DIACETYLTARTARIC and FATTY ACID ESTERS of GLYCEROL

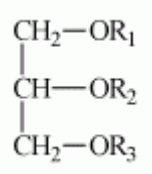
Prepared at the 57th JECFA (2001) and published in FNP 52 Add 9 (2001) superseding specifications prepared at the 51st JECFA and published in FNP 52 Add 6 (1998).An ADI of 0-50mg/kg bw was established at the 61st JECFA (2003)

These specifications cover the two products Diacetyltartaric and fatty acid esters of glycerol (INS 472e) and Tartaric, acetic and fatty acid esters of glycerol, mixed (INS 472f), which before 1998 had separate specifications, published in FNP 52 Add 5 (1997). The two specifications were combined at the 51st JECFA into one single set of specification, published in FNP 52 Add 6 (1998) under the name Diacetyltartaric and fatty acid esters of glycerol. As specifications no longer exist for Tartaric, acetic and fatty acid esters of glycerol, mixed, the ADI "not limited" was withdrawn at the 57th JECFA (2001).

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

DEFINITION The product consists of mixed glycerol esters of mono- and diacetyltartaric acid and fatty acids of food fats. 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.
Due to 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. The article of commerce may be further specified as to acid value, total tartaric acid content, free acetic acid content, saponification value, iodine value, free fatty acid content, solidification point of the free fatty acids.

Structural formula The major components are:



in which
1) one or two of the R groups is a fatty acid moiety
2) the other R groups are either
- diacetylated tartaric acid moiety
- monoacetylated tartaric acid moiety
- tartaric acid moiety
- acetic acid moiety
- hydrogen

DESCRIPTION From liquid to paste to wax-like solids (flakes or powder)

FUNCTIONAL USES Emulsifier

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)	Dispersible in cold and hot water, soluble in methanol and ethanol.
Test for 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.
Test for fatty acids (Vol. 4)	Passes test
Test for acetic acid (Vol. 4)	Passes test
Test for tartaric acid (Vol.4)	Passes test
Test for glycerol (Vol. 4)	Passes test

PURITY

Acids (Vol. 4)	Acids other than acetic, tartaric and fatty acids, shall not be detectable
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)
Free fatty acids (Vol. 4)	Not more than 3% as oleic acid
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%
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 on Volume 4, "Instrumental methods".

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.

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 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

Standard Curve

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. Set the instrument at zero with the blank, and then determine the absorbance of the four solutions of tartaric acid at 520 nm with a suitable spectrophotometer or a photoelectric colorimeter equipped with a 520-nm filter. From the data thus obtained, prepare a curve by plotting the absorbances on the ordinate against the corresponding quantities, in mg, of the tartaric acid on the abscissa.

Test 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 chloroform. Extract the liberated fatty acids with three successive 25-ml portions of water, and add the washings to the separator containing the water layer. Transfer the contents of the first separator to a 250-ml beaker, heat on a steam bath to remove traces of chloroform, 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.

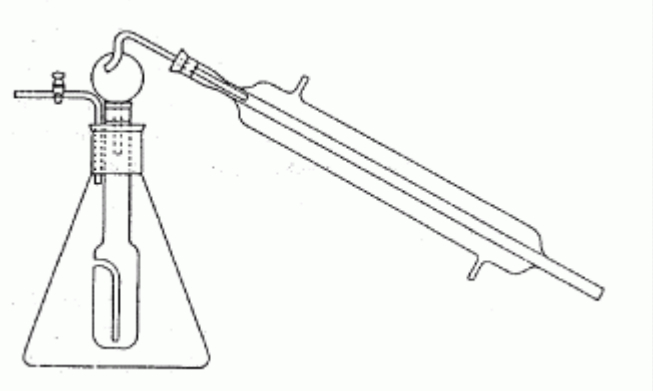
Procedure

Transfer 10.0 ml of Solution II prepared under Test Preparation into a 19- x

150-mm cuvette, and continue as directed under Standard Curve, beginning with "To each cuvette add 4.0 ml of a...". From the standard 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 for obtaining 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 (15 in 100) and 15 ml of water, swirl, let stand 1 min, and then 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 determined in the Assay for 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.



Modified Hortvet-Sellier Distillation Apparatus