

CALCIUM ALUMINIUM SILICATE

Prepared at the 28th JECFA (1984), published in FNP 31/2 (1984) and in FNP 52 (1992). Metals and arsenic specifications revised at the 57th JECFA (2001). A group ADI 'not specified' for silicon dioxide and certain silicates was established at the 29th JECFA (1985)

SYNONYMS Aluminium calcium silicate, calcium aluminosilicate, calcium silicoaluminate, sodium calcium silicoaluminate; INS No. 556

DEFINITION

Chemical names Calcium aluminosilicate

Assay Not less than 44% and not more than 50% of silicon dioxide (SiO₂)
Not less than 3% and not more than 5% of aluminium oxide (Al₂O₃)
Not less than 32% and not more than 38% of calcium oxide (CaO)
Not less than 0.5% and not more than 4% of sodium oxide (Na₂O)

DESCRIPTION Fine, white, free-flowing powder

FUNCTIONAL USES Anticaking agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Insoluble in water and ethanol

PURITY

Loss on ignition (Vol. 4) Not less than 14% and not more than 18% (ignition at 1000° to constant weight)

Loss on drying (Vol. 4) Not more than 10% (105°, 2 h)

Fluoride (Vol. 4) Not more than 50 mg/kg
Weigh 1 g of the sample to the nearest mg and proceed as directed in the Fluoride Limit Test (Method I or III).

Lead (Vol. 4) Not more than 5 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

METHOD OF ASSAY

Silicon dioxide:
Transfer 500 mg of the sample, previously dried at 105° for 2 h and weighed accurately, into a 250 ml beaker. Wash the walls of beaker with a few ml of water, then add 30 ml of 72% perchloric acid and 15 ml of hydrochloric acid.

Heat on a hot-plate until dense white fumes appear. Let cool. Add 15 ml of hydrochloric acid and reheat until dense fumes appear. Let cool, add 70 ml of water, and filter through Whatman No. 40 filter paper or equivalent. Wash the paper and the precipitate with hot water to remove perchloric acid. Then transfer the paper and the precipitate to a tared platinum crucible, and ignite at 900° to constant weight. Moisten the residue with a few drops of water, then add 15 ml of hydrochloric acid and 8 drops of sulfuric acid, and heat on a hotplate until white fumes of sulfur trioxide appear. Let cool. Add 5 ml of water, 10 ml of hydrofluoric acid (warning: toxic, corrosive, must not contact skin; work under fumehood) and 3 drops of sulfuric acid, then evaporate to dryness on a hotplate. Carefully heat over a flame until fumes of sulfur trioxide no longer appear. Then ignite at 900° to constant weight. The weight loss after the addition of hydrofluoric acid represents the weight of SiO₂ in the sample taken.

Aluminium oxide:

Fuse the residue obtained in the silicon dioxide determination with 2 g of potassium pyrosulfate for 5 min. Cool, dissolve the fusion in water, and dilute to 250 ml in a volumetric flask. Transfer 100 ml of the solution into a 600 ml-beaker, add 100 ml of water and 5 drops of bromotymol blue TS, and heat to as low boil. Add ammonium hydroxide, dropwise, until a blue colour appear, then boil the solution for 5 min. to expel the excess ammonia. Filter through Whatman No. 41, or equivalent filter paper, and wash the precipitate with six portions of a 1-in-50 hot ammonium chloride solution. Transfer the filter and precipitate into a tared platinum crucible, char the paper, and ignite over a Meker burner to constant weight. The weight of the residue, corrected for the ash content of the filter paper and multiplied by 2.5, represents the weight of Al₂O₃ in the original sample.

Calcium oxide:

To the combined filtrate and washings retained in the silicon dioxide determination, add, while stirring, about 30 ml of 0.05 M disodium ethylenediaminetetraacetate from a 50-ml burette. Then add 15 ml of sodium hydroxide TS and 300 g of hydroxynaphthol blue indicator, and continue the titration to a blue end point. Each ml of 0.05 M disodium ethylenediaminetetraacetate is equivalent to 2.804 mg of CaO.

Sodium oxide:

Transfer about 500 mg of the sample, previously dried at 105° for 2 h, and accurately weighed, into a tared platinum dish, and moisten with 8 to 10 drops of water. Add 25 ml of 70% perchloric acid and 10 ml of hydrofluoric acid (warning: toxic, corrosive, must not contact skin; work under fumehood) and heat on a hot plate in a hood until dense white fumes of perchloric acid appear. Add 10 ml of hydrofluoric acid (warning: toxic, corrosive, must not contact skin; work under fumehood), heat again to dense white fumes, and dissolve the residue in sufficient water to make 250 ml. Set a suitable flame photometer to a wavelength of 589 nm. Adjust the instrument to zero transmittance against water, then adjust it to 100% transmittance with a standard solution containing 200 µg of sodium, in the form of the chloride, per ml. Read the percent transmittance of three other standard solutions containing 50, 100 and 150 µg each of sodium per ml, and plot the standard curve as % transmittance vs. concentration of sodium. Place a portion of the sample solution in the photometer, read the percent transmittance in the same

manner, and by reference to the standard curve determine the concentration (C) of sodium, in T per ml in the sample solution. Calculate the quantity, in mg, of Na₂O in the sample taken by the formula:

$$250 \times C \times \frac{1.348}{1000} - F$$

where F is the quantity of sodium oxide equivalent to any sodium sulfate present in the sample, is found as follows:

Correction for sodium sulfate content:

Weigh accurately 12.5 g of the sample, previously dried at 105° for 2 h and stir it with 240 ml of water for at least 5 min. with a high speed mixer. Transfer the mixture into a 250-ml graduated cylinder, and wash the mixer container with water, adding the washings to the cylinder to make 250 ml. Stopper the cylinder, invert it several times to mix the sample, and determine the conductivity of the slurry using a suitable conductance bridge assembly. By means of a standard curve, obtained from solutions containing 50, 100, 200 and 500 mg of sodium sulfate per 100 ml, determine the concentration (C), in mg per 100 ml, of sodium sulfate in the sample slurry, and calculate the correction factor (F) by the formula:

$$F = 0.437 \times 2.5 \times C \times \frac{w}{W}$$

where w is the weight of the sample taken for the sodium oxide determination, and W is the weight of the sample taken for the preparation of the slurry.