# MONOGLYCERIDE CITRATE

Prepared at the 18th JECFA (1974), published in FNP 4 (1978) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004). ADI not evaluated at the 18th JECFA (1974)

**SYNONYMS** Citric acid ester of glyceryl monooleate

**DEFINITION** A mixture of glyceryl monooleate and its citric acid monoester,

manufactured by the reaction of glyceryl monooleate with citric acid under

controlled conditions

Structural formula

$$\mathrm{CH_2-OR_1}$$
  
|  
 $\mathrm{CH-OR_2}$   
|  
 $\mathrm{CH_2-OR_3}$ 

where R<sub>1</sub> represents oleic acid moiety and R<sub>2</sub> and R<sub>3</sub> a citric acid moiety or

hydrogen

**DESCRIPTION** Soft, white to ivory coloured, waxy solid with a lard-like consistency and a

bland odour

FUNCTIONAL USES Synergist and solubilizer for antioxidants and flavours

CHARACTERISTICS

**IDENTIFICATION** 

Solubility (Vol. 4) Insoluble in water; soluble in ethanol

Tests for fatty acids

(Vol. 4)

Passes tests

Test for citrate (Vol. 4) Passes tests

Test for glycerol (Vol. 4) Passes tests

**PURITY** 

Water (Vol. 4) Not more than 0.2% (Karl Fischer Method)

Sulfated ash (Vol. 4) Not more than 0.3%

Test 1 g of the sample

Acid value (Vol. 4) Not less than 70 and not more than 100

Saponification value

(Vol. 4)

Not less than 260 and not more than 265

Total citric acid Not less than 14.0 and not more than 17.0%

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** 

#### Total citric acid

## Standard Solution:

Transfer about 35 mg of sodium citrate dihydrate, accurately weighed, into a 100-ml volumetric flask. Dissolve and dilute to volume with water, and mix. Calculate the concentration (C) in  $\mu$ g per ml of citric acid in the final solution by the formula:

$$C = 1000 \, x \, \frac{0.65333 \, W}{100}$$

where W is the weight, in mg, of the sodium citrate dihydrate taken, and 0.6533 is a factor converting sodium citrate dihydrate to citric acid.

## Sample Solution:

Transfer about 150 mg of the sample accurately weighed, into a saponification flask. Add 50 ml of 4% alcoholic potassium hydroxide solution, and reflux for 1 h. Acidify the reaction mixture with hydrochloric acid to a pH of 2.8 - 3.2, transfer to a 400-ml beaker, and evaporate to dryness on a steam bath. Quantitatively transfer the contents of the beaker into a separator, using no more than 50 ml of water. Extract with three 50-ml portions of petroleum ether (b.p. 30-60°) discarding the extracts. Transfer the water layer to a 100-ml volumetric flask, dilute to volume with water, and mix.

## Procedure:

Pipet 2.0 ml each of the Standard Solution and of the Sample Solution into separate 40-ml graduated centrifuge tubes. Add 2 ml of a 1 in 2 sulfuric acid solution and 11 ml of water to each tube. Boil for 3 min, cool, and add 5 ml of bromine TS to each tube. Dilute to the 20-ml mark, allow to stand for 10 min, and centrifuge. Transfer 4.0 ml of each solution into separate 19 x 110mm test tubes. Add 1 ml of water, 0.5 ml of a 1 in 2 sulfuric acid solution, and 0.3 ml of 1 M potassium bromide, and shake. Add 0.3 ml of 1.5 N potassium permanganate, shake, and allow to stand for 2 min. Add 1 ml of a saturated solution of ferrous sulfate, shake, allow to stand for 2 min. Dilute to 10 ml with water. Add 10.0 ml of n-hexane (previously washed with sulfuric acid, followed by a water wash, and then dried over anhydrous sodium sulfate), shake vigorously for 2 min and centrifuge at a low speed for 1 min. Transfer 5.0 ml of the hexane extract into a 20 x 145-mm tube containing 10.0 ml of sodium sulfide solution (4 g of Na<sub>2</sub>S · 9 H<sub>2</sub>O in 100 ml of water), and shake vigorously briefly (3 oscillations only). Centrifuge the mixture at low speed for 1 min.

Immediately determine the absorbance of each aqueous layer in a 1-cm cell at 450 nm with a suitable spectrophotometer, using a reagent blank in the reference cell. Calculate the quantity, in mg, of citric acid in the sample

taken by the formula:

$$0.1C \times \frac{A_u}{A_s}$$

C is as defined under Standard Solution,  $A_{\text{u}}$  is the absorbance of the final solution from the Sample Solution, and  $A_{\text{s}}$  is that of the final solution from the Standard Solution.