AOAC SMPR 2015.009

Standard Method Performance Requirements[SM] (SMPRs) for Estimation of Total Phenolic Content Using the Folin-C Assay

Intended Use: Dispute Resolution and Routine Surveillance

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 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*[SM] or AOAC *Official Methods of Analysis*[SM], and can be used as acceptance criteria for verification at user laboratories.

2 Applicability

Estimation of total soluble phenolic content in dietary supplement raw materials and finished products using the Folin-C assay for comparison within same matrices.

3 Analytical Technique

Any Folin-C reagent-based technique that estimates total phenolic content and meets the following method performance requirements is/are acceptable.

4 Definitions

Dietary ingredients.—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 {Federal Food Drug and Cosmetic Act §201(ff) [U.S.C. 321 (ff)]}.

Dietary supplements.—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.

Quantitative method.—Method of analysis which response is the amount of the analyte measured either directly (enumeration in a mass or a volume), or indirectly (color, absorbance, impedance, etc.) in a certain amount of sample.

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 relative standard deviation (SD_R); or % reproducibility relative standard deviation ($\%RSD_R$).

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

Table 1. Method performance requirements[a]

		Gallic acid equivalent (w/w)	
Single-laboratory validation	Analytical range	5–500 ppm	
	Limit of quantitation (LOQ)	≤5 ppm	
	Recovery	80–110%	
	Repeatability (RSD_r)	3–5 ppm	≤9%
		>5 ppm	≤7%
Multi-laboratory validation	Reproducibility (RSD_R)	3–5 ppm	≤13%
		>5 ppm	≤10%

[a] Other calibrations would be acceptable.

5 Method Performance Requirements

See Table 1.

6 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include a blank, and check standards at the lowest point and midrange point of the analytical range. Evaluate matrix and possible interfering components.

7 Reference Material(s)

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed. Available at http://www.eoma.aoac.org/app_f.pdf.

NIST Guidance Document

NIST 3287 Blueberry Fruit Powder

NIST 3281 Cranberry Fruit Powder

NIST https://www-s.nist.gov/srmors/viewTable.cfm?tableid=79

8 Validation Guidance

Appendix D: *Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed. Available at http://www.eoma.aoac.org/app_d.pdf.

Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed. Available at http://www.eoma.aoac.org/app_f.pdf.

Appendix K: *Guidelines for Dietary Supplements and Botanicals*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed. Available at: http://www.eoma.aoac.org/app_k.pdf.

9 Maximum Time-to-Result

None

Approved by the AOAC Stakeholder Panel on Dietary Supplements (SPDS). Final Version Date: June 15, 2015. Effective Date: March 19, 2015.