DIACETYLTARTARIC and FATTY ACID ESTERS of GLYCEROL

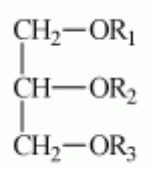
Prepared at the 71st JECFA (2009) and published in FAO JECFA Monographs 7 (2009) superseding specifications prepared at the 57th JECFA (2001) and published in the Combined Compendium of Food Additive Specifications, FAO JECFA Monographs 1 (2005). An ADI of 0-50 mg/kg bw was established at the 61st JECFA (2003).

SYNONYMS Diacetyltartaric acid esters of mono- and diglycerides; DATEM; Tartaric, acetic and fatty acid esters of glycerol, mixed; Mixed acetic and tartaric acid esters of mono and diglycerides of fatty acids; INS No. 472e

DEFINITION The product consists of mixed glycerol esters of mono- and diacetyltartaric acid and fatty acids from edible fats and oils. It is made by the interaction of diacetyltartaric anhydride and mono- and diglycerides of fatty acids in the presence of acetic acid, or by interaction of acetic anhydride and mono- and diglycerides of fatty acids in the presence of tartaric acid.
Because of inter- and intramolecular acyl-group exchange, both methods of production lead to the same essential components, the distribution of which depends on the relative proportions of the basic raw materials, on temperature, and on reaction time. The product may contain small amounts of free glycerol, free fatty acids, and free tartaric and acetic acids.

C.A.S. numbers 308068-42-0
100085-39-0

Structural formula



in which one or two of the R groups is a fatty acid moiety and the other R groups are either:

- diacetylated tartaric acid moiety
- monoacetylated tartaric acid moiety
- tartaric acid moiety
- acetic acid moiety
- hydrogen

DESCRIPTION Liquid, paste, or wax-like solid

FUNCTIONAL USES Emulsifier

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Dispersible in cold and hot water; soluble in methanol, ethanol, acetone, and ethyl acetate.

1,2-diols To a solution of 500 mg in 10 ml methanol, add dropwise, lead

	acetate TS. A white, flocculent, insoluble precipitate is formed.
Fatty acids (Vol. 4)	Passes test (See under “Specific methods, Fats, Oils, and Hydrocarbons; Identification Tests for Functional Groups; Test A: Methyl Esters of Fatty Acids”)
Acetic acid (Vol. 4)	Passes test (See under “Specific methods, Fats, Oils, and Hydrocarbons; Identification Tests for Functional Groups”)
Tartaric acid (Vol.4)	Passes test (See under “Specific methods, Fats, Oils, and Hydrocarbons; Identification Tests for Functional Groups”)
Glycerol (Vol. 4)	Passes test (See under “Specific methods, Fats, Oils, and Hydrocarbons; Identification Tests for Functional Groups”)
PURITY	
Acids (Vol. 4)	Acids other than acetic, tartaric and fatty acids, shall not be detectable (See under “Specific methods, Fats, Oils and Hydrocarbons; Identification Tests for Functional Groups”)
Sulfated ash (Vol. 4)	Not more than 0.5% determined at 800±25º Test 5 g of sample (Method I for solids; Method II for liquids) (See under, “General methods, Inorganic Components; Ash”)
Acid value (Vol. 4)	Not less than 40 and not more than 130 (See under, “Specific methods, Fats, Oils and Hydrocarbons”)
Total acetic acid	Not less than 8% and not more than 32% after hydrolysis See description under TESTS
Total tartaric acid	Not less than 10% and not more than 40% after saponification See description under TESTS
Total glycerol	Not less than 11% and not more than 28 % after saponification See description under TESTS
Free glycerol (Vol. 4)	Not more than 2.0% Prepare the aqueous extracts as directed under the "Procedure for 1-Monoglyceride (see under, "Specific methods, Fats, Oils and Hydrocarbons; 1-Monoglyceride and Free Glycerol Content") and proceed as directed under the "Procedure for Glycerol".
Lead (Vol. 4)	Not more than 2 mg/kg Determine using an AAS/ICP-AES 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 (under "General Methods, Metallic Impurities").

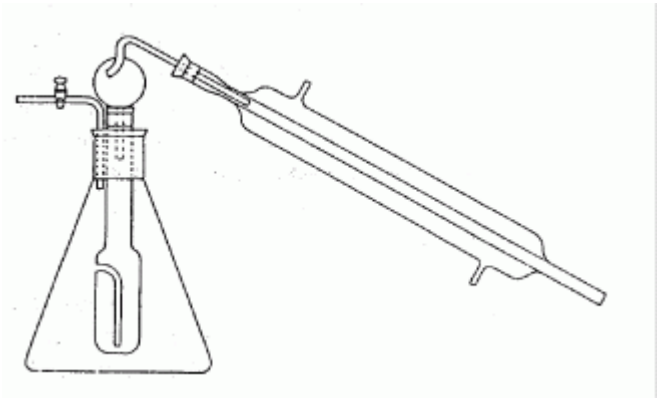
TESTS

PURITY TESTS

Total acetic acid

Apparatus

Assemble a modified Hortvet-Sellier distillation apparatus as shown in the figure, using a sufficiently large (approximately 38- x 203-mm) inner Sellier tube and large distillation trap.



Modified Hortvet-Sellier Distillation Apparatus

Procedure

Transfer 4 g of sample, accurately weighed, into the inner tube of the assembly, and insert the tube in the outer flask containing about 300 ml of recently boiled hot water. To the sample cautiously add 10 ml of approximately 4N perchloric acid [35 ml (60 g) of 70% perchloric acid in 100 ml of water], and connect the inner tube to a water-cooled condenser through the distillation trap. Distil by heating the outer flask so that 100 ml of distillate is collected within 20 to 25 min. Collect the distillate in 100-ml portions, add phenolphthalein TS to each portion, and titrate with 0.5N sodium hydroxide. Continue the distillation until a 100-ml portion of the distillate requires no more than 0.5 ml of 0.5N sodium hydroxide for neutralization. (CAUTION: Do not distil to dryness.) Calculate the weight, in mg, of volatile acids in the sample taken by the formula V x *e*, in which V is the total volume, in ml, of 0.5N sodium hydroxide consumed in the series of titrations and *e* is the equivalence factor 30.03.

Total tartaric acid

Sample Preparation

Transfer about 4 g of the sample, accurately weighed, into a 250-ml Erlenmeyer flask, and add 80 ml of approximately 0.5N potassium hydroxide and 0.5 ml of phenolphthalein TS. Connect an air condenser at least 65 cm in length to the flask, and heat the mixture on a hot plate for about 2.5 h. Add to the hot mixture approximately 10% phosphoric acid until it is definitely acid to congo red test paper. Reconnect the air condenser, and heat until the fatty acids are liquefied and clear. Cool and then transfer the mixture into a 250-ml separator with the aid of small portions of water and hexane. Extract the liberated fatty acids with three

successive 25-ml portions of hexane and collect the extracts in a second separatory funnel. Wash the combined hexane extracts with two 25-ml portions of water and add the washings to the first separatory funnel containing the water layer. Transfer the contents of the first funnel to a 250-ml beaker, heat on a steam bath to remove traces of hexane, filter through acid-washed, fine-texture filter paper into a 500-ml volumetric flask, and finally dilute to volume with water (Solution I). Pipet 25.0 ml of this solution into a 100-ml volumetric flask, and dilute to volume with water (Solution II). Retain the rest of Solution I for the determination of Total glycerol.

Standard solutions and blank

Transfer 100 mg of reagent-grade tartaric acid, accurately weighed, into a 100-ml volumetric flask, dissolve it in about 90 ml of water, add water to volume, and mix well. Transfer 3.0-. 4.0-, 5.0-, and 6.0-ml portions into separate 19- x 150-mm matched cuvettes, and add sufficient water to make 10.0 ml. To each cuvette add 4.0 ml of a freshly prepared 1 in 20 solution of sodium metavanadate and 1.0 ml of acetic acid. (NOTE: Use these solutions within 10 min after colour development.) Prepare a blank in the same manner, using 10 ml of water in place of the tartaric acid solutions.

Sample solution

Transfer 10.0 ml of Solution II into a 19- x 150-mm cuvette and add 4.0 ml of a freshly prepared 1 in 20 solution of sodium metavanadate and 1.0 ml of acetic acid. (NOTE: Use this solution within 10 min after colour development.)

Analysis

Set the suitable spectrophotometer at zero with the blank. Then determine the absorbance of the four Standard solutions of tartaric acid and the Sample solution at 520 nm. From the data thus obtained, prepare a standard curve by plotting the absorbances on the ordinate against the corresponding quantities, in mg, of the tartaric acid on the abscissa. Then from the curve, determine the weight, in mg, of tartaric acid in the final dilution, multiply this by 20, and divide the result by the weight of the original sample to give the percentage of tartaric acid.

Total glycerol

Transfer 5.0 ml of Solution I prepared in the test for Total tartaric acid into a 250-ml glass-stoppered Erlenmeyer or iodine flask. Add to the flask 15 ml of glacial acetic acid and 25.0 ml of periodic acid solution, prepared by dissolving 2.7 g of periodic acid (H_5IO_6) in 50 ml of water, adding 950 ml of glacial acetic acid, and mixing thoroughly; protect this solution from light. Shake the mixture for 1 or 2 min, allow it to stand for 15 min, add 15 ml of potassium iodide solution (150 mg/ml) and 15 ml of water, swirl, and let stand 1 min. Titrate the liberated iodine with 0.1N sodium thiosulfate, using starch TS as the indicator. Perform a residual blank titration using water in place of the sample. The corrected volume is the number of ml of 0.1N sodium thiosulfate required for the glycerol and the tartaric acid in the sample represented by the 5 ml of Solution I. From the percentage of the tartaric acid determined in the test for Total tartaric acid, calculate the volume of 0.1N sodium thiosulfate required for the tartaric acid in the titration. The difference between

the corrected volume and the calculated volume required for the tartaric acid is the number of ml of 0.1N sodium thiosulfate consumed due to the glycerol in the sample. One ml of 0.1N sodium thiosulfate is equivalent to 2.303 mg of glycerol and to 7.505 mg of tartaric acid.