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Iron ores — Determination of the moisture content of a lot

Minerais de fer — Détermination de l'humidité d'un lot

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 1, *Sampling*.

This fifth edition cancels and replaces the fourth edition (ISO 3087:2011), which has been technically revised. The main changes compared with the previous edition are as follows:

— the existing two 105 °C moisture determination methods have been confirmed to serve as reference methods;

— alternative moisture determination methods are now allowed if they can be shown to result in equivalent moisture contents as the reference methods;

— the weighing device readability requirement has been changed from 0,05 % to 0,01 % equivalent of test portion mass;

— Clause 9 has been revised;

— Annex D has been updated with new example reports.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Currently, large tonnages of iron ore are traded internationally and a small error in the measured moisture content [mass fraction (%)] of a lot has a considerable effect on the commercial transaction. The correct determination of moisture content of a lot is, therefore, a matter of importance for both the purchaser and the vendor.

This document does not address the determination of the hygroscopic moisture content of a test sample for chemical analysis. If the hygroscopic moisture content is required to be determined, reference should be made to ISO 2596:2006.

Iron ores — Determination of the moisture content of a lot

1 Scope

This document specifies a method for the determination of the moisture content of a lot of iron ore. This method is applicable to all iron ores, whether natural or processed.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3085, *Iron ores — Experimental methods for checking the precision of sampling, sample preparation and measurement*

ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling*

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

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp
— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Dry the test portion in air at 105 °C and measure the loss in mass. Express the moisture content as the mass loss relative to the original mass of the sample as a mass fraction (%).

5 Apparatus

5.1 Drying pan, with a smooth surface, free from contamination and capable of accommodating the specified quantity of a test portion in a layer of nominal thickness not greater than 31,5 mm.

5.2 Drying oven, equipped with a temperature indicator and control apparatus capable of regulating the temperature at any point in the oven at 105 °C ± 5 °C and so designed as to maintain this temperature with a current of air to ensure efficient drying but without any loss of sample, and fitted with a fan that allows for both the circulation and change of air.

5.3 Weighing device, with readability equivalent to at least 0,01 % of the Table 1 minimum test portion mass. The weighing device should be protected from the influence of heat.

The capacity of the weighing device shall be enough for the combined mass of the drying pan and the initial mass of the test portion.

6 Samples

Test samples that have been taken and prepared in accordance with ISO 3082 shall be used. The mass of a test portion, in relation to its nominal top size, is specified in Table 1, in accordance with ISO 3082:2017, 10.6.

The nominal top size of the moisture test sample shall be 31,5 mm or less. Samples with a nominal top size greater than 31,5 mm shall be crushed prior to extraction of test samples for moisture determination. When it is difficult to conduct crushing and dividing owing to a sample being adhesive or excessively wet, the sample may be partially dried in accordance with the procedure in Annex A.

For convenience, the test portion of mass 10 kg for ore of particle size less than 31,5 mm may be divided into two portions, each of which is subjected to moisture measurement. In calculating the results, the mean of the two values of initial mass and the mean of the two values of the drying loss in mass should be used.

Table 1 — Minimum mass of test portion

Nominal top size of test portion mm		Minimum mass of test portion kg
Over	Up to and including	
22,4	31,5	10
10,0	22,4	5
—	10,0	1

7 Procedure

7.1 Number of moisture measurements

Carry out one moisture measurement per test portion on the number of test portions specified in Table 2, in accordance with the conditions of preparation of the test sample.

In order to minimize losses of moisture to the atmosphere, it is necessary to perform all the initial weighings of the test portions as quickly as possible after obtaining those test portions.

Table 2 — Number of test portions

Preparation of test sample	Number of partial samples per lot	Number of test portions to be tested
From gross sample	—	4 per gross sample
From partial sample	2	4 per partial sample
	3 to 7	2 minimum per partial sample
	≥ 8	1 minimum per partial sample
From increment	—	1 minimum per increment

7.2 Reference methods

7.2.1 General

The moisture determination shall be conducted in accordance with 7.2.2 or, optionally for ores with 8 % or more of combined water, in accordance with 7.2.3.

7.2.2 Normal reference method

Apply the following procedure.

a) Spread the test portion in a layer of nominal thickness not greater than 31,5 mm in the tared drying pan (5.1) and determine the total mass immediately. Record the total mass, the mass of the drying pan, the initial mass of the test portion (m_1) and the numerical value of 0,05 % of m_1. To reduce drying time, it is recommended that the layer-thickness of the sample be kept as low as possible. It should be specified for particular ores by check experiments carried out beforehand.

b) Place the drying pan with the test portion in the drying oven (5.2) set at 105 °C, and maintain this temperature for not less than 4 h. Remove the drying pan with the test portion from the drying oven and weigh it immediately while still hot in order to minimize any reabsorption of moisture. Otherwise, weigh the test portion after cooling in air in a container having a close-fitting airtight lid. In each case, report the method of weighing.

c) Once more, place the drying pan with the test portion in the drying oven, heat for a further 1 h, and then repeat the weighing.

d) Repeat the procedure described in item c) until the difference in mass between subsequent measurements becomes 0,05 % or less of the initial mass of the test portion. Record the total mass and the mass of the test portion after each additional hour of drying (m_2 and onwards).

7.2.3 Optional reference method for ores of high combined water content

For ores containing 8 % or more combined water, the following procedure may be applied.

a) Spread the test portion in a layer of nominal thickness not greater than 31,5 mm in the tared drying pan (5.1) and determine the total mass immediately. Record the total mass, the mass of the drying pan and the initial mass of the test portion (m_1).

b) Place the drying pan with the test portion in the drying oven (5.2) set at 105 °C and maintain this temperature for not less than 24 h. Remove the drying pan with the test portion from the drying oven and weigh it immediately while still hot in order to minimize any reabsorption of moisture. Otherwise, weigh the test portion after cooling in air in a container having a close-fitting airtight lid. Record the total mass and the mass of the test portion after drying (m_2). In each case, report the method of weighing.

7.3 Alternative methods

7.3.1 General

Alternative methods refer to the potential use of different technology, such as ovens that do not rely on convection drying alone, or to procedures that potentially differ in terms of oven temperature (up to 140 °C) and time from what is described in 7.2, but which do not deviate from the conditions stipulated in Clause 6 and 7.1 (minimum mass of test portion and number of portions to be tested).

Such alternative methods or modifications for determining the moisture content are allowed where check experiments carried out beforehand, which demonstrate satisfactorily to all parties concerned that an equivalent moisture result is obtained when compared to the applicable reference method specified in 7.2. This demonstration shall include ISO 3086 bias testing using a relevant bias of no more than 0,05 % moisture and where the confidence interval includes the zero. Further, the precision of the alternative method shall be demonstrated by ISO 3085 to be the same or better than achieved by the applicable reference method.

If the sample is subsequently to be used for multiple use, the chemistry, LOI and size distribution of the dried remains shall be checked by means of ISO 3086 to ensure they are not different to dried remains from equivalent reference moisture testing methods. If any changes are identified, the alternative method shall be discarded.

7.3.2 Adjustment of drying time only

In the case where an alternative method changes only the drying times specified in 7.2.2 in order to eliminate repeat weighings for verification of constant mass, then it is only necessary to demonstrate equivalence of precision and moisture result.

8 Verification

Regular checking of apparatus and procedures is essential to verify the test results. Checks shall be carried out prior to the commencement of a routine test in accordance with this document and at regular intervals thereafter. The frequency of checking is a matter for each laboratory to determine. A detailed record of all verification activities shall be maintained for the following items:

— sprinkled water measurement:

  — volumenometer;

— rainfall measurement:

  — rain gauge;

— moisture test:

  — oven temperature/temperature regulation;

  — circulation and change of air in oven;

  — weighing device.

9 Calculation and expression of results

9.1 Test portion

The result of the determination of the moisture content, w_i, expressed as a mass fraction (%), for each test portion, is given by Formula (1) and reported to the second decimal place:

$$w_i = \frac{m_1 - m_2}{m_1} \times 100 \quad (1)$$

where

m_1 is the initial mass, in grams, of the test portion;

m_2 is the mass, in grams, of the test portion after drying.

An illustration of moisture determination of a random test portion is shown in an example test report in Table D.1.

9.2 Lot

9.2.1 The moisture content of a lot is given by one of the Formulae (2) to (6), as the occasion may demand, and reported to the first decimal place.

Sprinkled water and/or rainwater over iron ore during loading and/or unloading operation, i.e. moisture added after/before the point where sampling has taken place, shall be corrected in accordance with the procedure specified in Annex B.

9.2.2 When moisture determination is conducted on the gross sample from the lot, the moisture of the lot is determined as follows.

When the range of the four test results does not exceed 1,3*r*, as given in Table 3, the arithmetic mean $\bar{w}$ of the four results shall be the moisture content, expressed as a mass fraction (%), of the lot as given by Formula (2):

$$\bar{w} = \frac{w_1 + w_2 + w_3 + w_4}{4} \tag{2}$$

where w_1, w_2, w_3 and w_4 are the results of the determinations of the moisture contents, expressed as a mass fraction (%), on each of the four test portions.

When the range of the four test results exceeds 1,3*r*, as given in Table 3, the median shall be taken as the moisture content of the lot. The median of four test results is defined as the mean of the two non-extreme test results.

Table 3 — Repeatability limit of moisture determination on the gross sample

Average of moisture content $\bar{w}$ mass fraction (%)	Repeatability limit r[a] mass fraction (%)	Repeatability limit 1,3r mass fraction (%)
$\bar{w} \leq 3$	0,20	0,26
$3 < \bar{w} \leq 6$	0,25	0,33
$6 < \bar{w}$	0,31	0,40

[a] The theoretical background of the repeatability limit is shown in Annex C.

An illustration of moisture determination of a gross sampled lot with four test portions, where the range of the four moistures do not exceed 1,3r, is shown in an example test report in Table D.2.

An illustration of moisture determination of a gross sampled lot with four test portions, where the range of the four moistures exceed 1,3*r*, is shown in an example test report in Table D.3.

9.2.3 When mass-basis sampling has been performed and moisture determination is conducted on each partial sample, the weighted mean, $\overline{\overline{w}}$, of the results from all the partial samples, considering the number of increments for each partial sample, shall be the moisture content, expressed as a mass fraction (%), of the lot, as given by Formula (3):

$$\overline{\overline{w}} = \frac{\sum_{i=1}^{k} N_i \bar{w}_i}{\sum_{i=1}^{k} N_i} \tag{3}$$

where

k is the number of partial samples;

N_i is the number of increments in the ith partial sample;

$\bar{w}_i$ is the result of the determination of the moisture content, expressed as a mass fraction (%), of the ith partial sample, in accordance with Table 2, using either four or two as the number of test portions.

When partial samples are used and four test portions are conducted in any partial sample, the criteria established in 9.2.2 may be applied.

Illustrations of moisture determination of mass-basis sampled lots with two, four and eight partial samples are shown in the example test reports given in Annex D in Tables D.4, D.6 and D.8, respectively.

If it is impracticable to sample the lot as a whole, or desirable to sample a lot in separate parts of unequal mass as in the case of time-basis sampling, the moisture content of each part shall be determined independently and the weighted mean, $\overline{\overline{w}}$, of the results, expressed as a mass fraction (%), of the lot calculated from the individual results using Formula (4):

$$\overline{\overline{w}} = \frac{\sum_{i=1}^{k} m_i \bar{w}_i}{\sum_{i=1}^{k} m_i} \tag{4}$$

where

k is the number of partial samples;

m_i is the mass of the ith part;

$\bar{w}_i$ is the result of the determination of the moisture content, expressed as a mass fraction (%), of the ith part.

Illustrations of moisture determination of time-basis sampled lots with two, four and eight partial samples are shown in the example test reports given in Annex D, in Tables D.5, D.7 and D.9, respectively.

9.2.4 When moisture determination is conducted on each increment during mass basis sampling, the arithmetic mean, $\bar{w}$, of the results for all increments obtained in accordance with 9.1 shall be the moisture content, expressed as a mass fraction (%), of the lot as given by Formula (5):

$$\bar{w} = \frac{\sum_{i=1}^{n} w_i}{n} \tag{5}$$

where

n is the number of increments;

w_i is the result of the determination of the moisture content, expressed as a mass fraction (%), of the ith increment.

An illustration of moisture determination of a mass-basis sampled lot where moisture is individually determined for each of 30 increments, is shown in an example test report in Table D.10.

When moisture determination is conducted on each increment during time basis sampling, the weighted mean, $\overline{\overline{w}}$, of the results for all increments obtained in accordance with 9.1 shall be the moisture content, expressed as a mass fraction (%), of the lot as given by Formula (6):

$$\overline{\overline{w}} = \frac{\sum_{i=1}^{k} m_i w_i}{\sum_{i=1}^{k} m_i} \tag{6}$$

where

