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## **CITRIC AND FATTY ACID ESTERS OF GLYCEROL (TENTATIVE)**

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## CITRIC AND FATTY ACID ESTERS OF GLYCEROL (TENTATIVE)

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### Information required:

- *A validated method for the determination of total citric acid content*
- *Performance characteristics (method validation data) of the citric acid determination method*
- *Data on the total citric acid content, in at least five batches of products currently available in commerce, determined using the above method.*

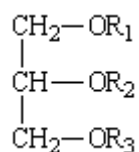
### SYNONYMS

Citric acid esters of mono- and di-glycerides, citroglycerides, CITREM; INS No. 472c

### DEFINITION

Citric and fatty acid esters of glycerol (CITREM) consists of mixed esters of citric acid and edible fatty acids with glycerol. It may contain free fatty acids, glycerol, citric acid and mono- and diglycerides, in minor quantities. The mono- and di- glycerides may include either one or two edible fatty acids from C12:0 to C18:0, mainly the saturated palmitic (C16:0) and stearic (C18:0) acids. It may also contain minor amounts of other fatty acids such as myristic (C14:0), oleic (C18:1), linoleic (C18:2) and arachidic acid (C20:0). CITREM is obtained by esterification of glycerol with citric acid and edible fatty acids, or by reaction of a mixture of mono- and diglycerides of edible fatty acids, with citric acid. CITREM may be partially or wholly neutralized with sodium hydroxide or potassium hydroxide.

### Structural formula



Where at least one of R<sub>1</sub>, R<sub>2</sub> or R<sub>3</sub> represents a citric acid moiety, one represents a fatty acid moiety and the remainder may represent citric acid, fatty acid or hydrogen.

<b>DESCRIPTION</b>	White to ivory coloured, oily to waxy material.
<b>FUNCTIONAL USES</b>	Stabilizer, emulsifier, dough conditioner, antioxidant synergist
<b>CHARACTERISTICS</b>	
<b>IDENTIFICATION</b>	
<u>Solubility</u> (Vol. 4)	Insoluble in water; soluble in oils and fats; insoluble in ethanol
<u>Test for fatty acids</u> (Vol. 4)	Passes test
<u>Test for citric acid</u>	Information required
<u>Test for glycerol</u> (Vol. 4)	Passes test
<b>PURITY</b>	
<u>Sulfated ash</u> (Vol. 4)	Non-neutralized products: not more than 0.5% Partially or wholly neutralized products: not more than 10%; test 2 g of the sample (Method I)
<u>Free glycerol</u> (Vol. 4)	Not more than 4%
<u>Total glycerol</u>	8-33%  See description under TESTS
<u>Total citric acid</u>	13-50%  (Information required)
<u>Total fatty acid</u>	37-81% See description under TESTS

Lead (Vol. 4)

Not more than 2 mg/kg.

(Not more than 0.1 mg/kg for use in infant formula and formula for special medical purposes intended for infants)

Determine using a method appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the methods described in Volume 4 (under “General Methods, Metallic Impurities”).

**TESTS**

**PURITY TESTS**

Total glycerol

CITREM is hydrolyzed, glycerol in the aqueous phase is oxidized using known excess of sodium periodate in a strongly acid medium and the unreacted periodate is back titrated using standard sodium thiosulfate solution.

Procedure:

Accurately weigh about 2 g of the sample into a saponification flask, add 50 ml of 0.5 M ethanolic potassium hydroxide, and reflux for 30 min.

To a 1-L volumetric flask add 99 ml  $\pm$  0.2 ml of chloroform using a burette and add 25 ml of glacial acetic acid using a graduated cylinder. Quantitatively transfer the content of the saponification flask to the volumetric flask, using three 25 ml portions of water. Add about 500 ml of water further, and shake vigorously for about 1 min. Dilute to volume with water, stopper, mix thoroughly and set aside for separation of layers.

Pipet 50 ml of acetic periodic acid TS into a series of 400 ml beakers. Prepare two blanks by adding 50 ml of water to each. Pipet 50 ml of the aqueous layer into one of the 400 ml beakers containing 50 ml of acetic periodic acid TS; shake gently to mix; cover with watch glass,

and allow to stand 30 min but not longer than 1.5 h. Add 20 ml of 15% potassium iodide solution, shake gently to mix, and allow to stand at least 1 min. but not more than 5 min. Do not allow to stand in bright or direct sunlight. Add 200 ml of water and titrate with 0.1 N sodium thiosulfate. Use a variable speed electric stirrer to keep the solution thoroughly mixed. Continue the titration to the disappearance of the brown iodine colour from the aqueous layer. Add 2 ml of starch TS and continue the titration to the disappearance of iodine from the tiny chloroform layer separated during titration and the disappearance of the blue iodine-starch complex colour from the aqueous layer. Read the burette to the nearest 0.01 ml. Treat the blanks in the same way as the sample.

#### Calculation

$$\% \text{ total glycerol} = [(B - S) \times N \times 2.302 \times 900] / (W \times 50)$$

where

- B volume of 0.1 N sodium thiosulfate used for the blank, ml
- S volume of 0.1 N sodium thiosulfate used for the sample, ml
- N exact normality of 0.1 N sodium thiosulfate
- W mass of sample, g

#### Total citric acid

*Information required*

#### Total fatty acid

Principle: This method measures total fatty acids by extracting with diethyl ether.

#### Procedure

Weigh accurately 5 g of the sample into a 250-ml round-bottomed flask, add 50 ml of potassium hydroxide, ethanolic, TS, and reflux for 1 h on a boiling water bath.

Quantitatively transfer the contents of the saponification flask to a 1,000 ml separating funnel, using three 25 ml portions of water, and add 5 drops of methyl orange indicator solution.

Cautiously add 50% hydrochloric acid until the colour of solution changes to orange red. Add 1 ml of excess acid. Shake well to mix the contents and separate the fatty acids.

Cool to room temperature and extract the separated fatty acids with three 100 ml portions of diethyl ether. Combine the extracts, and

wash with 50 ml portions of 10% sodium chloride solution until the washed sodium chloride solution becomes neutral.

Dry the ether solution with anhydrous sodium sulfate. Then evaporate off ether on a steam bath, leave additional 10 min on the steam bath, and weigh the residue. This is the weight of the total fatty acids.

Calculation:

$$\text{Total Fatty acids \%} = \frac{\text{mass of fatty acids g} \times 100}{\text{mass of sample g}}$$