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GLYCEROL ESTER OF WOOD ROSIN

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GLYCEROL ESTER OF WOOD ROSIN

Prepared at the 86th JECFA and published in FAO JECFA Monographs 22 (2018), superseding specifications prepared at the 77th JECFA (2013) and published in FAO JECFA Monographs 14 (2013). 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 (GEWR) is a complex mixture of glycerol di- and tri- esters of resin acids from wood rosin, with a residual fraction of glycerol monoesters. In addition, neutrals (non-acidic saponifiable and unsaponifiable substances) and residual free resin acids are present. Wood rosin is obtained by the solvent extraction of aged pine stumps, followed by a liquid-liquid solvent refining process. Refined wood rosin is 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 components are dehydroabietic and abietic acids. GEWR is produced by esterifying the resin acids with food grade glycerol. The product is then purified by steam stripping or by direct 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, plasticizer (in chewing gum bases).

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 acids 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 method 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 acids

The ester groups in the glycerol esters of wood rosin are reduced with a metal hydride to form a mixture of corresponding resin alcohols and glycerol which are analyzed by gas chromatography (Vol. 4). The characteristic chromatogram shows predominant peaks for abietic and dehydroabietic alcohols.

Apparatus

- Gas Chromatograph equipped with a flame ionization detector.
- Centrifuge: table top, capable of achieving 3200 rpm

Standards and reagents

- Internal Standard (1,4-Butanediol: >99%
- Toluene
- Sodium Vitride Reagent [(Sodium bis(2-methoxyethoxy) aluminium dihydride], 70% in toluene:(~ 3.5 mol/l)

Sodium Vitride solution:

Pipet 10.0 ml of sodium vitride reagent 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.

Procedure

Sample preparation

Weigh 250-300 mg sample into a 25 ml Erlenmeyer flask containing a Teflon coated stirrer bar. Pipet 5.0 ml toluene into the flask and stir until sample is dissolved. Pipet 5.0 ml of sodium vitiride solution into the flask, stopper the flask and stir for 30 min. 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 toluene layer into the gas chromatograph operating under the following conditions and record the chromatogram. Compare with the chromatogram shown below to verify the approximate retention order of the resin alcohols.

Chromatographic conditions

- Column: DB-1 methyl silicone (bonded and crosslinked) wide-bore capillary (15 m x 0.53 mm i.d.,1.5 µm).
- Injector: Flash vaporization injector
- Flow rates: Carrier Gas (He): 30 ml/min at 63 psi, Hydrogen: 30 ml/min and
- Air: 240 ml/min
- Temperatures: Column: Isothermal, 190º; Injector: 250º , and Detector: 250º

Gas chromatography of glycerol

Standards and reagents

- Glycerol: >99%
- 1,4-Butanediol (Internal standard): >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

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.

Procedure

Sample preparation

Proceed as in the sample preparation for the analysis of resin acids until the centrifugation step. Using a pipet or 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 to the remaining aqueous layer in the centrifuge, 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 following 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 standard.

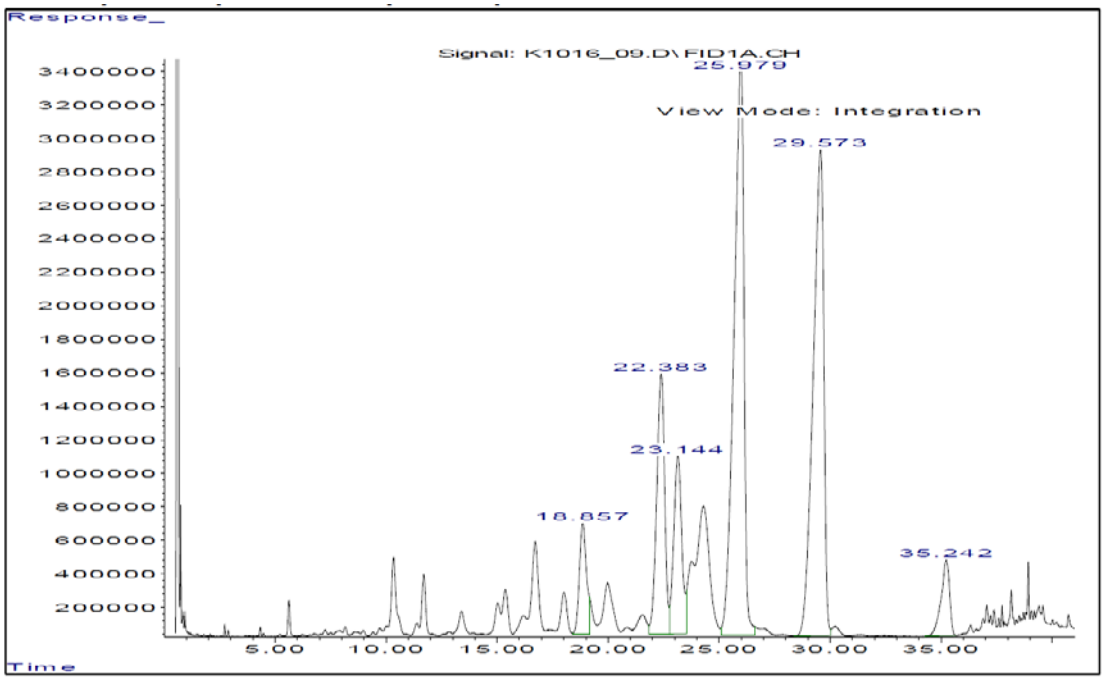
Chromatographic conditions

- Column: DB-WAX polyethyleneglycol (bonded and cross-linked), wide bore capillary (15 m x 0.53 mm i.d., 1.0 µm)
- Flow rates: Carrier Gas (He): 30 ml/min at 60 psi, Hydrogen: 30 ml/min and
- Air: 240 ml/min
- Temperatures: Column: Programmed, 120 to 200º at 6º /min; Injector: 250º,
- and Detector: 250º

Gas chromatography of resin acids in GEWR (determined as alcohols)

Typical GC-FID chromatogram of a GEWR sample. Retention times correspond to pimaric (18.8 min), isopimaric (22.4 min), palustric (23.1 min), dehydroabietic (25.9 min), abietic (29.6 min), and neoabietic (35.2 min) alcohols. This is a product derived from a plant-based source which can demonstrate significant variability and relative intensities.

Gas chromatogram



FTIR Spectrum of Glycerol esters of wood rosin