

Analysis of iron ores —

Part 3: Method for the determination of combined water content

[ISO title: Iron ores — Determination of combined water
content — Karl Fischer titrimetric method]

UDC 622.341.1:620.113:[543.812.4:543.24]: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 07-1999

The following BSI references relate to the work on this standard:
Committee reference ISM/58
Draft for comment 86/38438 DC

ISBN 0 580 16886 7

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	1
6 Sampling and samples	2
7 Procedure	2
8 Expression of results	3
9 Test report	5
Annex A Flowsheet on the procedure for the acceptance of analytical values for test samples	9
Annex B Derivation of repeatability and permissible tolerance equations	10
Annex C Graphical presentation of precision data obtained by international analytical trial	10
Figure 1 — Apparatus for the Karl Fischer measurement (two-furnaces-in-series type) (Example)	6
Figure 2 — Apparatus for the Karl Fischer measurement (interchangeable furnace type) (Example)	7
Figure 3 — Karl Fischer absorption cell (Example)	8
Figure 4 — Least-squares fit of precision against X for combined water	10
Table 1 — Mass of test portion	2
Table 2 — Combined water contents of test samples	10
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 7335:1987 “*Iron ores — Determination of combined water content — Karl Fischer titrimetric 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.

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
	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 5661:1987 <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 385, 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 385 is BS 846 “*Specification for burettes*”.

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.

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the combined water content of iron ores, using Karl Fischer solution.

This method is applicable to a concentration range of 0,05 to 10 % (m/m) of combined water in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

NOTE The term "combined water" means that part of the water content of an iron ore that can be removed only at temperatures substantially higher than 100 to 110 °C.

2 References

ISO 385, *Laboratory glassware — Burettes.*

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

Liberation of adventitious hygroscopic moisture by heating the predried test portion at 105 °C in a furnace in a stream of dry nitrogen. Successive heating up to 950 °C in another furnace and collection of the liberated combined water in ethylene glycol-methanol mixture.

Measurement of combined water content by titration with Karl Fischer solution using electrometric detection of the end point.

4 Reagents

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

4.1 Desiccant. Silica gel, self-indicating granules.

4.2 Desiccant. Anhydrous magnesium perchlorate [$\text{Mg}(\text{ClO}_4)_2$] of size 0,8 to 1,25 mm or other suitable desiccant.

NOTE Magnesium perchlorate is a powerful oxidant and must not be allowed to come into contact with organic materials. When exhausted, it should be washed down the sink with a copious flow of water.

4.3 Nitrogen. A supply of filtered, predried, oil-free nitrogen containing less than 10 µl of oxygen per litre at a pressure of approximately 35 kPa above atmospheric pressure.

4.4 Ethylene glycol ($\text{OHCH}_2\text{CH}_2\text{OH}$)-methanol (CH_3OH) mixture (1 + 1). This mixture should be tested each time before use for the amount of free moisture it contains. If it contains more than 0,05 % (m/m) of free moisture, it shall not be used.

NOTE In place of this mixture, anhydrous ethylene glycol may be used. In this case, Karl Fischer solution must contain the appropriate amount of methanol.

4.5 Karl Fischer solution (2,5 to 3,0 mg H_2O /ml). This solution shall be standardized on the day of use by one of the following substances:

- a) a standard water methanol solution; this solution shall previously be standardized by use of the following b), c) or d);
- b) water added by means of a microsyringe;
- c) citric acid monohydrate [$\text{C}(\text{OH})(\text{COOH})(\text{CH}_2\text{COOH})_2 \cdot \text{H}_2\text{O}$];
- d) disodium tartrate dihydrate [$(\text{CHOH COONa})_2 \cdot 2\text{H}_2\text{O}$].

5 Apparatus

A suitable apparatus for the determination is illustrated in Figure 1 (two-furnaces-in-series type) or in Figure 2 (interchangeable furnace type).

5.1 Gas flowmeter, capable of measuring a flow rate up to 250 ml/min. If a pressure drop over a constriction is used as a means of measuring flow rate, the manometer liquid shall be a non-volatile oil.

5.2 Drying towers (t_1 and t_2), of 250 ml capacity, packed respectively with desiccant (4.1) and (4.2) to dry the stream of nitrogen entering the heating tube.

5.3 Heating furnaces (f_1 and f_2). Electric furnaces in series as in Figure 1 or movable furnaces which can be located around, and removed from, the heating tube as in Figure 2. The furnaces shall be capable of maintaining temperatures of 105 ± 2 °C and 950 ± 20 °C, respectively, over a minimum tube length of about 150 mm, by controlling the electric current. Furnace temperatures shall be measured at the upper surface of the heating tube by means of a pyrometer or thermometer.

5.4 Heating tube. A silica tube as shown in Figure 1, of about 30 mm inside diameter, about 800 mm length, and provided with a long silica pushing rod. Another example (Figure 2) is about 350 mm long, closed at one end and fitted with a thin inner tube of about 8 mm external diameter, through which gas flows towards the outlet.