GLYCEROL ESTER OF WOOD ROSIN

Chemical and Technical Assessment

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1. Summary

This Chemical and Technical Assessment summarizes data and information on Glycerol ester of wood rosin (GEWR) submitted to JECFA by the sponsor in December 2012. GEWR is used as an emulsifier/density adjustment agent for flavouring oils in beverages and as a plasticizing agent in chewing gum base.

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. Besides these esters, GEWR also contains neutrals (non-acidic saponifiable and unsaponifiable substances) and residual free resin acids. Wood rosin 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 resin acid fraction of the wood rosin is a complex mixture of isomeric diterpenoid monocarboxylic acids having the typical empirical formula C20H30O2, of which the main component is abietic acid. The refined wood rosin is esterified with a food-grade glycerol to produce GEWR.

GEWR was previously considered by the Committee at its eighteenth, twentieth, thirty-third, thirty-seventh, forty-fourth, forty-sixth, seventy-first and seventy-fourth meetings. At its forty-sixth meeting (1996), an ADI of 0–25 mg/kg bw for GEWR was established based on multiple toxicological studies.[1] At its seventy-first meeting (2009), this ADI was extended to a group ADI of 0–25 mg/kg bw covering both glycerol ester of gum rosin (GEGR) and GEWR.[2] At the seventy-fourth meeting (2011), the group ADI for GEGR and GEWR was withdrawn, and a temporary group ADI for GEGR and GEWR of 0-12.5 mg/kg bw was eatablished pending the availability of additional compositional information on GEWR as well as the full reports of the 90-day toxicity studies on GEGR.[3] At the seventy-seventh meeting (2013), the temporary group ADI was withdrawn as the requested toxicity studies on GEGR were not submitted, and an ADI of 0-25 mg/kg bw for GEWR was re-established based on the submitted compositional information.

2. Description

Rosin is a natural product derived from pine trees and consists of a complex mixture of mutually soluble organic compounds. Rosins are produced commercially by one of the following three methods: (1) solvent extraction of aged and ground pine stumps (wood rosin), (2) extraction of crude tall oil as a byproduct of the Kraft pulping process for making paper followed by acidification and fractional distillation (tall oil rosin) and, (3) tapping the living tree to collect oleoresin followed by distillation to yield turpentine and a resinous substance (gum rosin). Because, gum, tall oil and wood rosins are obtained from pine trees, they are similar in composition. All rosins consist of free organic acids, called resin acids, as well as non-acidic saponifiable and unsaponifiable substances (neutrals). The composition can vary with the source of the pine tree and processing conditions. The glycerol esters of rosins are formed by the esterification of the source rosin (gum, tall oil or wood) with glycerol under inert atmosphere and at high temperatures resulting in either glycerol ester of gum rosin (GEGR), glycerol ester of tall oil rosin (GETOR) or glycerol ester of wood rosin (GEWR).

GEWR is a hard, yellow to pale amber-coloured solid material. GEWR is soluble in acetone but is insoluble in water. The Chemical Abstracts Services (CAS) Registry number assigned to GEWR is 8050-30-4.

3. Manufacturing

3.1 Raw material

Aged stumps of *P. palustris* and *P. elliottii* are used as the source of wood rosin. *P. palustris* and *P. elliottii* have been jointly used as the feedstock for wood rosin production since the beginning of the industry in the early 1900's.4-6 *P. palustris* and *P. elliottii* are the only Pinus species that produce stumpwood with the unique combination of decay-resistance, rosin yield, and highly refinable rosin, that are necessary for sustainable wood rosin production. These two species occur naturally in overlapping regions of the southeastern United States.[7-8] The availability of two suitable species in the same harvesting area ensures an abundant and readily accessible feedstock. A mixture of the two species is a fundamental characteristic of wood rosin production.

3.2 Production of wood rosin

Wood rosin is obtained by the solvent extraction of aged pine stumps followed by a liquid-liquid solvent refining process to separate undesirable substances and further standardize the final raw material to help assure consistency and compositional purity. The refined wood rosin is composed of approximately 90% resin acids and approximately 10% neutrals.

3.3 Production of GEWR

Refined wood rosin is pumped into a batch-type reactor, and esterified with food-grade glycerol. The reaction is allowed to proceed until samples of esterified material meet the desired product specifications (e.g., acid number, color, and softening point). The GEWR is then purified with steam stripping or by direct countercurrent steam distillation. After cooling, the GEWR is subjected to filtration, and the hot resin is fed into a pastilles-making unit and cooled to room temperature. The purified GEWR pastilles are freshly packed into plastic bags, which are immediately sealed to protect against ageing and oxidation.

4. Chemical characterization

4.1 Composition

The wood rosin starting material is composed of resin acids and neutrals. The sponsor reported that the resin acids content was quantified using an industry standard potentiometric titration method and averaged 88.0% (range 86.9-89.3%). The expected ranges based on the normal variation in the acid number of the refined wood rosin are 83-91% for resin acids. The submitted results are well within the expected ranges.

The resin-acid fraction of the wood rosin is a complex mixture of isomeric diterpenoid monocarboxylic acids having the empirical formula of C20H30O2, of which the primary component is abietic acid. The resin acid fraction also includes dehydroabietic acid (another main component), as well as communic acid, isopimaric acid, pimaric acid, sandoracopimaric acid, palustric acid, neoabietic acid and other unclassified acids. The structures of the principal resin acids are shown in Figure 1.

GEWR is a complex mixture of glycerol di- and tri- esters of resin acids from wood rosin, with a residual fraction of glycerol monoester. A representative molecular structure of a glycerol triester of resin acid, glycerol triester of abietic acid, is shown in Figure 2. The sponsor submitted information on the composition of GEWR based on the analysis of five production samples of GEWR. GEWR

samples were fractionated by solid phase extraction (SPE) then analyzed by high temperature/high resolution GC analysis using a capillary column specified for triglycerides.[9] GEWR was composed mainly of glycerol di- and tri- glycerol esters (78.3-83.9%) and neutrals (11.4-17.6%), with lesser amounts of glycerol monoesters (1.5-3.2%) and free resin acids (2.3-2.8%). The neutrals fraction of GEWR can be organized into three classes: (1) monoterpene neutrals, (2) diterpene neutrals, and (3) high molecular weight neutrals (monoterpene alcohol esters of rosin, fatty alcohol ester of rosin and diterpene alcohol esters of rosin).

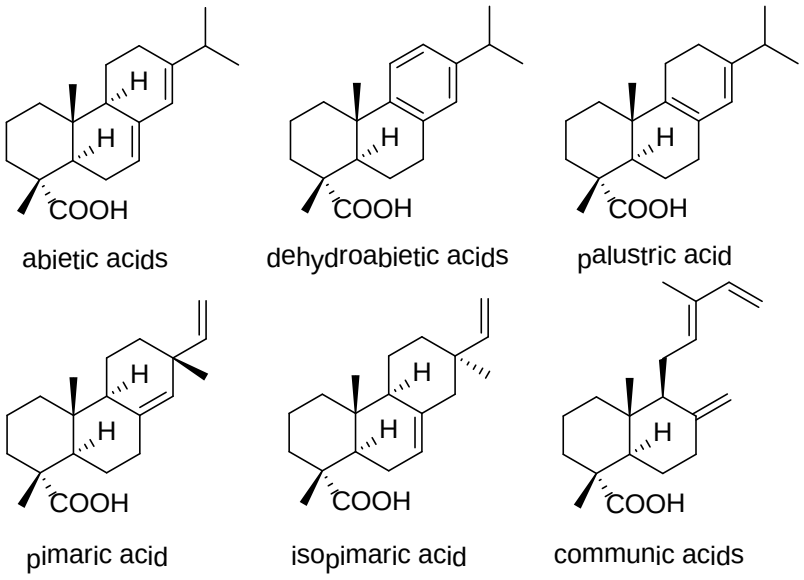

Figure 1 - Principal resin acids structures

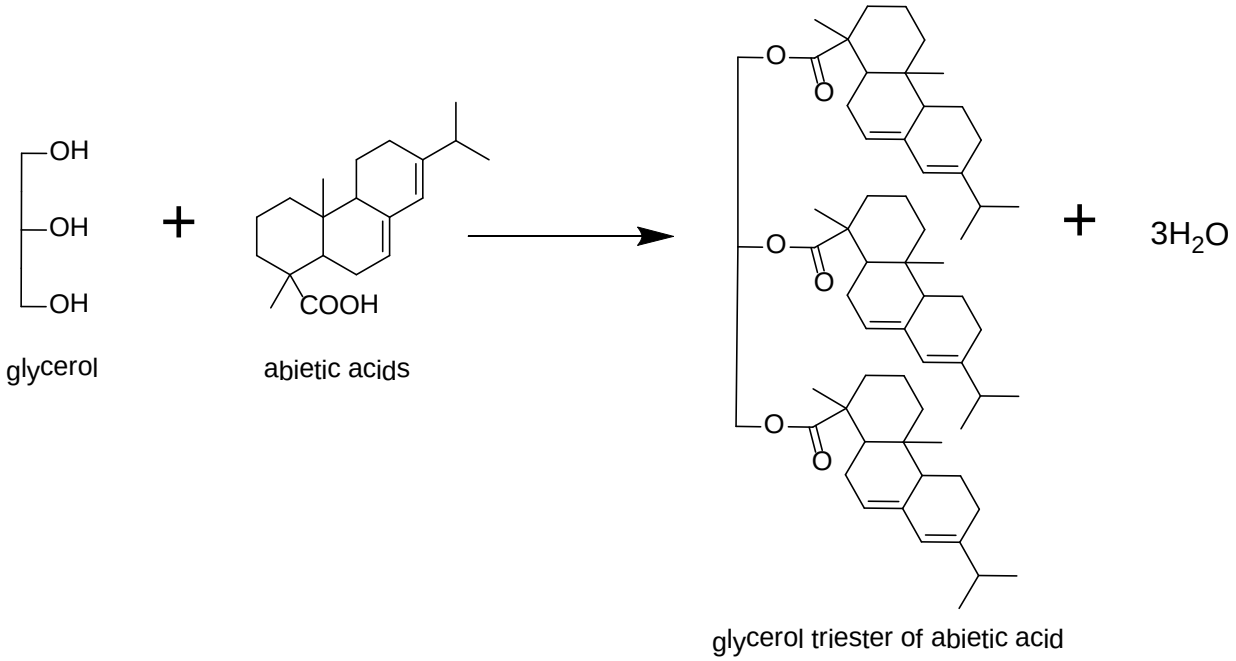

Figure 2 - Glycerol triester of abietic acid

4.2 Identification

GEWR is soluble in acetone and is insoluble in water. The presence of major resin acids, abietic and dehydroabietic acids, and glycerol which compose glycerol esters of resin acids in GEWR is confirmed by GC analysis. For the differentiation of GETOR, a sulfur test is also established.

4.3 Impurities (including degradation products)

The specifications monograph contains a maximum limit for lead of not more than 1 mg/kg.

4.4 Analytical methods

Analytical methods used to support the specifications for GEWR are based on general tests in the FAO Combined Compendium of Specifications (FAO JECFA Monographs 1, vol. 4, 2006) for identy and purity (e.g., solubility, IR spectrum, absence of sulfur, and lead). Methods for determining acid number and ring and ball softening point are also included. GC analysis of glycerol and resin alcohols, abietic alcohol and dehydroabietic alcohol, generated by reductive reaction of GEWR was used for identification of glycerol and major resin acids, abietic acid and dehydroabietic acid, which compose glycerol esters of resin acids in GEWR.[10]

5. Functional use

GEWR is used as an emulsifier/density adjustment agent for flavouring oils in beverages. Beverage emulsions consist of a continuous aqueous phase in which a discontinuous oil phase is dispersed in the form of microscopic droplets. They are characterized as oil-in-water emulsions, typically having an opaque or cloudy appearance. This appearance is typically associated by the consumer with that of natural fruit juice. Beverage emulsions are thermodynamically unstable two-phase systems that have a tendency to separate into two immiscible liquids. One of the approaches used to control the beverage emulsion stability is to minimize the density contrast between the oil phase and the aqueous phase with the use of "weighting" agents. Weighting agents, such as GEWR, are typically lipophilic components that serve to increase the density of the oil phase.

GEWR is also used in chewing gum base as a plasticizing agent.

6. Reactions and fate in foods

When used as prescribed, GEWR is essentially chemically and biologically inert and no reaction in foods and no effect on other nutrients are expected. As the carboxylic group of the resin acids in the rosin is attached to a sterically hindered tertiary carbon, esterification of this hindered carboxyl group generally requires higher temperatures and generally more drastic conditions than for other carboxylic acids. The steric effects are also responsible for the resistance of the resin-acid ester linkage to cleavage by water, acid, and alkali and explains the stability of the glycerol ester in the gastrointestinal tract with only a minor fraction undergoing partial hydrolysis.

7. References

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