

Quicklime, hydrated lime and natural calcium carbonate —

Part 102: Methods for chemical analysis

ICS 91.100.10

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by Technical Committee B/516, Cement and lime, to Subcommittee B/516/11, Lime, upon which the following bodies were represented:

- Autoclaved Aerated Concrete Products Association
- British Lime Association
- Building Limes Forum
- Department of the Environment, Transport and the Regions — Building Research Establishment
- English Heritage
- Mortar Industry Association
- Quarry Products Association
- Co-opted members

The following bodies from the Technical Committee were also involved in the preparation of this standard:

- British Cement Association
- British Precast Concrete Federation Ltd.
- Cement Admixtures Association
- Cementitious Slag Makers Association
- County Surveyors Society
- Department of the Environment, Transport and the Regions — Highways Agency
- Environment Agency
- Society of Chemical Industry
- UK Quality Ash Association

This British Standard, having been prepared under the direction of the Sector Policy and Strategy Committee for Building and Civil Engineering, was published under the authority of the Sector Policy and Strategy Committee on 6 November 2001

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First published as BS 6463-2, February 1984
Second edition as BS 6463-102, 6 November 2001

The following BSI references relate to work on this British Standard:
Committee reference B/516/11
Draft for comment 99/105742 DC

ISBN 0 580 33315 9

Amendments issued since publication

Amd. No.	Date	Comments

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Foreword

This part of BS 6463 has been prepared by Subcommittee B/516/11, Lime, under the direction of Technical Committee B/516, Cement and lime. It supersedes BS 6463-2:1984 which is withdrawn.

BS 6463-2:1984 brought together standard methods of chemical analysis for quicklime, hydrated lime and natural calcium carbonate including those for use in connection with water treatment, agriculture, steel making and chemical processing.

In that edition, atomic absorption methods were permitted. Since 1984 the use of atomic spectrometry has been widely used in this field of activity. These methods require special attention to calibration using reference materials.

This edition describes alternative analytical methods for several elements. Where available and appropriate, atomic spectrometry using atomic absorption (AAS) is recommended, although classical methods are still used by many laboratories for the determination of certain analytes and parameters. Repeatability and reproducibility for the test methods are not yet available. However, when the necessary testing has been done, this standard will be updated accordingly.

Other atomic absorption spectrometric (AAS) methods are available in BS EN 12485, and include determinations of antimony, arsenic, mercury and selenium.

These methods are to be preferred over the classical methods and it is recommended that they are adopted in specifications as the reference methods.

NOTE There are other atomic spectrometric methods that can give the sensitivity, precision and accuracy required by this part of BS 6463, the results of which can be calibrated against the methods described in this part of BS 6463. However, if a standard specifies a reference to BS 6463-102, only methods actually given in this part of BS 6463 can be used in order to claim compliance with that standard.

A number of classical chemical methods that were given in BS 6463-2:1984 have now been incorporated into other standards, and are no longer included in this part of BS 6463. These methods, and the standards in which they are now given, are for:

- available lime (BS EN 459-2);
- silicon (BS EN 196-2);
- iron (BS EN 196-2);
- calcium (BS EN 12485);
- magnesium (BS EN 12485);
- water soluble calcium oxide and calcium hydroxide (BS EN 12485);
- water insoluble constituents (BS EN 12485).

This revision of BS 6463 is published in three parts, as follows:

- Part 101: *Methods for preparing samples for testing*;
- Part 102: *Methods for chemical analysis*;
- Part 103: *Methods for physical testing*.

Part 101 gives methods for sampling powdered, granular and lump quicklime and natural calcium carbonate, and of powdered hydrated lime, milk of lime and lime putty.

Part 103 gives the physical tests for quicklime, hydrated lime, lime putty and natural calcium carbonate, of which the tests for quicklime, hydrated lime and lime putty were previously included in BS 6463-3 and BS 6463-4.

A British Standard does not purport to include all 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 to iv, pages 1 to 21 and a back cover.

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1 Scope

This part of BS 6463 describes methods for the chemical analysis of quicklime, hydrated lime and natural calcium carbonate. It describes both classical methods and methods which use atomic absorption spectrometry (AAS).

It gives classical chemical methods for:

- fluorine, matter insoluble in acetic acid, matter insoluble in hydrochloric acid;
- loss on ignition, neutralizing value;
- free water in hydrated lime and also in lime putty;
- total sulfur;
- carbon dioxide in carbonate.

It gives flame atomic absorption spectrometric (AAS) methods for aluminium, cadmium, chromium, cobalt, copper, iron, lead, magnesium, manganese, nickel, silicon, silver and zinc.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of this British Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. For undated references, the latest edition of the publication referred to applies.

BS 1752:1983, *Specification for laboratory sintered or fritted filters including porosity grading*.

BS 5898:1980, *Specification for principles of design and construction of volumetric glassware for laboratory use*.

BS 6463-101:1996, *Quicklime, hydrated lime and natural calcium carbonate — Part 101: Methods for preparing samples for testing*.

BS EN 12485, *Chemicals used for treatment of water intended for human consumption — Calcium carbonate, high-calcium lime and half-burnt dolomite — Test methods*.

BS EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods*.

BS ISO 3534-1:1993, *Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms*.

3 General requirements for testing

3.1 Number of tests

For each of the determinations in clauses 4 and 5, carry out two tests.

3.2 Reagents

Water shall conform to grade 2 of BS EN ISO 3696.

All reagents shall be of an analytical grade suitable for the method being used. For instrumental analysis, the instructions of the equipment manufacturer shall be observed. The content of the elements to be determined in the water or reagents used shall be negligible compared with the lowest concentration to be determined.

If commercially available AAS solutions are used as stock solutions, they shall be prepared for the purposes of analysis in accordance with the manufacturer's instructions.

The concentrated liquid reagents used in this standard shall have the densities shown in Table 1.

Table 1 — Densities of concentrated liquid reagents

Reagent	Density (ρ) at 20 °C g/ml
Acetic acid	1.05 to 1.06
Hydrochloric acid	1.16 to 1.19
Industrial methylated spirit	0.79
Nitric acid	1.40 to 1.42
Orthophosphoric acid	1.70 to 1.75
Sulfuric acid	1.84

NOTE The degree of dilution is always given as a volumetric sum, e.g. “dilute hydrochloric acid 1 + 2” means that 1 volume of concentrated hydrochloric acid is to be mixed with 2 volumes of water.

3.3 Volumetric glassware

Volumetric glassware shall be of class B as specified in BS 5898.

3.4 Preparation of the sample

Prepare the laboratory sample as described in BS 6463-101. Carry out all operations as quickly as possible to ensure that the sample is exposed to ambient air only for the minimum time that is necessary. Store the sample in an air-tight container of such a volume that the sample fills the container as completely as possible.

3.5 Expression of results

Express the results as the mean of two determinations.

Express results of value not less than 1.00 % to three significant figures. Express results of value less than 1.00 % to two significant figures. If the value of the result is less than 0.10 % express it to two significant figures where feasible.

4 Determination of major and minor constituents by classical methods

4.1 Determination of hydrochloric acid extractable fluoride

4.1.1 Principle

The sample is stirred with concentrated hydrochloric acid and the extracted fluorine is determined using a specific fluoride ion electrode which develops a potential proportional to the logarithm of the fluoride ion concentration in solution.

The method is applicable to quicklimes and hydrated limes, with fluorine contents in the range 20 mg/kg to 500 mg/kg. It is also applicable to natural calcium carbonates which are used for the neutralization of moderately acidic solutions ($\text{pH} > 1$).

NOTE The extractable fluoride content of a natural calcium carbonate should not be used to predict the levels of extractable fluoride in quicklimes and hydrated limes. This is due partly to the effects of the calcination and hydration reactions and partly to the volatility of fluorides at elevated temperatures.

4.1.2 Reagents

4.1.2.1 Sodium fluoride, NaF, dried at $(105 \pm 5)^\circ\text{C}$ for 1 h.

4.1.2.2 Fluoride, standard stock solution, with a concentration of fluoride ions of 500 $\mu\text{g/ml}$. Dissolve (1.105 ± 0.005) g of sodium fluoride (4.1.2.1) in water, make up to 1 000 ml in a volumetric flask and mix thoroughly. Store in a suitable container and under suitable conditions for up to 14 days if necessary (see note).

NOTE All fluoride solutions may be kept for limited periods providing they are placed in a cool dark cupboard in stoppered containers made of a material which has been shown not to interfere with the determination of fluorine. Laboratory grade polyethylene or polypropylene is usually suitable.

4.1.2.3 Fluoride, dilute solution, with a concentration of fluoride ions of 50 µg/ml. Pipette 50 ml of the standard stock fluoride solution (4.1.2.2) into a 500 ml volumetric flask. Make up to the mark with water and mix.

NOTE This solution can be kept for up to 14 days if stored as described (see note to 4.1.2.2).

4.1.2.4 Fluoride, dilute solution, with a concentration of fluoride ions of 5 µg/ml. Pipette 50 ml of fluoride solution (4.1.2.3) into a 500 ml volumetric flask. Make up to the mark with water and mix.

NOTE This solution can be kept for up to 14 days if stored as described (see note to 4.1.2.2).

4.1.2.5 Reference standard calcium carbonate, with less than 1 mg/kg of hydrochloric acid extractable fluorine.

4.1.2.6 Trisodium citrate dihydrate.

4.1.2.7 Trisodium citrate solution. Dissolve (147.0 ± 0.1) g of trisodium citrate dihydrate (4.1.2.6) in water and dilute to 1 l.

4.1.2.8 Hydrochloric acid, 1 + 19.

4.1.2.9 Fluoride standard solution, 10 µg in 80 ml. Pipette 2 ml of fluoride solution (4.1.2.4) into a polyethylene beaker. Add (0.20 ± 0.01) g of calcium carbonate (4.1.2.5), 18 ml of distilled water, 40 ml of trisodium citrate solution (4.1.2.7) and 20 ml of hydrochloric acid 1 + 19 (4.1.2.8). Add a magnetic follower and stir.

NOTE This solution can be kept for up to 48 h, if stored as described (see note to 4.1.2.2).

4.1.2.10 Fluoride standard solution, 100 µg in 80 ml. Pipette 20 ml of fluoride solution (4.1.2.4) into a polyethylene beaker. Add (0.20 ± 0.01) g of calcium carbonate (4.1.2.5), 40 ml of trisodium citrate solution (4.1.2.7) and 20 ml of hydrochloric acid 1 + 19 (4.1.2.8). Add a magnetic follower and stir.

NOTE This solution can be kept for up to 48 h, if stored as described (see note to 4.1.2.2).

4.1.3 Apparatus

4.1.3.1 Plastics beaker.

4.1.3.2 Magnetic follower.

4.1.3.3 Millivoltmeter, with an expandable scale and a specific ion electrode for fluorine, containing a lanthanum fluoride crystal across which a potential develops proportional to the fluoride ion concentration in solution. This potential is measured against a calomel reference electrode.

4.1.4 Preparation of sample solution

Accurately weigh (0.200 ± 0.005) g of sample into a plastics beaker (4.1.3.1). Add 25 ml of hydrochloric acid 1 + 19 (4.1.2.8) and a magnetic follower (4.1.3.2). Stir for 15 min and add 15 ml of distilled water and 40 ml of trisodium citrate solution (4.1.2.7).