STEARYL MONOGLYCERIDYL CITRATE

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FNP 52 (1992). Metals and arsenic specifications revised at the 55th
JECFA (2000). No ADI was allocated at the 26th JECFA (1982*

SYNONYMS   Stearyl monoglyceride citrate

DEFINITION   Prepared by the controlled chemical reaction from citric acid,
monoglycerides, and fatty acids (obtained by the glycerolysis of edible fats
and oils), and stearyl alcohol.

DESCRIPTION   Soft, off-white to tan, waxy solid having a lard-like consistency

FUNCTIONAL USES Emulsifier

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)   Soluble in chloroform and in ethylene glycol; insoluble in water

PURITY

Water (Vol. 4)   Not more than 0.25% (Karl Fischer Method)

Sulfated ash (Vol. 4)   Not more than 0.1%

Acid value (Vol. 4)   Not more than 52

Acids   Acids other than citric and fatty acids (such as acetic acid, succinic acid,
fumaric acid, tartaric acid, lactic acid) shall not be detectable

Saponification value   Not less than 215 and not more than 255
See description under TESTS

Total citric acid   Not less than 15% and not more than 18%
See description under TESTS

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

Saponification value   Transfer about 1 g of the sample, accurately weighed, into a 250-ml
Erlenmeyer flask, and add 25 ml of ethylene glycol, 35 ml of 0.5 N ethanolic
potassium hydroxide, and a few glass beads. Reflux for one h, using a
water condenser, then rinse the condenser with water and cool. Add 1 ml of
phenolphthalein TS and titrate with 0.5 N hydrochloric acid. Perform a blank

determination but do not reflux. The difference between the volumes, in ml of 0.5 N hydrochloric acid consumed in the actual test and in the blank titration, multiplied by 28.05 and divided by the weight, in g, of the sample taken, is the saponification value.

Total citric acid	

Reagents
- Brominating solution: Dissolve 19.84 g of potassium bromide, 5.44 of potassium bromate and 12 g of sodium metavanadate, $NaVO_3$ in water by warming, and dilute to 1,000 ml with water. Filter if necessary.
- Ferrous Sulfate Solution: Dissolve 44 g of ferrous sulfate, $FeSO_4 \cdot 7H_2O$, in 1 N sulfuric acid, dilute to 100 ml with 1 N sulfuric acid. Use within 5 days of preparation.
- Sulfide Solution: On the day of use, dissolve 4 g of thiourea in 100 ml of a 1 in 50 solution of sodium borate, $Na_2B_4O_7 \cdot 10H_2O$, and add 2 ml of sodium sulfide TS. Wait 30 min after the addition of the sodium sulfide TS before using.
- Standard Solution: Transfer about 50 mg of sodium citrate dihydrate, accurately weighed, into 500 ml volumetric flask, dissolve and dilute to volume with water, and mix. Transfer 15 ml of this solution into a 100 ml volumetric flask, dilute to volume with water, and mix. Calculate the concentration (C), in μg per ml of citric acid in the final solution by the formula (15 x 1,000 x 0.6533W)/(100 x 500), in which W is the weight, in mg of the sodium citrate taken, and 0.6533 is the factor converting sodium citrate dihydrate to citric acid.

Sample solution
Transfer about 250 mg of the sample, accurately weighed, to a 250-ml extraction flask, and add 15 ml of 0.5 N sodium hydroxide, 5 ml of ethanol and a few glass beads. Connect the flask with a water-cooled condenser, and reflux for 3 h. Immediately cool and neutralize to phenolphthalein TS with 0.5 N hydrochloric acid, then place the flask in an ice-bath and add 5 ml of sulfuric acid TS. Transfer the solution in a 125-ml separator, extract with three 40 ml portions of chloroform, and then extract the combined chloroform extracts in a 250-ml separator with three 10 ml portions of 0.5 N sulfuric acid adding the acid extracts to a second 250-ml separator. Wash the combined acid extracts with two 60 ml portions of chloroform, and discard the chloroform washes. Filter the acid solution into a 500-ml volumetric flask, neutralize slowly with 6 N sodium carbonate, and dilute to volume with water. Transfer 10 ml of this solution into a 100-ml volumetric flask, dilute to volume with water, and mix. Each ml of the final solution contains approximately 10 μg of citric acid.

Procedure
Pipet 2 ml each of the Standard Solution and of the Sample Solution into separate 40- or 45-ml glass-stoppered centrifuge tubes, and add 3 ml of water to each tube. Place 5 ml of water in a third tube for the reagent blank. Place the tubes in a ice-bath, add 5 ml of sulfuric acid TS, mix thoroughly, and allow to stand for exactly 5 min. Remove the tubes from the ice-bath, and allow them to come to room temperature during the next 5 min. To each tube add 5 ml of the Brominating Solution, then insert the stoppers, invert the tubes once or twice, and heat in a water bath at 30° for 20 min. Remove the tubes, add 1.5 ml of Ferrous Sulfate Solution, invert, again, and allow to stand for 5 min, shaking occasionally to ensure complete reduction of the

excess free bromine in the tubes. Add 6.5 ml of petroleum ether, shake for 2 or 3 min, and remove the water layer with a syringe. Wash the ether solutions with 15 ml of water, then remove the water and filter the ether extracts into the original centrifuge tubes which have been previously rinsed with the Sulfide Solution. Filter each ether extract through a tight plug of glass wool onto which has been placed a sufficient amount of anhydrous sodium sulfate to remove the last traces of water from the ether. Place 5.0 ml of the filtrate in a clean dry centrifuge tube, add 3 ml of Sulfide Solution, shake vigorously for 1.5 min and centrifuge. Decant about 0.5 ml of the supernatant ether layer from each tube, then carefully transfer the ether solutions into 1-cm cells and determine the absorbance of the extracts obtained from the Standard Solution and the Sample Solution at 500 nm with a suitable spectrophotometer, using the reagent blank in the reference cell.

Calculate the quantity, in mg of citric acid in the sample taken by the formula $5C \times A_u/A_s$, in which C is the exact concentration in μg per ml of citric acid in the Standard Solution, A_u is the absorbance of the solution from the Sample Solution, and A_s is the absorbance of the solution from the Standard Solution.