ALUMINIUM SULFATE (ANHYDROUS)

Prepared at the 55th JECFA (2000) and published in FNP 52 Add 8 (2000), superseding tentative specifications prepared at the 22nd JECFA (1978) and published in FNP 7 (1978) and in FNP 52 (1992). No ADI was allocated at the 22nd JECFA (1978).

SYNONYMS INS No. 520

DEFINITION

Chemical names	Aluminium sulfate
C.A.S. number	10043-01-3
Chemical formula	$Al_2(SO_4)_3$
Formula weight	342.13
Assay	Not less than 99.5% on the ignited basis

DESCRIPTION White powder, shining plates, or crystalline fragments; odourless

FUNCTIONAL USES Firming agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)	Freely soluble in water, insoluble in ethanol
pH (Vol. 4)	2.9 or above (5% solution)
Test for aluminium (Vol. 4)	Passes test
Test for sulfate (Vol. 4)	Passes test

PURITY

Loss on ignition (Vol. 4)	Not more than 5% (about 500°, 3 h)
Alkalis and alkaline earths (Vol. 4)	To a boiling solution of 2 g of the sample in 150 ml of water add a few drops of methyl red TS. Then add ammonia TS until the colour of the solution just changes to a distinct yellow. Add hot water to restore the original volume, and filter while hot. Evaporate 75 ml of the filtrate to dryness, and ignite to constant weight. Not more than 4 mg of residue remains (about 0.4%).
Fluoride	Not more than 30 mg/kg

See description under TESTS

Lead

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"

Selenium (Vol. 4)

Not more than 30 mg/kg
Test 0.2 g of the sample as directed in the Limit Test (Method II)

TESTS

Fluoride

Lime Suspension
Carefully slake about 56 g of low-fluoride calcium oxide (about 2 mg/kg F) with 250 ml of water, and add 250 ml of 60% perchloric acid slowly and with stirring. Add a few glass beads, and boil to copious fumes of perchloric acid, then cool, add 200 ml of water, and boil again. Repeat the dilution and boiling once more, cool, dilute considerably, and filter through a fritted glass filter, if precipitated silicon dioxide appears. Pour the clear solution, with stirring, into 1000 ml of a 1 in 10 sodium hydroxide solution, allow the precipitate to settle, and siphon off the supernatant liquid. Remove the sodium salts from the precipitate by washing 5 times in large centrifuge bottles, shaking the mass thoroughly each time. Finally, shake the precipitate into a suspension and dilute to 2000 ml. Store in paraffin-lined bottles and shake well before use. 100 ml of this suspension should give no appreciable fluoride blank when evaporated in a platinum dish and treated as directed below under "Procedure", beginning with "Ignite briefly at 600^{o},...".

Distillation Apparatus
Connect a 125-ml distillation flask with a condenser. Equip also with a thermometer and a capillary tube, both of which must extend into the liquid. Connect a small dropping funnel or a steam generator to the capillary tube. Support the flask on an asbestos mat with a hole which exposes about one-third of the flask to the flame. To minimize the distillation blank resulting from fluoride leached from the glassware, the distillation apparatus should be treated as follows: treat the glassware with hot 10% sodium hydroxide solution, followed by flushing with tap water and rinsing with distilled water. At least once daily, treat in addition by boiling down 15-20 ml of a 1 in 2 sulfuric acid solution until the still is filled with fumes; cool, pour off the acid, treat again with 10% sodium hydroxide solution, and rinse thoroughly.

Procedure
Add to the distilling flask 1.67 g of the sample, accurately weighed, and 25 ml of a 1 in 2 sulfuric acid solution. Distil until the temperature reaches 160^{o}, then maintain at 160^{o} to 165^{o} by adding water from the funnel, collecting 300 ml of distillate. Oxidize the distillate by the cautious addition of 2 or 3 ml of fluoride-free 30% hydrogen peroxide

(to remove sulfites), allow to stand for a few min, and evaporate in a platinum dish with an excess of Lime Suspension. Ignite briefly at 600°, then cool and wet the ash with about 10 ml of water. Cover the dish with a watch glass, and cautiously introduce under cover just sufficient 60% perchloric acid to dissolve the ash. Add the contents of the dish through the dropping funnel of a freshly treated distilling apparatus (the distilling flask should contain a few glass beads), using a total of 20 ml of 60% perchloric acid for dissolving the ash and transferring the solution. Add 10 ml of water and a few drops of a 1 in 2 silver perchlorate solution through the dropping funnel. Continue as directed in the Limit Test, Method I, *Thorium Nitrate Colorimetric Method*, beginning with "Distil until the temperature reaches 135° ...".

METHOD OF ASSAY Weigh accurately about 4 g of the sample, transfer into a 250-ml volumetric flask. Dissolve in water, dilute to volume with water, and mix. Pipet 10 ml of this solution into a 250-ml beaker, add 25.0 ml of 0.05 M disodium ethylenediaminetetra-acetate, and boil gently for 5 min. Cool, and with continuous stirring add in the order given: 20 ml of pH 4.5 buffer solution (77.1 g of ammonium acetate and 57 ml of glacial acid in 1000 ml), 50 ml of ethanol, and 2 ml of dithizone TS. Titrate with 0.05 M zinc sulfate until the colour changes from green-violet to rose-pink, and perform a blank determination, substituting 10 ml of water for the sample. Each ml of 0.05 M disodium ethylenediaminetetraacetate is equivalent to 8.553 mg of $Al_2(SO_4)_3$.