

AOAC SMPR[®] 2024.001 *Standard Method Performance Requirements (SMPRs[®]) for Selected Pesticides 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 uses SMPRs in its core science programs in which they are 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 AnalysisSM* and AOAC *Performance Tested MethodsSM* programs. Additionally, AOAC SMPRs 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 pesticides (Table 1) in natural color additives manufactured from crop-based sources (Tables 2 and 3). Table 1 provides the required list of pesticides. Additional compounds could be included in the method.

3 Analytical Technique

Any analytical technique(s) that measures the analyte of interest and meets the following method performance requirements is/are acceptable. More than one analytical technique may be needed.

4 Definitions

Certified Reference Material (CRM).—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).—Lowest level of analyte in a test sample that can be quantified at a specified level of precision.

Multiresidue method (MRM).—Method able to distinguish, followed by identification and/or quantification of, more than one pesticide residue in one analysis.

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 4.

6 System Suitability Tests and/or Analytical Quality Control

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

7 Validation Guidance

Validation must be conducted on the final processed color additive (not intermediates or raw materials) using at least one representative matrix from the aqueous extract and dry powder categories and one from each of the two subcategories (oleoresin and other) of the solvent extract category listed in Table 2.

Validation must be conducted at the target LOQ and at 10x LOQ.

“Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis” (2023) Latimer, Jr., G.W. (Ed.), *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed., <https://doi.org/10.1093/9780197610145.005.004>

“Appendix F: Guidelines for Standard Method Performance Requirements” (2023) Latimer, Jr., G.W. (Ed.), *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed., <https://doi.org/10.1093/9780197610145.005.006>

“Appendix K: Guidelines for Dietary Supplements and Botanicals” (2023) Latimer, Jr., G.W. (Ed.), *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed., <https://doi.org/10.1093/9780197610145.005.011>

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

European Commission Guidance Document (2021) *Analytical Quality Control and Method Validation Procedures for Pesticide Residues Analysis in Food and Feed* (SANTE 11312/2021)

8 Reference Materials

Refer to “Annex F: Development and Use of In-House Reference Materials” in “Appendix F: Guidelines for Standard Method Performance Requirements” (2023) Latimer, Jr., G.W. (Ed.), *Official Methods of Analysis of AOAC INTERNATIONAL*, 22nd Ed., <https://doi.org/10.1093/9780197610145.005.006>

Methods submitted for this SMPR should utilize pesticide CRMs for instrument calibration and quality control of method when available from an ISO/IEC 17034:2016 accredited provider.

9 Maximum Time-to-Results

None.

Approved by AOAC Working Group on Color Additives from Natural Sources (Pesticide Residues Subgroup) on January 12, 2024. Final Version Date: December 5, 2023. Effective Date: January 12, 2024.

Table 1. Selected pesticides

CAS No.	Name	CAS No.	Name
30560-19-1	Acephate	35554-44-0	Imazalil
135410-20-7	Acetamiprid	138261-41-3	Imidacloprid
131860-33-8	Azoxystrobin	173584-44-6	Indoxacarb
149877-41-8	Bifenazate	36734-19-7	Iprodione
82657-04-3	Bifenthrin	91465-08-06	Lambda-cyhalothrin
188425-85-6	Boscalid	330-55-2	Linuron
69327-76-0	Buprofezin	121-75-5	Malathion
63-25-2	Carbaryl	374726-62-2	Mandipropamid
133-06-2	Captan ^a	57837-19-1	Metalaxyl
10605-21-7	Carbendazim	10265-92-6	Methamidophos
500008-45-7	Chlorantraniliprole	16752-77-5	Methomyl (Lannate)
122453-73-0	Chlorfenapyr (Alert)	161050-58-4	Methoxyfenozide
2921-88-2	Chlorpyrifos	88671-89-0	Myclobutanil
210880-92-5	Clothianidin	116714-46-6	Novaluron
120116-88-3	Cyazofamid	1113-02-6	Omethoate
68359-37-5	Cyfluthrin	52645-53-1	Permethrin (<i>cis</i> and <i>trans</i>)
52315-07-8	Cypermethrin	51-03-6	Piperonyl butoxide
121552-61-2	Cyprodinil	41198-08-7	Profenofos
333-41-5	Diazinon	24579-73-5	Propamocarb
119446-68-3	Difenoconazole	60207-90-1	Propiconazole
35367-38-5	Diflubenzuron	175013-18-0	Pyraclostrobin
60-51-5	Dimethoate (Cygon)	96489-71-3	Pyridaben
110488-70-5	Dimethomorph	53112-28-0	Pyrimethanil
165252-70-0	Dinotefuran	95737-68-1	Pyriproxyfen (F)
153233-91-1	Etoxazole	124495-18-7	Quinoxifen
114369-43-6	Fenbuconazole	187166-40-1	Spinetoram
126833-17-8	Fenhexamid	148477-71-8	Spirodiclofen
39515-41-8	Fenpropathrin	203313-25-1	Spirotetramat
120068-37-3	Fipronil	946578-00-3	Sulfoxaflor
158062-67-0	Fonicamid	107534-96-3	Tebuconazole
272451-65-7	Flubendiamide	148-79-8	Thiabendazole
131341-86-1	Fludioxonil	111988-49-9	Thiacloprid
239110-15-7	Fluopicolide	153719-23-4	Thiamethoxam
658066-35-4	Fluopyram	141517-21-7	Trifloxystrobin
951659-40-8	Flupyradifurone	68694-11-1	Triflumizole
76674-21-0	Flutriafol		
907204-31-3	Fluxapyroxad		

^a If method is not able to reliably determine parent compound but is able to analyze corresponding metabolite (THPI), it will be considered by the AOAC expert review panel.

Table 2. Matrix categories

Category	Subcategory	Representative matrices
Aqueous extracts		Grape color extract Grape skin extract (enocianina) Fruit juice Vegetable juice Spirulina extract Butterfly pea flower extract Annatto extract
Dry powders		Dehydrated beets (beet powder) Paprika Turmeric Butterfly pea flower extract Fruit juice Vegetable juice
Solvent extracts	Oleoresin	Turmeric oleoresin Paprika oleoresin
	Other	Tomato lycopene extract; tomato lycopene concentrate Carrot oil Annatto extract

Table 3. Target matrices

Matrix ^a	21 CFR listing ^b	E No. ^c	Manufacturing process	Source
Annatto extract	73.30	E160b	Alkaline alcoholic or aqueous solvent extraction	Annatto seed
Dehydrated beets (beet powder)	73.40	E162	Dehydration of beets	Beets
Beta-carotene	73.95	E160a	Natural, not synthetic, product	Yellow, orange, and green leafy fruits and vegetables; algae; fungi
Grape color extract	73.169	E163	Aqueous extraction	Concord grapes
Grape skin extract (enocianina)	73.170	E163	Aqueous extraction	Grapes
Fruit juice	73.250		Aqueous extraction or dried powder	Fruit
Vegetable juice	73.260		Aqueous extraction or dried powder	Vegetables
Carrot oil	73.300		Hexane extraction from carrots	Carrots
Paprika	73.340	E160c	Ground dried pod of capsicum	Pod of capsicum
Paprika oleoresin	73.345	E160c	Solvent extraction from paprika	Capsicum
Spirulina extract	73.530		Aqueous extraction	Arthrospira platensis
Tomato lycopene extract; tomato lycopene concentrate	73.585	E160d	Ethyl acetate extraction from tomato pulp	Tomato
Turmeric	73.600	E100	Ground rhizome of <i>Curcuma longa</i> L.	Rhizome of curcuma
Turmeric oleoresin	73.615		Solvent extraction of turmeric	Rhizome of curcuma
Butterfly pea flower extract	73.69		Aqueous extraction	Dried butterfly pea flowers

^a Natural source only.

^b U.S. regulations describing color additive: <https://www.ecfr.gov/current/title-21/chapter-I/subchapter-A/part-73>.

^c E No. = Europe numbers, which are codes for substances used as food additives, including those found naturally in many foods, for use within the European Union (EU) and European Free Trade Association (EFTA).

Table 4. Method performance requirements

LOQ, mg/kg (ppm)	0.01–0.1
Analytical range	$0.5 \times \text{LOQ}$ to $100 \times \text{LOQ}$
Recovery, %	60–120 ^a
RSD _r , %	≤20
RSD _R , %	≤30

^a In exceptional cases, average recoveries outside the above range can be accepted, but shall lie within 50–130% and only when the precision criteria for RSD_r are met.