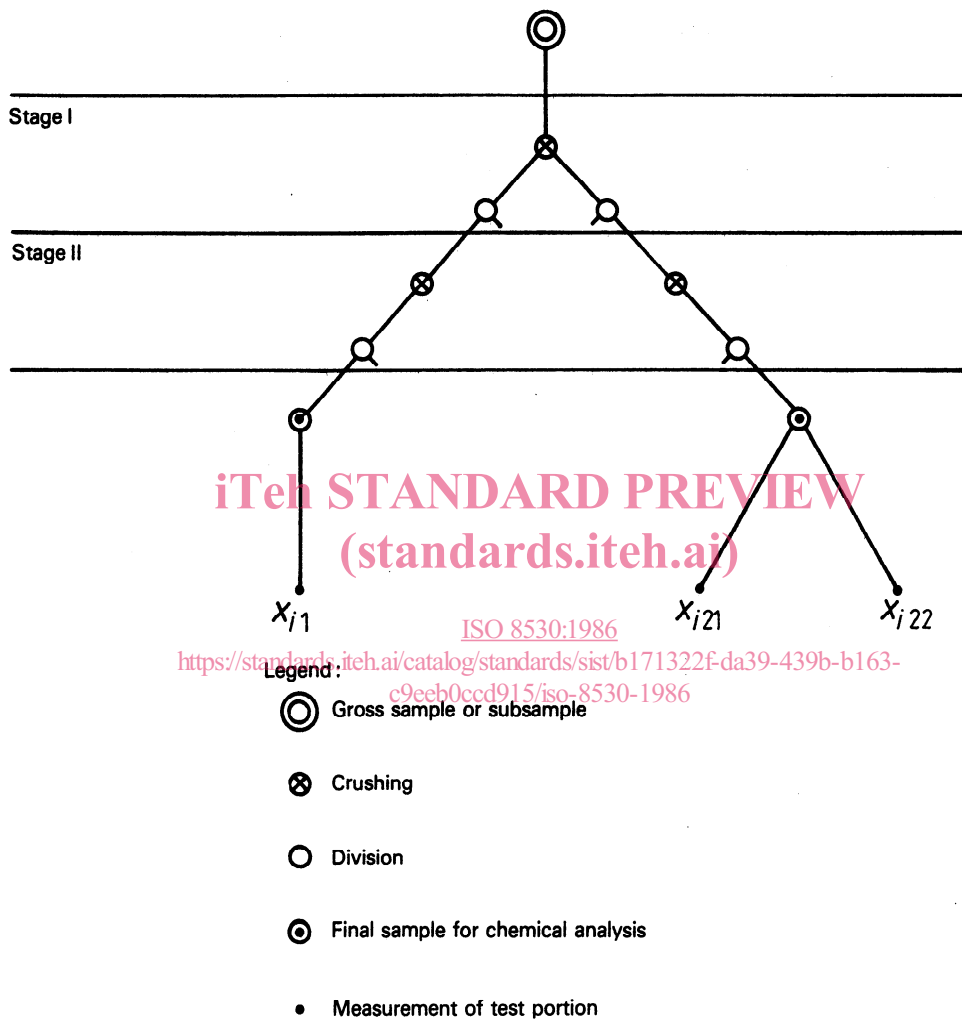


Figure 2 — Flow diagram for sample division in two stages (example)

Table – Data sheet for experiment of sample division (example)

Designation of experiment:					
Type of ore: (for example manganese ore)					
Name of method: (for example method specified in 4.1)					
Dates of experiment:					
Gross sample No.	Quality characteristic (for example % Mn)				
	x_{i1}	x_{i21}	x_{i22}	R_1	R_2
1.					
2.					
.					
.					
k.					
Sum				$\sum x_{i21} - x_{i22} $	$\sum x_{i1} - x_{i21} $ or $\sum x_{i1} - x_{i22} $
Mean range				\bar{R}_1	\bar{R}_2

Calculation

$$\hat{\sigma}_M^2 = (\bar{R}_1/1,128)^2 = \dots\dots\dots$$

$$\hat{\sigma}_D = \sqrt{(\bar{R}_2/1,128)^2 - \hat{\sigma}_M^2} = \dots\dots\dots$$

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