

# Quicklime, hydrated lime and natural calcium carbonate —

## Part 2: Methods of chemical analysis

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# Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Cement Gypsum Aggregates and Quarry Products Standards Committee (CAB/-) to Technical Committee CAB/5 upon which the following bodies were represented:

- British Aggregate Construction Materials Industries
- Cement Makers' Federation
- County Surveyors' Society
- Department of the Environment (Building Research Establishment)
- Department of the Environment (Property Services Agency)
- Dry Lining and Partition Association Limited
- Gypsum Products Development Association
- Institution of Structural Engineers
- National Federation of Building Trades Employers
- National Federation of Plastering Contractors
- Plasterers' Craft Guild
- Royal Institute of British Architects
- Scottish Master Plasterers' Association
- Society of Chemical Industry

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

- Chemical Industries Association Limited
- Mortar Producers' Association Limited
- Water Authorities

This British Standard, having been prepared under the direction of the Cement Gypsum Aggregates and Quarry Products Standards Committee, was published under the authority of the Board of BSI and comes into effect on 29 February 1984

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# Contents

|                                                                                      | Page               |
|--------------------------------------------------------------------------------------|--------------------|
| Committees responsible                                                               | Inside front cover |
| Foreword                                                                             | ii                 |
| 1 Scope                                                                              | 1                  |
| 2 Reporting                                                                          | 1                  |
| 3 Reagents                                                                           | 1                  |
| 4 Determination of silicon                                                           | 4                  |
| 5 Determination of iron                                                              | 4                  |
| 6 Determination of aluminium                                                         | 5                  |
| 7 Determination of manganese                                                         | 5                  |
| 8 Determination of copper                                                            | 6                  |
| 9 Determination of calcium                                                           | 7                  |
| 10 Determination of magnesium                                                        | 7                  |
| 11 Determination of fluoride                                                         | 8                  |
| 12 Determination of matter insoluble in acetic acid                                  | 9                  |
| 13 Qualitative test for acetic acid soluble heavy metals                             | 9                  |
| 14 Qualitative test for acetic acid soluble barium                                   | 9                  |
| 15 Determination of carbon dioxide                                                   | 10                 |
| 16 Determination of loss on ignition                                                 | 10                 |
| 17 Determination of sulphur                                                          | 11                 |
| 18 Determination of neutralizing value calculated in terms of calcium oxide          | 11                 |
| 19 Determination of free water (or moisture) in hydrated lime and lime putty         | 12                 |
| 20 Determination of available lime                                                   | 13                 |
| 21 Determination of soluble salts                                                    | 13                 |
| Figure 1 — Determination of carbon dioxide                                           | 14                 |
| Figure 2 — Determination of free water (or moisture) in hydrated lime and lime putty | 15                 |
| Publications referred to                                                             | Inside back cover  |

## Foreword

This British Standard has been prepared under the direction of the Cement, Gypsum, Aggregates and Quarry Products Standards Committee in order to bring together standard methods of test for quicklime, hydrated lime and natural calcium carbonate.

This standard incorporates and revises the test methods contained in BS 890 which will be revised in due course to refer to them. Although the standard deals primarily with methods of test for lime products used in the construction industry, methods used in other fields, e.g. water treatment, food, agriculture and the steel and chemical industries, have been included.

This standard will be published in four Parts, as follows.

- *Part 1: Methods of sampling;*
- *Part 2: Methods of chemical analysis;*
- *Part 3: Physical test methods for quicklime;*
- *Part 4: Physical test methods for hydrated lime and lime putty.*

Part 2 gives methods for the chemical analysis of quicklime, hydrated lime, lime putty and natural calcium carbonate. The methods given are considered to be the standard methods but atomic absorption methods can be used for the determination of, for example, silicon iron, magnesium and copper.

The methods are written to be compatible as far as practicable with those given in the British Pharmacopoeia (1980), the European Pharmacopoeia (1969) and the Food Chemical Codex (1981).

Except in the cases of Figure 1 and Figure 2 it has been considered unnecessary to itemize the standard laboratory apparatus required for the tests in this standard.

Safety requirements have not been included and individual laboratories carrying out the tests should have their own safe working procedures when dealing with hazardous materials.

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 16, 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

This Part of BS 6463 describes methods for the chemical analysis of quicklime, hydrated lime, lime putty and natural calcium carbonate.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

## 2 Reporting

The results of the analyses shall be quoted as “as received” or “dried at 105 °C”.

## 3 Reagents

**3.1 General.** Unless otherwise stated, all reagents used shall be of analytical reagent quality. In **3.2**, only the concentrated reagents are shown. Dilutions of acids and other liquid reagents are given as (1 + *n*) which indicates that one volume of the concentrated reagent shall be added to *n* volumes of water and mixed. Water shall comply with BS 3978 or shall be of an equivalent standard.

Solutions of solid reagents shall be filtered if the solution is not clear.

Reagents shall be as described in **3.2** to **3.4**. They shall be prepared by the methods given or, alternatively, proprietary reagents of equivalent quality shall be used.

### 3.2 General reagents

**3.2.1 Acetic acid**, glacial,  $\rho = 1.05$  g/L.

**3.2.2 Ammonia solution**,  $\rho = 0.880$  g/L.

**3.2.3 Ammonium chloride**

**3.2.4 Ammonium chloridelammonia buffer solution.** Dissolve 62.5 g ammonium chloride in water. Add 570 mL ammonia solution and dilute to 1 L with water. Mix.

**3.2.5 Ascorbic acid solution.** Dissolve 0.25 g of ascorbic acid in 60 mL of ethanol and dilute to 250 mL with water. Mix thoroughly.

**3.2.6 Barium chloride solution.** Dissolve 125 g of barium chloride in 1 L of water.

**3.2.7 Barium hydroxide solution.** Add 1 L of barium chloride solution to 1 L of sodium hydroxide solution (40 g/L) and mix in an aspirator fitted with a soda-lime guard tube. Allow any slight precipitate to settle before use.

**3.2.8 Bromine water (1 + 24).** Add 20 mL of bromine to 480 mL of water. Shake carefully in a glass stoppered bottle.

**3.2.9 Buffer pH 3.5 (for determination of heavy metals).** Mix 6.4 mL sodium acetate solution and 93.6 mL acetic acid (1 + 7.6). Dilute to 1 L with water. Adjust to pH 3.5 by adding more of either sodium acetate solution or acetic acid (1 + 7.6).

**3.2.10 Buffer pH 6.0 (for determination of aluminium).** Dissolve 257 g of ammonium acetate, approximately 500 mL of water and 10 mL glacial acetic acid. Dilute to 1 L with water and mix thoroughly.

**3.2.11 Calcium carbide**

**3.2.12 Calcium carbonate**

**3.2.13 Calcium sulphate**,  $\text{CaSO}_4 \cdot \text{H}_2\text{O}$ .

**3.2.14 Calcium sulphate solution.** Add 50 g of calcium sulphate to 1 L of water and stir for at least 1 h. Filter immediately before use.

**3.2.15 Chloroform**

**3.2.16 Cupferron solution.** Dissolve 6 g of cupferron in water at 5 °C to 8 °C. Dilute to 100 mL with water (1 mL of this solution precipitates 10 mg  $\text{Fe}^{3+}$  and 8 mg  $\text{Ti}^{4+}$ ).

**3.2.17 Dipyrityl solution.** Dissolve 0.2 g of 2,2-dipyrityl in 100 mL of 7.3 g/L hydrochloric acid (1 + 160).

**3.2.18 Diquinolyl reagent solution.** Dissolve 0.03 g of 2,2-diquinolyl in 100 mL of *n*-hexanol that has been freshly distilled from solid sodium hydroxide.

**3.2.19 Ethanol (or industrial methylated spirits)<sup>1)</sup>**

<sup>1)</sup> It should be noted that the use of industrial methylated spirits is governed by the Methylated Spirits Regulations 1983 (S.I. 1983, No. 252).

**3.2.20 Hydrochloric acid**,  $\rho = 1.18$  g/L.

**3.2.21 Hydrofluoric acid**,  $\rho = 1.13$  g/L.

**3.2.22 Hydroquinone solution**. Dissolve 1 g of hydroquinone in 100 mL of ethanol.

**3.2.23 Hydroxyammonium chloride**

**3.2.24 Hydroxyammonium chloride solution**. Dissolve 25 g of hydroxyammonium chloride in approximately 80 mL of water. Filter if necessary and dilute to 100 mL with water.

If any appreciable amounts of copper are present in the solution, extract with successive 10 mL portions of a 0.01 % solution of dithizone in carbon tetrachloride until there is no more violet colouration due to copper, hence no change in the green colour of the dithizone solution. Then extract the solution with carbon tetrachloride until all colour has been removed from the solution.

**3.2.25 N-hexanol**

**3.2.26 Nitric acid**,  $\rho = 1.42$  g/L.

**3.2.27 Perchloric acid**,  $\rho = 1.13$  g/L.

**3.2.28 Phosphoric acid solution**. Add 200 mL of orthophosphoric acid ( $\rho = 1.75$  g/L) to 700 mL of water and dilute to 1 L.

**3.2.29 Phosphorus pentoxide**

**3.2.30 Potassium hydrogen sulphate**

**3.2.31 Potassium periodate**

**3.2.32 Sodium acetate solution**. Dissolve 136 g of sodium acetate (trihydrate) in water and dilute to 1 L with water.

When the solution is to be used for the determination of copper and any appreciable amounts of copper are present, purify by extraction with 0.01 % dithizone solution in carbon tetrachloride as described for hydroxyammonium chloride solution.

**3.2.33 Sodium diethyldithiocarbamate solution**. Dissolve 1 g of sodium diethyldithiocarbamate in 1 L of water.

**3.2.34 Sodium hydroxide**

**3.2.35 Sodium hydroxide solution**. Dissolve 160 g of sodium hydroxide in 1 L of water.

**3.2.36 Sodium nitrite solution**. Dissolve 2 g of sodium nitrite in 100 mL of water.

**3.2.37 Sodium sulphate solution**. Dissolve 150 g of sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) in 1 L of water.

**3.2.38 Sodium sulphate/alcohol wash**. To 80 mL of water, add 10 mL of industrial methylated spirits<sup>2)</sup> and 10 mL of sodium sulphate solution.

**3.2.39 Sucrose**. Granulated sugar.

**3.2.40 Sulphuric acid**,  $\rho = 1.84$  g/L.

**3.2.41 Thioacetamide**

**3.2.42 Thioacetamide solution**. To 0.2 mL of a 40 g/L solution of thioacetamide, add 1 mL of a mixture of 4 mL sodium hydroxide solution, 10 mL water and 20 mL glycerin. Heat on a water bath for 20 s. Prepare immediately before use.

**3.2.43 Triethanolamine solution**

**3.2.44 Trisodium citrate solution**. Dissolve 294 g of trisodium citrate in 1 L of water.

### 3.3 Standard solutions

**3.3.1 Aluminium, standard stock solution**, 1 mL  $\equiv$  1 000  $\mu\text{g}$  Al. Dissolve 17.5830 g of  $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  in 500 mL of water. Add 5 mL of hydrochloric acid ( $\rho = 1.18$  g/L) and dilute to 1 L in a volumetric flask.

**3.3.2 Aluminium, standard working solution**, 1 mL  $\equiv$  10  $\mu\text{g}$  Al. Dilute 10 mL of the standard stock solution to 1 L with water in a volumetric flask.

**3.3.3 Calcium, standard solution (0.01M)**. 1 mL  $\equiv$  400  $\mu\text{g}$  Ca. Dissolve 1.0000 g of calcium carbonate, previously dried at 105 °C, in a slight excess of hydrochloric acid (1 + 4), boil to expel carbon dioxide, cool and dilute to 1 L in a volumetric flask.

<sup>2)</sup> It should be noted that the use of industrial methylated spirits is governed by the Methylated Spirits Regulations 1983 (S.I. 1983, No. 252).

**3.3.4 Copper, standard stock solution,** 1 mL  $\equiv$  200  $\mu$ g Cu. Dissolve 0.1000 g of pure copper in 3 mL of nitric acid, add 1 mL of sulphuric acid (1 + 1) and evaporate to fuming. Allow to cool, dissolve the residue in water and dilute to 500 mL in a volumetric flask.

**3.3.5 Copper, standard working solution,** 1 mL  $\equiv$  1  $\mu$ g Cu. Dilute 5 mL of the standard stock solution to 1 L with water in a volumetric flask.

**3.3.6 EDTA (0.05M).** Dissolve 18.60 g of ethylenediamine-tetra-acetic acid disodium salt in 1 L of water. Store in a polyethylene bottle. Standardize against standard calcium solution.

**3.3.7 EDTA (0.01M).** Dissolve 3.72 g of ethylenediamine-tetra-acetic acid disodium salt in 1 L of water. Store in a polyethylene bottle. Standardize against standard calcium solution.

**3.3.8 Fluoride, standard stock solution,** 1 mL  $\equiv$  100  $\mu$ g F. Dissolve 0.220 g sodium fluoride in water, dilute to 1 L in a volumetric flask and mix thoroughly. Store in a polyethylene bottle.

**3.3.9 Fluoride, dilute solution,** 1 mL  $\equiv$  10  $\mu$ g F. Pipette 10 mL of the standard stock solution into a 100 mL volumetric flask. Dilute to the mark and mix. Prepare this solution on the day the sample is to be analysed.

**3.3.10 Fluoride, working solution,** 1 mL  $\equiv$  1  $\mu$ g F. Pipette 10 mL of dilute solution into a 100 mL volumetric flask. Dilute to the mark and mix. Prepare this solution on the day the sample is to be analysed.

**3.3.11 Hydrochloric acid (M).** Dilute 90 mL of hydrochloric acid to 1 L with water. Standardize against sodium carbonate using screened methyl orange as indicator.

**3.3.12 Hydrochloric acid (0.5M).** Dilute 45 mL of hydrochloric acid to 1 L with water. Standardize against sodium carbonate using screened methyl orange as indicator.

**3.3.13 Hydrochloric acid (0.1M).** Dilute 50 mL of hydrochloric acid (M) to 500 mL with water in a volumetric flask.

**3.3.14 Iron, standard stock solution,** 1 mL  $\equiv$  500  $\mu$ g Fe. Dissolve 0.500 g of pure iron in 50 mL of hydrochloric acid (1 + 1). Transfer to a 1 L volumetric flask and dilute to the mark with water.

**3.3.15 Iron, standard working solution,** 1 mL  $\equiv$  50  $\mu$ g Fe. Dilute 25 mL of the standard stock solution to 250 mL with water in a volumetric flask.

**3.3.16 Lead, standard stock solution,** 1 mL  $\equiv$  100  $\mu$ g Pb. Dissolve 0.1598 g of lead nitrate  $\text{Pb}(\text{NO}_3)_2$  in 100 mL of water containing 1 mL of nitric acid. Dilute to 1 L in a volumetric flask and mix thoroughly.

**3.3.17 Lead, standard working solution,** 1 mL  $\equiv$  1  $\mu$ g Pb. Dilute 1 mL of the standard stock solution to 100 mL in a volumetric flask with water on the day it is to be used.

**3.3.18 Manganese, standard working solution,** 1 mL  $\equiv$  100  $\mu$ g Mn. Dissolve 0.1436 g of potassium permanganate in 250 mL of water, reduce with a very slight excess of sulphur dioxide gas dissolved in water to 500 mL in a volumetric flask.

**3.3.19 Sodium hydroxide (0.5M).** Dissolve 20 g of sodium hydroxide in water and make up to 1 L with water. Standardize against hydrochloric acid (0.5M) using phenolphthalein as indicator.

### 3.4 Indicator solutions

**3.4.1 Bromophenol blue.** To 100 mL of ethanol, add 3 mL of sodium hydroxide solution (0.1M) and 0.2 g of bromophenol blue. Dilute to 500 mL with water.

**3.4.2 Calcon<sup>3)</sup>**

**3.4.3 Calcon triturate.** Grind with a mortar and pestle 1 g of Calcon with 99 g of sodium chloride previously dried at 105 °C.

**3.4.4 Methyl orange screened with xylene cyanol.** Dissolve 0.2 g of methyl orange in 100 mL of water and add this solution to a solution of 0.4 g xylene cyanol in 10 mL of industrial methylated spirits and 90 mL of water.

**3.4.5 Methyl red.** Dissolve 0.5 g of methyl red in 100 mL of water.

**3.4.6 Phenolphthalein.** Dissolve 0.5 g of phenolphthalein in 50 mL of industrial methylated spirits and dilute to 100 mL with water.

**3.4.7 Mordant black II.<sup>4)</sup>** Grind with a mortar and pestle 0.2 g of mordant black II with 100 g of sodium chloride previously dried at 105 °C. Store in a sealable container.

<sup>3)</sup> Available commercially as, e.g. Solochrome dark blue (C.I. 15705). See BS 4123.

<sup>4)</sup> Available commercially as, e.g. Solochrome black. See BS 4123.