

METHODS OF TESTING REFRACTORY MATERIALS

Part 2. Chemical analysis (wet methods)

Section 2.3. Chemical analysis of magnesites and dolomites

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The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

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A complete list of British Standards, numbering over 9,000, fully indexed and with a note of the contents of each, will be found in the BSI Catalogue which may be purchased from BSI Sales Department. The Catalogue may be consulted in many public libraries and similar institutions.

This standard makes reference to the following British Standards:

- BS 410. Test sieves.
- BS 507. Ethanol.
- BS 1583. One-mark pipettes.
- BS 1752. Laboratory sintered or fritted filters.
- BS 1792. One-mark volumetric flasks.
- BS 2734. Boiling flasks (narrow-necked), conical, flat bottom and round bottom.
- BS 3591. Industrial methylated spirits.
- BS 3978. Water for laboratory use.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following BSI references relate to the work on this standard:
Committee references RPE/1 and RPE/1/1
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CO-OPERATING ORGANIZATIONS

The Refractory Products Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government department and scientific and industrial organizations:

- *British Ceramic Research Association
British Chemical Plant Manufacturers' Association
- *British Steel Industry
Chemical Industries Association
Coke Oven Managers' Association
Combustion Engineering Association
- *Council of British Manufacturers of Petroleum Equipment
Engineering Equipment Users' Association
- *Gas Council
Glass Manufacturers' Federation
- *Ministry of Defence, Navy Department
- *National Federation of Clay Industries
Society of British Gas Industries
- *Society of Glass Technology
Society of Industrial Furnace Engineers
- *Water-tube Boilermakers' Association

The Government department and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Cast Iron Research Association
British Investment Casters' Technical Association
Electricity Council, the Central Electricity Generating Board
and the Area Boards in England and Wales
Institute of Petroleum
Institution of Gas Engineers

METHODS OF TESTING REFRACTORY MATERIALS

Part 2. Chemical analysis (test methods)

Section 2.3. Chemical analysis of magnesites and dolomites

FOREWORD

This section of Part 2 is based on well established procedures for the analysis of magnesites and dolomites which have been in use in various industrial laboratories for a number of years. It does not cover the determination of alkalis in dolomite, which can conveniently be achieved by classical methods, but this aspect will receive further consideration.

METHODS

1. SCOPE

This British Standard describes the methods for the determination of loss on ignition and of silica, ferric oxide, titania, manganese oxide, chromic oxide, alumina, lime and magnesia in magnesites and dolomites, and of alkali metal oxides in magnesites, suitable for industrial refractories. The methods are, in general, not sufficiently sensitive for the analysis of special high-purity magnesias.

2. PRINCIPLE

2.1 Loss on ignition. The loss on ignition is determined at $1025 \pm 25^\circ\text{C}$.

2.2 Determination of silica, ferric oxide, titania, manganese oxide, chromic oxide, alumina, lime and magnesia. The sample is decomposed with hydrochloric acid and the silica separated by coagulation and filtration. The crucible containing this silica is ignited and weighed before and after treatment with hydrofluoric and sulphuric acids. The silica remaining in the filtrate is subsequently determined by a spectrophotometric method, based on the formation of molybdenum blue, in a portion of the solution used for the determination of ferric oxide, titania, manganese oxide, chromic oxide, alumina, lime and magnesia.

The residue from the silica is fused in sodium carbonate and boric acid and the cold melt dissolved in the filtrate from the silica. After dilution to a standard volume, the ferric oxide is determined spectrophotometrically* with 1:10-phenanthroline, the titania with hydrogen peroxide, the manganese oxide with potassium periodate and the chromic oxide with diphenylcarbazide for very

* The preparation of calibration graphs is described in Appendix A.

low chrome contents and as the chrome-EDTA complex for materials containing more than about 0.1 % of chromic oxide. For the alumina determination, the ammonia group oxides in a measured volume of the solution are precipitated with ammonia, to remove most of the magnesia, the precipitate is dissolved in hydrochloric acid, the ferric and titanium ions are removed by a cupferron/chloroform solvent extraction and the determination is completed volumetrically with ethylenediamine-tetra-acetic acid (EDTA) and zinc, using dithizone as indicator. Lime is determined on a measured volume of the solution to which triethanolamine is added to complex interfering elements; a known volume of standard EGTA, sufficient to complex all the calcium, is then added and the magnesium is then precipitated with potassium hydroxide. The excess EGTA is titrated with calcium solution, calcein being used as indicator. Magnesia is determined volumetrically on a measured volume of the solution, after removal of ferric, manganese and most of the titanium ions by a sodium diethyldithiocarbamate/chloroform solvent extraction. Alumina and any remaining titanium ions are complexed with triethanolamine and the magnesium is titrated, in a strongly ammoniacal solution containing ammonium chloride, with CDTA. If more than 1 % of alumina is present, this and the titania are removed by a cupferron/chloroform solvent extraction. The titration also includes the titration for lime, which must be allowed for.

2.3 Determination of alkalis. For magnesites, the sample is decomposed by hydrofluoric and perchloric acids to remove the silica. A further evaporation with perchloric acid removes traces of fluoride; the residue is dissolved in hydrochloric acid and the solution diluted to a standard volume. Alkalis are determined using a flame photometer.

For dolomites, alkalis cannot be determined by this method because of interference by calcium.

3. REAGENTS

3.1 Unless otherwise stated, all reagents shall be of analytical grade, where available, and distilled water shall be used throughout the analysis*. The following reagents are required.

3.2 Solid reagents

(1) *Aluminium sulphate.*

(2) *Ammonium chloride.*

(3) *Boric acid.*

(4) *Calcein indicator.* Mix, by grinding together, 0.2 g of calcein, 0.12 g of thymolphthalein and 20 g of potassium chloride.

* See BS 3978, 'Water for laboratory use'.

- (5) *Mordant black 3*. Mix, by grinding together, 0.5 g of Mordant black 3* and 20 g of sodium chloride.
- (6) *Potassium chloride*.
- (7) *Potassium periodate*.
- (8) *Silica*. A quartz or sand of known high purity; not less than 99 % SiO_2 .
- (9) *Sodium carbonate*. Anhydrous.
- (10) *Sodium chloride*.
- (11) *Thymolphthalein*.

3.3 Solutions

- (1) *Acetic acid*. Glacial.
- (2) *Acetic acid* (1 + 1). Add 500 ml of the acetic acid (glacial) to 500 ml of water and mix.
- (3) *Acetone*.
- (4) *Ammonia*. $d = 0.88$ (18N approx.).
- (5) *Ammonia* (1 + 1). Add 500 ml of the ammonia solution ($d = 0.88$) to 500 ml of water and mix.
- (6) *Ammonium acetate* (100 g/l approx.). Dilute 140 ml of the acetic acid (glacial) to 1700 ml with water and add carefully, with stirring, 140 ml of the ammonia solution ($d = 0.88$). Mix, cool and adjust to pH 6.0–6.5, with either acetic acid or ammonia solution.
- (7) *Ammonium acetate buffer*. Add, with stirring, 120 ml of the acetic acid (glacial) to 500 ml of water, followed by 74 ml of the ammonia solution ($d = 0.88$). Cool, dilute to 1 litre and mix.
- (8) *Ammonium ceric nitrate* (10 g/l). Dissolve 2.5 g of ammonium ceric nitrate in about 200 ml of water, cautiously add 7 ml of the sulphuric acid ($d = 1.84$), cool, dilute to 250 ml and mix.
- (9) *Ammonium molybdate* (80 g/l). Dissolve 80 g of ammonium molybdate in water, filter if necessary, dilute to 1 litre and mix. Store in a polythene† bottle. Discard after 4 weeks, or earlier if any appreciable deposit is observed.
- (10) *Ammonium nitrate* (10 g/l). Dilute 10 ml of the nitric acid ($d = 1.42$) to about 200 ml. Add the ammonia solution (1 + 1) until the solution is faintly alkaline to bromophenol blue. Cool and dilute to 1 litre; heat to about 80 °C before use.
- (11) *Bromophenol blue* (1 g/l). Grind 0.1 g of bromophenol blue with 1.5 ml of sodium hydroxide solution (4 g/l), dilute to 100 ml with water and mix.

* Proprietary brands are known as Solochrome black 6B and Eriochrome blue-black B.

† It should be noted that the name 'polythene' is equivalent to the name 'polyethylene'.