

1 Version 6, 08/24/2020

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3 **Standard Method Performance Requirements (SMPRs) for Characterization and**  
4 **Quantitation of Monoesters 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 Methods<sup>SM</sup>* or *AOAC Official*  
18 *Methods of Analysis<sup>SM</sup>* 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 monoesters of glycerol esters of wood rosin  
24 (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 Any analytical technique(s) that measures the analyte of interest and meets the  
30 following method performance requirements is/are acceptable. All peaks of monoester  
31 components present above the LOQ in the final analysis must be identified and  
32 quantitated.

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34 **4. Definitions**

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

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39 *Repeatability.*—Variation arising when all efforts are made to keep conditions constant  
40 by using the same instrument and operator (in the same laboratory) and repeating  
41 during a short time period. Expressed as the repeatability standard deviation ( $SD_r$ ); or  
42 % repeatability relative standard deviation ( $\%RSD_r$ ).

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44 *Reproducibility.*—Variation arising when identical test materials are analyzed in different

laboratory by different operators on different instruments. The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SD<sub>R</sub>); or % reproducibility relative standard deviation (% RSD<sub>R</sub>).

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

## 5. Method Performance Requirements

**Table 1. Method Performance Table for Glycerol Monoesters from GEWR**

Limit of quantitation (LOQ)	0.5% <sup>a</sup>
Analytical range	0.5–10% <sup>a</sup>
Recovery	95–105%
Repeatability (RSD <sub>r</sub> )	3%
Reproducibility (RSD <sub>R</sub> )	4.4%

<sup>a</sup>Percent refers to the concentration of a molecule in the GEWR product, e.g., 1% = 0.01 g molecule X per g GEWR.

## 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 and purity of peaks, preferably by MS. NIST Mass Spectral Library can be helpful.

(d) Method developer should explain how response factors are estimated when a direct standard is not available.

(e) Purity of the internal standard should be >95%.

## 7. Validation Guidance

Appendix F: *Guidelines for Standard Method Performance Requirements, Official Methods of Analysis of AOAC INTERNATIONAL* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA ([http://www.eoma.aoac.org/app\\_f.pdf](http://www.eoma.aoac.org/app_f.pdf))

Validation studies should include at least 4 GEWR products produced from different pine species to include, but not be limited to, *Pinus halepensis*, *Pinus brutia*, *Pinus palustris*, and *Pinus elliottii*.

## 8. Reference materials

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

83 INTERNATIONAL Official Methods of Analysis (2012). Available at:  
84 [http://www.eoma.aoac.org/app\\_f.pdf](http://www.eoma.aoac.org/app_f.pdf)

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

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