AOAC SMPR 2016.003

Standard Method Performance Requirements (SMPRs®) for Quantitation of Curcuminoids

Intended Use: Reference method for cGMP compliance

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 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

The method will be able to separate and quantify each individual curcuminoid (curcumin, demethoxycurcumin, and bisdemethyoxycurcumin) in turmeric [*Curcuma longa* Linn.] dietary ingredients and dietary supplement finished products containing turmeric, alone or in combination with other dietary ingredients.

3 Analytical Technique

Any analytical technique(s) that measures the analytes of interest and meets the following method performance requirements is/are acceptable.

4 Definitions

Analytes:

Curcumin.—IUPAC name: (1E,6E)-1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione. CAS No. 458-37-7. *See* Figure 1 for molecular structure.

Demethoxycurcumin.—IUPAC name: (1E,6E)-1-(4-hydroxy-3-methoxyphenyl)-7-(4-hydroxyphenyl)hepta-1,6-diene-3,5-dione. CAS No. 24939-17-1. *See* Figure 2 for the molecular structure of demethoxy-curcumin.

Bisdemethoxycurcumin.—IUPAC name: (1E,6E)-1,7-bis(4-hydroxyphenyl)hepta-1,6-diene-3,5-dione. CAS No. 24939-16-0. *See* Figure 3 for molecular structure.

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 supplement*s.—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.

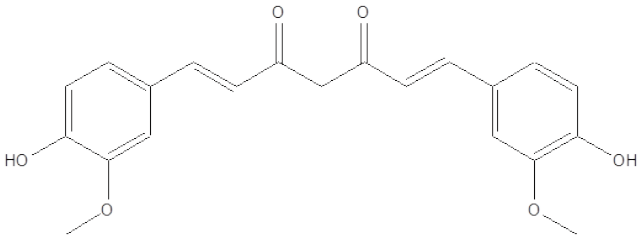

Figure 1. Molecular structure of curcumin.

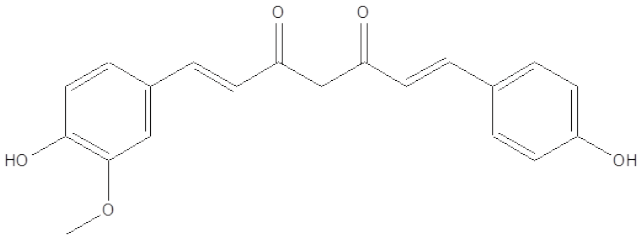

Figure 2. Molecular structure of demethoxycurcumin.

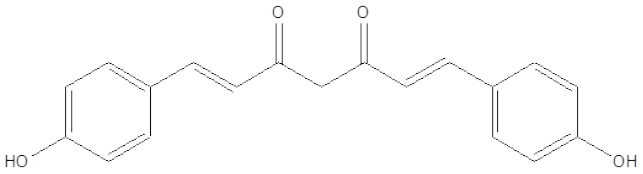

Figure 3. Molecular structure of bisdemethoxycurcumin.

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

Table 1. Method performance requirements

Parameter	Requirement	
Limit of quantitation (LOQ), %	≤0.1	
Recovery, %	95–110	
Analytical range, %	≤0.1–50	>50
RSD_r, %	≤5	≤3
RSD_R, %	≤8	≤5

Table 2. Curcuminoids in the presence of other dietary ingredients, for example:

Piper nigrum
Zingiber officinale (ginger)
Capsicum annuum (cayenne pepper)
β-carotene
Lutein
Lycopene
Zeaxanthin
Divalent cations (calcium and magnesium) Ca^{+2}

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

5 Method Performance Requirements

See Table 1.

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.

7 Reference Material(s)

Curcumin USP Reference Standard (Cat. No. 1151855)

Demethoxycurcumin USP Reference Standard (Cat. No. 1173100)

Bisdemethoxycurcumin USP Reference Standard (Cat. No. 1075305)

Curcuminoids USP Reference Standard (Cat. No. 1151866)

NIST SRM 3299 *Curcuma longa* L. (Turmeric) Rhizome

NIST SRM 3300 *Curcuma longa* L. (Turmeric) Rhizome Extract

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* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA, (http://www.eoma.aoac.org/app_f.pdf)

Table 3. Matrixes

Dried plant material
Extracts (purified curcuminoids)
Tablets
Capsules
Softgel capsules
Powders
Tinctures
Liquids

8 Validation Guidance

For methods based on UV, all compounds in Table 2 must be evaluated for interference.

All target analytes and all matrixes listed in Table 3 shall be evaluated. One analyte per matrix is acceptable provided all three analytes are represented in the complete evaluation.

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

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

Appendix K: *Guidelines for Dietary Supplements and Botanicals*, *Official Methods of Analysis* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_k.pdf)

9 Maximum Time-to-Result

None

Approved by AOAC Stakeholder Panel on Dietary Supplements (SPDS). Final Version Date: March 17, 2016. Effective Date: March 17, 2016.