AOAC SMPR® 2018.004

Standard Method Performance Requirements (SMPRs®) for Determination of *trans* Resveratrol in Dietary Supplements and Dietary Ingredients

Intended Use: Quality Assurance and Compliance to Current Good Manufacturing Practices

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 written and adopted by AOAC stakeholder panels composed of representatives from the industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC expert review panels in their evaluation of validation study data for methods being considered for *Performance Tested Methods*℠ or AOAC *Official Methods of Analysis*℠, and can be used as acceptance criteria for verification at user laboratories. [Refer to Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA.]

2 Applicability

The method must be specific for *trans* resveratrol quantitation in the presence of the *cis* isomer in dietary supplements and dietary ingredients as listed in Table 1.

3 Analytical Technique

Any analytical technique that meets the following method performance requirements is acceptable.

4 Definitions

Analytical range.—Includes all steps of the analytical procedure including sample preparation and further dilutions.

Dietary ingredient.—A vitamin; a mineral; an herb or other botanical; an amino acid; a dietary substance for use by man to supplement the diet by increasing total dietary intake; or a concentrate, metabolite, constituent, extract, or combination of any of the above dietary ingredients. {United States Federal Food Drug and Cosmetic Act §201(ff) [U.S.C. 321 (ff)]}

Dietary supplement.—A product intended for ingestion that contains a "dietary ingredient" intended to add further nutritional value to (supplement) the diet. Dietary supplements may be found in many forms such as tablets, capsules, softgels, gelcaps, liquids, or powders.

Limit of quantitation (LOQ).—The minimum concentration or mass of analyte in a given matrix that can be reported as a quantitative result.

Raw materials.—Fresh, dried, or cut plant materials.

Recovery.—The fraction or percentage of spiked analyte that is recovered 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 and repeating during a short time period. Expressed as the repeatability standard deviation (SD_r); or % repeatability relative standard deviation (%RSD_r).

Reproducibility.—The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SD_R); or % reproducibility relative standard deviation (%RSD_R).

trans Resveratrol.—IUPAC name: 5-[(*E*)-2-(4-hydroxyphenyl) ethenyl]benzene-1,3-diol. CAS No. 501-36-0. *See* Figure 1 for chemical structure.

5 Method Performance Requirements

See Tables 2 and 3.

6 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blank check samples, and check standards at the lowest point and midrange point of the analytical range. A control sample must be included. The method must be specific for *trans* resveratrol.

7 Reference Material(s)

See Table 4.

NIST *Vaccinium* spp. berries (freeze dried, extract, or oral dosage form) available in small amounts

Sigma CRM 76511

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements*, 20th Ed. of the *Official Methods of Analysis of AOAC INTERNATIONAL* (2016). Available at: http://www.eoma.aoac.org/app_f.pdf

8 Validation Guidance

Appendix D: *Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis*, 20th Ed. of the *Official Methods of Analysis of AOAC INTERNATIONAL* (2016). Available at: http://www.eoma.aoac.org/app_d.pdf

Appendix K: *Guidelines for Dietary Supplements and Botanicals,* 20th Ed. of the *Official Methods of Analysis of AOAC INTERNATIONAL* (2016). Also at: *J. AOAC Int*. **95**, 268(2012); DOI: 10.5740/jaoacint.11-447 and available at: http://www.eoma.aoac.org/app_k.pdf

9 Maximum Time-to-Determination

No maximum time.

Approved by the AOAC Stakeholder Panel on Dietary Supplements (SPDS). Final Version Date: March 16, 2018.

Table 1. Examples of dietary supplements and dietary ingredients
Powders
Tablets
Capsules
Liquids
Softgels
Extracts

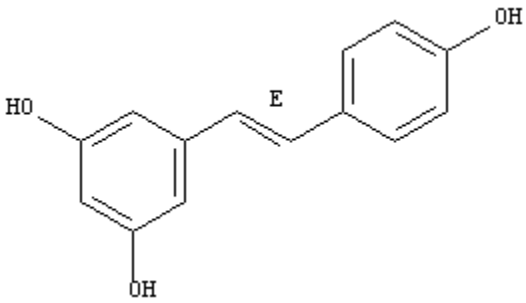

Figure 1. Chemical structure of resveratrol.

Table 2. Analytical range and LOQ based on matrix	
Parameter	
Analytical range, % (w/w)[a]	0.01–100
Limit of quantitation, % (w/w)	0.01
[a] Range may be narrower depending on the analytical matrix.	

Table 3. Method performance requirements as a function of range			
	Acceptance criteria		
Parameter	<1%	1–50%	>50–100%
Recovery, %	85–115	>97–103	98–102
RSD_r, %	≤7.5	≤5	≤2
RSD_R, %	≤10	≤8	≤3

Table 4. Reference material(s)			
Source	*trans*-Resveratrol	*cis*-Resveratrol	Resveratrol d-4
Alkemist Labs	4963S		
Cresent Chemical Co.	CA16811600		
LGC	CDX-00018089-500		
European Pharmacopoeia Reference Standard	Y000111194		
Phytolab	89539		
Santa Cruz Biotechnology	sc-200808	sc-205254	
Selleckhem	S 1396		
Sigma-Aldrich	R5010		
TLC Pharmceutical Standards	R-079001	R-079002	R-079002
Toronto Research Chemicals	R150000	R150005	R150001
USP	162105		