SODIUM IRON (III) ETHYLENEDIAMINETETRAACETATE, TRIHYDRATE

Prepared at the 53rd JECFA (1999) and published in FNP 52 Add 7 (1999), superseding specifications prepared at the 41st JECFA (1993), published in FNP 52 Add 2 (1993). Evaluated as safe for use in supervised food fortification programmes at the 53rd JECFA (1999)

SYNONYMS Ferric sodium edetate, sodium iron EDTA, sodium feredetate

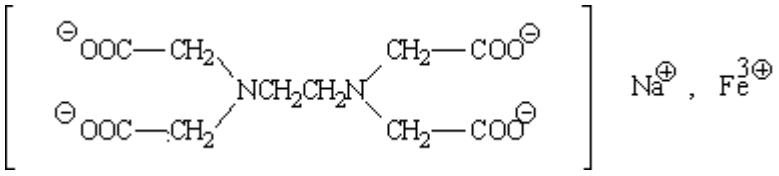
DEFINITION

Chemical names: Sodium [[N,N'-ethanediylbis[N-(carboxymethyl) glycinato]] (4-)] ferrate(1-); sodium [(ethylenedinitrilo) tetraacetato]ferrate(1-); Sodium iron (III) ethylenediaminetetraacetate

C.A.S. number: 15708-41-5

Chemical formula: $C_{10}H_{12}FeN_2NaO_8 \cdot 3H_2O$

Structural formula:



Formula weight: 421.09 (trihydrate)

Assay: Not less than 12.5% and not more than 13.5% iron, calculated on the basis of the trihydrate. Not less than 65.5% and not more than 70.5% EDTA, calculated on the basis of the trihydrate.

DESCRIPTION Light yellow coloured powder that is relatively stable and unaffected by storage

FUNCTIONAL USES Nutrient supplement

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4): Soluble in water

Test for iron (Vol. 4): Passes test

Test for sodium (Vol. 4): Passes test

PURITY

pH (Vol. 4): 3.5 - 5.5 (1 in 100 soln)

Water-insoluble matter — Not more than 0.1%
Weigh accurately 5 g of sample and transfer into 100 ml water. Stir until dissolved. Place a filter paper (1 µm porosity, maximum) in a Gooch crucible (3.5-4.0 cm) and seat the paper by applying vacuum while washing with water. Dry the crucible at 175° for 15 min, cool in a desiccator, and weigh. Pour sample solution through the crucible and wash with three successive 10 ml portions of water. Dry the crucible at 110° for one hour. Cool in a desiccator and weigh the crucible. Calculate as percentage.

Nitrilotriacetic acid — Not more than 0.1%
See description under TESTS

Arsenic (Vol. 4) — Not more than 1 mg/kg
Test 3 g of the sample as directed in the Limit test (Method II)

Lead (Vol. 4) — Not more than 1 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

Nitrilotriacetic acid

Mobile Phase Add 10 ml of a 1 in 4 solution of tetrabutylammonium hydroxide in methanol to 200 ml of water, and adjust with 1 M phosphoric acid to a pH of 7.5 ± 0.1. Transfer the solution to a 1000-ml volumetric flask, add 90 ml of methanol, dilute with water to volume, mix, filter through a membrane filter (0.5-µg or finer porosity), and degas.

Ammonium hydroxide blank solution Add 0.5 ml of ammonium hydroxide to a 10 ml volumetric flask. Dilute with water to volume.

Stock Standard Solution Transfer about 100 mg of nitrilotriacetic acid, accurately weighed, to a 10-ml volumetric flask, and add 0.5 ml of ammonium hydroxide, and mix. Dilute to volume, and mix.

Standard Preparation Transfer 1.0 g of the sample to a 100-ml volumetric flask. Add 100 µl of the *Stock Standard Solution,* dilute with water to volume, and mix. Sonicate, if necessary, to achieve a complete solution.

Test Preparation Transfer 1.0 g of the sample to a 100-ml volumetric flask. Add 100 µl of *Ammonium hydroxide blank solution* and dilute with water to volume, and mix. Sonicate, if necessary, to achieve complete solution.

Chromatographic System (see High-Pressure Liquid Chromatography, in General Methods (Guide to JECFA Specifications), FNP 5/Rev. 2 (1991)). The chromatograph is equipped with a 254-nm detector and a 4.6-nm × 15-cm column that contains 5- 10-µm porous microparticles of silica to which is bonded octylsilane (Zorbax 8 or equivalent). The flow rate is about 2 ml/min. Chromatograph three replicate injections of the *Standard Preparation,* and

record the peak responses as directed under *Procedure.* The relative standard deviation is not more than 2.0%, and the resolution factor between nitrilotriacetic acid and sodium iron EDTA is not less than 4.0.

Procedure Separately inject equal volumes (about 50 µl) of the *Standard Preparation* and the *Test Preparation* into the chromatograph, record the chromatograms, and measure the responses for the major peaks. The retention times are about 3.5 min for nitrilotriacetic acid and 11.9 min for sodium iron EDTA. The response of the nitrilotriacetic acid peak of the *Test Preparation* does not exceed the difference between the nitrilotriacetic acid peak responses obtained from the *Standard Preparation* and the *Test Preparation.*

METHOD OF ASSAY

Iron

Dissolve the sample (approx. 0.5 g accurately weighed) in distilled water (40 ml) in an iodine flask. Add concentrated hydrochloric acid (20 ml) mix, add potassium iodide (3 g) and then allow to stand for 5 min.
Titrate the liberated iodine with standardised 0.1 M sodium thiosulfate, using starch solution as indicator. Avoid vigorous mixing during the titration. Perform a blank, omitting the sample.

$$\%Fe = \frac{(Ts - Tb) \times M \times 0.05585 \times 100}{W}$$

where
Ts = sample titre in ml
Tb = blank titre in ml
0.05585 = atomic weight of iron × 10^{-3}
M = molarity of sodium thiosulfate
W = sample weight (g)

EDTA

Reagents

(1) 0.25 M calcium acetate solution, standardized - Weigh and transfer 44.0 g reagent grade calcium acetate monohydrate to a 1 L volumetric flask; add water to dissolve and fill to the mark. Weigh accurately 2.0 to 2.1 g of reagent grade EDTA acid into each of three 250-ml conical flasks. Add 150 ml water and adjust to pH 11-12 (pH paper may be used) with 50% sodium hydroxide solution. Add about 30 mg of hydroxynaphthol blue indicator and titrate with calcium acetate to a sharp red endpoint.

$$\text{molarity} = \frac{\text{weight of EDTA acid (g)} \times 1000}{\text{ml titrant} \times 292.24}$$

(2) Triethanolamine, reagent grade. (3) Hydroxynaphthol Blue indicator. (4) 50 % sodium hydroxide solution.

Procedure

Accurately weigh 0.8 - 1.0 g of sample into a 250-ml beaker. Add 75 ml of distilled water to dissolve. Adjust the pH to 9.0 by dropwise addition of triethanolamine. Then adjust to pH 12.5 - 13.0 by addition of 50% aqueous

NaOH. The solution should be clear and colourless. Add about 30 mg hydroxynaphthol blue indicator and titrate with 0.25 M calcium acetate solution to a red endpoint.

$$\% \text{ EDTA acid} = \frac{\text{ml } Ca(OAc)_2 \text{ x molarity x } 292.24}{10 \text{ x sample weight (g)}}$$