

Analysis of iron ores —

Part 13: Method for the determination of arsenic content: spectrophotometric method

[ISO title: Iron ores — Determination of arsenic content —
Molybdenum blue spectrophotometric method]

UDC 622.341.1:[620.113:622.7.016.34]:622.349.71:[535.243:622.346.2]:620.1

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Iron and Steel Standards Committee (ISM/-) to Technical Committee ISM/58, upon which the following bodies were represented:

British Steel Industry
Institution of Mining and Metallurgy

The following body was also represented in the drafting of the standard, through subcommittees and panels:

British Ceramic Research Ltd.

This British Standard, having been prepared under the direction of the Iron and Steel Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 January 1989

© BSI 10-1999

The following BSI references relate to the work on this standard:
Committee reference ISM/58
Draft for comment 86/38439 DC

ISBN 0 580 16884 0

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

	Page
Committees responsible	Inside front cover
National foreword	ii
1 Scope and field of application	1
2 References	1
3 Principle	1
4 Reagents	1
5 Apparatus	2
6 Sampling and samples	2
7 Procedure	2
8 Expression of results	4
9 Test report	5
Annex A Flowsheet on the procedure for the acceptance of analytical values for test samples	7
Annex B Derivation of repeatability and permissible tolerance equations	8
Annex C Graphical presentation of precision data obtained by international analytical trial	9
Figure 1 — Arsenic distillation apparatus	6
Figure 2 — Least-squares fit of precision against X for arsenic	9
Table 1 — Aliquot portions of distillate	3
Table 2 — Test samples for interlaboratory tests	8
Publications referred to	Inside back cover

National foreword

This Part of BS 7020 has been prepared under the direction of the Iron and Steel Standards Committee. It is identical with ISO 7834:1987 “*Iron ores — Determination of arsenic content — Molybdenum blue spectrophotometric method*” published by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

In British Standards it is current practice to use the spelling “sulphur” etc., instead of “sulfur”, etc.

Wherever the words “International Standard” appear, referring to this standard, they should be read as “Part of BS 7020”.

Cross-references

International Standard	Corresponding British Standard
ISO 1042:1981	BS 1792:1982 <i>Specification for one-mark volumetric flasks</i> (Identical) BS 5660 <i>Methods of sampling iron ores</i>
ISO 3081:1986	Part 1:1987 <i>Manual method of increment sampling</i> (Identical)
ISO 3082:1987	Part 2:1987 <i>Mechanical method of increment sampling and sample preparation</i> (Identical)
ISO 3083:1986	BS 5561:1987 <i>Method for preparation of samples of iron ores by manual means</i> (Identical)
ISO 7764:1985	BS 7020 <i>Analysis of iron ores</i> Part 1:1988 <i>Method for the preparation of pre-dried test samples for chemical analysis</i> (Identical)

The Technical Committee has reviewed the provisions of ISO 648, to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard.

A related British Standard to ISO 648 is BS 1583 “*Specification for one-mark pipettes*”.

The reference to ISO 5725 is for information only.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

WARNING — Attention is drawn to the toxic nature of arsenic and its solutions and of other reagents used in this method and to the need to take particular care in the handling and disposal of solutions.

1 Scope and field of application

This International Standard specifies a molybdenum blue spectrophotometric method for the determination of the arsenic content of iron ores. This method is applicable to a concentration range of 0,000 1 to 0,1 % (*m/m*) (1 to 1 000 µg/g) of arsenic in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

2 References

- ISO 648, *Laboratory glassware — One-mark pipettes*.
 ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.
 ISO 3081, *Iron ores — Increment sampling — Manual method*.
 ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method*.
 ISO 3083, *Iron ores — Preparation of samples — Manual method*.
 ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*.

3 Principle

Decomposition of a test portion by sintering with sodium peroxide and leaching with water and hydrochloric acid.

Transfer of the solution to a distillation flask, evaporation of part of the solution, treatment with potassium bromide and hydrazine sulfate, followed by adjustment of the acidity. Distillation of arsenic trichloride and collection of the distillate in nitric acid.

Evaporation to dryness and baking at controlled temperature, followed by treatment with ammonium molybdate-hydrazine reagent to form an arseno-molybdenum blue complex.

Spectrophotometric measurement of the absorbance at approximately 840 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only deionized water or water of equivalent purity.

NOTE In order to obtain reliable values at the lowest levels of arsenic content in test samples (< 20 µg/g), reagents should be selected or purified so that the value for the absorbance of the blank test measured in a cell of 20 mm optical path length is not greater than 0,025, equivalent to 1 µg of arsenic. In particular, nitric acid may need to be redistilled, or the apparatus cleaning procedures (5.2) may need to be applied more rigorously.

4.1 Sodium peroxide (Na_2O_2), fine powder.

4.2 Potassium bromide (KBr).

4.3 Hydrazine sulfate ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{SO}_4$).

4.4 Hydrochloric acid (ρ 1,16 to 1,19 g/ml).

4.5 Nitric acid (ρ 1,4 g/ml), diluted 1 + 1.

4.6 Sulfuric acid (ρ 1,84 g/ml), free from phosphorus.

4.7 Ammonium molybdate [$(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$], solution, 10 g/l.

To 400 ml of water in a 1 litre beaker, add carefully, with stirring, 133 ml of sulfuric acid (4.6). Cool, add $10 \pm 0,1$ g of ammonium molybdate and dissolve with stirring. Transfer to a 1 litre volumetric flask or stoppered measuring cylinder, dilute to the mark with water and mix.

4.8 Hydrazine sulfate ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{SO}_4$) solution, 0,15 g/l.

4.9 Molybdate-hydrazine reagent

To 70 ml of water in a 100 ml volumetric flask, add $10 \pm 0,1$ ml of ammonium molybdate solution (4.7) and 10 ml of hydrazine sulfate solution (4.8). Dilute to the mark with water and mix. Prepare freshly for each series of tests.

4.10 Arsenic, standard solution A, 200 µg/ml.

Dry several hundred milligrams of arsenic trioxide (As_2O_3) at 105 °C for 1 h. Dissolve 0,132 g of the dried product in 2 ml of 40 g/l sodium hydroxide solution, add 30 ml of water, neutralize with sulfuric acid (4.6) diluted 1 + 9, using methyl orange indicator, and add 4 g of sodium hydrogen carbonate. Dilute in a volumetric flask to 500 ml with water and mix.

1 ml of standard arsenic solution A contains 200 µg of arsenic.

4.11 Arsenic, standard solution B, 5 µg/ml.

Transfer by pipette 25 ml of standard arsenic solution A (4.10) to a 1 litre volumetric flask, dilute to the mark with water and mix.

1 ml of standard arsenic solution B contains 5 µg of arsenic.