

BS ISO 13312:2017



BSI Standards Publication

Iron ores — Determination of potassium — Flame atomic absorption spectrometric method

National foreword

This British Standard is the UK implementation of ISO 13312:2017. It supersedes BS ISO 13312:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

A list of organizations represented on this committee can be obtained on request to its secretary.

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© The British Standards Institution 2017.
Published by BSI Standards Limited 2017

ISBN 978 0 580 93912 9

ICS 73.060.10

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2017.

Amendments/corrigenda issued since publication

Date	Text affected
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INTERNATIONAL
STANDARD

ISO
13312

Third edition
2017-03

**Iron ores — Determination of
potassium — Flame atomic absorption
spectrometric method**

*Minerais de fer — Dosage du potassium — Méthode par
spectrométrie d'absorption atomique dans la flamme*



Reference number
ISO 13312:2017(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 on 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 the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 13312:2006), of which it constitutes a minor revision with the following changes:

- in [7.2](#), a new sentence to make reference to ISO 2596 has been included;
- in [9.2.4](#), [Formula \(7\)](#) and the relevant descriptions have been modified to harmonize this subclause across all International Standards for which ISO/TC 102/SC 2 is responsible;
- in [5.4](#), [5.5](#), [8.4.2](#) and [9.2.1](#), the footnotes have been moved to the appropriate place.

Iron ores — Determination of potassium — Flame atomic absorption spectrometric method

WARNING — This document may involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of the mass fraction of potassium in iron ores.

This method is applicable to mass fractions of potassium between 0,002 5 % and 0,52 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

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 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

The test portion is decomposed by treatment with hydrochloric acid and hydrofluoric acid, followed by evaporation to dryness. The residue is wetted and the evaporation is repeated with a new portion of hydrochloric acid. The residue is dissolved with hydrochloric acid followed by appropriate dilution. The solution is aspirated into the air/acetylene flame of the atomic absorption apparatus.

The absorbance value obtained for potassium is measured in comparison with those obtained from calibration solutions.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only water that complies with Grade 2 of ISO 3696.

Reagents are to be selected or purified for the lowest possible blank value.

5.1 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml.

5.2 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (mass fraction), or ρ 1,19 g/ml, 48 % (mass fraction).

5.3 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 2.

5.4 Background solution.

Dissolve 43 g of high-purity iron oxide powder in 500 ml of hydrochloric acid (5.1). Allow to cool and dilute with water to 1 000 ml.

Instead of iron oxide, the use of metallic iron with a suitable oxidant is permitted. The alkali content of the oxidant shall be low.

5.5 Potassium, standard solution, 20 μg K/ml.

Pulverize about 3 g of high-purity potassium chloride in an agate mortar, dry in an oven at 105 °C to 110 °C for 2 h, and allow to cool to room temperature in a desiccator. Dissolve 1,907 g in water, dilute with water to 1 000 ml in a volumetric flask and mix.

Transfer 10,0 ml of this solution to a 500 ml volumetric flask, dilute with water to volume and mix.

NOTE Glass equipment can be used.

Store this standard solution in a plastic bottle.

1 ml of this standard solution contains 20 μg of potassium.

6 Apparatus

Ordinary laboratory equipment, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042, respectively, and the following.

6.1 Polytetrafluoroethylene (PTFE) beakers, of capacity 100 ml, provided with PTFE covers.

6.2 PTFE-coated magnetic stirring bars.

6.3 PTFE digestion bomb.

6.4 Plastic pipettes.

6.5 Plastic volumetric flasks and storage bottles.

6.6 Magnetic stirring hotplates.

NOTE Platinum vessels can be used instead of PTFE beakers.

Except where stated, glass equipment should be avoided, as it could contaminate the solutions.

To obtain reliable values, the equipment should be cleaned and checked as follows.

- a) Rinse all volumetric ware, including the pipettes used for preparing the calibration solutions, with dilute hydrochloric acid (5.3) before use. Check the calibration regularly or as needed.
- b) Clean PTFE vessels and stirring bars by stirring with 50 ml of dilute hydrochloric acid (5.3) and by heating for 15 min. Discard the rinsings and conduct a blank test in each vessel in turn, exactly as specified in 8.3. If any absorbance value is above the limit specified in 8.3, the cleaning procedure should be repeated or acid reagents of a higher purity should be used. At no stage should the stirring bars be handled with the fingers.
- c) Platinum vessels, exclusively used for potassium analysis according to this document, can be cleaned by the same method as the PTFE vessels [see b)]. Otherwise, they should be pre-cleaned by fusion with lithium tetraborate or lithium borate, until the absorbance readings fall to those for the lithium salt alone.
- d) Rinse storage bottles with dilute hydrochloric acid (5.3) before use.

6.7 Atomic absorption spectrometer.

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the air/acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the burner is in operation.

The atomic absorption spectrometer shall meet the following criteria.

- a) Minimum sensitivity: The absorbance of the most concentrated calibration solution (see 8.4.4) shall be at least 0,25.
- b) Graph linearity: The slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be less than 0,7 of the value of the slope for the bottom 20 % of the concentration range determined in the same way.
- c) Minimum stability: The standard deviation of the absorbance of the most concentrated calibration solution and that of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, shall be less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated calibration solution.

The use of a strip-chart recorder and/or digital readout device is recommended to evaluate criteria a), b) and c) and for all subsequent measurements.

NOTE Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into an air/acetylene flame of a premix burner.

— hollow cathode lamp, mA	10
— wavelength, nm	766,5
— air flow-rate, l/min	10
— acetylene flow-rate, l/min	2

In systems where the values shown for gas flow-rates do not apply, the ratio of the gas flow-rates can still be a useful guideline.

7 Sampling and samples

7.1 Laboratory sample

For analysis, use a laboratory sample of $-100\ \mu\text{m}$ particle size which has been taken and prepared in accordance with ISO 3082. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of $-160\ \mu\text{m}$.

NOTE A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

7.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test sample at $105\ \text{°C} \pm 2\ \text{°C}$ as specified in ISO 7764. (This is the predried test sample.)

For ores having significant content of combined water or oxidizable compounds, an air-equilibrated test sample shall be prepared in accordance with ISO 2596.

8 Procedure

8.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with [Annex A](#), independently, on one predried test sample.

NOTE The expression “independently” means that the second and any subsequent result are not affected by the previous result(s). For the particular analytical method, this condition implies that the repetition of the procedure is carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

8.2 Test portion

Taking several increments, weigh, to nearest 0,000 2 g, 0,2 g to 0,5 g (depending on the potassium concentration) of the predried test sample obtained in accordance with [7.2](#).

The test portion should be taken and weighed quickly to avoid reabsorption of moisture.

8.3 Blank test and check test

Before proceeding to the treatment of test portions, ensure that the cleaning procedures conducted in [6.6](#) (see list items), together with the quality of the reagents being used, have produced a blank test value for the potassium determination not greater than the equivalent of 0,002 % (mass fraction) potassium in the ore.

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in [7.2](#).

Where the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents are from the same reagent bottles.

Where the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that, in either case, no significant

changes in the analytical procedure will become necessary. Where a certified reference material is not available, a reference material may be used (see 9.2.4).

8.4 Determination

8.4.1 General

To prevent contamination during analysis, the following precautions shall be taken:

- a) finger contact with sample, solutions and stirring bars shall be avoided;
- b) mouth suction of pipettes shall not be permitted.

8.4.2 Decomposition of the test portion

Transfer the test portion (see 8.2) to a 100 ml PTFE beaker (6.1). See Note in 6.6.

Moisten it with a few drops of water, then add 10 ml of hydrochloric acid (5.1) and 10 ml of hydrofluoric acid (5.2). Add a PTFE-coated magnetic stirring bar (6.2) and cover with a PTFE cover. Adjust the temperature of the magnetic stirring hotplate (6.6) so that a temperature of about 98 °C will be maintained in water in a covered PTFE beaker. Heat, with stirring, for 45 min or until no further dissolution of the test portion occurs. Remove the cover, stop the stirrer, leaving the bar in the solution, and evaporate to dryness. Add 5 ml of hydrochloric acid (5.1) and evaporate again to dryness. Dissolve the salts in 5 ml of hydrochloric acid (5.1) and 40 ml of water, and transfer to a 100 ml one-mark plastic volumetric flask (6.5). Dilute to volume with water and mix.

NOTE If any significant amount of residue remains, conduct the digestion process in a stirred PTFE digestion bomb (6.3) for 45 min at 160 °C.

8.4.3 Treatment of the solution

If the concentration of potassium is too high, it is necessary to dilute the test solution. Transfer, using a plastic pipette (6.4), y ml of the test solution to a 100 ml one-mark plastic volumetric flask, add $0,1 \times (100 - y)$ ml of the background solution (5.4), dilute with water to volume and mix (see Table 1).

A diluted test solution shall be measured together with a diluted blank test solution, containing the same amount of background solution as the test solution. Prepare the diluted blank test solution as follows: pipette y ml of the blank test solution into a 100 ml one-mark plastic volumetric flask, add $0,1 \times (100 - y)$ ml of the background solution, dilute to volume with water and mix.

Table 1 — Dilution guide for test solution

Potassium mass-fraction range %	Aliquot from 100 ml y
0,002 5 to 0,060	—
0,060 to 0,20	30,0
0,20 to 0,52	10,0

8.4.4 Preparation of the set of calibration solutions

From the potassium standard solution (5.5), prepare calibration solutions as follows.

Using plastic pipettes, transfer 0 ml; 2,0 ml; 5,0 ml; 10,0 ml; 15,0 ml of potassium standard solution (5.5), respectively, to 100 ml one-mark plastic volumetric flasks. Add, using a plastic pipette, 10 ml of the background solution (5.4) to each, dilute with water to volume and mix. These calibration solutions cover the concentration range 0 µg K/ml to 3 µg K/ml and contain 3 000 µg Fe/ml.

Store the calibration solutions in plastic bottles.

8.4.5 Adjustment of atomic absorption spectrometer

Optimize the response of the instrument as specified in 6.7. Set the wavelength of potassium (766,5 nm) to obtain minimum absorbance. After 10 min preheating of the burner, adjust the fuel flow and burner position to obtain maximum absorbance, while aspirating the most concentrated calibration solution (see 8.4.4). Aspirate water and the calibration solution to establish that the absorbance reading is not drifting, and then set the reading for water to zero absorbance.

8.4.6 Atomic absorption measurements

Aspirate the calibration and test solutions or diluted test solutions in order of increasing absorption, starting with the blank test solution, or diluted blank test solution, and the zero calibration solution. When a stable response is obtained for each solution, record the readings. Aspirate the test solutions or diluted test solutions at the proper points in the calibration series and record their readings. Aspirate water between each calibration and test solution. Repeat the measurements at least twice.

Obtain the net absorbance of each calibration solution by subtracting the average absorbance of the zero calibration solution. In a similar manner, obtain the net absorbance of the test solution or diluted test solution by subtracting the absorbance of the corresponding blank test solution.

Prepare calibration graphs by plotting the net absorbance values of the calibration solutions against the concentration, in micrograms of potassium per millilitre (the test solution or, if diluted, the diluted test solution is the final test solution).

Convert the net absorbance of the final test solution to micrograms of potassium per millilitre by means of the calibration graph.

With concentration readings, the calculation should be made from absorbances to permit checking of the graph's linearity and the blank test value.

9 Expression of results

9.1 Calculation of mass fraction of potassium

The mass fraction of potassium, expressed as a percentage, is calculated to five decimal places for mass fractions higher than 0,01 %, and to six decimal places for mass fractions lower than 0,01 %, using [Formula \(1\)](#):

$$w_K = \frac{\rho_M}{m_1 \times 100} \quad (1)$$

where

w_K is the mass fraction of potassium, expressed as a percentage, of the test sample;

ρ_M is the concentration, in micrograms per millilitre, of potassium in the final test solution;

m_1 is the mass, in grams, of test sample represented in 100 ml of the final test solution ([8.4.6](#)), calculated from the following formula:

$$m_1 = \frac{m \times V}{100}$$

m being the mass, in grams, of the test portion (see [8.2](#));

V being the volume, in millilitres, of the aliquot taken in [8.4.3](#). If no dilution has been made, $V = 100$.

9.2 General treatment of results

9.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by the following regression formulae:

NOTE Additional information is given in [Annex B](#) and [Annex C](#).

$$R_d = 0,024\ 3\ X^{0,565\ 3} \quad (2)$$

$$P = 0,044\ 0\ X^{0,590\ 8} \quad (3)$$

$$\sigma_d = 0,008\ 6\ X^{0,565\ 3} \quad (4)$$

$$\sigma_L = 0,014\ 3\ X^{0,596\ 9} \quad (5)$$

where X (i.e. w_K) is calculated as follows:

- for the within-laboratory [Formula \(2\)](#) and [Formula \(4\)](#), the arithmetic mean of the duplicate values;
- for the between-laboratories [Formula \(3\)](#) and [Formula \(5\)](#), the arithmetic mean of the final results ([9.2.5](#)) of the two laboratories;

R_d is the independent duplicate limit;

P is the permissible tolerance between laboratories;

σ_d is the independent duplicate standard deviation;

σ_L is the between-laboratories standard deviation.

9.2.2 Determination of analytical result

Having computed the independent duplicate results according to [Formula \(1\)](#), compare them with the independent duplicate limit (R_d), using the procedure given in [Figure A.1](#), and obtain the final laboratory result μ (see [9.2.5](#)).

9.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final results reported by two laboratories. The assumption is that both laboratories followed the same procedure as described in [9.2.2](#).

Compute the following quantity using [Formula \(6\)](#):

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad (6)$$

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

μ_{12} is the mean of the final results.

Substitute μ_{12} for X in [Formula \(3\)](#) and calculate P .

If $|\mu_1 - \mu_2| \leq P$, the final results are in agreement.

9.2.4 Check for trueness

The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM) or a reference material (RM) (see last paragraph of 8.3). Calculate the analytical result (μ_c) for the RM/CRM using the procedures in 8.1 and 8.2, and compare it with the reference or certified value A_c . There are two possibilities:

- a) $|\mu_c - A_c| \leq C$, in which case, the difference between the reported result and the reference/certified value is statistically insignificant.
- b) $|\mu_c - A_c| > C$, in which case, the difference between the reported result and the reference/certified value is statistically significant.

where

μ_c is the analytical result for the certified reference material;

A_c is the certified/reference value for the CRM/RM;

C is a value dependent on the type of CRM/RM used.

Certified reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35.

C shall be calculated using [Formula \(7\)](#):

$$C = 2 \sqrt{\frac{s_{Lc}^2 + \frac{s_{Wc}^2}{n_{Wc}}}{N_c} + \sigma_L^2 + \frac{\sigma_d^2}{n}} \quad (7)$$

where

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 carried out on the CRM/RM;

σ_L and σ_d are as defined in [9.2.1](#).

The following procedure should be used when the information on the reference material certificate is incomplete:

- if there are sufficient data to enable the between-laboratories standard deviation to be estimated, delete the expression s_{Wc}^2 / n_{Wc} and regard s_{Lc} as the standard deviation of the laboratory means;
- if the certification has been made by only one laboratory or if the interlaboratory results are missing, use [Formula \(8\)](#):

$$C = 2 \sqrt{2\sigma_L^2 + \frac{\sigma_d^2}{n}} \quad (8)$$

A CRM certified by only one laboratory should be avoided unless it is known to have an unbiased certified value.

9.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations specified in [Annex A](#), calculated to five decimal places for mass fractions of potassium higher than 0,01 % and to six decimal places for mass fractions lower than 0,01 %. For mass fractions higher than 0,01 %, the value is rounded off to the third decimal place as specified in a), b) and c). In a similar manner, with the ordinal numbers increased by one, the value for mass fractions lower than 0,01 % is rounded off to the fourth decimal place.

- a) If the figure in the fourth decimal place is less than 5, it is discarded and the figure in the third decimal place is kept unchanged.
- b) If the figure in the fourth decimal place is 5 and there is a figure other than 0 in the fifth decimal place, or if the figure in the fourth decimal place is greater than 5, the figure in the third decimal place is increased by one.
- c) If the figure in the fourth decimal place is 5 and the figure 0 is in the fifth decimal place, the 5 is discarded and the figure in the third decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

9.3 Oxide factor

The oxide factor, expressed as a percentage, is given by [Formula \(9\)](#):

$$w_{K_2O} = 1,2046 \times w_K \quad (9)$$

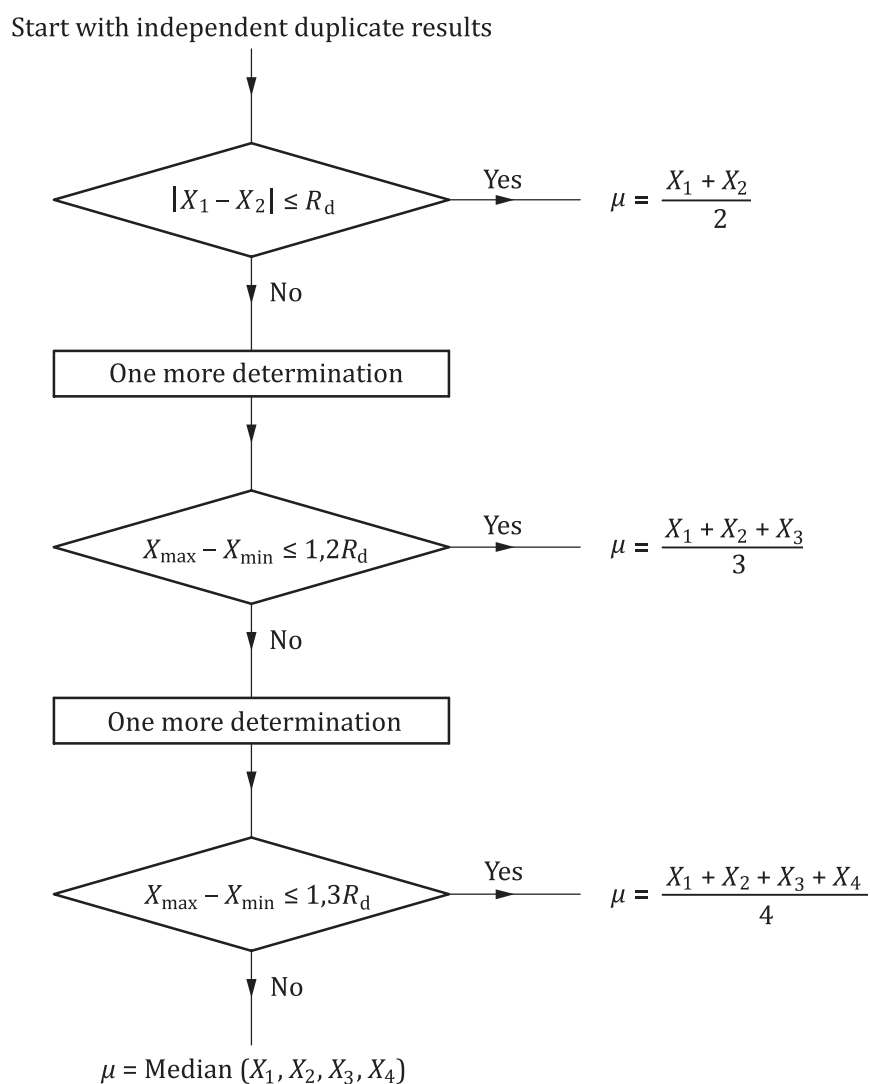
10 Test report

The test report shall include the following information:

- a) the name and address of the testing laboratory;
- b) the date of issue of the test report;
- c) a reference to this document, i.e. ISO 13312;
- d) the details necessary for the identification of the sample;
- e) the result of the analysis;
- f) the reference number of the result;
- g) any characteristics noticed during the determination and any operations not specified in this document which may have had an influence on the results, either for the test sample or for the certified reference material(s).

Annex A (normative)

Flowsheet of the procedure for the acceptance of analytical values for test samples



Key

R_d permissible tolerance within a laboratory (repeatability)

Figure A.1 — Flowsheet of the procedure for the acceptance of analytical values for test samples

Annex B (informative)

Derivation of repeatability and permissible tolerance formulae

The regression formulae in [9.2.1](#) were derived from the results of international analytical trials carried out in 1976/1979 on five iron ore samples, involving 39 laboratories in nine countries.

The graphical treatment of the precision data is given in [Annex C](#).

The test samples used are listed in [Table B.1](#).

Table B.1 — Mass fraction of potassium in test samples

Sample	Mass fraction of potassium %
Dampier	0,002 5
Schefferville	0,026 4
Haksberg, concentrate	0,074 1
Malmberget	0,216
Grangesberg	0,511

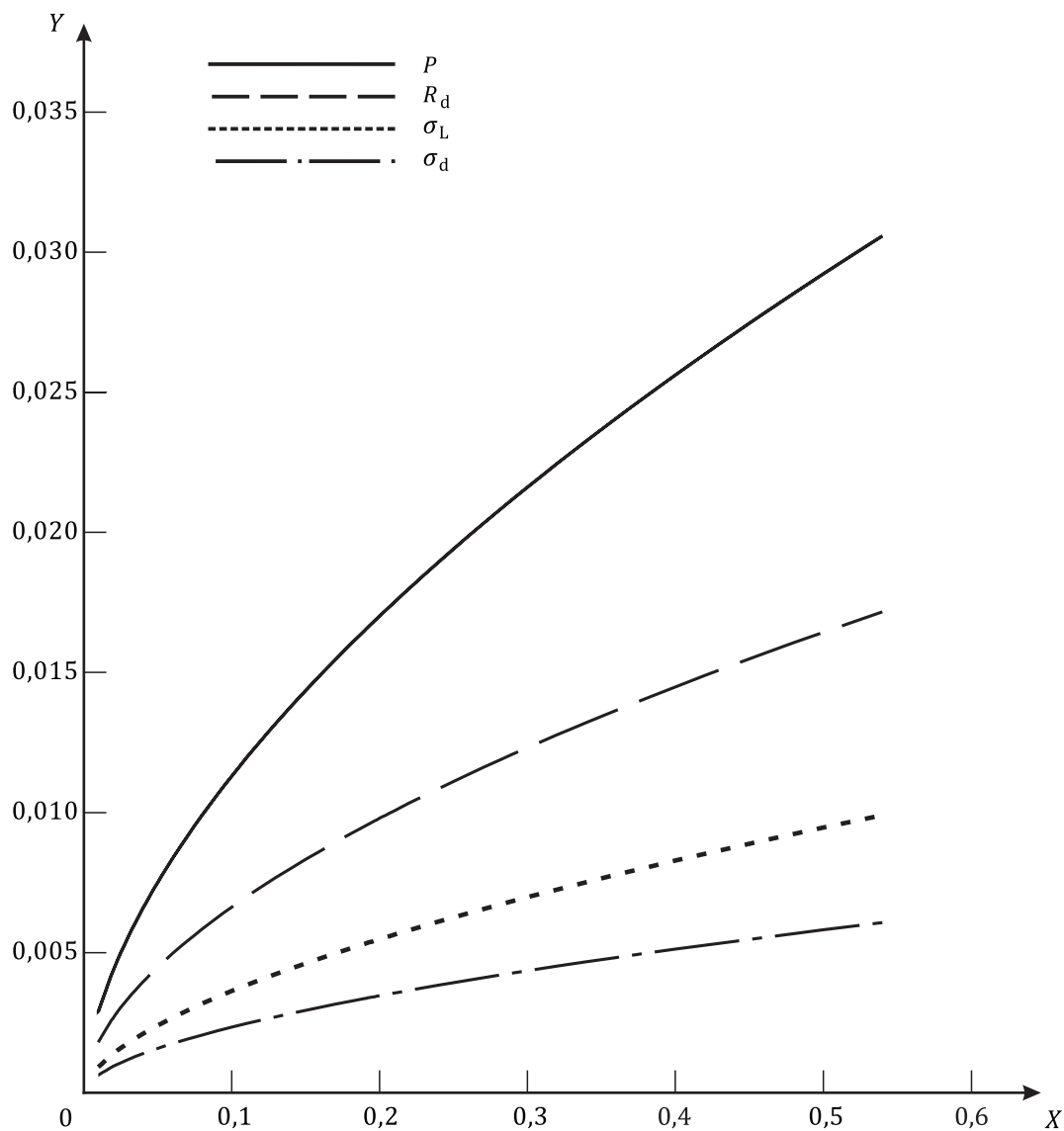
NOTE 1 A report of the international trial and a statistical analysis of the results (Document ISO/TC 102/SC 2 N 509E, June 1979) is available from the secretariat of ISO/TC 102/SC 2.

NOTE 2 The statistical analysis was performed in accordance with the principles embodied in ISO 5725-2.

Annex C (informative)

Precision data obtained by international analytical trials

NOTE [Figure C.1](#) is a graphical presentation of the formulae in [9.2.1](#).



Key

X mass fraction of potassium, %

Y precision, %

Figure C.1 — Least-squares fit for precision against X for potassium

Bibliography

- [1] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*
- [2] ISO Guide 35, *Reference materials — General and statistical principles for certification*

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