

1 Version 2, 08/24/2020

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

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6 Intended Use: Global reference method

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8 **1. Purpose**

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

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21 **2. Applicability**

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23 Characterization and quantitation of component fractions of glycerol esters of wood
24 rosin (GEWR) from *Pinus halepensis*, *Pinus brutia*, *Pinus palustris*, and *Pinus elliottii* (and
25 potentially other pine species).

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27 **3. Analytical Technique**

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29 GPC or other analytical technique that measures the components of interest and
30 meets the following method performance requirements is acceptable. The components
31 of interest include free resin acids, glycerol monoesters, and neutrals. Free resin acids
32 include all unreacted organic acids present in the GEWR product; glycerol monoesters
33 include all monoesters present in the GEWR product; and neutrals include all non-
34 saponifiable and non-acidic saponifiable components, mainly monoterpenes, diterpenes
35 and high molecular weight neutrals, in the GEWR product.

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38 **4. Definitions**

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40 *Limit of quantitation (LOQ).*—LOQ is the lowest level of analyte in a test sample that can
41 be quantified at a specified level of precision.

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43 *Repeatability.*—Variation arising when all efforts are made to keep conditions constant
44 by using the same instrument and operator (in the same laboratory) and repeating

45 during a short time period. Expressed as the repeatability standard deviation (SD_r); or
46 % repeatability relative standard deviation (% RSD_r).

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

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54 **Recovery.**—The fraction or percentage of analyte that is measured when the test sample
55 is analyzed using the entire method.

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57 **5. Method Performance Requirements**

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59 **Table 1. Method Performance Table for Component Fractions in GEWR**

	Free resin acids	Monoesters	Neutrals
Limit of quantitation (LOQ)	0.01% ^a	0.5% ^a	0.01% ^a
Analytical range	0.01%–5% ^a	0.5%–5% ^a	0.01%–20% ^a
Recovery/Accuracy	90–107%	95–105%	90–107%
Repeatability (RSD_r)	5.3%	3%	5.3%
Reproducibility (RSD_R)	8%	4.4%	8%

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^aPercent refers to the mass fraction of the component in the GEWR product, e.g., 1% = 0.01 g/g.

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62 **6. System Suitability Tests and/or Analytical Quality Control**

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

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(b) Retention time should be stable to $\pm 1\%$.

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

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68 **7. Validation Guidance**

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70 Appendix F: *Guidelines for Standard Method Performance Requirements, Official*
71 *Methods of Analysis of AOAC INTERNATIONAL* (2016) 20th Ed., AOAC INTERNATIONAL,
72 Rockville, MD, USA (http://www.eoma.aoc.org/app_f.pdf)

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74 Validation studies should include at least 4 GEWR products produced from different
75 pine species to include, but not be limited to, *P. palustris*, *P. elliotii*, *P. halpensis*, and *P.*
76 *brutia*.

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78 **8. Reference materials**

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80 Refer to Annex F: *Development and Use of In-House Reference Materials* in [Appendix](#)
81 [F: Guidelines for Standard Method Performance Requirements](#), 19th Edition of the AOAC

82 INTERNATIONAL Official Methods of Analysis (2012). Available at:
83 http://www.eoma.aoac.org/app_f.pdf

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85 **9. *Maximum Time-to-Results***

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87 None.