
**Iron ores — Determination of arsenic
content — Hydride generation atomic
absorption spectrometric method**

*Minerais de fer — Dosage de l'arsenic — Méthode par spectrométrie
d'absorption atomique à génération d'hydrure*

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

Iron ores — Determination of arsenic content — Hydride generation atomic absorption spectrometric method

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

1 Scope

This International Standard specifies a hydride generation atomic absorption spectrometric method for the determination of the arsenic content of iron ore.

This International Standard is applicable to mass fractions of arsenic between 0,000 66 % and 0,020 15 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 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 Guide 35:2006, *Reference materials — General and statistical principles for certification*

3 Principle

The test portion is decomposed by treatment with hydrochloric and nitric acid. The residue is treated with sodium peroxide and sodium carbonate. Potassium iodide reduces As(V) to As(III) and ascorbic acid masks the effect of iron in the solution. At optimum acidity of around 26 %, the solution and sodium borohydride react to generate gaseous arsenic hydride. An inert carrier gas is used to transport the arsenic hydride to a quartz tube atomizer. The equipment is set to measure the absorbances at 197,3 nm. The absorbances of the test and calibration solutions, including those of certified or other reference materials, are compared to determine the arsenic content.

4 Reagents

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

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

4.1 Sodium peroxide (Na₂O₂).

4.2 Sodium carbonate (Na_2CO_3), anhydrous powder.

4.3 Sodium bicarbonate (NaHCO_3).

4.4 Sodium hydroxide (NaOH).

4.5 Iron oxide (Fe_2O_3).

4.6 Arsenic trioxide (As_2O_3).

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

4.8 Nitric acid, ρ 1,42 g/ml.

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

4.10 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2+98.

4.11 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 12+88.

4.12 Sodium hydroxide solution, 40 g/l.

Dissolve 2 g of sodium hydroxide to 10 ml of water; dilute to 50 ml.

4.13 Sodium borohydride solution, 8 mg/ml.

Dissolve 0,5 g of sodium hydroxide to 10 ml of water, then dissolve 0,8 g of sodium borohydride [$\text{NaBH}_4 > 95 \% (m/m)$] in the 50 ml beaker contained sodium hydroxide solution; dilute to 100 ml. This solution shall be freshly prepared and used immediately.

4.14 Potassium iodide solution, 100 mg/ml.

Dissolve 10 g of potassium iodide [$\text{KI} > 98,5 \% (m/m)$] in 20 ml of water. Dilute to 100 ml.

4.15 Ascorbic acid solution, 100 mg/ml.

Dissolve 10 g of ascorbic acid [$\text{C}_6\text{H}_8\text{O}_6 > 99,5 \% (m/m)$] in 20 ml of water. Dilute to 100 ml. This solution shall be freshly prepared and used immediately.

4.16 Background solution, 10 mg Fe/ml.

Dissolve 1,43 g of iron oxide (4.5) [minimum purity 99,99 % (m/m)] in 30 ml of hydrochloric acid (4.7). Evaporate until a syrupy consistency is obtained, cool, add 10 ml of hydrochloric acid (4.9), and transfer the solution to a 100 ml one-mark volumetric flask. Dilute to volume with water and mix.

4.17 Arsenic standard solution A, 100 $\mu\text{g/ml}$.

Dry several hundred milligrams of arsenic trioxide [$\text{As}_2\text{O}_3 > 99,95 \% (m/m)$] at 105 °C for 1 h. Dissolve 0,132 g of the dried product in 2 ml of sodium hydroxide solution (4.12), add 30 ml of water, neutralize with hydrochloric acid (4.10), using methyl orange (4.21) indicator, and add 4 g of sodium bicarbonate (4.3). Dilute in a volumetric flask to 1 000 ml with water and mix.

1 ml of standard arsenic solution A contains 100 μg of arsenic.

4.18 Arsenic standard solution B, 1 µg/ml.

Transfer 10 ml of arsenic standard solution A (4.17) to a 1 l volumetric flask, dilute to the mark with water, and mix.

1 ml of standard arsenic solution B contains 1 µg of arsenic.

4.19 Arsenic standard solution C, 0,1 µg/ml.

Transfer 10 ml of arsenic standard solution B (4.18) to a 100 ml volumetric flask, dilute to the mark with water, and mix.

1 ml of standard arsenic solution C contains 0,1 µg of arsenic.

4.20 Methyl orange, indicator.**4.21 Methyl orange solution, 1 mg/ml.**

Dissolve 0,10 g of methyl orange in 20 ml of water. Dilute to 100 ml.

5 Apparatus

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

5.1 Atomic absorption spectrometer, equipped with an arsenic hollow cathode lamp or electrodeless discharge lamp.

WARNING — Follow the manufacturer's instructions to avoid possible explosion hazards for igniting and extinguishing the air-acetylene flame and possible burning for hot electric furnace. Wear tinted safety glasses whenever the atomic absorption spectrometer is in operation. Good ventilation of the flame is necessary to prevent poison by arsenic hydride.

The atomic absorption spectrometer used in this method shall meet the following criteria.

- Minimum sensitivity: The absorbance of the highest concentration calibration solution (see 7.4.3) is at least 0,25.
- Graph linearity: The slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) is not less than 0,7 of the value of the slope for the bottom 20 % of the concentration range determined in the same way.
- 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, are less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated 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.

Parameters will vary with each instrument. The parameters in Annex B were successfully used in several laboratories and they can be used as guidelines. An air-acetylene flame or electric furnace capable of reaching temperatures of 900 °C to 1 000 °C is used.

5.2 Hydride generator, continuous or discontinuous injection sample hydride generator.

An example of continuous injection sample hydride generator is given in Figure 1, and an example of discontinuous injection sample hydride generator is given in Figure 2.

5.3 Quartz tube atomizer (T type).

An example of a quartz tube atomizer is given in [Figure 3](#).

5.4 Muffle furnace.

5.5 Zirconium crucible, of minimum capacity 25 ml.

5.6 Pipettes.

5.7 Volumetric flasks and storage bottles.

5.8 Carrier gas supply, inert gas.

6 Sampling and samples

6.1 Laboratory sample

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

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

6.2 Preparation of predried test samples

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

7 Procedure

7.1 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 is not affected by the previous result(s). For this 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.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 1 g, approximately 0,5 g of the predried test sample obtained in accordance with [6.2](#).

7.3 Blank test and check test

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out 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 [6.2](#).

Where the analysis is carried out on several samples at the same time, the blank value can be represented by one test, provided that the procedure is the same and the reagents used 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 can 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 can be used.

7.4 Determination

7.4.1 Decomposition of the test portion

Transfer the test portion to a 250 ml beaker. Moisten with a few millilitres of water, and add 30 ml of hydrochloric acid (4.7) and 1 ml of nitric acid (4.8). Cover with a watch-glass and heat gently until most of the test portion is decomposed. Increase the heat and digest just below boiling until no further attack is apparent. Evaporate the solution to near-dryness, cool, add 10 ml hydrochloric acid (4.9), and dissolve salt. Transfer the solution obtained to a 100 ml volumetric flask, dilute to the mark with water, and mix.

7.4.2 Treatment of the residue

If a residue is found in the solution, it should be treated by filtering the solution through a medium-texture filter paper into a 250 ml beaker. Carefully remove all adhering particles with a rubber-tipped glass rod and transfer to the filter, washing three times with dilute hydrochloric acid (4.10), then with hot water until the paper is free of iron. Transfer the paper and residue to a zirconium crucible. Retain the filtrate. Dry and ash the filter paper and residue at a low temperature. Add the mixture of 0,2 g sodium peroxide (4.1) and 0,4 g sodium carbonate (4.2) to the residue, heating gently, then ignite it in a muffle furnace at 700 °C for 10 min. Cool, dissolve the cooled melt with 5 ml of hydrochloric acid (4.10), and transfer into the retained filtrate.

The test portion should be taken and weighed quickly to avoid re-absorption of moisture. During evaporation, the watch glass can be moved a little.

7.4.3 Treatment of the solution

Transfer an appropriate aliquot of this solution (see Table 1) or certified reference material solution to a 100 ml one-mark volumetric flask, add adequate volumes of background solution (4.16) so that the contents of the iron in the test solution are matched to 1,5 mg/ml, then add 10 ml of ascorbic acid solution (4.15), 10 ml of potassium iodide solution (4.14), and hydrochloric acid (4.11) to the mark and mix. Stand the solutions for 3 h, then use for determination. Carry out determination of the blank test portion at the same time.

Table 1 — Dilution guide for test solution

Arsenic content range %	Aliquot from 100 ml ml
0,000 66 to 0,001	20
0,001 to 0,01	10
0,01 to 0,020 15	2

NOTE The dilutions will provide arsenic contents falling within the range of the calibration solutions. For instruments having high sensitivity, smaller portions of the test solution may be preferable. Avoid aliquot portions of less than 2 ml by marking a preliminary dilution. Treat the blank test solution similarly.

7.4.4 Preparation of the set of calibration solutions

From the arsenic standard solution C (4.19), prepare calibration solutions as follows.

Using pipettes, transfer 0 ml, 0,1 ml, 0,5 ml, 1 ml, 3 ml, 4 ml, 6 ml, 10 ml, 20 ml, 30 ml, and 40 ml of arsenic standard solution C (4.19), respectively, to 100 ml one-mark volumetric flasks. Add 15 ml of the background solution (4.16) and 10 ml of ascorbic acid solution (4.15) and 10 ml of potassium iodide solution (4.14) to each, then add hydrochloric acid (4.11) to the flasks until the mark and mix. Stand for at least 3 h, then use for determination.

NOTE 1 The detection limit of the method can vary because of differences between instruments (atomic absorption spectrometers and hydride generators). For instruments with high sensitivity, a calibration solution with low arsenic content can be used.

NOTE 2 Where the arsenic content in a sample is known to be within a certain range, the set of calibration solutions can be varied to provide good results within that range.

7.4.5 Adjustment of the atomic absorption spectrometer

Optimize the response of the instrument as specified in 5.1. Set the wavelength for arsenic (193,7 nm) and align the position of the quartz tube to obtain minimum absorbance. Fit the instrument, adapter, and connectors of the hydride generator and adjust in accordance with the manufacturer's instructions. Light the appropriate flame or heat electric furnace to the appropriate temperature. After several minutes of preheating, adjust the flow of carrier gas to the appropriate flow rate. Adjust correlative parameters to obtain maximum absorbance while injecting the calibration solution (7.4.3) of highest arsenic content (not less than 0,25).

7.4.6 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 measurement at least twice.

Obtain the net absorbance of each calibration solution by subtracting the average absorbance of the zero calibration. 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 the net absorbance values of the calibration solutions against the concentration, in micrograms of arsenic per millilitre (the test solution or, if diluted test solution is the final test solution).

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

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

8 Expression of results

8.1 Calculation of mass fraction of arsenic content

The mass fraction of arsenic, expressed as a percentage, is calculated to four decimal places for mass fractions higher than 0,001 %, and to five decimal places for mass fractions lower than 0,001 %, using Formula (1).

$$W_{\text{As}}(\%) = \frac{C \times V}{m_1} \times 10^{-7} \quad (1)$$

where

W_{As} is the mass fraction of arsenic of the test sample, expressed as a percentage;

C is the concentration of arsenic in the final test solution, in ng/ml;

V is the volume of arsenic in the final test solution ($V = 100$), in ml;

m_1 is the mass, in grams, of test sample represented in 100 ml of the final test solution (see [7.4.5](#)), calculated from Formula (2).

$$m_1 = \frac{m \times V_1}{100} \quad (2)$$

where

M is the mass of laboratory test portion, in g;

V_1 is the volume of aliquot of test solution, in ml.

8.2 General treatment of results

8.2.1 Repeatability and permissible tolerance

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

$$R_d = 0,729X^2 + 0,2779X + 0,000007 \quad (3)$$

$$P = 1,6279X^2 + 0,4193X + 0,00009 \quad (4)$$

$$\sigma_d = -0,2604X^2 + 0,0993X + 0,000002 \quad (5)$$

$$\sigma_L = -0,581\ 4X^2 + 0,149\ 8X + 0,000\ 03 \quad (6)$$

where

- X is the mass fraction of arsenic, expressed as a percentage, of the predried test sample;
- within-laboratory [Formulae (3) and (5)]: the arithmetic mean of the duplicate values;
- between-laboratory [Formulae (4) and (6)]: the arithmetic mean of the final results (8.2.5) of the two laboratories;
- R_d is the permissible tolerance within a laboratory (repeatability);
- P is the permissible tolerance between laboratories;
- σ_d is the within-laboratory standard deviation;
- σ_L is the between-laboratories standard deviation.

8.2.2 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 Annex A, and obtain the final laboratory result (see 8.2.5).

8.2.3 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 8.2.2.

Compute the following quantity:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad (7)$$

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 (6) and calculate P .

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

8.2.4 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 the fourth paragraph in 7.3). Calculate the analytical result (μ_c) for the CRM/RM 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 CRM/RM;
 A_c is the reference/certified 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 35:2006.

For a CRM/RM certified by an interlaboratory test programme, C shall be calculated as follows:

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

where

- s_c is the standard deviation of laboratory means (each data for calculating the standard deviation is the average data in each certifying laboratory) of the CRM/RM;
 N_c is the number of certifying laboratories;
 n is the number of replicate determinations carried out on the CRM/RM.

For CRMs certified by only one laboratory, C shall be calculated as follows:

$$C = \sqrt{2\sigma_L^2 + \frac{\sigma_r^2}{n}} \quad (9)$$

CRMs certified by only one laboratory should be avoided unless they are known to have an unbiased certified value.

8.2.5 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 operation specified in [Annex A](#). The arithmetic mean of the acceptable analytical values, if the content of arsenic is larger than or equal to 0,001 %, calculated to the sixth decimal place, is rounded off to the fourth decimal place as follows:

- when the figure in the fifth decimal place is less than 5, it is discarded and the figure in the fourth decimal place is kept unchanged;
- when the figure in the fifth decimal place is 5 and there is a figure other than 0 in the sixth decimal place, or when the figure in the fifth decimal place is greater than 5, the figure in the fourth decimal place is increased by one;
- when the figure in the fifth decimal place is 5 and 0 is in the sixth decimal place, the 5 is discarded and the figure in the fourth 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.

The arithmetic mean of the acceptable analytical values, if the content of arsenic is less than 0,001 %, calculated to the seventh decimal place, is rounded off to the fifth decimal place as follows:

- when the figure in the sixth decimal place is less than 5, it is discarded and the figure in the fifth decimal place is kept unchanged;
- when the figure in the sixth decimal place is 5 and there is a figure other than 0 in the seventh decimal place, or when the figure in the sixth decimal place is greater than 5, the figure in the fifth decimal place is increased by one;
- when the figure in the sixth decimal place is 5 and 0 is in the seventh decimal place, the 5 is discarded and the figure in the fifth 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.

8.3 Oxide factor

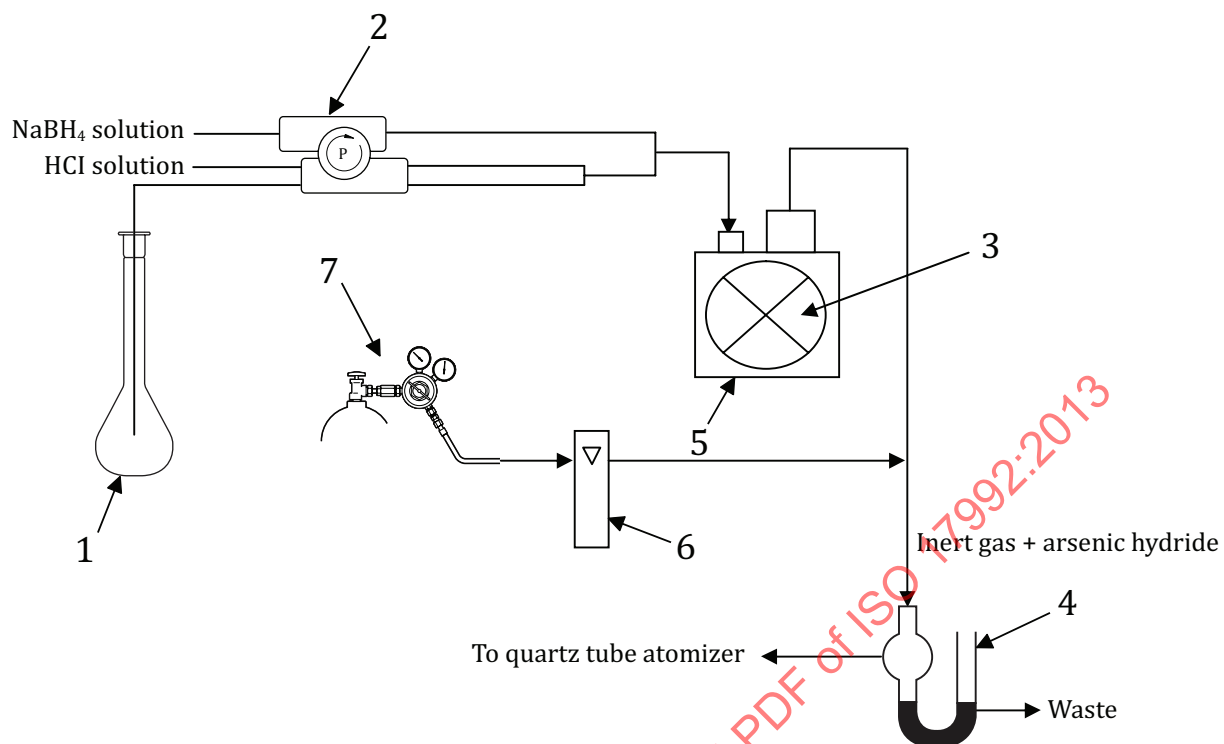
The oxide factor, expressed as a percentage, is given by Formula (10):

$$w_{As_2O_3} = 1,320\ 3 \times w_{As} \quad (10)$$

9 Test report

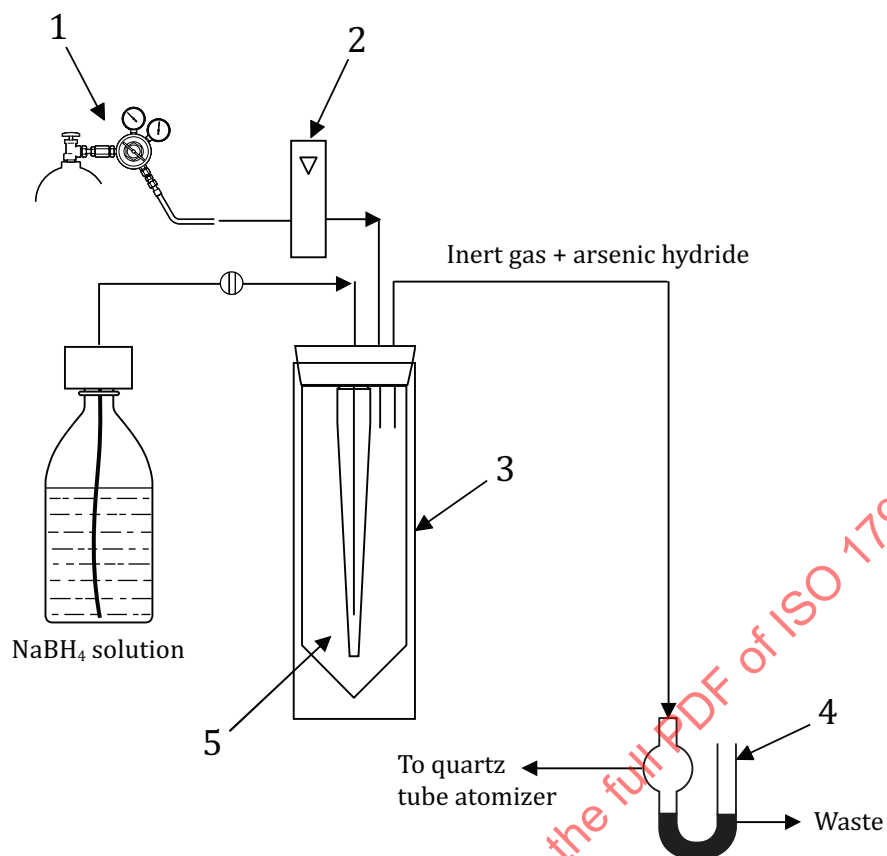
The test report shall include the following information:

- a) name and address of the testing laboratory;
- b) date of issue of the test report;
- c) a reference to this International Standard (i.e. ISO 17992);
- d) details necessary for the identification of the sample;
- e) result of the analysis;
- f) reference number of the result;
- g) any characteristics noticed during the determination and any operations not specified in this International Standard which can have had an influence on the result, for either the test sample or the certified reference material(s).

**Key**

- 1 sample solution
- 2 creep pump
- 3 reaction coil
- 4 gas-liquid separator
- 5 reaction tube
- 6 flow meter
- 7 carrier gas (inert gas)

Figure 1 — Example of continuous injection sample hydride generator



Key

- 1 carrier gas (inert gas)
- 2 flow meter
- 3 reaction flask
- 4 gas-liquid separator
- 5 sample solution

Figure 2 — Example of discontinuous injection sample hydride generator

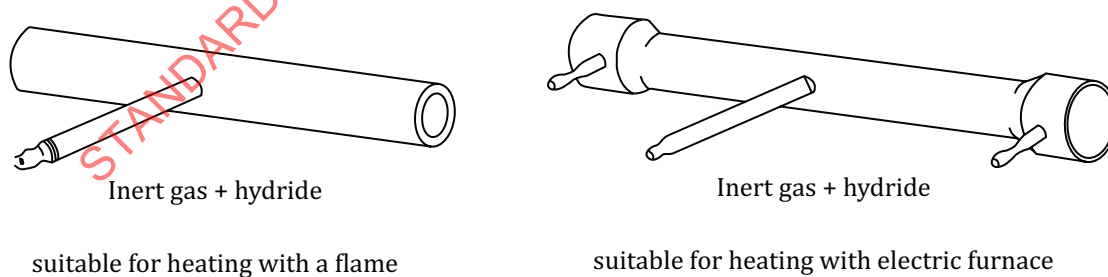
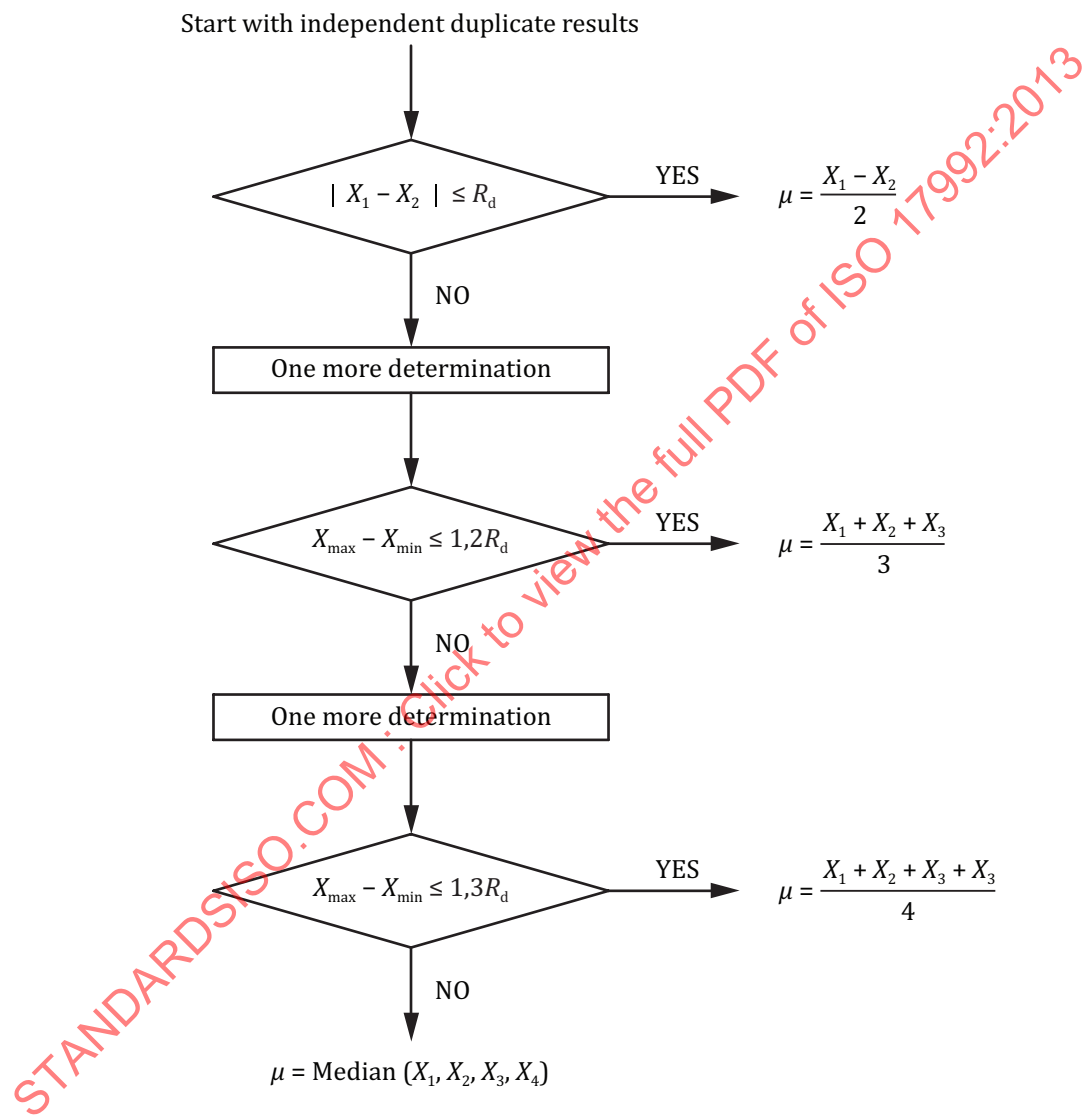


Figure 3 — Example of quartz tube atomizer (two types)

Annex A (normative)

Flow sheet of the procedure for the acceptance of analytical values for test sample


Key

R_d permissible tolerance within a laboratory (repeatability)

Figure A.1