GLYCEROL ESTER OF WOOD ROSIN

Prepared at the 77th JECFA (2013) and published in FAO JECFA Monographs 14 (2013), superseding tentative specifications prepared at the 74th JECFA (2011), published in FAO JECFA Monographs 11 (2011). An ADI of 0-25 mg/kg bw for glycerol ester of wood rosin was established at the 77th JECFA (2013).

SYNONYMS INS No. 445(iii)

DEFINITION Glycerol ester of wood rosin is a complex mixture of glycerol di- and tri-esters of resin acids from wood rosin, with a residual fraction of glycerol monoesters. Besides these esters, neutrals (non-acidic saponifiable and unsaponifiable substances) and residual free resin acids are present. It is obtained by the solvent extraction of aged pine stumps (*Pinus palustris* (longleaf) and *Pinus elliottii* (slash) species) followed by a liquid-liquid solvent refining process. The refined wood rosin composed of approximately 90% resin acids and approximately 10% neutrals. The resin acid fraction is a complex mixture of isomeric diterpenoid monocarboxylic acids having the typical empirical formula $C_{20}H_{30}O_2$, of which the main component is abietic acid. The substance is purified by steam stripping or by countercurrent steam distillation.

These specifications do not cover substances derived from gum rosin, an exudate of living pine trees, and substances derived from tall oil rosin, a by-product of kraft (paper) pulp processing.

C.A.S. number 8050-30-4

DESCRIPTION Hard, yellow to pale amber-coloured solid

FUNCTIONAL USES Emulsifier, density adjustment agent (flavouring oils in beverages), stabilizer, chewing gum base component

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Insoluble in water, soluble in acetone

Infrared absorption (Vol. 4) The infrared spectrum of a thin film of the sample (potassium bromide disc) corresponds with the typical infrared spectrum below

Sulfur test Negative
Weigh 40-50 mg of sample into a test tube and add 1- 2 drops of a 20% (w/v) solution of sodium formate. Place a strip of lead acetate test paper over the mouth of the test tube. Heat the tube until fumes are formed that contact the test paper. Continue heating for 2-5 min. The formation of a black spot of lead sulfide indicates the presence of sulfur-containing compounds. (Detection Limit: 50 mg/kg sulfur)

Gas chromatography of resin alcohols and glycerol	Passes test See description under TESTS

PURITY

Specific gravity (Vol. 4)	d (20, 25): Not less than 0.935 (50% solution in d-limonene)
Ring and ball softening point (Vol. 4)	Not less than 82° (see "Specific Methods, Glycerol Esters of Rosins")
Acid value (Vol. 4)	Between 3 and 9 (see "Specific Methods, Fats, Oils, and Hydrocarbons")
Lead (Vol. 4)	Not more than 1 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 the principles of the method described in Volume 4 (under "General Methods, Metallic Impurities").

TESTS

IDENTIFICATION TESTS

Gas chromatography of resin alcohols and glycerol

The complex ester groups in the glycerol esters of wood rosin are reduced by reaction with a metal hydride (sodium bis(2-methoxy-ethoxy) aluminium dihydride) in toluene solution to form a mixture of resin alcohols and glycerol. Excess reagent is then hydrolyzed with aqueous acid forming two phases. Gas chromatography of the toluene phase produces a characteristic chromatogram of the resin alcohols that abietyl and dehydroabietyl alcohols are predominates. Confirm the presence of two predominate peaks of both resin alcohols with the typical gas chromatogram below. Chromatography of the neutralized aqueous phase on a different column verifies the presence of glycerol.

Apparatus

Gas Chromatograph equipped with a flame ionization detector.
Recorder: 0 to 1 V
Analytical Balance: capable of weighing to the nearest 0.001 g
Centrifuge: table top, capable of achieving 3200 rpm

Reagents

Toluene, reagent grade.
Sodium Vitride™ Reagent [Sodium bis(2-methoxyethoxy) aluminium dihydride, pract. ~ 70% in toluene (~ 3.5 mol/l)] (Fluka Chemical Corp., Hauppage, NY, USA, or equivalent). Pipet 10.0 ml into a 100 ml volumetric flask. Dilute to volume with toluene and mix thoroughly.
Hydrolysis Solution: Slowly add 50 ml of concentrated sulfuric acid, reagent grade, to 200 ml distilled water while stirring in an ice bath. Cool to room temperature.
Phenolphthalein Solution: 1% in ethanol.
Sodium Hydroxide Solution: Dissolve 16 g of reagent grade NaOH in 70-80 ml of distilled water and cool to room temperature. Dilute to 100 ml with distilled water and mix thoroughly. Store in a polyethylene bottle.
1,4-Butanediol: 99+%

Glycerol: 99+%
Internal Standard Solution: weigh 0.1 g of 1,4-butanediol into a 100 ml volumetric flask. Dilute to volume with distilled water and mix thoroughly.
Glycerol Solution: weigh 0.1 g of 1,4-butanediol and 0.1 g glycerol into a 100 ml volumetric flask. Dilute to volume with distilled water and mix thoroughly.

Procedure I (Resin alcohols)
Weigh 250-300 mg sample into a 25 ml Erlenmeyer flask containing a stirring bar (Teflon coated, 1 inch). Pipet 5.0 ml toluene into the flask and stir magnetically until sample is dissolved. Pipet 5.0 ml of Sodium Vitride™ Reagent into the flask, cap, and stir for 30 min. Uncap and, while stirring, pipet 3.0 ml of Hydrolysis Solution into the flask. Continue stirring for 3 min. Transfer contents of flask to centrifuge tube (15 mL), stopper, and shake vigorously. Vent and centrifuge at 2800-3200 rpm for 5 min. Inject 0.5 µl of the upper layer into the gas chromatograph operating under the specified conditions and record the chromatogram. Compare with the chromatograms shown below to verify the approximate retention order of the resin alcohols.

Chromatography conditions
Column: DB-1 methyl silicone (bonded and crosslinked), 15 m, wide-bore capillary (0.53 mm i.d.), film thickness 1.5 µm, temperature range 60º to 300/320º (e.g., J & W Scientific Inc,. Cat. No. 125-1012). A direct flash vaporization injection port liner is recommended.
Flow Rates
Carrier Gas (He): 30 ml/min at 63 psi
Hydrogen: 30 ml/min
Air: 240 ml/min
Temperatures
Column: Isothermal, 190º
Inlet: 250º
Detector: 250º

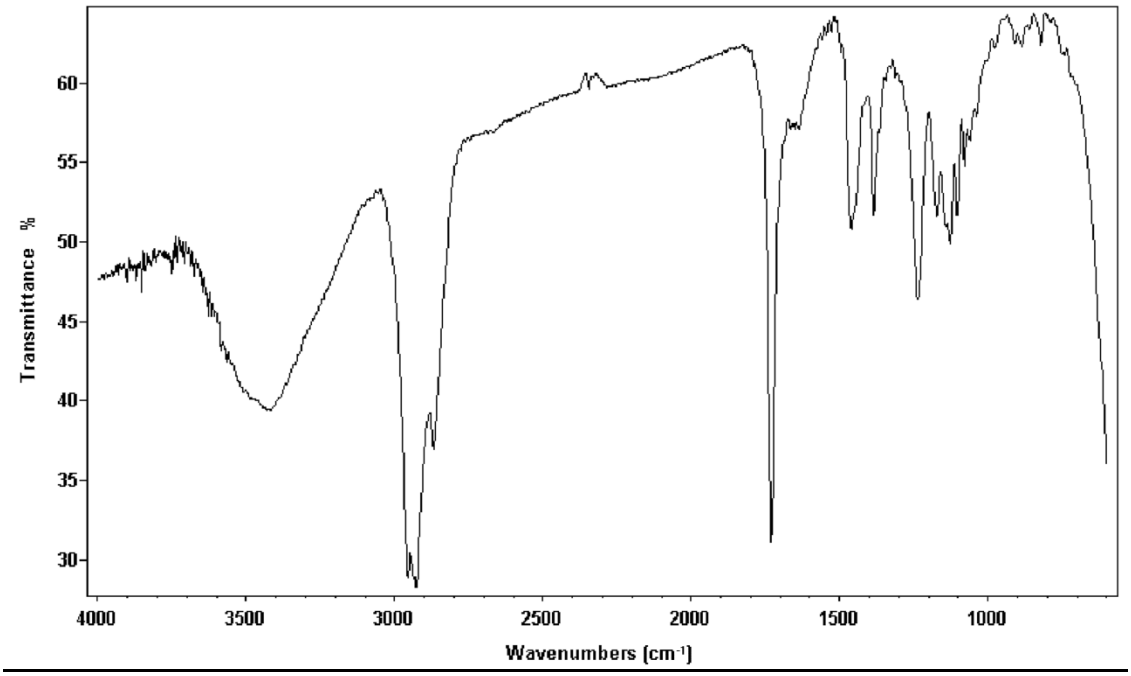
Procedure II (Glycerol)
Using a pipet or hypodermic syringe, remove the toluene layer and part of the aqueous layer leaving approximately 2 ml of the aqueous layer in the centrifuge tube. Add 1 drop of phenolphthalein solution and neutralize with the Sodium Hydroxide Solution. Aluminium salts will precipitate. Pipet 5 ml of the Internal Standard Solution into the tube, dilute to 15 ml with distilled water, stopper, shake, and then centrifuge at 2800-3200 rpm for 5 min. Inject 1 µl of the clear supernatant liquid into the gas chromatograph operating under the specified conditions and record the chromatogram.
Inject 1 µl of the Glycerol Solution and record the chromatogram. Measure the retention times of any observed peaks relative to 1,4-butanediol. Compare retention times to that of glycerol.

Chromatography conditions
Column: DB-WAX polyethyleneglycol (bonded and cross-linked), 15 m, wide bore capillary (0.53 mm i.d.), film thickness 1.0 µm, temperature range 20º to 230º (e.g., J & W Scientific Inc., Cat, No. 125-7012).
Flow Rates
Carrier Gas (He): 30 ml/min at 60 psi
Hydrogen: 30 ml/min
Air: 240 ml/min

Temperatures

Column: Programmed, 120 to 200º at 6º /min
Inlet: 250º
Detector: 250º

Infrared spectrum



NOTE: The IR spectrum for glycerol ester of wood rosin is referenced from the Food Chemicals Codex, 8th Edition, 2012, p. 506. Reprinted with permission from the US Pharmacopeial Convention, 12601 Twinbrook Parkway, Rockville, MD USA 20852.

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