

OCTENYL SUCCINIC ACID MODIFIED GUM ARABIC

Prepared at the 74th JECFA (2011) and published in the FAO Monographs 11 (2011), superseding specifications prepared at the 71st JECFA (2009), 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; INS No. 423
DEFINITION	Octenyl succinic acid modified gum arabic is produced by esterifying gum arabic (<i>Acacia seyal</i>), or gum arabic (<i>Acacia senegal</i>) 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	
<u>Solubility</u> (Vol. 4)	Freely soluble in water; insoluble in ethanol
<u>Precipitate formation</u>	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.
<u>pH</u> (Vol. 4)	3.5 to 6.5 (5% solution)
<u>Viscosity</u>	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	
<u>Degree of esterification</u>	Not more than 0.6% See description under TESTS
<u>Loss on drying</u> (Vol.4)	Not more than 15% (105°, 5h)
<u>Total ash</u> (Vol.4)	Not more than 10% (530°)
<u>Acid-insoluble ash</u> (Vol.4)	Not more than 0.5%
<u>Water-insoluble matter</u> (Vol. 4)	Not more than 1.0%
<u>Starch or dextrin</u>	Boil a 1 in 50 aqueous solution of the sample, add about 0.1 ml iodine TS. No bluish or reddish colour should be produced.

<u>Tannin-bearing gums</u>	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.
<u>Residual octenyl succinic acid</u>	Not more than 0.3% See description under TESTS
<u>Microbiological criteria</u> (Vol. 4)	<i>Salmonella</i> species: absent in 25 g <i>Escherichia coli</i> : absent in 1 g
<u>Lead</u> (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 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 (to nearest 0.1 mg), of the sample in a 150-ml beaker and wet it with a few ml of isopropanol. Pipette 25.0 ml of 2.5 N hydrochloric acid in isopropanol and stir the mixture for 30 min with a magnetic stirrer. Using a graduated measuring cylinder, add 100 ml of 90% isopropanol in water and stir for another 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 (check using 0.1 N silver nitrate). Transfer the filtrate to a 600-ml beaker, rinse the Buchner flask and bring to a 300-ml volume with distilled water. Place the beaker on top of a boiling water bath for 10 min, while stirring. While hot, titrate 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 Determine by HPLC on the 2-bromoacetophenone-derivatised methanolic extract of the sample.

Extraction and Preparation of Sample Solution

Accurately weigh 500 mg (to nearest 0.1 mg) of the sample in a 25 ml Erlenmeyer flask, add 15 ml of methanol, stopper the flask and shake it on a shaker overnight. Filter the extract using a filter paper, wash the residue, three times with 7 ml portions of methanol and combine the filtrate (about 80% of the OSA residues is extracted by this procedure). Add 1 ml of 0.16 N KOH in methanol to the combined filtrate. Dry the extract using a flash evaporator at 30°

and dissolve the residue in 2 ml of methanol. Pipette 0.5 ml of this solution into a reaction vial, add 0.5 ml of derivatisation 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 CH₃CN]. Add 2 ml CH₃CN to the reaction vial, cap the vial and heat at 80° for 30 min. Allow the vial to reach room temperature and analyse the reaction product by HPLC within 24 h.

HPLC Conditions:

Column: μ-Bondapack C18 or equivalent

Mobile Phase: Methanol and Water with gradient elution: 70% to 80% of methanol in water in 5 min

Flow rate: 1.5 ml/min

Detector: UV at 254 nm

Injection volume: 5 μl

Preparation of Standard Curve

Prepare a 105.14 mg/ml solution of octenyl succinic acid anhydride (available from Milliken Chemicals) in methanol (Solution A). Using a syringe draw 0.25 ml of Solution A, transfer into a 25-ml volumetric flask and dilute to mark with methanol (Solution B).

Prepare three working standards (Solution C1, C2 and C3) by transferring 0.5, 1 and 2 ml each of Solution B into three 50-ml round bottom flasks, add 1 ml of 0.16 N KOH in methanol to each flask, dry the solution using a flash evaporator at 30° and dissolve the residue in 2.0 ml of methanol. To 0.5 ml each of these solutions in reaction vials, add 0.5 ml each of derivatisation 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 CH₃CN]. Add 2 ml of CH₃CN to each vial, cap the vials and heat for 30 min at 80°. Allow the vials to reach room temperature and analyze by HPLC immediately. The amount of octenyl succinic acid in each 5-μl injection is as follows:

Solution C1: 0.2375 μg

Solution C2: 0.4750 μg

Solution C3: 0.9500 μg

Construct the standard curve using peak height against the amount of standard in the injected volume.

Inject 5-μl of prepared sample solution and read the amount of octenyl succinic acid in the injection from the standard curve.

Calculation

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

where

V is the amount of OSA in the injected volume; and

W is the weight of the sample (mg).

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