

SULFURIC ACID

Prepared at the 20th JECFA (1976), published in FNS 1B (1977) and in FNP 52 (1992). Metals and arsenic specifications revised at the 59th JECFA (2002). No ADI was allocated at the 20th JECFA (1976)

SYNONYMS

INS No. 513

DEFINITION

Chemical names	Sulfuric acid
C.A.S. number	7664-93-9
Chemical formula	H ₂ SO ₄
Formula weight	98.07
Assay	Not less than the minimum amount of H ₂ SO ₄ specified by the vendor

DESCRIPTION Clear, colourless or slightly brown, very corrosive oily liquid

FUNCTIONAL USES Acid

CHARACTERISTICS

IDENTIFICATION

<u>Solubility</u> (Vol. 4)	Miscible with water, with generation of much heat, and with ethanol
<u>Test for acid</u>	A 1 in 100 solution of the sample is acid to litmus paper.
<u>Test for sulfate</u> (Vol. 4)	Passes test

PURITY

<u>Total ash</u>	Not more than 0.02% (w/w) Weigh 5 g of the sample in a platinum or quartz dish. Evaporate to dryness on a sand bath, strongly ignite at the temperature of 450° - 550° to constant weight. The weight of the residue does not exceed 1 mg.
<u>Nitrate</u>	Not more than 10 mg/kg on H ₂ SO ₄ basis See description under TESTS
<u>Reducing substances</u>	Not more than 40 mg/kg as SO ₂ . Carefully dilute 8 g of the sample with about 50 ml of ice-cold water, keeping the solution cool during the addition. To the dilution add 0.10 ml of 0.1 N potassium permanganate. The solution remains pink for not less than 5 min.
<u>Chlorides</u> (Vol. 4)	Not more than 50 mg/kg 2 g of the sample meets the requirements of the Limit Test for Chloride, using 0.1 mg of chloride ion (Cl) in the control.
<u>Iron</u>	Not more than 200 mg/kg Transfer a volume equivalent to 5 g of the sample into about 25 ml of water contained in a 50-ml volumetric flask, cool, and dilute to volume. Dilute 1 ml

portion of the solution (100-mg sample) to 40 ml. Add about 30 mg of ammonium persulfate crystals and 10 ml of ammonium thiocyanate TS. Any red colour does not exceed that produced by 2 ml of Iron Standard solution (20 µg Fe) in an equal volume of solution containing the same quantities of the reagents used in the test.

Selenium (Vol. 4)

Not more than 20 mg/kg
0.3 g of the sample meets the requirements of the Limit Test for Selenium (Method II).

Lead (Vol. 4)

Not more than 2 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

PURITY TESTS

Nitrate

Standard Nitrate Solution:

Transfer 8.022 g of potassium nitrate, KNO_3 , previously dried at 105° for 1 h, into a 500-ml volumetric flask, dissolve it in water, dilute to volume and mix well. Slowly add from a buret 5 ml of this solution to 400 ml of reagent grade sulfuric acid, previously cooled to 5° , keeping the tip of the buret below the surface of the acid. After the solution has reached room temperature, transfer into a 500-ml volumetric flask, and dilute to volume with reagent grade sulfuric acid. Each ml contains 100 µg of HNO_3 .

Procedure:

Into each of two 100-ml Nessler tubes transfer 50 ml of reagent grade sulfuric acid, add slowly 5 ml of a freshly prepared 1 in 10 solution of ferrous sulfate, $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, mix with a glass rod, and cool in an ice bath to between 10° and 15° . To one tube of the cooled mixture add a 10-ml sample, previously cooled to between 10° and 15° , and dilute to the 100-ml mark with reagent grade sulfuric acid chilled to about the same temperature. Add the Standard Nitrate Solution, dropwise, from a micro-buret to the other tube, with frequent mixing, until the colour of the control solution to 100 ml and continue adding the Standard Nitrate Solution to as exact a match in colour intensity as possible when compared with the sample solution by looking down through the solutions against a white background illuminated by diffused light.

Compute the weight of H_2SO_4 in the weight of the sample from the concentration obtained by the Assay. Not more than 0.1 ml of the Standard Nitrate Solution is required for each gram of H_2SO_4 .

METHOD OF ASSAY

Transfer a 1-ml sample into a small, tared, glass-stoppered conical flask, insert the stopper, weigh accurately, and cautiously add about 30 ml of water. Cool the mixture, add methyl orange TS, and titrate with 1 N sodium hydroxide. Each ml of 1 N sodium hydroxide is equivalent to 49.04 mg of H_2SO_4 .