

BSI Standards Publication

Fly ash for concrete

Part 1: Definition, specifications and conformity criteria



BS EN 450-1:2012 BRITISH STANDARD

National foreword

This British Standard is the UK implementation of EN 450-1:2012. It supersedes BS EN 450-1:2005+A1:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/517/4, Additions for concrete.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Fly ash for concrete - Part 1: Definition, specifications and conformity criteria

Cendres volantes pour béton - Partie 1: Définition, spécifications et critères de conformité

Flugasche für Beton - Teil 1: Definition, Anforderungen und Konformitätskriterien

This European Standard was approved by CEN on 6 July 2012.

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EUROPEAN COMMITTEE FOR STANDARDIZATION COMITÉ EUROPÉEN DE NORMALISATION EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 450-1:2012) 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 450-1:2005+A1:2007.

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 89/106/EEC.

For relationship with EU Directive(s), see informative Annex ZA, which is an integral part of this document.

This document is supported by standards of the series EN 451 for test methods for the determination of free calcium oxide content and of the fineness by sieving.

The main changes with respect to the previous edition are listed below:

- extension of the permissible content of solid co-combustion materials;
- extension of the permissible content of liquid and gaseous co-combustion materials;
- fixed limits for loss on ignition;
- incorporation of the specifications from EN 450-1+A1:2007;
- general editorial revision.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The use of coal for electricity production results in the generation of large quantities of fly ash.

Different types of coal and the type of boiler used in this process produce different fly ashes, such as siliceous, silico-calcareous, or calcareous fly ashes with pozzolanic and/or latent hydraulic properties. All these types of fly ash are used in concrete production in some European countries, based on national experience and tradition.

Before use, fly ash may be subject to processing, for example by classification, selection, sieving, drying, blending, grinding or carbon reduction, to optimize its fineness, reduce its water demand or to improve other properties. Such processed fly ashes may conform to this document to which reference is made in such case. If they are out of the scope of this document, their suitability for use as Type II additions in concrete according to EN 206-1 may also be established from national standards or provisions or European Technical Approvals valid in the place of use of the concrete and which refer specifically to the use of the addition in concrete conforming to EN 206-1.

When using fly ashes conforming to this document, it should be noted that, apart from the effect from the pozzolanicity of the fly ash, certain properties of fresh and hardened concrete may be affected. Where relevant, such effects need to be considered in concrete mix design (see EN 206-1).

1 Scope

This European Standard specifies requirements for the chemical and physical properties as well as quality control procedures for siliceous fly ash, as defined in 3.2, for use as a type II addition for production of concrete conforming to EN 206-1. Fly ash according to this document may also be used in mortars and grouts.

Fly ash produced with other types or higher percentages of co-combustion materials than those provided for in Clause 4 is outside the scope of this European Standard.

It is beyond the scope of this European Standard to specify provisions governing the practical application of fly ash in the production of concrete, i.e. requirements concerning composition, mixing, placing, curing etc. of concrete containing fly ash. Regarding such provisions, reference should be made to other European or national standards for concrete, such as EN 206-1.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 196-1:2005, Methods of testing cement — Part 1: Determination of strength

EN 196-2:2005 Methods of testing cement — Part 2: Chemical analysis of cement

EN 196-3, Methods of testing cement — Part 3: Determination of setting times and soundness

EN 196-7, Methods of testing cement — Part 7: Methods of taking and preparing samples of cement

EN 197-1:2011, Cement — Part 1: Composition, specification and conformity criteria for common cements

EN 206-1, Concrete — Part 1: Specification, performance, production and conformity

EN 450-2:2005, Fly ash for concrete — Part 2: Conformity evaluation

EN 451-1, Method of testing fly ash — Part 1: Determination of free calcium oxide content

EN 451-2, Method of testing fly ash — Part 2: Determination of fineness by wet sieving

EN 933-10, Tests for geometrical properties of aggregates — Part 10: Assessment of fines — Grading of filler aggregates (air jet sieving)

EN 1015-3:1999, Methods of test for mortar for masonry — Part 3: Determination of consistence of fresh mortar (by flow table)

EN 1097-7, Tests for mechanical and physical properties of aggregates — Part 7: Determination of the particle density of filler — Pyknometer method

EN ISO 11885, Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICPOES) (ISO 11885)

EN 14588:2010, Solid biofuels — Terminology, definitions and descriptions

ISO 10694, Soil quality — Determination of organic and total carbon after dry combustion (elementary analysis)

ISO 29581-2, Cement — Test methods — Part 2: Chemical analysis by X-ray fluorescence

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1

type II addition

finely divided inorganic, pozzolanic or latent hydraulic material that may be added to concrete in order to improve certain properties or to achieve special properties

Note 1 to entry: See EN 206-1.

3.2

fly ash

fine powder of mainly spherical, glassy particles, derived from burning of pulverised coal, with or without co-combustion materials, which has pozzolanic properties and consists essentially of SiO_2 and Al_2O_3 and which:

- is obtained by electrostatic or mechanical precipitation of dust-like particles from the flue gases of the power stations; and
- may be processed, for example by classification, selection, sieving, drying, blending, grinding or carbon reduction, or by combination of these processes, in adequate production plants, in which case it may consist of fly ashes from different sources, each conforming to the definition given in this clause

Note 1 to entry: Municipal and industrial waste incineration ashes do not conform to the definition given in this clause.

3.3

test cement

selected brand of Portland cement of type CEM I, strength class 42,5 or higher, conforming to EN 197-1 to be used for carrying out the tests needed to evaluate conformity to the requirements of 5.3.2, 5.3.3, 5.3.5 and 5.3.6

3.4

particle density

average density of fly ash particles, including voids inside the particles

3.5

activity index

ratio (in percent) of the compressive strength of standard mortar bars, prepared with 75 % test cement plus 25 % fly ash by mass, to the compressive strength of standard mortar bars prepared with 100 % test cement, when tested at the same age

3.6

autocontrol

continuous statistical quality control of the fly ash based on the testing of samples taken by the producer or their agent at point(s) of release from the fly ash producing facility

3.7

control period

period of production and/or dispatch identified for the evaluation of the autocontrol test results

3.8

characteristic value

value of the required property outside of which lies a specified percentage, the percentile $P_{\mathbf{k}}$, of all the values of the population

3.9

single result limit value

value of a chemical or physical property which – for any single test result – in the case of an upper limit is not to be exceeded or in the case of a lower limit is, as a minimum, to be reached

3.10

allowable probability of acceptance CR

for a given sampling plan, the allowed probability of acceptance of fly ash with a characteristic value outside the specified minimum or maximum values

3.11

sampling plan

specific plan which states the (statistical) sample size(s) to be used, the percentile P_k , and the allowable probability of acceptance CR

3.12

spot sample

sample taken at the same time and from one and the same place, relating to the intended tests which can be obtained by combining one or more immediately consecutive increments

Note 1 to entry: See EN 196-7.

3.13

green wood

wood originating from trees, bushes and shrubs that is created when processing wood as cross-cut ends, planings, saw dust and shavings used in the form of dust, chips and pellets

4 Specific provisions for fly ash from co-combustion

4.1 Co-combustion materials

Fly ash from co-combustion as defined in 3.2 is obtained from pulverised coal fired simultaneously with at least one co-combustion material as listed in Table 1. The minimum percentage, by dry mass, of coal (K_c) shall be not less than 60 %, or 50 % if the co-combustion material is only from green wood (see 3.13). The maximum proportion of ash derived from co-combustion materials (M) shall not be greater than 30 % by dry mass when calculated from Formula (1).

The proportion of ash derived from co-combustion shall be calculated with Formula (1):

$$M = 100 (K_1 \times A_1 + K_2 \times A_2 ... K_n \times A_n) / (K_c \times A_c + (K_1 \times A_1 + K_2 \times A_2 ... K_n \times A_n))$$
 (1)

where

- M is the proportion of co-combustion ash in total fly ash, in % by mass;
- A_i is the ash content of co-combustion material no. i, in % by mass;
- *n* is the number of co-combustion materials being used;
- A_{c} is the ash content of coal, in % by mass;

 K_i and K_c are respectively the proportions of co-combustion material(s) and coal being fired;

and where

 $(K_c + K_1 + K_2 + ... K_0) = 1$ and $K_c \ge 0.60$, or 0.50 if the co-combustion material consists of green wood only.

In addition, if virtually ash free liquid and gaseous fuels are used as co-combustion materials, their percentage by net calorific value shall be determined and shall not exceed 40 % of the total net calorific value.

Higher percentages of virtually ash free liquid and gaseous fuel may be used during the start-up process of a power plant.

Table 1 — Types of co-combustion materials

1	Solid Bio Fuels conforming to EN14588:2010 including animal husbandry residues as defined in 4.5 and excluding waste wood as defined in 4.52, 4.132 and 4.174.			
2	Animal meal (meat and bone meal)			
3	Municipal sewage sludge			
4	Paper sludge			
5	Petroleum coke			
6	Virtually ash free liquid and gaseous fuels			

NOTE Other types of co-combustion materials not included in Table 1 might be subject to an ETA.

4.2 Establishment of suitability of fly ash from co-combustion

The suitability of fly ash obtained from combustion of coal with co-combustion materials given in Table 1 shall be established and documented by the producer. An initial co-combustion in the furnace using the highest intended amount of co-combustion material shall be performed. A representative sample of fly ash taken from this co-combustion (see Clause 7) shall be used to establish the suitability.

The suitability of fly ash obtained from co-combustion with one of the co-combustion materials specified in Table 1 is established if conformity to the requirements in 5.2, 5.3 and 5.4 has been proven.

5 Specifications

5.1 General

The chemical and physical requirements in 5.2 and 5.3 are specified as characteristic values. Conformity to a characteristic value is assessed by means of a statistical quality control procedure as described in Clause 8.

The test methods prescribed in this document are reference methods. Other test methods may be used provided they give results equivalent to those obtained with the reference method. In case of a dispute, only the reference method shall be used.

5.2 Chemical requirements

5.2.1 General

The chemical composition shall be expressed as proportions by mass of dry fly ash (see Clause 7 for a method of obtaining a sample of dry fly ash).

In performing the test methods for major elements described in EN 196-2:2005, Clause 13, the following modifications shall be observed:

- a) When heating the sample-peroxide mixture (EN 196-2:2005, 13.2) a furnace temperature of 550 °C shall be used and maintained for 60 min. Timing to start is when the temperature has recovered to 540 °C;
- b) When dispersing the sintered mass from the furnace (EN 196-2:2005, 13.2), proceed with the test even though the melting may not be clear;
- c) To decompose the evaporation residue (EN 196-2:2005, 13.7), add 2,0 g potassium bisulphate instead of the sodium carbonate/sodium chloride mixture.

5.2.2 Loss on ignition

The loss on ignition shall be determined in accordance with the principles of the method described in EN 196-2 but using an ignition time of 1 h, and shall fall within the limits of the categories specified below:

- Category A: not greater than 5,0 % by mass;
- Category B: not greater than 7,0 % by mass;
- Category C: not greater than 9,0 % by mass.

The purpose of this requirement is to limit the residue of unburnt carbon in the fly ash. It is sufficient, therefore, to show, through direct measurement of unburnt carbon residue, that the content of unburnt carbon falls within the limits of the categories specified above. The content of unburnt carbon shall be determined in accordance with ISO 10694.

NOTE As the magnitude of the loss on ignition may have an influence on the effect of air-entraining admixtures used for the manufacture of concrete resistant to freezing and thawing, the three loss on ignition categories defined in this document allow the user to take this into account by choosing the appropriate category for each particular application and exposure class, thus following the standards and/or regulations for concrete valid in the place of use.

5.2.3 Chloride

The content of chloride, expressed as Cl⁻, shall be determined in accordance with EN 196-2 and shall not be greater than 0,10 % by mass.

5.2.4 Sulphate (SO₃) content

The sulphate (SO₃) content, shall be determined in accordance with EN 196-2 and shall not be greater than 3,0 % by mass.

5.2.5 Free calcium oxide

The content of free calcium oxide shall be determined by the method described in EN 451-1. If the content of free calcium oxide is greater than 1,5 % by mass, the fly ash shall be tested for conformity to the requirements for soundness in 5.3.3.

5.2.6 Reactive calcium oxide

The content of reactive calcium oxide shall be calculated as stipulated in EN 197-1:2011, 3.1 and shall not exceed 10,0 % by mass.

If the total content of calcium oxide determined in accordance with 5.2.1 does not exceed 10,0 % by mass, the requirement for reactive calcium oxide shall be deemed to be satisfied.

5.2.7 Reactive silicon dioxide

In the initial type test for fly ash from co-combustion (4.2) the amount of reactive silicon shall be analysed in accordance with EN 197-1 and shall not be less than 25 % by mass. Fly ash obtained from combustion of pulverised coal only shall be deemed to satisfy this requirement.

5.2.8 Silicon dioxide (SiO₂), aluminium oxide (Al₂O₃) and iron oxide (Fe₂O₃)

The sum of the contents of silicon dioxide (SiO_2), aluminium oxide (Al_2O_3) and iron oxide (Fe_2O_3) shall be determined in accordance with EN 196-2, modified as indicated in 5.2.1, and shall not be less than 70 % by mass. Fly ash obtained from combustion of pulverised coal only shall be deemed to satisfy this requirement.

5.2.9 Total content of alkalis

The total content of alkalis shall be determined in accordance with EN 196-2 and calculated as Na_2O_{eq} and shall not exceed 5,0 % by mass. Fly ash obtained from combustion of pulverised coal only shall be deemed to satisfy this requirement.

5.2.10 Magnesium oxide

In the initial type test for fly ash from co-combustion (4.2), the content of magnesium oxide (MgO) shall be determined in accordance with EN 196-2 and shall not be greater than 4,0 % by mass. Fly ash obtained from combustion of pulverised coal only shall be deemed to satisfy this requirement.

5.2.11 Phosphate

The content of total phosphate (P_2O_5) shall be determined in accordance with ISO 29581-2 and shall not be greater than 5,0 % by mass. Fly ash obtained from combustion of pulverised coal only shall be deemed to satisfy this requirement.

In the initial type test for fly ash from co-combustion (4.2), the content of soluble phosphate (P_2O_5) shall be determined in accordance with the method described in Annex C and shall not be greater than 100 mg/kg.

5.3 Physical requirements

5.3.1 Fineness

The fineness of fly ash shall be expressed as the mass proportion in percent of the ash retained when sieved on a 0,045 mm mesh sieve. The fineness may be determined by wet sieving in accordance with EN 451-2 or by air jet sieving in accordance with EN 933-10 and shall fall within the limits of the categories specified below:

Category N: the fineness shall not exceed 40 % by mass, and it shall not vary by more than \pm 10 percentage points from the declared value.

Category S: the fineness shall not exceed 12 % by mass. The \pm 10 percentage points fineness variation limits are not applicable.

In case of dispute, the wet sieving method according to EN 451-2 shall be used as the reference method.

5.3.2 Activity index

Preparation of standard mortar bars and determination of the compressive strength shall be carried out in accordance with EN 196-1.

The activity index at 28 days and at 90 days shall not be less than 75 % and 85 %, respectively.

The test cement used, as defined in 3.3, is selected by the fly ash producer and is further characterised by its fineness and contents of tricalcium aluminate and alkalis as follows:

- Fineness (Blaine): at least 300 m²/kg;
- Tricalcium aluminate: 6 % by mass to 12 % by mass;
- Alkalis (Na₂O_{eq}): 0,5 % by mass to 1,2 % by mass.

NOTE The result of the activity index tests gives no direct information on the strength contribution of fly ash in concrete, nor is the use of the fly ash limited to the mixing ratio used in these tests.

5.3.3 Soundness

The soundness shall be determined on 30 % fly ash plus 70 % test cement (both by mass) in accordance with EN 196-3 and shall not be greater than 10 mm.

Where the free calcium oxide content of the fly ash determined in accordance with 5.2.5 does not exceed 1,5 % by mass, this requirement shall be deemed to be satisfied.

5.3.4 Particle density

The particle density shall be determined in accordance with EN 1097-7 and shall not deviate by more than 200 kg/m³ from the value declared by the producer.

5.3.5 Initial setting time

The initial setting time shall be determined on a 25 % fly ash plus 75 % test cement paste (both by mass) in accordance with EN 196-3 and shall not be more than twice as long as the initial setting time of a 100 % (by mass) test cement paste. The requirements regarding initial setting time specified in EN 197-1 shall be met by the test cement when tested alone. Fly ash obtained from combustion of pulverised coal only shall be deemed to satisfy this requirement.

5.3.6 Water requirement

The water requirement of fly ash of fineness category S shall be determined by the method described in Annex B and shall not be greater than 95 % of that for the test cement alone.

For fly ash of fineness category N this requirement does not apply.

5.4 Other requirements

5.4.1 Durability requirements

The composition and the performance of the fly ash shall be such that durable concrete (see EN 206-1) may be produced when using the fly ash. Fly ash conforming to the definition in 3.2 and fulfilling the chemical requirements in 5.2 and the physical requirements in 5.3 shall be deemed to satisfy the durability requirements.

In certain applications, particularly for concrete in severe environmental conditions, the choice of fly ash category may have an influence on the durability of concrete, e.g. freeze-thaw resistance and resistance to alkali aggregate reactions. In such cases, the choice of fly ash category shall follow the appropriate standards and/or regulations valid in the place of use.

5.4.2 Release of dangerous substances and emission of radioactivity

Fly ash shall not contain substances which, if released from concrete, are dangerous for health, hygiene and the environment (see Annex A).

Should there be any additional requirements regarding environmental compatibility from national laws, regulations and administrative provisions in the place of use of the fly ash concerning

- a) the fly ash,
- b) the leachate of concrete produced with fly ash, or
- c) the co-combustion material,

these are to be considered and conformity to the relevant regulations shall, where required, be evaluated before production.

5.5 Information to be supplied upon request

Information on the properties listed below shall be supplied to the user upon request:

- characteristics of the test cement;
- whether the fly ash is obtained from co-combustion, and, in the affirmative, declaration of conformity to this document, any standards or regulations valid in the place of use, and the results of suitability tests as required in 4.2 and 4.3;
- a typical chemical oxide composition of the fly ash comprising the contents of silicon dioxide (SiO₂), aluminium oxide (Al₂O₃), iron oxide (Fe₂O₃), determined in accordance with EN 196-2;
- the total content of alkalis determined in accordance with EN 196-2 and calculated as Na₂O_{eq};
- the water content for standard consistence of a co-combustion fly ash/test cement paste, as determined by EN 196-3 in connection with determining the initial setting time (see 5.3.5);
- the water requirement for category S fly ash.

6 Packaging and labelling

Fly ash may be delivered in suitable bulk-delivery transportation systems or in suitable packages.

Packages and documents related to bulk deliveries shall be marked with the name or identification mark of the production plant where the fly ash was manufactured.

NOTE For CE marking and labelling, ZA.3 applies.

7 Sampling

Spot samples, equally distributed over the production period, shall be taken at the point of release into a bulk-delivery transportation system or into packages, or, alternatively, directly from bulk-delivery transportation systems or packages, using the equipment and principles described in EN 196-7.

For the purpose of carrying out all the analyses and tests needed to show conformity or non-conformity to the requirements set out in Clause 5, a representative laboratory sample of dry fly ash of at least 0,5 kg is required. This sample shall be obtained by subdividing, such as quartering, a spot sample of at least 2 kg. The laboratory sample shall be dried in a well-ventilated oven at (105 ± 5) °C to constant weight and then cooled in a dry atmosphere.

8 Conformity criteria

8.1 General requirements

Conformity of fly ash to this document shall be continually evaluated on the basis of testing of spot samples. The properties, test methods and the minimum testing frequencies for the auto control testing by the producer are specified in Table 2.

Conformity of fly ash to this document shall be evaluated in accordance with EN 450-2.

- NOTE 1 For attestation of conformity for CE marking reference is made to Annex ZA.
- NOTE 2 EN 450-2 does not deal with acceptance inspection at delivery.

Table 2 — Properties, test methods and minimum testing frequencies for the auto control testing by the producer or his agent and the statistical assessment procedure

	1	2	3	4	5	6	7	8
Property		Test method	Autocontrol testing ^a			Conformity verification ^o		
			Minim	num testing f	requency	Statistical assessment procedure		
						Inspecti	on by	
			Routine situation	Initial period for a new fly ash	Initial test co- combustion	Variables ^e	Attributes	
1	Loss on ignition	EN 196-2	1/day ^d	2/day ^d		х		С
2	Fineness	EN 451-2/ EN 933-10	1/day ^d	2/day ^d		х		C/D
3	Free calcium oxide	EN 451-1	1/week ^g	2/week			x ^f	Р
4	Reactive calcium oxide ^h	EN 196-2	1/month	2/month			х	Р
5	Chloride	EN 196-2	1/month	2/month			х	Р
6	Sulphate content	EN 196-2	1/month	2/month			х	Р
7	Particle density	EN 1097-7	1/month	2/month			х	D
8	Activity index	EN 196-1	2/month	4/month			x ^f	Р
9	Soundness (if required) ^j	EN 196-3	1/week	2/week			х	Р
10	Sum of silicon dioxide, aluminium oxide and iron oxide ⁱ	EN 196-2	1/month	2/month			х	Р
11	Reactive silicon dioxide ^m	EN 197-1			х			Р
12	Alkalis ⁱ	EN 196-2	1/month	2/month			х	Р
13	Magnesium oxide ^m	EN 196-2			Х			Р
14	Soluble phosphate	Annex C			Х			Р
15	Total phosphate ^m	ISO 29581-2	1/month	2/month			Х	Р

Table 2 (continued)

16	Initial setting time ^m	EN 196-3	1/month	2/month		Х	Р
17	Water requirement n	Annex B	2/week	4/week		х	Р
18	Dangerous substances and emission of radioactivity ^k	1					

- A minimum of 10 samples shall be used for assessing the conformity and shall represent a period of not more than 12 months and not less than 1 month.
- b Where allowed in the relevant parts of EN 196 or EN 451, methods other than those indicated may be used in factory production control in agreement with the certification body provided they give results equivalent to those obtained with the reference method.
- The methods used to take and prepare samples shall be in accordance with EN 196-7.
- d Day of delivery.
- e If the data are not normally distributed then the method of assessment may be decided on a case by case basis.
- fulf the number of samples is at least one per week during the control period, the assessment may be made by variables.
- ^g If all results from the last 10 determinations are less than 0,5 % by mass then the test frequency may be reduced to 1/month. Should any single monthly result exceed 0,5 % by mass, or should a change in coal source occur for which there is no existing knowledge about the resulting fly ash, then the full test frequency shall be used again.
- h See 5.2.6.
- ¹ Tests to be performed on fly ash obtained from co-combustion of pulverised coal with co-combustion materials in order to satisfy requirements of 5.2.8 and 5.2.9 respectively. Tests on fly ash obtained from combustion of pulverised coal only are required to provide information on request.
- j Only if free calcium oxide exceeds 1,5 % by mass.
- k Only relevant when specifically required (see also 5.4.2).
- Depending on the requirement (see Notes 1 and 2 in ZA.1).
- m Tests to be performed on fly ash obtained from combustion of pulverised coal with co-combustion materials in order to satisfy the requirements of 5.2.7, 5.2.10, 5.2.11 and 5.3.5 respectively.
- n Test to be performed on fly ash of fineness category S only.
- P: Pass/fail C: Category D: Declared value

8.2 Statistical conformity criteria

8.2.1 General

Conformity shall be formulated in terms of a statistical criterion based on:

- a) the required characteristic values for chemical and physical properties as specified in 5.2 and 5.3;
- b) a percentile P_k of 10 % on which the required characteristic value is based;
- c) an allowable probability of acceptance CR (consumer's risk) of 5 %.

Conformity to the requirements shall be verified either by variables or by attributes, as described in 8.2.2 and 8.2.3 and as specified in Table 2.

A minimum of ten samples shall be used for assessing the conformity and shall represent a period of not more than twelve months and not less than one month.

8.2.2 Inspection by variables

For this inspection, the test results are assumed to be normally distributed.

Conformity is verified when Formulae (2) or (3), as relevant, are satisfied:

$$\overline{x} - k_{\mathsf{A}} \times s \ge L \tag{2}$$

or

$$\bar{x} + k_{\mathsf{A}} \times s \le U \tag{3}$$

where

- \bar{x} is the arithmetic mean of the totality of the auto control test results in the control period;
- s is the standard deviation of the totality of the auto control test results in the control period;
- k_{Λ} is the acceptability constant;
- L is the specified lower limit referred to in Clause 5;
- U is the specified upper limit referred to in Clause 5.

The acceptability constant k_A depends on the percentile P_k on which the characteristic value is based, on the allowable probability of acceptance CR and on the number n of the test results. Values of k_A are listed in Table 3.

Table 3 — Acceptability constant k_A (P_k = 10 %) at CR = 5 %

Number of test results n	$k_{A}^{}}$
20 to 21	1,93
22 to 23	1,89
24 to 25	1,85
26 to 27	1,82
28 to 29	1,80
30 to 34	1,78
35 to 39	1,73
40 to 44	1,70
45 to 49	1,67
50 to 59	1,65
60 to 69	1,61
70 to 79	1,58
80 to 89	1,56
90 to 99	1,54
100 to 149	1,53
150 to 199	1,48
200 to 299	1,45
300 to 399	1,42
≥ 400	1,40
	1

Values of $k_{\rm A}$ valid for intermediate values of n may also be used. These values may be calculated by a formula of Natrella /1/.

8.2.3 Inspection by attributes

The number $c_{\rm D}$ of test results below or above the characteristic value shall be counted and compared with an acceptable number $c_{\rm A}$, calculated from the number n of autocontrol test results and the percentile $P_{\rm k}$ as specified in Table 4.

Conformity is verified if Formula (4) is satisfied:

$$c_{\mathsf{D}} \le c_{\mathsf{A}} \tag{4}$$

The value of c_A depends on the percentile P_k on which the characteristic value is based, on the allowable probability of acceptance CR and on a number n of the test results. Values of c_A are listed in Table 4.

Number of test results n a	<i>c</i> _A			
20 to 39	0			
40 to 54	1			
55 to 69	2			
70 to 84	3			
85 to 99	4			
100 to 109	5			
≥ 110	0,075 (<i>n</i> – 30)			

Table 4 — Values of c_A (P_k = 10 %) at CR = 5 %

8.3 Single result conformity criteria

In addition to the statistical conformity criteria, conformity of test results to the requirements of this document requires that it shall be verified that each test result remains within the single result limit values specified in Table 5.

Table 5 — Limit values for single results

	Property	Single result limit values	
		7,0 % by mass (category A)	
1	Loss on ignition (upper limit value)	9,0 % by mass (category B)	
		11,0 % by mass (category C)	
2	Fineness (upper limit value)	45 % by mass (category N)	
۷	Fineness (upper limit value)	13 % by mass (category S)	
3	Fineness variation (lower and upper limit values)	± 15 percentage points from declared value (category N only)	
4	Chloride (upper limit value)	0,10 % by mass	
5	Free calcium oxide (upper limit value)	1,6 % by mass	
6	Reactive calcium oxide (upper limit value)	11,0 % by mass	
7	Sulphate content (upper limit value)	3,5 % by mass	
8	Silicon dioxide + aluminium oxide + iron oxide (lower limit value)	65 % by mass	
9	Total content of alkalis (upper limit value)	5,5 % by mass	
10	Total phosphate (upper limit value)	5,5 % by mass	
11	Soundness (upper limit value)	11 mm	
12	Activity index at 28 days (lower limit value)	70 %	
12	Activity index at 90 days (lower limit value)	80 %	
13	Particle density variation (lower and upper limit values)	± 225 kg/m ³ from declared value	
14	Initial setting time (upper limit value)	2,25 times the setting of the test cement alone	
15	Water requirement (upper limit value)	97 % (category S only)	

^a If the number of test results is n < 20 (for $P_k = 10$ %) a statistically based conformity criterion is not possible. Despite this, a criterion of $c_A = 0$ shall be used in cases where n < 20.

Annex A (normative)

Release of dangerous substances and emission of radioactivity

Materials used in products shall not release any dangerous substances in excess of the maximum permitted levels specified in a relevant European Standard for the material or permitted in the national regulations of the member state of destination".

NOTE See ZA.1 in Annex ZA.

Annex B

(normative)

Determination of the water requirement for Category S fly ash

B.1 Principle

The reduction in water required when fly ash is added to a mortar is measured by comparing the flow of a control and test mix.

B.2 Apparatus

- **B.2.1 Mixer**, conforming to EN 196-1:2005, 4.4.
- **B.2.2 Flexible scraper,** of rubber or plastics material, suitable for removing mortar adhering to the mixer blades and inside surfaces of the mixer bowl.
- **B.2.3** Balance, of sufficient capacity and capable of weighing to the nearest 1,0 g.
- B.2.4 Flow table, conforming to Annex A of EN 1015-3:1999 and associated mould and tamper.

B.3 Materials

- B.3.1 CEN standard sand, conforming to EN 196-1:2005, 5.1.
- **B.3.2** Test cement, as defined in 3.3.
- B.3.3 Water, deionised or distilled.

B.4 Mix proportions

The mix composition shall be as given in Table B.1

Table B.1 — Mix proportions

Material	Control mortar	Test mortar
Test cement	(450 ± 1) g	(315 ± 1) g
Fly ash	Nil	(135 ± 1) g
CEN standard sand	(1350 ± 5) g	(1350 ± 5) g
Water	(225 ± 1) g	Mass M to give a flow value of ±10 mm of that of the control mortar

B.5 Procedure

Use the mixing procedure described in EN 196-1:2005, 6.2, immediately on completion of mixing determine the flow of the mortar in accordance with EN 1015-3. Lift the mould from the table (60 ± 5) s after mixing has been completed and operate the table immediately. Operate the table 15-times in (15 ± 2) s and measure the flow.

Adjust the water content of the test mortar to obtain a flow value within \pm 10 mm of that of the control mortar.

The determination of consistence described in EN 1015-3 may be omitted.

B.6 Calculation

Calculate the water requirement of the fly ash as a percentage by the following formula:

$$W = M/225 \times 100$$
 (B.1)

where

W is water requirement;

M is the mass of water used in the test mortar (in g).

B.7 Reporting

Report the water requirement of the fly ash to the nearest 1 %.

Annex C

(normative)

Determination method on the content of soluble phosphate (expressed as P_2O_5)

C.1 Principle of the method

The object of the test is to determine the content of soluble phosphate in fly ash, expressed as phosphorus pentoxide (mg/kg). The content is determined by measuring the concentration of phosphorus in the filtrate of a suspension of de-mineralised water stirred with a fixed amount of fly ash at a constant pH of 8.5 ± 0.2 .

C.2 Sample for analysis

For this test the following sample is required: 250,00 g dry fly ash.

C.3 Reagents

- **C.3.1 Demineralised water**, with a conductivity of at most 100 μ S/m.
- C.3.2 Diluted hydrochloric acid (HCI), 1mol/l of analytical quality.
- **C.3.3 Diluted sodium hydroxide (NaOH)**, of analytical quality (concentration to be determined based on the behaviour of the fly ash).

C.4 Apparatus

The apparatus and tools mentioned in this paragraph shall be checked before use to assure the appropriate operation as well as the absence of disturbing elements that may influence the test results.

- **C.4.1 Calibrated analytical balance,** with a measuring range of at least 1 kg and a maximum inaccuracy of 0,01 g.
- C.4.2 pH-stationery, or pH-apparatus, with manual control.
- **C.4.3 Filtration equipment,** suitable for membrane filters of $0.2 \, \mu m$ and capable of handling 750 ml of material.
- **C.4.4** Membrane filters for the filter equipment, with a pore size of 0,2 μ m.
- C.4.5 Magnetic stirrer.
- C.4.6 Paper filter (medium).
- C.4.7 Polyethylene (PE) or glass bottles.

C.5 Procedure

- C.5.1 The test shall be performed at a temperature of the solution that may vary between 20 °C and 25 °C.
- **C.5.2** Rinse the container of the pH-stationery or the pH-apparatus (C.4.2) twice in succession with demineralised water (C.3.1). Place the analysis sample (C.2) in the container. Add 750,0 ml demineralised water (C.3.1).

Stir the suspension intensively for 1 min. Adjust the pH to 8,5 of the suspension manually with diluted hydrochloric acid (HCl) of 1M (C.3.2). Stir the suspension intensively for 10 min with a magnetic stirrer (C.4.5). Keep the pH of the suspension at $8,5 \pm 0,2$ by use of the pH-stationery or pH-apparatus (C.4.2) adding diluted hydrochloric acid (C.3.2). Write down the total added volume (ml) of hydrochloric acid (x).

In case the fly ash is slightly acidic, the pH shall be adjusted with diluted sodium hydroxide (NaOH) (C.3.3).

- **C.5.3** Filtrate (under vacuum) the suspension once through the paper filter (C.4.6) over a period of 6 min and thereafter without rinsing through the membrane filter $0.2 \mu m$ (C.4.4), over a period of 4 min.
- **C.5.4** The filtrate shall be stored in polyethylene (PE) or glass bottles (C.4.7).
- **C.5.5** Analyse the resulting filtrate according to EN ISO 11885, using inductively coupled plasma optical emission spectroscopy (ICPOES), or any other technique with at least the same accuracy. Determine the concentration P in mg/l (C).

C.6 Calculation

Calculate the content of soluble phosphate as follows:

$$U = \frac{(750 + x)}{250} \times C \times \frac{142}{62} \tag{C.1}$$

where

- U is the content of soluble phosphate (as P_2O_5) in the original sample (mg/kg);
- x is the added volume of hydrochloric acid (ml);
- C is the measured concentration of phosphorus (as P) in the filtrate (mg/l).

C.7 Report

The report shall contain at least the following information:

- information necessary for the identification of the analysis sample;
- origin and specification of the analysis sample;
- test date:
- added volume of hydrochloric acid and sodium hydroxide;
- content of soluble phosphate (as P₂O₅).

Annex ZA

(informative)

Clauses of this European Standard addressing the provisions of the EU Construction Product Directive.

ZA.1 Scope and relevant characteristics

This European Standard has been prepared under Mandate M/128 ("Products related to concrete, mortar and grout")¹⁾ given to CEN by the European Commission and the European Free Trade Association.

The clauses of this European Standard shown in this annex meet the requirements of the mandate given under the EU Construction Products Directive (89/106/EEC).

Conformity to these clauses confers a presumption of fitness of the fly ash covered by this annex for the intended uses indicated herein; reference shall be made to the information accompanying the CE marking.

WARNING — Other requirements and other EU Directives, not affecting the fitness for intended uses, can be applicable to fly ash falling within the scope of this European Standard.

NOTE 1 In addition to any specific clauses relating to dangerous substances contained in this standard, there may be other requirements applicable to the products falling within its scope (e.g. transposed European legislation and national laws, regulations and administrative provisions). In order to meet the provisions of the EU Construction Products Directive, these requirements need also to be complied with, <u>when and where</u> they apply.

NOTE 2 An informative database of European and national provisions on dangerous substances is available at the Construction web site on EUROPA (accessed through: http://ec.europa.eu/enterprise/construction/cpd-ds/.

This annex establishes the conditions for the CE marking of the fly ash intended for the uses indicated in Table ZA.1 and shows the relevant clauses applicable:

This annex has the same scope as Clause 1 of this standard and is defined by Table ZA.1.

¹⁾ As amended

Table ZA.1 — Relevant clauses and intended use

Essential Characteristics	Requirement clauses in this European Standard	Levels and/or classes	Notes
Activity index (compressive strength)	5.3.2	None	Requirement expressed in terms of lower limits at 28 and 90 days (in %)
			Pass/fail
Fineness	5.3.1	None	Requirements expressed in terms of upper limit for sieve residue (in % by mass) for each of two different categories and upper and lower variation limits (in percentage points) for one of these categories.
			Declaration of category
Soundness — Expansion	5.3.3	None	Requirement expressed in terms of upper limit (in mm). Deemed to satisfy if free CaO does no exceed 1,5 % by mass
			Pass/fail
Soundness — Free CaO	5.2.5	None	Requirement expressed in terms of upper limit (in % by mass)
	0.2.0		Pass/fail
Loss on ignition	5.2.2	None	Requirements expressed in terms of upper limits (in % by mass) fo each of three different categories.
Composition: Sum of contents	520	None	Declaration of category
Composition: Sum of contents of silicon dioxide, aluminium oxide and iron oxide	5.2.8	none	Requirement expressed in terms of lower limit (in % by mass) oxide
			Pass/fail
Composition: Total content of alkalis	5.2.9	None	Requirement expressed in terms of upper limit (in % by mass).
			Pass/fail
Composition: Reactive silicon dioxide	5.2.7	None	Requirement expressed in terms of lower limit (in % by mass)
			Pass/fail

Table ZA.1 (concluded)

Composition: Sulphate content	5.2.4	None	Requirement expressed in terms
			of upper limit (in % by mass)
			Pass/fail
Composition: Chloride	5.2.3	None	Requirement expressed in terms of upper limit (in % by mass)
			Pass/fail
Composition: Reactive calcium oxide	5.2.6	None	Requirement expressed in terms of upper limit (in % by mass). Deemed to satisfy if total content of CaO is below 10,0 % by mass
			Pass/fail
Composition: Magnesium oxide	5.2.10	None	Requirement expressed in terms of upper limit (in % by mass).
			Pass/fail
Composition: Soluble phosphate	5.2.11	None	Requirement expressed in terms of upper limit (in mg/kg)
			Pass/fail
Composition: Total phosphate	5.2.11	None	Requirement expressed in terms of upper limit (in % by mass)
			Pass/fail
Particle density	5.3.4	None	Requirement expressed as declared value (in kg/m³) with upper and lower tolerance limits
			Declared value
Initial setting time	5.3.5	None	Requirement expressed in terms of upper limit (in minutes) for deviation from the setting time of cement paste without fly ash. Pass/fail
Water requirement	5.3.6	None	Requirement expressed in terms of upper limit (in %) (only for fly ash of fineness category S)
			Pass/fail
Durability	5.4.1	None	Fly ash conforming to this European Standard is deemed to provide a durable concrete when other requirements for durability of concrete in relevant standards and/or regulations valid in the place of use are fulfilled.
Release of dangerous substances and emission of radioactivity	5.4.2	None	ZA.1 (Notes 1 and 2) and ZA.3

The requirement on a certain characteristic is not applicable in those Member States (MSs) where there are no regulatory requirements on that characteristic for the intended use of the product. In such cases, producers

placing their products on the market of these MSs are not obliged to determine nor declare the performance of their products with regard to this characteristic and the option "No performance determined" (NPD) in the information accompanying the CE marking (see ZA.3) may be used. The NPD option may not be used, however, where the characteristic is subject to a threshold level.

ZA.2 Procedure for attestation of conformity of fly ash

ZA.2.1 System of attestation of conformity

The system of attestation of conformity of the fly ash indicated in Table ZA.2, in accordance with the Commission decision 1999/469/EC of 25 June 1999 as amended by decision 01/596/EC of 8 January 2001 as given in Annex III of the mandate for "Products related to concrete, mortar and grout", is shown in Table ZA.2 for the indicated intended use and relevant level(s) or class(es):

Table ZA.2 — System of attestation of conformity

Product	Intended use	Level(s) or class(es)	Attestation of conformity system		
Additions Type II	For concrete, mortar and grout	-	1+		
System 1+: See Directive 89/106/EEC (CPD) Annex III.2.(i) with audit testing of samples.					

The attestation of conformity of the fly ashes in Table ZA.1 shall be based on the evaluation of conformity procedure indicated in Table ZA.3 resulting from application of the clauses of this or other European Standards indicated therein.

Table ZA.3 — Evaluation of conformity clauses to apply in addition to Clause 8 of this standard

	Content of the task	Evaluation of conformity clauses to apply	
Factory production control (F.P.C)	Parameters related to all relevant characteristics of Table ZA.1	EN 450-2:2005, 4.1 and 4.2	
Further testing of samples taken at factory	All relevant characteristics of Table ZA.1	EN 450-2:2005, 4.3	
Initial type testing	All relevant characteristics of Table ZA.1, except	EN 450-2:2005, 5.4 and 5.6	
	- release of dangerous substances and emission of radioactivity (see Figure ZA.1) ^a		
Initial inspection of factory and of F.P.C	Parameters related to all relevant characteristics of Table ZA.1	EN 450-2:2005, 5.5	
Continuous surveillance, assessment and approval of F.P.C.	Parameters related to all relevant characteristics of Table ZA.1	EN 450-2:2005, 5.2 and 5.3	
Audit testing of samples taken at factory	All relevant characteristics of Table ZA.1, except	EN 450-2:2005, 5.4	
	- release of dangerous substances and emission of radioactivity (see Notes 1 and 2 of ZA.1) ^a		
	control (F.P.C) Further testing of samples taken at factory Initial type testing Initial inspection of factory and of F.P.C Continuous surveillance, assessment and approval of F.P.C. Audit testing of samples	Factory production control (F.P.C) Parameters related to all relevant characteristics of Table ZA.1 Further testing of samples taken at factory Initial type testing All relevant characteristics of Table ZA.1, except - release of dangerous substances and emission of radioactivity (see Figure ZA.1) a Initial inspection of factory and of F.P.C Continuous surveillance, assessment and approval of F.P.C. Audit testing of samples taken at factory All relevant characteristics of Table ZA.1 Parameters related to all relevant characteristics of Table ZA.1 Parameters related to all relevant characteristics of Table ZA.1 All relevant characteristics of Table ZA.1 All relevant characteristics of Table ZA.1 Parameters related to all relevant characteristics of Table ZA.1 All relevant characteristics of Table ZA.1 All relevant characteristics of Table ZA.1 except - release of dangerous substances and emission of radioactivity (see Notes 1)	

ZA.2.2 EC Certificate of conformity

When compliance with the conditions of this Annex is achieved, the certification body shall draw up a certificate of conformity (EC Certificate of conformity), which entitles the producer to affix the CE marking. The EC certificate of conformity shall include:

- name, address and identification number of the certification body;
- name and address of the producer, or his authorised representative established in the EEA, and place of production;

NOTE The manufacturer may also be the person responsible for placing the product onto the EEA market, if he takes responsibility for CE marking.

- description of the product (fly ash for concrete conforming to this European standard);
- provisions to which the product conforms (i.e. Annex ZA of this European standard);
- particular conditions applicable to the use of the product (none as regards conformity);
- the number of the certificate;
- conditions of validity of the certificate, where applicable;
- name of, and position held by, the person empowered to sign the certificate.

The above mentioned EC certificate of conformity shall be presented in the language or languages accepted in the Member State in which the product is to be used.

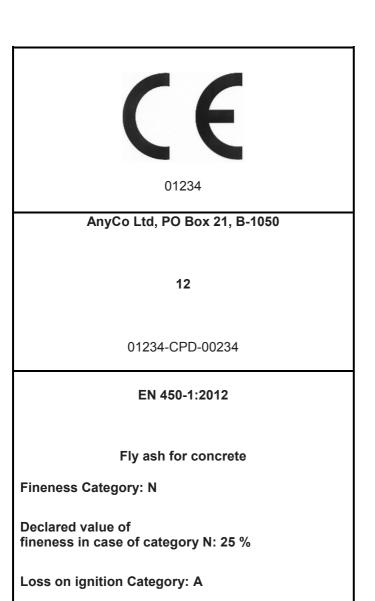
ZA.3 CE marking and labelling

The producer or his authorised representative established within the EEA is responsible for the affixing of the CE marking. The CE marking symbol to affix shall be in accordance with Directive 93/68/EEC and shall be shown on the accompanying commercial documents e.g. a delivery note (bulk fly ash) or on the packaging (packed fly ash). The following information shall accompany the CE marking symbol:

- a) identification number of the certification body;
- b) name or identifying mark and registered address of the producer;
- c) the last two digits of the year in which the marking is affixed;
- d) number of the EC Certificate of conformity;
- e) reference to this European Standard with version date;
- f) description of the product: i.e. fly ash for concrete;
- g) information on those relevant requirements listed in Table ZA.1 which are to be declared presented as:
 - 1) declared values and, where relevant, level or class (including "pass" for pass/fail requirements, where necessary) to declare for each requirement as indicated in Table ZA.1;
 - 2) "No performance determined" for characteristics where this is relevant;
 - 3) as an alternative, a standard designation which shows some or all of the relevant characteristics (where the designation covers only some characteristics, it will need to be supplemented with declared values for other characteristics as above).

The "No performance determined" (NPD) option may not be used where the characteristic is subject to a threshold level. Otherwise, the NPD option may be used when and where the characteristic, for a given intended use, is not subject to regulatory requirements in the Member State of destination.

Figure ZA.1 gives an example of the information to be given on the label, packaging and/or commercial documents.



Particle density: 2 300 kg/m³

Dangerous Substance: NL, F²⁾

CE conformity marking, consisting of the "CE"-symbol given in Directive 93/68/EEC.

Identification number of the certification body

Name or identifying mark and registered address of the producer

Last two digits of the year in which the marking was affixed

Certificate number

N°. of European standard with date of version

Description of product and information on regulated characteristics

Abbreviation of the name of the country where the fly ash complies with national regulation.

Figure ZA.1 — Example CE marking information

In addition to any specific information relating to dangerous substances shown above, the product should also be accompanied, when and where required and in the appropriate form, by documentation listing any other legislation on dangerous substances for which compliance is claimed, together with any information required by that legislation.

NOTE 1 European legislation without national derogations need not be mentioned.

NOTE 2 Affixing the CE marking symbol means, if a product is subject to more than one Directive, that it complies with all applicable Directives

²⁾ In accordance with the internationally recognised car plate code. To the abbreviation of country name, further information concerning the provisions may be added.

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- [2] CR 1901 Regional specifications and recommendations for the avoidance of damaging alkali-silica reactions in concrete

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