AOAC SMPR® 2023.004

Standard Method Performance Requirements (SMPRs®) for Selected Residual Solvents in Color Additives from Crop-Based Sources

Intended Use: Surveillance and Monitoring by Trained Technicians

1 Purpose

What: AOAC *Standard Method Performance Requirements* (SMPRs®) are voluntary consensus standards developed in accordance with the AOAC policy, "AOAC Due Process for Development of AOAC Non-Method Consensus Standards and Documents." SMPRs describe the scientific community's recommended minimum method performance characteristics and analytical requirements for a specific method-related intended use.

Who: Drafted by AOAC working groups, SMPRs are adopted by AOAC by a consensus of stakeholders affiliated with its integrated science programs and projects which are composed of volunteer subject matter experts representing academia, government, industry, and nonprofit sectors from around the world.

Use: AOAC SMPRs are used in the AOAC core science programs as a resource for AOAC method experts, including expert review panels, in the evaluation of validation study data for methods submitted to the AOAC *Official Methods of Analysis*SM and AOAC *Performance Tested Methods*SM programs. AOAC SMPRs also may be used to provide acceptance criteria for the verification of methods and serve as a resource to guide method development and optimization.

2 Applicability

Quantitative analysis of selected residual solvents (Table 1) in color additives from crop-based sources (Table 2).

3 Analytical Technique

Any analytical technique(s) that measures the analyte(s) of interest in the target matrix/matrices and meets the following method performance requirements is/are acceptable. More than one column chemistry may be needed; for example, if using a nonspecific detector, a second column confirmation is required. Nontargeted methods with the capability to detect multiple residues are preferred (e.g., mass spectrometric detection).

4 Definitions

Certified Reference Method (CRM).—A reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a reference material certificate issued by an authoritative body that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

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

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

Table 1. Selected residual solvents

Solvent	CAS No.
Methanol	67-56-1
Ethanol	64-17-5
Isopropanol (2-propanol)	67-63-0
N-butanol	71-36-3
Acetone	67-64-1
Hexanes[a]	110-54-3[b]
Heptanes[a]	142-82-5[c]
Ethyl acetate	141-78-6
Isobutyl acetate	110-19-0
Methyl ethyl ketone	78-93-3
Methyloxolane (methyltetrahydrofuran)	96-47-9
Methylene chloride	75-09-2
Ethylene dichloride (1,2-dichloroethane)	107-06-2
Trichloroethylene (1,1,2-trichloroethene)	79-01-6
Chloroform	67-66-3
Benzene	71-43-2
Toluene	108-88-3
Xylenes[a]	1330-20-7[d]
Diethyl ether	60-29-7
Methyl *tert*-butyl ether	1634-04-4

[a] Reported as sum of isomers.

[b] CAS No. for *n*-hexane [isomers include 2-methylpentane (107-83-5), 3-methylpentane (96-14-0), 2,3-dimethylbutane (79-29-8), and 2,2-dimethylbutane (75-83-2)].

[c] CAS No. for *n*-heptane [Isomers include 2-methylhexane (591-76-4), 3-methylhexane-racemic mixture (589-34-4), 2,2-dimethylpentane (590-35-2), 2,3-dimethylpentane-racemic mixture (565-59-3), 2,4-dimethylpentane (108-08-7), 3,3-dimethylpentane (562-49-2), 3-ethylpentane (617-78-7), and 2,2,3-trimethylbutane (464-06-2)].

[d] CAS No. for mixed xylene isomers [isomers include *o*-xylene (95-47-6), *m*-xylene (108-38-3), and *p*-xylene (106-42-3)].

Recovery.—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 (in the same laboratory) and repeating during a short time period. Expressed as the repeatability standard deviation (SD_r); or % repeatability relative standard deviation (% RSD_r).

Reproducibility.—Variation arising when identical test materials are analyzed in different laboratories 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_R); or % reproducibility relative standard deviation (% RSD_R).

5 Method Performance Requirements

See Table 3.

6 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blank check samples and appropriate check standards.

Table 2. Target matrices

CFR00	Target matrix	Manufacturing process	Source
73.30	Annatto extract	Solvent extraction	Annatto seed
73.40	Dehydrated beets (beet powder)[a]	Dehydration of beets	Beets
73.95	Beta-carotene	Natural, not synthetic, product	Yellow, orange, and green leafy fruits and vegetables; algae; fungi
73.125	Sodium copper chlorophyllin	Processed to add copper	Alfalfa
73.169	Grape color extract[a]	Aqueous extraction	Concord grapes
73.170	Grape skin extract (enocianina)[a]	Aqueous extraction	Grapes
73.250	Fruit juice[a]	Aqueous extraction	Fruit
73.260	Vegetable juice[a]	Aqueous extraction	Vegetables
73.300	Carrot oil	Solvent extraction from carrots	Carrots
73.340	Paprika[a]	Ground dried pod of capsicum	Pod of capsicum
73.345	Paprika oleoresin	Solvent extraction from paprika	Capsicum
73.500	Saffron[a]	Dried stigma of *Crocus sativus* L.	Dried stigma of *Crocus sativus* L.
73.530	Spirulina extract	Aqueous extraction	*Arthrospira platensis*
73.585	Tomato lycopene extract; tomato lycopene concentrate[a]	Ethyl acetate extraction from tomato pulp	Tomato
73.600	Turmerica	Ground rhizome of *Curcuma longa* L.	Rhizome of curcuma
73.615	Turmeric oleoresin	Solvent extraction of turmeric	Rhizome of curcuma

[a] Natural source only.

Table 3. Method performance requirements

Solvents	Analytical range, ppm		Recovery, %		LOQ, ppm	RSD_r, %	RSD_R, %
	Min.	Max.	<10 ppm	>10 ppm			
Methanol	2	100	50–150	75–125	2	≤20	≤30
Ethanol	2	400			2		
Isopropanol (2-propanol)	2	100			2		
N-butanol	2	100			2		
Acetone	2	100			2		
Hexanes[a]	2	100			2		
Heptanes[a]	2	100			2		
Ethyl acetate	2	100			2		
Methyl ethyl ketone	2	100			2		
Methyloxolane	2	100			2		
Methylene chloride	2	100			2		
Ethylene dichloride (1,2-dichloroethane)	2	100			2		
Trichloroethylene (1,1,2-trichloroethene)	2	100			2		
Chloroform	2	100			2		
Benzene	0.5	100			0.5		
Toluene	0.5	100			0.5		
Xylenes[a]	0.5	100			0.5		
Diethyl ether	0.5	100			0.5		
Methyl *tert*-butyl ether	0.5	100			0.5		

[a] Reported as sum of isomers.

7 Reference Materials

Refer to "Annex F: Development and Use of In-House Reference Materials" in "Appendix F: Guidelines for *Standard Method Performance Requirements*" (2023) *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed. Available at: https://academic.oup.com/book/45491

Residual solvent mixtures and internal standards (available in different chemical functional groups) are available from various commercial suppliers. It is recommended that for GC-MS methods, method developers use internal standards corresponding to functional groups as commercially available from suppliers.

8 Validation Guidance

Methods submitted for this SMPR should utilize CRMs for instrument calibration and quality control of methods when available from an ISO-accredited provider.

"Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis" (2023) *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed. Available at: https://doi.org/10.1093/9780197610145.005.004

"Appendix F: Guidelines for *Standard Method Performance Requirements*" (2023) *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed. Available at: https://doi.org/10.1093/9780197610145.005.006

"Appendix K: Guidelines for Dietary Supplements and Botanicals" (2023) *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed. Available at: https://doi.org/10.1093/9780197610145.005.011

U.S. Food and Drug Administration, *Bioanalytical Method Validation Guidance for Industry* (May 2018)

9 Maximum Time-to-Results

None.

Version 14: Final version, September 1, 2023. Approved by stakeholders interested in and affiliated with AOAC Working Group on Colors Additives from Natural Sources and the affiliated AOAC Community. Effective date: November 13, 2023.