ALUMINIUM AMMONIUM SULFATE

Prepared at the 29th JECFA (1985), published in FNP 34 (1986) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004). A group PTWI of 1 mg/kg bw for aluminium and its salts was established at the 67th JECFA (2006).

SYNONYMS Ammonium alum; INS No. 523

DEFINITION

Chemical names: Aluminium ammonium sulphate

C.A.S. number: 7784-25-0

Chemical formula: $AlNH_4(SO_4)_2 \cdot 12H_2O$

Formula weight: 453.32

Assay: Not less than 99.5% of $AlNH_4(SO_4)_2 \cdot 12H_2O$

DESCRIPTION Large, colourless crystals, white granules, or a powder; odourless

FUNCTIONAL USES Buffer, neutralizing agent, colour fixative

CHARACTERISTICS

IDENTIFICATION

Solubility: Freely soluble in water; insoluble in ethanol

Test for aluminium (Vol. 4): Passes test

Test for ammonium (Vol. 4): Passes test

Test for sulfate (Vol. 4): Passes test

PURITY

Alkalis and alkaline earths (Vol. 4 for TS solutions): Completely precipitate the aluminium from a boiling solution of 1 g of the sample in 100 ml of water by the addition of enough ammonia TS to render the solution distinctly alkaline to methyl red TS, and filter. Evaporate the filtrate to dryness, and ignite. The weight of the residue does not exceed 5 mg.

Fluoride: Not more than 30 mg/kg
See description under TESTS

Selenium (Vol. 4): Not more than 30 mg/kg
Test 0.2 g of the sample as directed in the Limit Test (Method II)

Lead

Not more than 3 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.”

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 until copious fumes of perchloric acid are evolved. 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 sodium hydroxide solution (1 in 10), allow the precipitate to settle, and siphon off the supernatant liquid. Remove the sodium salts from the precipitate by washing five 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. (Note: 100 ml of this suspension should give no appreciable fluoride blank when evaporated, distilled, and titrated as directed in the Limit Test, Method I *Thorium Nitrate Colorimetric Method*).

Procedure
Assemble the distilling apparatus as directed in the Limit Test (Method I*)*, and add to the distilling flask about 1.67 g of the sample, accurately weighed, and 25 ml of dilute sulfuric acid (1 in 2). 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 fluorine-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^{o}, then cool and wet the ash with about 10 ml of water. Cover the dish with a watch glass, and cautiously introduce under the cover just sufficient 60% perchloric acid to dissolve the ash. Add the contents of the dish through the dropping funnel of a freshly prepared distilling apparatus (the distilling flask should contain a few glass beads), using a total of 20 ml of the perchloric acid for dissolving the ash and transferring the solution. Add 10 ml of water and a few drops of silver perchlorate solution (1 in 2) through the dropping funnel, and continue as directed in the Limit Test Method I *Thorium Nitrate Colorimetric Method* beginning with "Distil until the temperature reaches 135^{o}." (See Volume 4)

METHOD OF ASSAY

Weigh accurately about 1 g of the sample, dissolve in 50 ml of water, add 50 ml of 0.05 M disodium EDTA and 20 ml of pH 4.5 buffer solution (77.1 g of ammonium acetate and 57 ml of glacial acetic acid in 1000 ml of solution), and boil gently for 5 min. Cool, and add 50 ml of ethanol and 2 ml of dithizone TS. Titrate with 0.05 M zinc sulfate to a bright rose-pink colour, and perform a blank determination. Each ml of 0.05 M disodium EDTA is equivalent to 22.67 mg of $AlNH_4(SO_4)_2 \cdot 12H_2O$.