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Iron ores — Guidelines for the use of certified reference materials (CRMs)

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National foreword

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Minerais de fer — Lignes directrices pour l'utilisation des matériaux de référence certifiés

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Foreword

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ISO 16042 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

Introduction

This International Standard is intended for use in conjunction with other International Standards for the chemical analysis of iron ores. It describes recommended procedures for the use of iron ore certified reference materials (CRMs) in verifying the trueness of chemical analysis. It is recommended that CRMs also be used for general quality assurance purposes in laboratories where the analysis of iron ores is carried out regularly.

It is also recommended that CRMs be, from time to time, introduced as unknowns in laboratory proficiency testing schedules.

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Iron ores — Guidelines for the use of certified reference materials (CRMs)

1 Scope

This International Standard describes recommended procedures for the use of CRMs that have been prepared and certified in accordance with ISO 11459. Such CRMs are used by laboratories to carry out the trueness tests as specified in the various International Standards for the chemical analysis of iron ores, to verify the analysis of shipment samples and the ongoing reliability of analytical results.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 8258:1991, *Shewhart control charts*

ISO 11459, *Iron ores — Certified reference materials — Preparation and certification for use in chemical analysis*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

ISO Guide 30, *Terms and definitions used in connection with reference materials*

ISO Guide 33, *Uses of certified reference materials*

ISO Guide 35, *Reference materials — General and statistical principles for certification*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 and ISO Guide 30 apply.

4 Principle

The uses of CRMs as specified in this document are as follows:

a) to verify the trueness of analysis of shipment samples that are used to determine the commercial value of a cargo or any specified quantity of iron ore sampled according to ISO 3082 and delivered from the vendor to the purchaser;

b) to demonstrate the ongoing ability of a laboratory to provide analysis of a defined accuracy and precision;

c) to test the proficiency of a laboratory to carry out the chemical analysis of iron ores.

The use of CRMs for the purpose of standardization of titrants, or calibration of analytical equipment, is specifically excluded.

Further uses of certified reference materials are outlined in ISO Guide 33.

5 Ore types

International Standards for the chemical analysis of iron ores specify that the certified reference material be of a mineralogical ore type similar to the sample whose chemical constituents are to be determined. The mass-fraction ranges of the elements to be determined between the CRM and the test sample should be as specified in Clause 6.

The contributing ore sources derived from single or multiple mines, blended to produce a traded iron ore, may exhibit widely varying characteristics. It is therefore recommended that producers of iron ore are pro-active in ensuring that the blend of iron ores produced for international trade is represented by a specific CRM. Ideally, each traded ore type should be represented, with elemental mass fractions close to typical.

Procedures for the certification of CRMs in general are given in ISO Guide 35, and procedures for the certification of iron ore CRMs in particular are given in ISO 11459.

6 Mass-fraction difference

The allowable absolute differences in mass fractions of various elements between the test sample and the CRM can be calculated using the following equation for elemental mass fractions up to 10 %:

$$R_{\mathrm{c}} = C_{\mathrm{s}} \pm \frac{(-10{,}771 \ln C_{\mathrm{s}} + 25{,}4) C_{\mathrm{s}}}{200}$$

where

R_{c} is the mass-fraction range;

C_{s} is the mass fraction of sample.

An example of the calculation of mass-fraction difference is given in Annex A.

It is recommended that, for the mass fraction of total iron (% Fe), the CRM should not vary by more than 0,5 % Fe from the mass fraction of the test sample.

7 Extraction of the test portion

Iron ore CRMs are usually sold in 100 g lots. Typically, for the purpose of analysis, a test portion of 0,38 g to 1,00 g in mass is used. The extraction of the test portion is basically a sampling exercise, which should be carried out on the full quantity of the unit mass by which the CRM is supplied. The extraction of test portions from a diminishing quantity out of one bottle is not recommended.

It is recommended that the total contents of the bottle of CRM prior to its first use be divided into portions that match the intended test portions. Each portion shall be packed in an inert plastic or glass sealed container, with a tightly fitting screw-cap plastic lid, preferably with a plastic insert in the top of the bottle, or a heavy-duty plastic bag or vial having a tightly fitting lid. Plastic containers and caps/inserts should be checked to ensure that the electrostatic properties of the plastic do not cause particles to stick selectively to the plastic, thereby causing segregation of the material. Bottles shall not have lids with cardboard inserts, as these can cause contamination of the samples. Samples shall not be packed in paper bags, as the paper can contaminate samples used for low levels of certain elements. Containers should be labelled as follows:

a) identification by name or number of the CRM;

b) the serial number of the bottle in which the CRM was supplied; should the CRM manufacturer not have supplied a bottle number, it is recommended that the laboratory give each bottle its own number;

c) the serial number of the test portion as extracted.

EXAMPLE JSS 820-2/100/5 represents the fifth portion extracted out of bottle 100 from CRM JSS 820-2.

The division of the CRM shall be effected by rotary sample division or riffle splitter division. In each case, the minimum increments represented in a test portion should be 15 to 20 in number.

To minimize bias in sampling, test portions shall be taken at random.

8 Two-bottle strategy

The number of replicate analyses carried out on a CRM shall be a minimum of two. It is recommended that a duplicate be derived from separate bottles of the CRM.

9 Alteration of the CRM

Alterations to the mass fractions of the various certified data of a CRM can occur in a number of ways. The strategy outlined in Clause 7 assists in avoiding this problem or at least limiting it to a single occurrence. The two-bottle strategy (Clause 8) assists in the detection of the above. Good housekeeping is a basic laboratory requirement and should be in evidence in all laboratories.

The following rules apply:

a) magnetic spatulas and magnetized equipment shall not be used;

b) electrostatically charged equipment shall be discharged or its use avoided;

c) containers used for storage of CRMs shall not exhibit electrostatic surface charges;

d) vibrating spatulas shall not be used for dispensing test portions;

e) CRMs shall not be stored in drying ovens;

f) platinum ware used for fusions shall be clean and shall not cause any contamination;

g) glassware used in analytical determinations shall be clean and not cause contamination;

h) exposure to acid and other vapours shall be avoided;

i) storage in places subject to vibrations shall be avoided.

In all cases, CRMs shall be stored in accordance with manufacturers' recommendations.

10 Shewhart control charts

For each CRM used on a regular basis within a laboratory, Shewhart control charts shall be constructed in accordance with ISO 8258. Separate charts should be constructed for bottle 1 and bottle 2 or the different bottles should be identifiable on the chart. Whilst the trueness test for each use of the CRM still applies, the use of the Shewhart chart is recommended to identify outliers, trends and runs along the principles outlined in ISO 8258:1991, Figure 2 “Tests for assignable causes”. The Shewhart chart should be updated for every newly completed analysis of the CRM.

11 Acceptance of analytical values

The result obtained for the certified reference material shall be such that the difference between this result and the certified value of the certified reference material is statistically insignificant. For a certified reference material that has been analysed by at least 10 laboratories using method(s) that are comparable in both accuracy and precision, the following condition may be used to test the significance of the difference:

$$\left|A_{\mathrm{c}} - A\right| \leqslant 2\sqrt{\frac{S_{\mathrm{Lc}}^2 + \frac{S_{\mathrm{wc}}^2}{n_{\mathrm{wc}}}}{N_{\mathrm{c}}} + \sigma_{\mathrm{L}}^2 + \frac{\sigma_{\mathrm{d}}^2}{n}}$$

where

A_{c} is the certified value;

A is the result or the mean of results obtained for the certified reference material;

S_{Lc} is the between-laboratories standard deviation of the certifying laboratories;

S_{wc} is the within-laboratory standard deviation of the certifying laboratories;

n_{wc} is the average number of replicate determinations in the certifying laboratories;

N_{c} is the number of certifying laboratories;

n is the number of replicate determinations on the reference material (it is recommended that n = 2 as a minimum);

σ_{L} is the between-laboratory standard deviation, as derived from the precision equations of an ISO/TC102/SC 2 analytical method;

σ_{d} is the within-laboratory standard deviation, as derived from the precision equations of the relevant analytical method.

If the condition is satisfied, i.e. if the left-hand side is less than or equal to the right-hand side, then the difference $A_{\mathrm{c}} - A$ is statistically insignificant; otherwise it is statistically significant.

If the difference is significant, the analysis shall be repeated simultaneously with an analysis of the test sample. If the difference is again significant, the procedure shall be repeated using a different certified reference material of the same type of ore.

If the range of the two values for the test sample is outside the independent duplicate limit calculated according to the International Standard being used, one or more additional tests shall be carried out.

Acceptability of the results for the test sample shall, in each case, be subject to the acceptability of the results for the certified reference material.

It is not recommended that reference materials that have only been certified by one laboratory be used.

12 Test report

The analytical results of a CRM used to verify the analysis of a shipment sample shall be reported, together with the data obtained on the shipment sample if requested by the trading parties. The laboratory shall keep traceable records of CRM test results.

Annex A
(informative)

Example of the calculation of mass-fraction difference

Substituting the analyte mass-fraction range with 0,235 %:

$$R_c = 0,235 \pm \frac{(-10,771 \times \ln(0,235) + 25,4) \times 0,235}{200} \%$$

Solving for the natural logarithm of 0,235:

$$R_c = 0,235 \pm \frac{(-10,771 \times (-1,488\,2) + 25,4) \times 0,235}{200} \%$$

Solving the numerator:

$$R_c = 0,235 \pm \frac{9,634\,6}{200} \%$$

Solving for the mass-fraction range gives:

$$R_c = 0,235 \pm 0,048\ \%$$

Thus, at an analyte mass fraction of 0,235 %, the CRM mass fraction should preferably lie between 0,187 % and 0,283 %.

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