Diacetyl Tartaric Acid Esters of Mono- and Diglycerides

DATEM

CAS: [91052-83-4]

INS: 472e CAS: [100085-39-0]

DESCRIPTION

Diacetyl Tartaric Acid Esters of Mono- and Diglycerides occur over a range in appearance from sticky, viscous liquids through a fatlike consistency to a waxy solid, depending on the iodine value of the oils or fats used in their manufacture. They are the reaction product of partial glycerides of edible oils, fats, or fat-forming fatty acids with diacetyl tartaric anhydride. The diacetyl tartaroyl esters are miscible in all proportions with oils and fats. They are soluble in most common fat solvents, in methanol, in acetone, and in ethyl acetate, but are insoluble in other alcohols, in acetic acid, and in water. They are dispersible in water and resistant to hydrolysis for moderate periods of time. The pH of a 3% dispersion in water is between 2 and 3.

Function Emulsifier.

REQUIREMENTS

Identification Add, dropwise, lead acetate TS to a solution of 500 mg of sample in 10 mL of methanol. A white, flocculent, practically insoluble precipitate forms.
Assay for Tartaric Acid Between 17.0 and 20.0 g of tartaric acid ($C_4H_6O_6$) per 100 g of sample after saponification.
Acetic Acid Between 14.0 and 17.0 g of acetic acid (CH_3COOH) per 100 g of sample after hydrolysis.
Acid Value Between 62 and 76.
Fatty Acids (Total) Not less than 56.0 g of total fatty acids per 100 g of sample after hydrolysis.
Glycerin Not less than 12.0 g of glycerin ($C_3H_8O_3$) per 100 g of sample after hydrolysis.
Lead Not more than 2 mg/kg.
Residue on Ignition Not more than 0.01%.
Saponification Value Between 380 and 425.

TESTS

Assay for Tartaric Acid

Standard Reference 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- × 150-mm matched cuvettes, and add sufficient water to make 10.0 mL. Add 4.0 mL of a freshly prepared 1:20 sodium metavanadate solution and 1.0 mL of glacial acetic acid to each cuvette.

> **Note**: Use these solutions within 10 min after color development.

Prepare a blank in the same manner, using 10 mL of water in place of the tartaric acid solutions. Set a suitable spectropho-

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

Assay Preparation Transfer about 4 g of sample, accurately weighed, into a 250-mL Erlenmeyer flask, and add 80 mL of 0.5 *N* potassium hydroxide and 0.5 mL of phenolphthalein TS. Connect an air condenser at least 65 cm long to the flask, and heat the mixture on a hot plate for about 2.5 h. Remove the air condenser and add approximately 10% phosphoric acid to the hot mixture until it is definitely acid to congo red test paper. Reconnect the air condenser, and heat until the fatty acids are liquified and clear. Cool, and 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 separator. Wash the combined hexane extracts with two 25-mL portions of water, and add the washings to the separator containing the water layer. Retain the combined hexane extracts for the determination of total fatty acids. Transfer the contents of the first separator 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 *Glycerin* (below).

Procedure Transfer 10.0 mL of *Solution II* into a 19- × 150-mm cuvette, and continue as directed under *Standard Reference Curve*, beginning with "add 4.0 mL of a freshly prepared 1:20 sodium metavanadate solution. . . ." From the reference curve, determine the weight, in milligrams, of tartaric acid in the final dilution, multiply this by 20, and divide the result by the weight of the original sample to obtain the percentage of tartaric acid.

Acetic Acid Determine as directed under *Volatile Acidity*, Appendix VII, using a 4-g sample, accurately weighed, and 30.03 as the equivalence factor (*e*).

Acid Value Transfer about 1 g of sample, accurately weighed, into a 125-mL Erlenmeyer flask. Prepare a solvent by mixing 1 volume of hexane with 4 volumes of methanol, adding phenol red TS, and neutralizing, if necessary. Dissolve the sample in about 25 mL of this solvent by gently warming, if necessary. Titrate the solution with 0.1 *N* methanolic potassium hydroxide to a light red endpoint. Perform a blank determination (see *General Provisions*) using a 25-mL portion of the solvent, and make any necessary correction. Calculate the acid value by the formula

$$56.1V \times N/W,$$

in which V is the volume, in milliliters, of the methanolic potassium hydroxide and N is the normality; and W is the weight, in grams, of the sample taken.

Fatty Acids (Total) Dry the combined hexane extracts of fatty acids obtained in the *Assay for Tartaric Acid* by shaking with a few grams of anhydrous sodium sulfate. Filter the

solution into a tared, 250-mL beaker, evaporate the hexane on a steam bath, cool, and weigh.

Glycerin Prepare periodic acid solution 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). Transfer 5.0 mL of *Solution I*, prepared in the *Assay for Tartaric Acid* (above), into a 250-mL glass-stoppered Erlenmeyer or iodine flask. Add 15 mL of glacial acetic acid and 25.0 mL of periodic acid solution to the flask, shake the mixture for 1 or 2 min, allow it to stand for 15 min, add 15 mL of a 15:100 potassium iodide solution and 15 mL of water, swirl, let it stand for 1 min, and then titrate the liberated iodine with 0.1 *N* sodium thiosulfate, using starch TS as the indicator. Perform a *Residual Blank Titration* (see *General Provisions*) using water in place of sample, and make any necessary correction. The corrected volume is the number of milliliters of 0.1 *N* sodium thiosulfate required for the glycerin 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 0.1 *N* 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 milliliters of 0.1 *N* sodium thiosulfate consumed because of the glycerin in the sample. One milliliter of 0.1 *N* sodium thiosulfate is equivalent to 2.303 mg of glycerin and to 7.505 mg of tartaric acid.

Lead Determine as directed for *Method II* in the *Atomic Absorption Spectrophotometric Graphite Furnace Method* under *Lead Limit Test*, Appendix IIIB, using a 10-g sample.

Residue on Ignition Determine as directed under *Residue on Ignition*, Appendix IIC, igniting a 10-g sample.

Saponification Value Determine as directed under *Saponification Value*, Appendix VII, using about 2 g of sample, accurately weighed.

> **Note**: Add 5 to 10 mL of water to samples and blanks before saponification; otherwise, sufficient salts precipitate during saponification to cause serious bumping and spattering.

Packaging and Storage Store in well-closed containers.