

Standard Method Performance Requirements (SMPRs®) for Identification and Quantitation of Selected Pesticide Residues in Dried Cannabis Materials

Intended Use: Consensus-Based Reference Method

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 MethodsSM* or *AOAC Official Methods of AnalysisSM*, and can be used as acceptance criteria for verification at user laboratories.

2 Applicability

Method, or a suite of methods, to identify and quantify selected pesticide residues (Table 1) in dried cannabis plant materials.

3 Analytical Technique

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

4 Definitions

Dried plant material.—Dried whole or milled flower plant material from *Cannabis* sp. and its hybrids.

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

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

Parts per million (PPM).—Also expressed as milligrams of analyte per kilogram of dried plant materials (as received).

Quantitative method.—Method of analysis where 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.

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

Reproducibility.—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 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.

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*, 21st Ed. of the *Official Methods of Analysis of AOAC INTERNATIONAL* (2019). 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*, 21st Ed. of the *Official Methods of Analysis of AOAC INTERNATIONAL* (2019). Available at http://www.eoma.aoac.org/app_d.pdf

Appendix F: *Guidelines for Standard Method Performance Requirements*, 21st Ed. of the *Official Methods of Analysis of AOAC INTERNATIONAL* (2019). Available at http://www.eoma.aoac.org/app_f.pdf

Appendix K: *Guidelines for Dietary Supplements and Botanicals*, 21st Ed. of the *Official Methods of Analysis of AOAC INTERNATIONAL* (2019). Available at http://www.eoma.aoac.org/app_k.pdf

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

European Commission Guidance Document on *Analytical Quality Control and Method Validation Procedures for Pesticide Residues and Analysis in Food and Feed* (SANTE/11813/2017)

9 Maximum Time-to-Result

None.

Approved by the AOAC Stakeholder Panel on Strategic Food Analytical Methods (SPSFAM). Final Version Date: August 26, 2018.

Table 1. Pesticides

Compound	CAS No.	Lowest action level, ppm	Target LOQ, ppm ^a
Abamectin (Avermectins: B1a & B1b)	71751-41-2	0.05	0.025
Acephate	30560-19-1	0.1	0.05
Acequinocyl	57960-19-7	0.1	0.05
Acetamiprid	135410-20-7	0.1	0.05
Aldicarb	116-06-3	0.1	0.05
Allethrin	584-79-2	0.1	0.05
Ancymidol	12771-68-5	0.1	0.05
Azadirachtin	108168-76-9	0.1	0.05
Azoxystrobin	131860-33-8	0.02	0.01
Benzovindiflupyr	1072957-71-1	0.1	0.05
Bifenazate	149877-41-8	0.01	0.005
Bifenthrin	82657-04-3	0.01	0.005
Boscalid	188425-85-6	0.1	0.05
Buprofezin	69327-76-0	0.1	0.05
Captan	133-06-2	0.05	0.025
Carbaryl	63-25-2	0.2	0.1
Carbofuran	1563-66-2	0.1	0.05
Chlorantraniliprole	500008-45-7	0.2	0.1
Chlordane	57-74-9	0.1	0.05
Chlorfenapyr	122453-73-0	0.1	0.05
Chloromequat chloride	999-81-5	0	0.005 ^b
Chlorpyrifos	2921-88-2	0.1	0.05
Clofentezine	74115-24-5	0.1	0.05
Clothianidin	21088-92-5	0	0.005 ^b
Coumaphos	56-72-4	0.1	0.05
Cyantraniliprole	736994-63-1	0	0.005 ^b
Cyfluthrin (Baythroid)	68359-37-5	0.01	0.005
Cypermethrin	52315-07-8	0.05	0.025
Cyprodinil	121552-61-2	0	0.005 ^b
Daminozide	1596-84-5	0.05	0.025
Deltamethrin	52918-63-5	0	0.005 ^b
Diazinon	333-41-5	0.1	0.05
Dichlorvos	62-73-7	0.1	0.05
Dimethoate	60-51-5	0.1	0.05
Dimethomorph	110488-70-5	2	1
Dinotefuran	165252-70-0	0	0.005 ^b
Dodemorph	1593-77-7	0	0.005 ^b
Endosulfan I (alpha)	959-98-8	0	0.005 ^b
Endosulfan II (beta)	33213-65-9	0	0.005 ^b
Endosulfan sulfate	1031-07-8	0	0.005 ^b

Table 1. (continued)

Compound	CAS No.	Lowest action level, ppm	Target LOQ, ppm ^a
Ethephon	16672-87-0	0	0.005 ^b
Ethoprophos (Prophos)	13194-48-4	0.1	0.05
Etofenprox	80844-07-1	0.1	0.05
Etoxazole	153233-91-1	0.01	0.005
Etridiazole (Terrazole)	2593-15-9	0	0.005 ^b
Fenhexamid	126833-17-8	0.1	0.05
Fenoxycarb	79127-80-3	0.1	0.05
Fenpyroximate (mix of isomers)	111812-58-9	0.1	0.05
Fensulfthion	115-90-2	0	0.005 ^b
Fenthion	55-38-9	0	0.005 ^b
Fenvalerate (Sanmarton)	51630-58-1	0	0.005 ^b
Fipronil	120068-37-3	0.1	0.05
Flonicamid	158062-67-0	0.1	0.05
Fludioxonil	131341-86-1	0.02	0.01
Fluopyram	658066-35-4	0	0.005 ^b
Flurprimidol	56425-91-3	0	0.005 ^b
Hexythiazox	78587-05-0	0.1	0.05
Imazalil	35554-44-0	0.01	0.005
Imidacloprid	138261-41-3	0.01	0.005
Iprodione	36734-19-7	0	0.005 ^b
Kinoprene	37882-31-8	0	0.005 ^b
Kresoxim-methyl	143390-89-0	0.1	0.05
Malathion	121-75-5	0.05	0.025
Metalaxyl	57837-19-1	0.2	0.1
Methiocarb	2032-65-7	0.1	0.05
Methomyl	16752-77-5	0.4	0.2
Methoprene	40596-69-8	0	0.005 ^b
Methyl parathion	298-00-0	0.1	0.05
Mevinphos	7786-34-7	0.1	0.05
MGK-264	113-48-4	0.2	0.1
Myclobutanil	88671-89-0	0.01	0.005
Naled (Systhane) (Dibrom)	300-76-5	0.1	0.05
Novaluron	116714-46-6	0	0.005 ^b
Oxamyl	23135-22-0	0.5	0.25
Paclobutrazol	76738-62-0	0.05	0.025
Pentachloronitrobenzene (Quintozene)	82-68-8	0.2	0.1
Permethrin (mix of isomers)	52645-53-1	0.04	0.02
Phenothrin (d-phenothrin)	26002-80-2	0	0.005 ^b
Phosmet (Imidan)	732-11-6	0.02	0.01
Phosmet (oxon)	3735-33-9	0.2	0.1

Table 1. (continued)

Compound	CAS No.	Lowest action level, ppm	Target LOQ, ppm ^a
Piperonyl butoxide	51-03-6	1	0.5
Pirimicarb	23103-98-2	0	0.005 ^b
Prallethrin (mix of isomers)	23031-36-9	0.1	0.05
Propiconazole (tilt)	60207-90-1	0.1	0.05
Propoxur (Baygon)	114-26-1	0.1	0.05
Pyraclostrobin	175013-18-0	0	0.005 ^b
Pyrethrin (mix of isomers)	8003-34-7	0.5	0.25
Pyridaben	96489-71-3	0.1	0.05
Resmethrin	10453-86-8	0	0.005 ^b
Spinetoram	187166-40-1	0.1	0.05
Spinosad (mixture of A and D)	168316-95-8	0.06	0.03
Spirodiclofen	148477-71-8	0	0.005 ^b
Spiromesifen	283594-90-1	0.01	0.005
Spirotetramat	203313-25-1	0.02	0.01
Spiroxamine	118134-30-8	0.1	0.05
Tebuconazole	107534-96-3	0.01	0.005
Tebufenozide	112410-23-8	0	0.005 ^b
Teflubenzuron	83121-18-0	0	0.005 ^b
Tetrachlorvinphos	961-11-5	0	0.005 ^b
Tetramethrin	7696-12-0	0	0.005 ^b
Thiacloprid	111988-49-9	0.1	0.05
Thiamethoxam	153719-23-4	0.05	0.025
Thiophanate-methyl	23564-05-8	0	0.005 ^b
Trifloxystrobin	141517-21-7	0.01	0.005

^a AOAC expert review panel can consider LOQs that are higher than the target LOQ based on its judgement.

^b The AOAC Cannabis Working Group recommended an LOQ of 0.005 ppm for pesticides that do not have a regulatory imposed action level.

Table 2. Method performance requirements

Parameter	Requirement
LOQ, ppm ^a	Specified in Table 1
Analytical range, ppm	LOQ to 100 LOQ specified in Table 1 ^a

^a LOQ determined as 50% of the lowest maximum tolerance level in a July 2018 survey of U States and Canada.

Table 3. Method performance requirements for pesticides in Table 1

Parameter	Requirement
Recovery, %	60–120
RSD _r , %	≤20
RSD _R , %	≤30