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Additions for concrete

Ajout pour béton

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 71, *Concrete, reinforced concrete and pre-stressed concrete*, Subcommittee SC 3, *Concrete production and execution of concrete structures*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Silica fume consists of mainly spherical particles of amorphous silicon dioxide smaller than 10^{-6} m and is highly pozzolanic. It is collected by filters as a by-product of the smelting process to produce silicon metal and ferro-silicon alloys. It can be supplied as collected from the filters (undensified), after treatment to increase its bulk density (densified), or as a slurry. Silica fume from more than one furnace, filter or intermediate storage silo will normally be blended in the production plant.

Many years of research and practical experience have demonstrated that silica fume which satisfies the requirements in this document has highly pozzolanic properties and can be used to produce concrete with improved properties in both the fresh and hardened states. Silica fume is normally used in combination with a plasticizer and/or superplasticizer.

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 countries, based on national experience and tradition.

Before use, fly ash can 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 can conform to this document to which reference is made in such a case. If they are out of the scope of this document, their suitability for use as Type II additions in concrete according to ISO 22965-2 can also be established from national standards or provisions valid in the place of use of the concrete and which refer specifically to the use of the addition in concrete conforming to ISO 22965-2.

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 can be affected. Where relevant, such effects need to be considered in concrete mix design (see ISO 22965-2).

Blast-furnace slag is classified into two types, air-cooled blast-furnace slag and granulated blast-furnace slag, according to the cooling process after the molten slag of approximately 1 500 °C is removed from the furnace. Granulated slag is made by rapidly chilling molten slag, such as by water jet, into a granulated glassy material, which is used for ground granulated blast-furnace slag, a material for slag cement. The amorphous glassy granulated slag has hydraulicity. When finely ground into ground granulated blast-furnace slag, it also demonstrates hardening and strength-developing properties (latent hydraulicity), as the slag itself undergoes hydration, similarly to cement, in the co-presence of cement (an alkaline stimulant) and water.

Additions for concrete

1 Scope

This document specifies requirements for the properties for silica fume, siliceous fly ash and ground granulated blastfurnace slag for use as a type II addition for production of concrete conforming to ISO 22965. Additions according to this document can also be used in mortars and grouts.

This document applies to the silica fume which is a by-product of the smelting process used to produce silicon metal and ferro-silicon alloys.

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

Ground granulated blastfurnace slag containing any added materials other than grinding aids and/or gypsum (calcium sulfate) is not within the scope of this document.

It is not within the scope of this document to specify provisions for the practical application of additions in the production of concrete, mortar or grout, i.e. requirements concerning composition, mixing, placing, curing, etc.

NOTE Some rules are given in ISO 22965-2, e.g. provisions on general suitability and use of additions. Guidance on batching, control of additions content and the use of the *k*-value concept are also given in ISO 22965-2.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 679, *Cement — Test methods — Determination of strength*

ISO 9277, *Determination of the specific surface area of solids by gas adsorption — BET method*

ISO 9286, *Abrasive grains and crude — Chemical analysis of silicon carbide*

ISO 9597, *Cement — Test methods — Determination of setting time and soundness*

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

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 16559, *Solid biofuels — Terminology, definitions and descriptions*

ISO 19596, *Admixtures for concrete*

ISO 20290-1, *Aggregates for concrete — Test methods for mechanical and physical properties — Part 1: Determination of bulk density, particle density, particle mass-per-volume and water absorption*

ISO 22965-2, *Concrete — Part 2: Specification of constituent materials, production of concrete and compliance of concrete*

ISO 29581-1, *Cement — Test methods — Part 1: Analysis by wet chemistry*

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

EN 196-6, *Methods of testing cement — Part 6: Determination of fineness*

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

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

EN 413-2:2005, *Masonry cement — Part 2: Test methods*

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)*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

3.1
activity index
ratio (in percent) of the compressive strength of a mortar where a specific percentage of the cement is replaced with the addition, relative to the compressive strength of the reference mortar specimens made from the test cement, tested at the same age

3.2
characteristic value
value having a prescribed probability of not being attained in a hypothetical unlimited test series

Note 1 to entry: Equivalent to "fractile" which is defined in ISO 3534-1:1993.

[SOURCE: ISO 8930:1987]

3.3
densified
state of silica fume that has been treated to increase the bulk density by particle agglomeration, the bulk density typically being above 450 kg/m^3

3.4
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;
- can 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 can consist of fly ashes from different sources, each conforming to the definition given in this document

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

3.5
granulated blastfurnace slag
vitrified material made by rapid cooling of a slag melt of suitable composition, obtained by smelting iron ore in a blastfurnace, consisting of at least two thirds by mass of glassy slag and possessing hydraulic properties when suitably activated

Note 1 to entry: Rapid cooling includes quenching in water (granulation) and projecting through water and air (pelletization).

3.6
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

3.7
ground granulated blastfurnace slag
fine powder made by drying and grinding granulated blastfurnace slag where gypsum can be added and sulphur trioxide (SO_3) can be added up to 4,0 % of mass

3.8
particle density
average density of addition particles, including voids inside the particles

3.9
production plant
facility used by a manufacturer for the production and processing of addition

Note 1 to entry: Processing of addition includes selection, slurrifying, blending or densifying of addition.

3.10
silica fume
set of very fine particles of amorphous silicon dioxide collected as a by-product of the smelting process used to produce silicon metal and ferro-silicon alloys

Note 1 to entry: Silica fume can be processed, for example by classification, selection, blending, densifying, or slurrifying, or by a combination of these processes, in adequate production plants. Such processed silica fume can consist of silica fumes from different sources, each conforming to the definition given in this document.

Note 2 to entry: Other names used for silica fume are condensed silica fume and microsilica.

3.11
silica fume slurry
slurry
homogeneous, pH-regulated liquid suspension of silica fume in water, typically with a dry content of 50 % by mass, corresponding to about 700 kg of silica fume per cubic metre of slurry

3.12
spot sample
sample taken within a short period of time and at a fixed point from within a larger quantity, relating to the intended tests

Note 1 to entry: It can be obtained by combining one or more immediately consecutive increments.

3.13
test cement
Portland cement, conforming to ISO 22965, to be used for carrying out the tests needed to evaluate conformity

Note 1 to entry: Test cement is selected by the manufacturer and is further characterized by its fineness and contents of tricalcium aluminate and alkalis as follows:

- Fineness (Blaine): at least 300 m^2/kg when determined in accordance with EN 196-6 (or equivalent);

- Tricalcium aluminate: 6 % to 12 % when determined in accordance with EN 196-2 (or equivalent);
- Alkalis (Na_2O eqv): 0,4 % to 1,2 % when determined in accordance with ISO 29581-1 or ISO 29581-2 (or equivalent);
- Cement 28-day strength: 42,5 or higher.

3.14
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 ISO 22965.

3.15
undensified
state of silica fume taken directly from the collection filter, the bulk density typically being in the range 150 kg/m^3 to 350 kg/m^3

4 Specifications

4.1 General

The chemical and physical requirements are specified as characteristic values. Conformity to a characteristic value is assessed by means of statistical control procedures (see Clause 6).

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

4.2 Silica fume

4.2.1 General

The properties in 4.2.2.1 to 4.2.3.1 are specified as proportions by mass of dry silica fume. The laboratory samples shall be dried in a ventilated oven at (105 ± 5) °C to constant mass and then cooled in a dry atmosphere.

4.2.2 Chemical requirements

4.2.2.1 Silicon dioxide

The content of silicon dioxide, SiO_2, as determined by the method described as reference method in ISO 29581-1 or ISO 29581-2 shall be not less than 85 % by mass.

4.2.2.2 Elemental silicon

The content of elemental silicon, Si, determined according to ISO 9286, shall not be greater than 0,4 % by mass.

4.2.2.3 Free calcium oxide

The content of free calcium oxide, free CaO, as determined by the method described in EN 451-1 (or equivalent), shall not be greater than 1,0 % by mass.

4.2.2.4 Sulfate

The sulfate content, as determined by the method described in ISO 29581-1 or ISO 29581-2 (or equivalent) and expressed as total content of SO_3, shall not be greater than 3,0 % by mass.

4.2.2.5 Total content of alkalis

The total content of alkalis determined by the method described in ISO 29581-1 or ISO 29581-2 and calculated as "Na_2O equivalent" shall be declared.

NOTE Different national provisions adopt different principles but, in general, only a small proportion of alkalis in silica fume are considered to contribute to alkali silica reaction (see CEN Report CR 1901).

4.2.2.6 Chloride

The total content of chloride, calculated in accordance with the method described in ISO 29581-1 or ISO 29581-2, shall not be greater than 0,3 % by mass. If the Cl^- content is above 0,10 % by mass, the upper limit for its characteristic value shall be declared by the manufacturer.

4.2.2.7 Loss on ignition

The loss on ignition, as determined in accordance with the method described in ISO 29581-1 or ISO 29581-2, but using an ignition time of 1 h, shall not be greater than 5,0 % by mass.

4.2.3 Physical requirements

4.2.3.1 Specific surface area

The specific surface area, as determined by nitrogen adsorption according to the method given in ISO 9277, shall not be less than 15 000 m^2/kg, nor more than 35 000 m^2/kg.

4.2.3.2 Dry mass content in slurry

The dry mass content shall not deviate from the value declared by the supplier by more than ±2 % by mass of the slurry when determined by drying a representative sample of at least 5 g of slurry in a well ventilated oven at (105 ± 5) °C to constant mass. Constant mass is considered to be reached when successive weightings at least 1 h apart during drying at (105 ± 5) °C do not differ by more than 0,2 %.

4.2.3.3 Activity index

The activity index is determined as the ratio (in percent) of the compressive strength of standard mortar bars, prepared with 90 % test cement plus 10 % silica fume per mass of total binder, to the compressive strength of standard mortar bars prepared with 100 % test cement, when tested at the same age.

Preparation of standard mortar bars and determination of the compressive strength shall be carried out in accordance with the method described in ISO 679. The mortar containing silica fume shall be mixed with an amount of superplasticizer (conforming to ISO 19596) so that the mortar has a consistency equivalent to the reference mortar when tested by the flow table method given in EN 413-2 (or equivalent).

The activity index shall be at least 100 % when tested at a mortar age of 28 days.

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

4.3 Fly ash

4.3.1 Chemical requirements

4.3.1.1 General

The chemical composition shall be expressed as proportions by mass of dry fly ash.

In performing the test methods for major elements described in ISO 29581-1:2009, Clause 13, the following modifications shall be observed:

a) When heating the sample-peroxide mixture (ISO 29581-1:2009, 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 (ISO 29581-1:2009, 13.2), proceed with the test even though the melting may not be clear;

c) To decompose the evaporation residue (ISO 29581-1:2009, 13.7), add 2,0 g potassium bisulphate instead of the sodium carbonate/sodium chloride mixture.

4.3.1.2 Loss on ignition

The loss on ignition shall be determined in accordance with the principles of the method described in ISO 29581-1 or ISO 29581-2 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 can 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.

4.3.1.3 Chloride

The content of chloride, expressed as total content of Cl^-, shall be determined in accordance with ISO 29581-1 or ISO 29581-2 and shall not be greater than 0,10 % by mass.

4.3.1.4 Sulphate (SO_3) content

The sulphate content, expressed as total content of SO_3, shall be determined in accordance with ISO 29581-1 or ISO 29581-2 and shall not be greater than 3,0 % by mass.

4.3.1.5 Free calcium oxide

The content of free calcium oxide shall be determined by the method described in EN 451-1 (or equivalent). 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 4.3.2.3.

4.3.1.6 Reactive calcium oxide

The content of reactive calcium oxide shall be calculated and shall not exceed 10,0 % by mass.

Reactive calcium oxide (CaO) is defined as fraction of the calcium oxide which, under normal hardening conditions, can form calcium silicate hydrates or calcium aluminate hydrates. To calculate this fraction, the total calcium oxide content (see ISO 22981-1) is reduced by the fraction corresponding to calcium carbonate ($CaCO_3$), based on the measured carbon dioxide (CO_2) content (see ISO 22981-1), and the fraction corresponding to calcium sulfate ($CaSO_4$), based on the measured sulfate (SO_3) content (see ISO 22981-1) after subtraction of the SO_3 taken up by alkalis.

If the total content of calcium oxide determined in accordance with 4.3.1.1 does not exceed 10,0 % by mass, the requirement for reactive calcium oxide shall be deemed to be satisfied.

4.3.1.7 Reactive silicon dioxide

The amount of reactive silicon shall not be less than 25 % 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 (see 4.3.3.2) the amount of reactive silicon shall be determined and the result shall be declared.

Reactive silicon dioxide (SiO) is defined as fraction of the silicon dioxide which is soluble after treatment with hydrochloric acid (HCl) and with boiling potassium hydroxide (KOH) solution. The quantity of reactive silicon dioxide is determined by subtracting from the total silicon dioxide content (see ISO 29581-1 or ISO 29581-2) the fraction contained in the residue insoluble in hydrochloric acid and potassium hydroxide (see ISO 29581-1 or ISO 29581-2), both on a dry basis.

4.3.1.8 Silicon dioxide (SiO_2), aluminium oxide (Al_2O_3) and iron oxide (Fe_2O_3)

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 ISO 29581-1 or ISO 29581-2, modified as indicated in 4.3.1.1, and shall not be less than 70 % by mass. Fly ash obtained from combustion of pulverized coal only shall be deemed to satisfy this requirement.

4.3.1.9 Total content of alkalis

The total content of alkalis shall be determined in accordance with ISO 29581-1 or ISO 29581-2 and calculated as Na_2O_{eq} and shall not exceed 5,0 % by mass. Fly ash obtained from combustion of pulverized coal only shall be deemed to satisfy this requirement.

4.3.1.10 Magnesium oxide

In the initial type test for fly ash from co-combustion (see 4.3.3.2), the content of free magnesium oxide (periclase) shall be determined and shall not be greater than 5,0 % by mass. Fly ash obtained from combustion of pulverized coal only shall be deemed to satisfy this requirement.

4.3.1.11 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 pulverized coal only shall be deemed to satisfy this requirement.

In the initial type test for fly ash from co-combustion (see 4.3.3.2), 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.

4.3.1.12 Moisture content

The moisture content shall be determined with the principles of the method described in ISO 29581-1 and shall not be greater than 1,0 % by mass.

4.3.2 Physical requirements

4.3.2.1 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 equivalent) or by air jet sieving in accordance with EN 933-10 (or equivalent) and shall fall within the limits of the categories specified below:

Category N: the fineness shall not exceed 40 % by mass and/or Blaine fineness ≥250 m^2/kg, and it shall not vary by more than ±10 percentage points from the declared value.

Category S: the fineness shall not exceed 12 % by mass and/or Blaine fineness ≥450 m^2/kg. The ±10 percentage points fineness variation limits are not applicable.

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

4.3.2.2 Activity index

Preparation of standard mortar bars and determination of the compressive strength shall be carried out in accordance with ISO 679. The activity index is determined as the ratio (in percent) of the compressive strength of standard mortar bars, prepared with 75 % test cement plus 25 % fly ash by mass of total binder, to the compressive strength of standard mortar bars prepared with 100 % test cement, when tested at the same age.

The activity index at 28 days and at 91 days shall not be less than 75 % and 85 %, respectively.

The test cement used, as defined in 3.13, is selected by the fly ash producer and is characterised by its fineness and contents of tricalcium aluminate and alkalis.

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.

4.3.2.3 Soundness

The soundness shall be determined on 30 % fly ash plus 70 % test cement (both by mass) in accordance with ISO 9597 and shall not be greater than 10 mm.

Where the free calcium oxide content of the fly ash determined in accordance with 4.3.1.5 does not exceed 1,5 % by mass, this requirement shall be deemed to be satisfied.

4.3.2.4 Particle density

The particle density shall be determined in accordance with ISO 20290-1 and shall not deviate by more than 200 kg/m^3 from the value declared by the producer.

4.3.2.5 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 ISO 9597 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 (or equivalent) shall be met by the test cement when tested alone. Fly ash obtained from combustion of pulverized coal only shall be deemed to satisfy this requirement.

4.3.2.6 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.

4.3.3 Specific provisions for fly ash from co-combustion

4.3.3.1 Co-combustion materials

Fly ash from co-combustion as defined in 3.4 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 % in case of co-combustion of materials other than clean, not contaminated biomass (see 3.6). 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):

$$M = 100\,(K_1 \times A_1 + K_2 \times A_2 \ldots K_n \times A_n) \,/\, [K_\mathrm{c} \times A_\mathrm{c} + (K_1 \times A_1 + K_2 \times A_2 \ldots K_n \times A_n)] \qquad (1)$$

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 $(K_\mathrm{c} + K_1 + K_2 + \ldots K_n) = 1$.

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.

NOTE 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 ISO 16559 including animal husbandry residues and excluding waste wood.
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

4.3.3.2 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 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 4.3.1, 4.3.2 and 4.3.3 has been proven.

4.4 Ground granulated blastfurnace slag

4.4.1 General

The main constituent shall be granulated blastfurnace slag, as defined in 3.7. Its chemical composition shall consist of at least two-thirds by mass of the sum of calcium oxide (CaO), magnesium oxide (MgO) and silicon dioxide (SiO_2). The remainder shall be aluminium oxide (Al_2O_3) together with small amounts of other compounds. The ratio by mass (CaO + MgO)/(SiO_2) shall exceed 1,0 or the ratio by mass (CaO + MgO + Al_2O_3)/(SiO_2) shall exceed 1,6.

Ground granulated blastfurnace slag conforming to this document shall contain no added materials except gypsum, SO_3, and/or grinding aids to assist in the manufacture. For the total quantity of gypsum or SO_3, see Table 2. The total quantity of grinding aid shall not exceed 1,0 % and the organic content of any grinding aid(s) shall not exceed 0,2 % (both by mass of the ground granulated blastfurnace slag). Grinding aids shall not promote corrosion of the reinforcement or impair the properties of the ground granulated blastfurnace slag or the concrete, mortar or grout, made from it.

4.4.2 Chemical requirements

The chemical properties of the ground granulated blastfurnace slag shall conform to the requirements in Table 2. The method in Annex A shall be used for determining the moisture content of ground granulated blastfurnace slag.

Table 2 — Chemical requirements given as characteristic values

Property	Test reference	Requirements[a]
Magnesium oxide	ISO 29581-1 or ISO 29581-2	≤10 %
Sulfide	ISO 29581-1 or ISO 29581-2	≤2,0 %
Sulfate[b]	ISO 29581-1 or ISO 29581-2	≤4,0 %[b]
Loss on ignition, corrected for oxidation of sulfide[c]	ISO 29581-1 or ISO 29581-2	≤3,0 %
Chloride[d]	ISO 29581-1 or ISO 29581-2	≤0,10 %
Moisture content	Annex A	≤1,0 %

[a] Requirements are given by mass of the ground granulated blastfurnace slag.

[b] The maximum value of SO_3 when gypsum is added is 4,0 %.

[c] When testing loss of ignition the heating temperature shall be 700 °C.

[d] Ground granulated blastfurnace slag may contain more than 0,10 % chloride but in that case the maximum chloride content, as a value not to be exceeded, shall be stated on the packages or the documents (see Clause 7).

4.4.3 Physical requirements

4.4.3.1 Fineness

The specific surface area determined in accordance with the air permeability method specified in EN 196-6, shall be not less than 275 m^2/kg.

4.4.3.2 Requirements when combined with the test cement

4.4.3.2.1 Test cement

The test cement shall be a Portland cement, for instance according to ISO 22965, and shall be selected by the ground granulated blastfurnace slag manufacturer, as defined in 3.13.

4.4.3.2.2 Initial setting time

When determined in accordance with ISO 9597, the initial setting time of a combination (by mass) of 50 % of ground granulated blastfurnace slag with 50 % of test cement, shall not be more than twice as long as that of the test cement on its own.

4.4.3.2.3 Activity index

The activity index shall be expressed as the ratio (in percent) of the compressive strength of the combination (by mass) of 50 % of ground granulated blastfurnace slag with 50 % of test cement, to the compressive strength of the test cement on its own. The compressive strengths shall be determined in accordance with ISO 679 and the water:combination ratio and the water:cement ratio shall both be 0,50.

The activity index at 7 days and at 28 days shall be not less than 45 % and 70 % respectively. When testing at 91 days is required, the activity index shall be according to the provisions in the place of use.

NOTE The activity index gives no direct information on the strength contribution of ground granulated blastfurnace slag in concrete, nor is the use of the ground granulated blastfurnace slag limited to the mixing ratio used in the activity index test.

4.5 Durability requirements

The composition and the performance of the addition shall be such that durable concrete may be produced when using it. Addition conforming to this document is deemed to satisfy the durability requirements, provided that other requirements for durability of concrete in relevant standards and/or regulations valid in the place of use are fulfilled.

In certain applications, particularly for concrete in severe environmental conditions, the choice of addition 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 addition shall follow the appropriate standards and/or regulations valid in the place of use.

4.6 Release of dangerous substances and emission of radioactivity

Relevant national or regional regulations applicable in the place of use of the addition, or the leachate of concrete produced with the addition, shall be identified and documented before production.

Inhalation should be avoided by using respiratory protection during dust generating operations.

4.7 Information to be supplied upon request

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

a) general:

- characteristics of the test cement, for instance initial setting time, 7-day and 28-day strength;
- total content of alkalis determined in accordance with ISO 29581-1 or ISO 29581-2, or other method agreed between manufacturer and user, and expressed as equivalent sodium oxide;
- activity index determined as specified in 4.2.3.3, 4.3.2.2 or 4.4.3.2.3, as relevant;

b) silica fume:

- a typical chemical composition of silica fume comprising the contents of silicon dioxide (SiO_2), elemental silicon (Si), free calcium oxide (CaO), sulfate (SO_3), and chloride (Cl^-) determined as specified in 4.2.2;
- specific surface area determined in accordance with ISO 9277;
- dry mass content in slurry determined as specified in 4.2.3.2;

c) fly ash:

- whether fly ash is obtained from co-combustion and the results of suitability tests as required in 4.3.3.2;
- a typical chemical oxide composition of fly ash comprising the contents of silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), iron oxide (Fe_2O_3), determined in accordance with ISO 29581-1 or ISO 29581-2;
- the water content for standard consistence of a co-combustion fly ash/test cement paste, as determined by ISO 9597 in connection with determining the initial setting time (see 5.3.5);
- the water requirement for category S fly ash;

d) ground granulated blastfurnace slag:

- chemical oxide composition of ground granulated blastfurnace slag, comprising the contents of magnesium oxide (MgO), sulfide, sulfate and chloride;
- loss of ignition, corrected for oxidation of sulfide;

— moisture content.

The format and the basis on which the information on the properties is declared, shall be as agreed between the manufacturer and user.

5 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 (or equivalent).

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 4, a representative laboratory sample of the addition 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.

6 Conformity control and evaluation of conformity

Guidance on conformity control and evaluation of conformity are given in Annex D.

7 Packaging, labelling and marking

Additions may be delivered in suitable packages or by means of suitable bulk-delivery transportation systems. The following information shall be marked on the packages or in the case of bulk delivery, on the documents:

a) type of addition and, where appropriate, additional product identification;

b) form of delivery: undensified, densified or slurry (stating the dry mass content in percent);

c) quantity of addition shall be marked on each package and in the delivery note;

d) name or identification mark of the production plant where the addition was manufactured;

e) date of packing or shipment;

f) chloride content, if it is above 0,10 % by mass;

g) number and year of this document.

Annex A
(normative)

Method of determining the moisture content of ground granulated blastfurnace slag

A.1 Principle

The moisture content is determined by drying a sample in an oven until constant mass is achieved.

A.2 Apparatus

A.2.1 Balance, capable of weighing to an accuracy of 0,001 g.

A.2.2 Shallow container, of about 20 g capacity.

A.2.3 Electric oven, with natural ventilation controlled at (110 ± 5) °C.

A.2.4 Desiccator, containing dried magnesium perchlorate.

A.3 Procedure

Weigh (to the nearest 0,001 g), 10 g ± 1 g of ground granulated blastfurnace slag into the shallow container that has been previously dried and weighed. Place the container in the electric oven for 1 h. Remove the container and contents and allow to cool in the desiccator to room temperature and then weigh it. Repeat the heating and cooling cycle until constant mass is achieved, i.e. when the difference between two successive weighings is less than 0,005 g.

A.4 Calculation

Calculate the moisture content, C, of the sample as a percentage, from Formula (A.1):

$$C = 100 \times (m_1 - m_2) / m_2 \tag{A.1}$$

where

m_1 is the mass of the initial sample;

m_2 is the mass of the dried sample (in g).

A.5 Report

Report the moisture content as a percentage, to the nearest 0,1 %.

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 ISO 679:2009, 4.6.2.

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 EN 1015-3:1999, Annex A, (or equivalent) and associated mould and tamper.

B.3 Materials

B.3.1 ISO reference sand, conforming to ISO 679:2009, 5.1.

B.3.2 Test cement, as defined in 3.3.

B.3.3 Water, deionized 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
ISO standard sand	(1 350 ± 5) g	(1 350 ± 5) g
Water	(225 ± 1) g	Mass, m_w, to give a flow value of ±10 mm of that of the control mortar

B.5 Procedure

Use the mixing procedure described in ISO 679:2009, 6.2, immediately on completion of mixing determine the flow of the mortar in accordance with EN 1015-3 (or equivalent). 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 ± 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, W, as a percentage with Formula (B.1):

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

where m_w 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 in fly ash (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 µS/m.

C.3.2 Diluted hydrochloric acid (HCl), 1 mol/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 µm and capable of handling 750 ml of material.

C.4.4 Membrane filters for the filter equipment, with a pore size of 0,2 µm.

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 demineralized water (C.3.1). Place the analysis sample (C.2) in the container. Add 750,0 ml demineralized 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 ± 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 µm (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 ISO 11885, using inductively coupled plasma optical emission spectroscopy (ICP-OES), 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 given in Formula (C.1):

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

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 P_2O_5).