

# Method of testing fly ash

## Part 1. Determination of free calcium oxide content

The European Standard EN 451-1 : 1994 has the status of a British Standard

UDC 666.952.2 : 620.1 : 661.842.22

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## 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/516/101, Pozzolanas, upon which the following bodies were represented:

- British Aggregate Construction Materials Industries
- British Cement Association
- British Precast Concrete Federation
- British Ready Mixed Concrete Association
- Cementitious Slag Makers' Association
- Department of the Environment (Building Research Establishment)
- Department of Transport
- Electricity Association
- Federation of Civil Engineering Contractors
- Quality Ash Association

This British Standard, having been prepared under the direction of the Sector Board for Building and Civil Engineering, was published under the authority of the Standards Board and comes into effect on 15 September 1995

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The following BSI references relate to the work on this standard:  
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## National foreword

This Part of BS EN 451 has been prepared by Subcommittee B/516/101 and is the English language version of EN 451-1 : 1994 *Method of testing fly ash - Part 1: Determination of free calcium oxide content* published by the European Committee for Standardization (CEN). This standard has been prepared as part of a package of standards harmonized within the member countries of CEN to support the essential requirements of the Construction Products Directive.

BS EN 451-1 describes a method for the determination of free calcium oxide in fly ash by extraction with a mixture of ethyl acetoacetate and butan-2-ol followed by titration with hydrochloric acid using bromophenol blue as indicator. The method has been used successfully for the determination of free calcium oxide in Portland cements but there is limited experience in the UK of applying the method to fly ashes.

There is a technical distinction between the various states in which calcium oxide may be present in ash, only that present as free calcium oxide having the potential to cause long-term unsoundness in concrete. Calcium oxide present as part of the glassy phase in ash, or otherwise bound in chemical compounds, does not lead to unsoundness.

BS EN 451-1 supports BS EN 450 *Fly ash for concrete - Definitions, requirements and quality control*. The national foreword to BS EN 450 explains the relationship between BS EN 450 and BS 3892 : Part 1. In particular, it is noted that whilst both standards specify ashes arising from pulverized bituminous or other hard coals, BS EN 450 also permits ashes from sub-bituminous coals on a national basis. Ashes from such sub-bituminous coals, which do not occur in the UK, may contain free calcium oxide. However, the likelihood of free calcium oxide being present in ashes from bituminous or other hard coals such as are specified in BS 3892 : Part 1 are remote.

Hence whilst both BS EN 450 and BS 3892 : Part 1 include a requirement for the content of total calcium oxide, only the former includes a requirement for free calcium oxide. An autocontrol maximum of 1.0 % is specified, although a value of up to 2.5 % is permitted provided that the ash complies with the requirement for soundness specified in BS EN 450.

### Cross-references

Publication referred to	Corresponding British Standard
EN 196-7 : 1989	BS EN 196-7 : 1992 <i>Methods of testing cement Part 7. Methods of taking and preparing samples of cement</i>
ISO 3534-1 : 1993	BS ISO 3534 <i>Statistics. Vocabulary and symbols</i> BS ISO 3534-1 : 1993 <i>Probability and general statistical terms</i>

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NORME EUROPÉENNE  
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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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**CEN**

European Committee for Standardization  
Comité Européen de Normalisation  
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

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**Foreword**

This European Standard was drawn up by the Technical Committee CEN/TC 104, Concrete, the Secretariat of which is held by DIN.

The preparatory work was carried out by WG 4 of CEN/TC 104 since June 1988 in which the following countries participated: Austria, Belgium, Denmark, France, Germany, Ireland, Italy, Netherlands, Norway, Portugal, Sweden and United Kingdom.

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

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 March 1995, and conflicting national standards shall be withdrawn at the latest by March 1995.

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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.

EN 196-7 : 1989	<i>Methods of testing cement - Methods of taking and preparing samples of cement</i>
ISO 565 : 1990	<i>Test sieves - Woven metal wire cloth, perforated plate and electroformed sheet - Nominal sizes of openings</i>
ISO 3534-1 : 1993	<i>Statistics - Vocabulary and symbols - Part 1: Probability and general statistical terms</i>

## 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, are expressed in absolute percentages by mass.

### 3.3 Expression of masses and volumes

Express masses in grams to the nearest 0,0001 g and volumes from the burette in millilitres to the nearest 0,05 ml.

## 4 Reagents

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

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

4.2 *Butan-2-ol*.

4.3 *Propan-2-ol*.

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

4.5 *Hydrochloric acid* (0,100 M).

## 5 Apparatus

5.1 *Balance*, capable of weighing to an accuracy of 0,0001 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 subsample 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 (4.1) and 80 ml butan-2-ol (4.2). Fit the flask with the spiral reflux condenser (5.6) and the absorption tube (5.7) and boil for 1 h. Filter the warm mixture through the filter crucible (5.8). Wash the residue with propan-2-ol (4.3) until the filtrate reaches a volume of 50 ml. If the filtrate is cloudy, reject it and repeat the extraction with a new weighed portion of the subsample.

Add a few drops of bromophenol blue indicator (4.4) to the filtrate and titrate with hydrochloric acid (4.5) until the colour changes to yellow. Record the volume  $V$  of hydrochloric acid used.

### 6.3 Calculations

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

$$W_{\text{CaO}} = \frac{28,04 \times C \times V}{m \times 1000}$$

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).



## List of references

See national foreword.

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