BRITISH STANDARD

BS EN
451-1:2003

Method of testing fly ash —

Part 1: Determination of free calcium oxide content

The European Standard EN 451-1:2003 has the status of a British Standard

ICS 91.100.30

National foreword

This British Standard is the official English language version of EN 451-1:2003. It supersedes BS EN 451-1:1995 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee B/516, Cement and lime, to Subcommittee B/516/101, Additions for concrete, which has the responsibility to:

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English version

Method of testing fly ash - Part 1: Determination of free calcium oxide content

Méthode d'essai des cendres volantes - Partie 1: Détermination de la teneur en oxyde de calcium libre

Prüfverfahren für Flugasche - Teil 1: Bestimmung des freien Calciumoxidgehalts

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Foreword

This document (EN 451-1:2003) has been prepared by Technical Committee CEN/TC 104 “Concrete and related products”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2004, and conflicting national standards shall be withdrawn at the latest by June 2004.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

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1 Scope

This European Standard describes the procedure for the determination of free calcium oxide content in fly ash.

The standard describes the reference procedure. If other methods are used it shall be shown that they give results equivalent to those obtained by the reference method.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 450, *Fly ash for concrete - Definitions, requirements and quality control*

ISO 565, *Test sieves — Metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings*.

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

3 General

3.1 Number of determinations

Carry out twice the procedure for determining the free calcium oxide content.

If the difference between two determinations is more than twice the repeatability standard deviation, repeat the test and take the mean of the closest values.

3.2 Repeatability and reproducibility

The standard deviations of repeatability and reproducibility, as defined in ISO 3534-1, are expressed in absolute percentages by mass.

3.3 Expression of masses and volumes

Express masses in grams to the nearest 0,000 1 g and volumes from the burette in millilitres to the nearest 0,05 ml.

4 Reagents

4.1 General

Use only reagents of analytical grade and distilled water, or water of equal purity.

4.2 Butanoic acid, 3-oxo-ethyl ester (= ethyl acetoacetate).

4.3 Butan-2-ol.

4.4 Propan-9-ol.

4.5 Indicator (0,1 g of bromophenol blue in 100 ml of ethanol).

4.6 Hydrochloric acid (0,100 M).

5 Apparatus

5.1 **Balance**, capable of weighing to an accuracy of 0,000 1 g or better

5.2 **Test sieve**, with 0,063 mm sieve cloth conforming to ISO 565.

5.3 **Mortar and pestle**, or similar equipment for grinding.

5.4 **Desiccator**, containing a drying agent, e.g. silica gel.

5.5 **Flask**, 250 ml.

5.6 **Spiral reflux** condenser:

5.7 **Absorption tube**, filled with sodium hydroxide on an inorganic carrier (to protect the contents of the flask and the condenser from reacting with atmospheric carbon dioxide).

5.8 **Filter crucible**, with pore sizes of 0,004 mm to 0,010 mm.

NOTE Alternatively also a filter funnel, in which a filter paper with fine pores of a diameter of approximately 0,002 mm and a filter paper with medium pores of a diameter of approximately 0,007 mm can be placed, may be used.

6 Procedure

6.1 Preparation of sample

Subdivide the laboratory sample, prepared in accordance with 6.2.2 of EN 450 by a suitable method to obtain a subsample of about 100 g. Pass this subsample through the test sieve (5.2). Grind any residue in the mortar (5.3) until all the subsample passes through the sieve completely. Homogenize the total subsample and place it in the desiccator (5.4) until tested.

6.2 Determination

Place a weighed portion of 1,0 g to 1,5 g of the sub-sample prepared in accordance with 6.1 into the 250 ml flask (5.5) and add a mixture of 12 ml butanoic acid, 3-oxo-ethyl ester (4.2) and 80 ml butan-2-ol (4.3). Fit the flask with the spiral reflux condenser (5.6) and the absorption tube (5.7) and boil for 3 h. Filter the warm mixture through the filter crucible (5.8). Wash the residue with 50 ml propan-2-ol (4.4). If the filtrate is cloudy, reject it and repeat the extraction with a new weighed portion of the sub sample.

Add a few drops of bromophenol blue indicator (4.5) to the filtrate and titrate with hydrochloric acid (4.6) until the colour changes to yellow.

Record the volume V of hydrochloric acid used.

6.3 Calculations

The free calcium oxide content (W_{CaO}), expressed as a percentage by mass of the dry fly ash, shall be calculated from the following equation:

$$W_{\mathrm{CaO}} = \frac{28{,}04 \quad C \quad V}{m \quad 1\,000} \quad 100 \qquad (1)$$

where

C is the concentration (in mol/l) of the hydrochloric acid solution;

V is the volume (in ml) of hydrochloric acid solution used for titration;

m is the weighed proportion (in g) of the dried fly ash.

7 Results

The mean value of two determinations, calculated to two decimal places and expressed to one decimal place, shall be taken as the free calcium oxide content of the sample.

8 Repeatability and reproducibility

The standard deviation for repeatability is 0,02 % by mass (provisional value).

The standard deviation for reproducibility is 0,04 % by mass (provisional value).

Bibliography

EN 196-7:1989, *Methods of testing cement - Methods of taking and preparing samples of cement*.

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