AOAC SMPR® 2020.016

Standard Method Performance Requirements (SMPRs®) for Characterization and Quantitation of Component Fractions from Glycerol Esters of Wood Rosin (GEWR)

Intended Use: Global Reference Method

1 Purpose

AOAC SMPRs describe the minimum recommended performance characteristics to be used during the evaluation of a method. The evaluation may be an on-site verification, a single-laboratory validation, or a multi-site collaborative study. SMPRs are drafted by AOAC working groups composed of representatives from industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. Approved by AOAC, AOAC SMPRs may be used for method development, are used by AOAC expert review panels in their evaluation of validation study data for method being considered for *Performance Tested Methods*℠ or AOAC *Official Methods of Analysis*℠, and can be used as acceptance criteria for verification at user laboratories.

2 Applicability

Characterization and quantitation of component fractions of glycerol esters of wood rosin (GEWR) from *Pinus halepensis*, *Pinus brutia*, *Pinus palustris*, and *Pinus elliottii* (and potentially other pine species). The components of interest include free resin acids, glycerol monoesters, and neutrals. Free resin acids include all unreacted organic acids present in the GEWR product; glycerol monoesters include all monoesters present in the GEWR product; and neutrals include all nonsaponifiable and nonacidic saponifiable components, mainly monoterpenes, diterpenes, and high-molecular-weight neutrals, in the GEWR product.

3 Analytical Technique

GPC or other analytical technique that measures the components of interest and meets the following method performance requirements is acceptable.

4 Definitions

Limit of quantitation (LOQ).—Lowest level of analyte in a test sample that can be quantified at a specified level of precision.

Recovery.—Fraction or percentage of analyte that is measured when the test sample is analyzed using the entire method.

Repeatability.—Variation arising when all efforts are made to keep conditions constant by using the same instrument and operator (in the same laboratory) and repeating during a short time period. Expressed as repeatability standard deviation (SD_r); or % repeatability relative standard deviation (%RSD_r).

Table 1. Method performance for component fractions in GEWR

	Free resin acids	Monoesters	Neutrals
LOQ, %	0.01[a]	0.5[a]	0.01[a]
Analytical range, %	0.01–10[a]	0.5%–10[a]	0.01–20[a]
Recovery/ accuracy, %	90–107	95–105	90–107
RSD_r, %	5.3	3	5.3
RSD_R, %	8	4.4	8

[a] Percent refers to the mass fraction of component in the GEWR product, e.g., 1% = 0.01 g/g.

Reproducibility.—Variation arising when identical test materials are analyzed in different laboratories by different operators on different instruments. The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as reproducibility standard deviation (SD_R); or % reproducibility relative standard deviation (%RSD_R).

5 Method Performance Requirements

See Table 1.

6 System Suitability Tests and/or Analytical Quality Control

(a) Suitable methods will include blanks and appropriate check standards.

(b) Retention time should be stable to ±1%.

(c) Method developer should provide proof of identity of peaks.

7 Validation Guidance

Appendix F: *Guidelines for Standard Method Performance Requirements* (2019) *Official Methods of Analysis of AOAC INTERNATIONAL,* 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www. eoma.aoac.org/app_f.pdf)

Validation studies should include at least four GEWR products produced from different pine species to include, but not be limited to, *P. palustris*, *P. elliottii*, *P. halpensis*, and *P. brutia*.

8 Reference Materials

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements* (2019) *Official Methods of Analysis of AOAC INTERNATIONAL,* 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www. eoma.aoac.org/app_f.pdf)

9 Maximum Time-to-Results

None.

Developed by AOAC Working Group on Glycerol Esters of Wood Rosins (GEWR). Approved by AOAC GEWR stakeholders on December 15, 2020.

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