OCTENYL SUCCINIC ACID MODIFIED GUM ARABIC

New specifications prepared at the 71st JECFA (2009) and published in FAO JECFA Monographs 7 (2009). A temporary ADI "not specified" was established at the 71st JECFA (2009.

SYNONYMS Gum arabic hydrogen octenylbutandioate; Gum arabic hydrogen octenylsuccinate; OSA modified gum arabic; OSA modified gum acacia

DEFINITION Octenyl succinic acid modified gum arabic is produced by esterifying gum arabic (*Acacia seyal*), or gum arabic (*Acacia senegal*) in aqueous solution with not more than 3% of octenyl succinic acid anhydride. It is subsequently spray dried.

C.A.S. number 455885-22-0

DESCRIPTION Off-white to light tan, free flowing powder

FUNCTIONAL USES Emulsifier

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Freely soluble in water; insoluble in ethanol

Precipitate formation Add 0.2 ml of dilute lead subacetate TS to 10 ml of a cold 1:50 aqueous solution. A white, flocculent precipitate forms immediately.

pH (Vol. 4) 3.5 to 6.5 (5% solution)

Viscosity Not more than 30 cP (5% solution, 25°)
Add 95 ml of water to a beaker. Place a magnetic stir bar into the water and while stirring add 5 g of the sample. Stir on medium speed for 2 h. Measure viscosity on Brookfield LV viscometer, or equivalent, using spindle number 3 at 30 rpm (factor = 40).

PURITY

Degree of esterification Not more than 0.6%
See description under TESTS

Loss on drying (Vol.4) Not more than 15% (105°, 5h)

Total ash (Vol.4) 10% (530°)

Acid-insoluble ash (Vol.4) Not more than 0.5%

Water-insoluble matter (Vol. 4) Not more than 1.0%

Starch or dextrin Boil a 1 in 50 aqueous solution of the sample, add about 0.1 ml iodine TS. No bluish or reddish color should be produced.

Tannin-bearing gums

To 10 ml of a 1 in 50 aqueous solution of the sample add about 0.1 ml ferric chloride TS. No blackish coloration or blackish precipitate should be formed.

Residual octenyl succinic acid

Not more than 0.3%
See description under TESTS

Microbiological criteria (Vol. 4)

Salmonella species: absent in 25 g
Escherichia coli: absent in 1 g

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 principles of methods described in Volume 4 (under “General Methods, Metallic Impurities”).

TESTS

PURITY TESTS

Degree of esterification

Principle: The degree of esterification is determined by the amount of alkali consumed after acidification and thorough washing of the sample.

Procedure

Weigh 5.0 g of sample in a 150-ml beaker and wet it with a few ml of isopropanol. Add, by pipette 25 ml of 2.5 N hydrochloric acid in isopropanol and stir for 30 min on a magnetic stir plate. Add 100 ml of 90% isopropanol in water from a graduated cylinder and stir for 10 min. Filter the sample through a Buchner funnel and wash the filter cake with 90% isopropanol in water until the filtrate is negative for chloride ions checked by 0.1 N silver nitrate. Transfer the filter cake to a 600-ml beaker, rinse the Buchner funnel and bring to a 300-ml volume with distilled water. Place for 10 min in a boiling water bath while stirring and titrate while hot with 0.1 N sodium hydroxide using phenolphthalein TS as an indicator.

Calculation

$$\text{Degree of esterification} = \frac{0.162 \times A}{1 - 0.210 \times A}$$

where
A is milliequivalents of sodium hydroxide required per 1g of the sample.

Residual octenyl succinic acid

Principle: HPLC method on 2-bromacetophenone-derivatised methanolic extract of the sample.

Extraction and Preparation of Sample Solution

Extract about 500 mg of the sample, accurately weighed, with 15 ml of methanol overnight under constant shaking. Filter the extraction mixture, wash the precipitate on the filter three times with 7 ml portions of methanol and combine all filtrates (about 80% of the residuals are extracted by this procedure). Add 1 ml of 0.16 N KOH in methanol to the extract. Dry the extract using a flash evaporator

at 30° and dissolve the residue in 2 ml of methanol. Take 0.5 ml of residue solution to the reaction vial and add 0.5 ml of derivative reagent [2.8 g of 2-p-dibromoacetophenone and 0.28 g of 1,4,7,10,13,16-hexaoxacyclooctadecane (18-Crown-6) in 50 ml CH_3CN]. Add 2 ml CH_3CN to the reaction vial, cap it and heat for 30 min at 80°. Cool the reaction solution to room temperature and use it within 24 h.

Liquid Chromatography Analysis

- Column: Micro-Bondapack C18 (Waters) or equivalent, 20°
- Mobile Phase: Gradient elution of 70% to 80% methanol in water in 5 min
- Flow rate: 1.5 ml/min
- Detector: UV at 254 nm, attenuation 0.16 AUFS
- Injection volume: 5 µl

Preparation of Standard Curve

Prepare a 0.5 M solution of octenyl succinic acid anhydride (available from Milliken Chemical) (Solution A). With a syringe take 0.25 ml of Solution A and transfer into a 25-ml volumetric flask. Dilute to the mark with methanol (Solution B). Prepare three standards by transferring 0.5, 1 and 2 ml of Solution B into three 50-ml round bottom flasks and adding to each 1 ml of 0.16 N KOH in methanol. Dry each solution using a flash evaporator at 30° and dissolve the residue in 2.0 ml of methanol (Solution C1, C2 and C3). Place 0.5 ml of the residue solution in the reaction vial and add 0.5 ml derivative reagent [2.8 g of 2-p-dibromoacetophenone and 0.28 g of 1,4,7,10,13,16-hexaoxacyclooctadecane (18-Crown-6) in 50 ml of CH_3CN]. Add 2 ml of CH_3CN to the reaction vials, cap them and heat for 30 min at 80°. Cool to room temperature and inject 5 µl into the Liquid Chromatograph (the derivative should be used immediately). The amount of residuals in each of the 5-µl injections are the following:
for Solution C1 0.2375 µg
for Solution C2 0.4750 µg
for Solution C3 0.9500 µg
Plot peak height from Liquid Chromatograph Chart versus µg of residuals per 5 ml of solution.

Calculation

Using the peak height of the unknown sample from the Liquid Chromatograph Chart, determine the level of residuals (calculated as octenyl succinic acid) in the injected volume from the standard curve.

$$\% \text{ Residual octenyl succinic acid } = \frac{300 \times V}{W}$$

where
V is the value from the graph; and
W is the weight of the sample (mg).

NOTE: The formula is corrected to 100% recovery by dividing with 0.80, so that 240/0.80 = 300.