

BS ISO 17992:2013



BSI Standards Publication

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

bsi.

...making excellence a habit.
AVA Documents™
Document Sharing Hub

National foreword

This British Standard is the UK implementation of ISO 17992:2013.

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.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2013. Published by BSI Standards Limited 2013

ISBN 978 0 580 79546 6

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 30 September 2013.

Amendments issued since publication

Date	Text affected
------	---------------

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





COPYRIGHT PROTECTED DOCUMENT

© ISO 2013

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Principle	1
4 Reagents	1
5 Apparatus	3
6 Sampling and samples	4
6.1 Laboratory sample.....	4
6.2 Preparation of predried test samples.....	4
7 Procedure	4
7.1 Number of determinations.....	4
7.2 Test portion.....	4
7.3 Blank test and check test.....	4
7.4 Determination	5
8 Expression of results	7
8.1 Calculation of mass fraction of arsenic content.....	7
8.2 General treatment of results	7
8.3 Oxide factor	10
9 Test report	10
Annex A (normative) Flow sheet of the procedure for the acceptance of analytical values for test sample	13
Annex B (informative) Parameters of some instruments	14
Annex C (informative) Derivation of repeatability and permissible tolerance formulae	16
Annex D (informative) Precision data obtained by international analytical trials	17

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 (Na_2O_2).

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.