STEARYL TARTRATE

Prepared at the 55th JECFA (2000) and published in FNP 52 Add 8 (2000), superseding specifications prepared at the 46th JECFA (1996) and published in FNP 52 Add 4 (1996). No ADI was established, but a level of use of 0 - 500 mg/kg in flour was accepted at the 9th JECFA (1965).

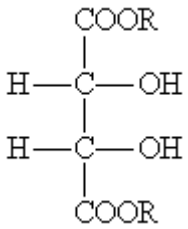
SYNONYMS Stearyl palmityl tartrate; INS No. 483

DEFINITION The product of the esterification of tartaric acid with commercial stearyl alcohol, which consists essentially of stearyl and palmityl alcohols; consists mainly of diester, with minor amounts of monoester and of unchanged starting materials

Chemical names: Main components are distearyl tartrate, dipalmityl tartrate and stearylpalmityl tartrate

Chemical formula: Distearyl tartrate: $C_{40}H_{78}O_6$
Dipalmityl tartrate: $C_{36}H_{70}O_6$
Stearylpalmityl tartrate: $C_{38}H_{74}O_6$

Structural formula



Where R = $(CH_2)_{17}CH_3$ or $(CH_2)_{15}CH_3$

Formula weight: Distearyl tartrate: 655.06
Dipalmityl tartrate: 598.95
Stearylpalmityl tartrate: 627.00

Assay: Not less than 90% of total ester content corresponding to an ester value within the range of 163 to 180

DESCRIPTION Cream-coloured unctuous substance

FUNCTIONAL USES Flour treatment agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4): Insoluble in water, insoluble in cold ethanol, soluble in hot ethanol

Melting range (Vol. 4): 67 - 77°
Hydroxyl value (Vol. 4): 200 to 220

Test for tartrate (Vol. 4): Passes test

PURITY

Sulfated ash (Vol. 4) — Not more than 0.5%
Test 2 g of the sample (Method I)

Total tartaric acid — Not less than 18% and not more than 35%
See description under TESTS

Unsaponifiable matter — Not less than 77% and not more than 83%
See description under TESTS

Acid value — Not more than 6
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 preparations may be based on the principles of method described in Volume 4, “Instrumental Methods”.

TESTS

PURITY TESTS

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 standard 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).

Procedure

Transfer 10.0 ml of Solution II prepared under Test Preparation into a 19 x 150-mm 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 solution at 520 nm with a suitable spectrophotometer or a photoelectric colorimeter equipped with a 520-nm filter. 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 to obtain the percentage of tartaric acid.

Unsaponifiable matter

This procedure determines those substances frequently found dissolved in fatty materials that cannot be saponified by alkali hydroxides but that are soluble in ordinary fat solvents.

Procedure

Weigh accurately 5.0 g of the sample into a 250-ml flask, add a solution of 2 g potassium hydroxide in 40 ml of alcohol, and boil gently under a reflux condenser for 1 h. Transfer the contents of the flask to a glass-stoppered extraction cylinder (approximately 30 cm in length, 3.5 cm in diameter, and graduated at 40, 80, and 130 ml). Wash the flask with sufficient alcohol to make a volume of 40 ml in the cylinder, and complete the transfer with warm and then cold water until the total volume is 80 ml. Finally, wash the flask with a few ml of petroleum ether, add the washings to the cylinder, cool the contents of the cylinder to room temperature, and add 50 ml of petroleum ether.

Insert the stopper, shake the cylinder vigorously for at least 1 min, and allow both layers to become clear. Siphon the upper layer as completely as possible without removing any of the lower layer, collecting the ether fraction in a 500-ml separator. Repeat the extraction and siphoning at least six times with 50-ml portions of petroleum ether, shaking vigorously each time. Wash the combined extracts, with vigorous shaking, with 25-ml portions of 10% alcohol until the wash water is neutral to phenolphthalein, and discard the washings. Transfer the ether extract to a tared beaker, and rinse the separator with 10 ml of ether, adding the rinsings to the beaker.

Evaporate the ether on a steam bath just to dryness, and dry the residue to constant weight, preferably at 75^{o} to 80^{o} under a vacuum of not more than 200 mm of Hg , or at 100^{o} at ambient pressure for 30 min. Cool in a desiccator, and weigh to obtain the uncorrected weight of unsaponifiable matter.

Determine the quantity of fatty acids in the residue as follows: Dissolve the residue in 50 ml of warm alcohol (containing phenolphthalein TS and previously neutralized with sodium hydroxide to a faint pink colour), and

titrate with 0.02N sodium hydroxide to the same colour. Each ml of 0.02N sodium hydroxide is equivalent to 5.659 mg of fatty acids, calculated as oleic acid. Subtract the calculated weight of fatty acids from the weight of the residue to obtain the corrected weight of unsaponifiable matter in the sample.

Acid value

Weigh accurately about 1 g of the sample and dissolve in about 20 ml of hot 95% ethanol, previously neutralized in the presence of 0.5 ml of phenolphthalein TS. Cool the solution, then neutralize by titration with 0.01N ethanolic potassium hydroxide.

$$\text{Acid value} = \frac{0.561 \times \text{titration value (ml)}}{\text{sample weight (g)}}$$

Retain the neutralized solution for the Assay.

METHOD OF ASSAY

Add exactly 50 ml of 0.1N ethanolic potassium hydroxide solution to the neutralized solution obtained above from the determination of the acid value and bring to a steady boil for 2 min. Cool the solution and titrate the excess of potassium hydroxide with B ml of 0.1N hydrochloric acid. Perform a blank determination (= A ml of 0.1N hydrochloric acid).

$$\text{Ester value} = \frac{5.61 \times (A - B)}{\text{weight of sample (g)}}$$