Designation: D8399 – 23

Standard Test Method for Multi-residue Analysis of Pesticides in Dried Cannabis and Hemp Raw Materials Using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)[1]

This standard is issued under the fixed designation D8399; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method allows for the concentration determination of the pesticides listed in Table 1 and shall apply to any dried raw material from a cannabis plant (Note 1, Note 2) regardless of the type of cannabis plant from which it was derived (**1**, **2**).[2] For the sake of brevity, the term "cannabis" shall be used from now on to refer to any type of cannabis plant including those which can be classified as hemp. The procedure includes sub-sampling a ground, homogenous sample, liquid-solid extraction with acetonitrile:acetic acid (100:1, v:v), solid phase extraction with C18 SPE media, dilution in 3 mM ammonium formate in water with 0.1 % formic acid and 3 mM ammonium formate in methanol with 0.02 % formic acid in a 20:80 ratio (v:v) and analysis by LC-MS/MS. This procedure encompasses the entire process from sample preparation to analyte quantitation encompassing a range of 0.005 µg/g to 0.500 µg/g.

NOTE 1—For this test method, dried raw material from a cannabis plant includes one or more of inflorescence, leaves, or stems.

NOTE 2—Certain jurisdictions or regulations may require specific parts of the plant to be included or excluded for analysis and those regulations will take precedence for the selection of plant parts.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 Table 1 lists the analytes measured by this test method.

1.4 No recommendations found within this test method shall preclude observance of federal, state, or local regulations, which may be more restrictive or have different requirements.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1193 Specification for Reagent Water

D8245 Guide for Disposal of Resin-Containing Cannabis Raw Materials and Downstream Products

D8270 Terminology Relating to Cannabis

D8282 Practice for Laboratory Test Method Validation and Method Development

3. Terminology

3.1 *Definitions*—For general terms related to cannabis, refer to Terminology D8270.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blank, n*—a cannabis-only sample prepared from the same homogenous cannabis blend used to prepare the calibration curve, without the addition of internal standard working solution (ISWS).

3.2.2 *blank-0, n*—a cannabis-only sample prepared from the same homogenous cannabis blend used to prepare the calibration curve, with the addition of ISWS.

3.2.3 *blank-S, n*—a solvent blank prepared from only the mobile phases, without cannabis or analytes.

3.2.4 *growth regulator, n*—a class of chemical compounds used to modulate the growth and development of a crop.

3.2.5 *pesticide, n*—a product or substance used as a means to reduce or mitigate pests such as insects, fungus, bacteria, and weeds.

[1] This test method is under the jurisdiction of ASTM Committee D37 on Cannabis and is the direct responsibility of Subcommittee D37.03 on Laboratory.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 List of Measureable Analytes

Abamectin	Daminozide	Imidacloprid	Pyraclostrobin
Acephate	Deltamethrin	Iprodione	Pyrethrins
Acequinocyl	Diazinon	Kresoxim Methyl	Pyridaben
Acetamiprid	Dichlorvos	Malathion	Resmethrin
Aldicarb	Dimethoate	Metalaxyl	Spinetoram
Allethrin	Dimethomorph	Methiocarb	Spinosad
Azadirachtin	Dinotefuran	Methomyl	Spirodiclofen
Azoxystrobin	Dodemorph	Methoprene	Spiromesifen
Benzovindiflupyr	Ethoprophos	Mevinphos	Spirotetramat
Bifenazate	Etofenprox	Myclobutanil	Spiroxamine
Bifenthrin	Etoxazole	Naled	Tebuconazole
Boscalid	Fenoxycarb	Novaluron	Tebufenozide
Buprofezin	Fenpyroximate	Oxamyl	Teflubenzuron
Carbaryl	Fensulfothion	Paclobutrazol	Tetrachlorvinphos
Carbofuran	Fenthion	Permethrins	Tetramethrin
Chlorantraniliprole	Fenvalerate	Phenothrin	Thiacloprid
Chlorpyrifos	Fipronil	Phosmet	Thiamethoxam
Clofentezine	Flonicamid	Piperonylbutoxide	Thiophanate Methyl
Clothianidin	Fludioxonil	Pirimicarb	Trifloxystrobin
Coumaphos	Fluopyram	Prallethrin	
Cyantraniliprole	Hexythiazox	Propiconazole	
Cyprodinil	Imazalil	Propoxur	

Pyrethrins = Pyrethrin I and II
Spinetoram = Spinetoram J and L
Spinosad = Spinosyn A and D

3.2.5.1 *Discussion*—May be used in gardens, crops, woodlands or recreational areas, these are fungicides, insecticides, herbicides, and biocontrols approved for use by regulatory authorities to control pests harmful to plants, humans, or animals.

3.3 *Abbreviated Terms, Acronyms, and Initialisms*

3.3.1 *HPLC, n*—high performance liquid chromatography

3.3.2 *ICS, n*—independent check standard

3.3.3 *ISWS, n*—internal standard working solution

3.3.4 *LOD, n*—limit of detection

3.3.5 *LOQ, n*—limit of quantitation

3.3.6 *MCS, n*—master calibration standard

3.3.7 *MS, n*—mass spectrometer

3.3.8 *MS/MS, n*—tandem mass spectrometry

3.3.9 *QMS, n*—quality management system

3.3.10 *SPE, n*—solid phase extraction

3.3.11 *SRM, n*—selective reaction monitoring

4. Summary of Test Method

4.1 The quantitative analysis of pesticides in dried cannabis is performed by liquid-solid extraction into acetonitrile:acetic acid (100:1, v:v) followed by solid phase extraction and dilution prior to quantitative HPLC-MS/MS analysis.

4.2 Pesticides are identified by retention time and by selective reaction monitoring (SRM) transitions. An SRM transition consists of a pseudo-molecular ion, selected in quadrupole one, and a product ion, selected in quadrupole three. Pseudo-molecular ions are fragmented to product ions in quadrupole two (collision cell). The product ion selected in quadrupole three is transmitted to the detector of the mass spectrometer to produce a signal, resulting in a peak for the pesticide in the chromatogram. Pesticides are quantitated using the designated quantitative SRM transition. The final result reported for each sample lists the concentration in cannabis.

5. Significance and Use

5.1 The analysis and reporting of pesticide content in all forms of cannabis raw material that is grown or processed or both, with the intent of ingestion or consumption, is required to address health and safety concerns, satisfy testing and labeling requirements, and meet the regulatory guidelines of various jurisdictions where cannabis has been legalized for ingestion or consumption for medicinal or recreational purposes, or both. This test method is useful in providing quantitative results for the analytes listed in Table 1, which is a subset of the pesticide testing requirements found in regulatory documents for cannabis, not limited to and including Canada, many U.S. states where legalization has occurred, and the European Pharmacopeia (**1-4**). This test method may be appropriate for additional pesticide and growth regulator analytes, which may be added to those of Table 1 provided validation is performed in accordance with Practice D8282. Analytes may be removed from Table 1 without additional validation.

6. Interferences

6.1 Contaminants present in solvents, reagents, glassware, and other apparatus or co-extracted from matrix, producing discrete artifacts or elevated baselines, have the potential to cause method interferences. All of these materials are demonstrated to be free from interferences by analyzing blank matrix samples under the same conditions as samples. A blank sample is used to evaluate potential interferences for the internal standards while a blank-0 sample is used to evaluate potential interferences for the analytes. A blank-S sample is used to evaluate potential interferences caused by LC contamination or analyte carryover.

6.2 Contaminants that are co-extracted from the sample have the potential to cause method interferences. The extent of matrix interferences can vary considerably from sample source depending on variations of the sample matrix.

7. Apparatus

7.1 *Analytical Balance*—Any analytical balance capable of readability down to 0.01 mg.

7.2 *Grinder/Homogenizer*—Any grinder capable of grinding dried cannabis raw materials to a powder form.

7.2.1 Cryogenic grinding is the preferable technique to provide a homogeneous, ground sample.

7.3 *Solvent Dispenser*—Any solvent dispenser capable of dispensing 5 mL ± 0.1 mL.

7.4 *Multi-tube Vortexer*—Any vortex mixer capable of mixing multiple 15 mL tubes at high speed.

7.5 *Centrifuge*—Any centrifuge capable of holding 15 mL tubes and operating at 5000 r/min ± 500 r/min (4700 RCF ± 470 RCF).

7.6 *LC-MS/MS System:*

7.6.1 *Liquid Chromatography (LC) System*—A complete LC system, including gradient pump, temperature-controlled autosampler, and column heater is required in order to analyze samples. Any LC that is capable of performing at the flows, pressures, controlled temperatures, sample volumes, and requirements of the standard shall be considered suitable for use.

7.6.2 *Tandem Mass Spectrometer (MS/MS) System*—A MS/MS system capable of selective reactive monitoring (SRM) analysis shall be considered suitable for use.

7.7 *Analytical Column*—Any column (Note 3) that achieves adequate resolution and results may be used.

7.8 *Tri-mode (WCX, WAX, RP) Guard Column* (See Note 3.)

NOTE 3—A reversed-phase analytical column, 2.1 mm × 100 mm, 2.2 µm, 120 Å, containing a polar embedded stationary phase, and a reversed-phase guard column (2.1 mm × 10 mm, 5 µm, 120 Å), containing embedded weak anion and cation exchange groups in the stationary phase were used to develop this method.

7.9 *Vortex Mixer*—Any vortex mixer capable of vortex mixing 15 mL tubes at high speed.

8. Reagents and Materials

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D1193.

8.3 *Acetic Acid,* LC-MS grade, or equivalent.

8.4 *Acetonitrile,* LC-MS grade, or equivalent.

8.5 *Ammonium Formate,* LC-MS grade, or equivalent.

8.6 *Dried cannabis raw material*.

8.7 *Formic Acid,* LC-MS grade, or equivalent.

8.8 *Methanol,* LC-MS grade, or equivalent.

8.9 *Pesticides Reference Material Solution,* 96 components in acetonitrile, or equivalent.

8.10 *C18,* SPE cartridges.

8.11 *Autosampler Vials,* amber silanized glass, 2 mL.

9. Hazards

9.1 All work with solvents shall be carried out in a fume hood while personal protection equipment is worn, including gloves, safety glasses or goggles, and a lab coat.

9.2 Several solvents are used in this test method, including methanol and acetonitrile. Check their safety datasheet to identify specific hazards. See Guide D8245. Follow local regulations for proper disposal of spent chemicals.

10. Calibration and Standardization

10.1 The mass spectrometer shall be calibrated per manufacturer specifications before analysis. In order to obtain valid and accurate analytical values within the confidence limits, the following procedures shall be followed when performing the test method.

10.2 *Calibration and Standardization:*

10.2.1 This test method uses calibration samples (CAL) and independent check samples (ICS) prepared in a blend of high-THCA, balanced THCA-CBDA, and high-CBDA dried cannabis and subsequently extracted into solution.

10.2.1.1 The blended cannabis is designed to include high levels of naturally occurring cannabinoids from multiple cultivars, resulting in a sample that is as close to the average cannabis sample as possible.

10.2.2 Seven (7) calibration levels and three (3) ICS levels shall be prepared, each containing the analytes listed in Table 1. Additional calibration and ICS levels may be added as deemed necessary. Stock solutions are typically available as 100 µg/mL to 1000 µg/mL in either methanol or acetonitrile and may contain 0.1 % formic acid. Prepare an appropriate volume of two (2) master calibration standard (MCS) solutions in amber, silanized glass vials by combining the analytical reference standards and diluting to a concentration of 8.33 µg/mL with acetonitrile. The two MCS solutions shall be prepared using stocks from different suppliers, different lots, or different vials/ampules. One MCS solution is to be used for preparation of the CAL solutions and the other for preparation of the ICS solutions. MCS solutions with fewer analytes may be prepared provided that the analyte concentrations remain the same. Pesticide reference standard solutions shall be stored according to the manufacturers' instructions and used prior to

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

the expiration date stated by the manufacturer. MCS solutions, CAL solutions, and ICS solutions shall be stored at –20 °C or lower and replaced every six (6) months.

10.2.3 Prepare spiking solutions by diluting the MCS solution in amber, silanized glass vials as described in Table 2, or equivalent.

10.2.4 Prepare individual internal standard stock solutions for daminozide-d_4 (IS-1), myclobutanil-d_4 (IS-2), diazinon-d_5 (IS-3), piperonylbutoxide-d_9 (IS-4), and deltamethrin-d_5 (IS-5) at 100 µg/mL by weighing 0.1 mg of each internal standard to a vial and dissolving in 1.0 mL of acetonitrile.

10.2.5 Prepare an internal standard working solution (ISWS) at a concentration of 1.0 µg/mL of each internal standard by combining 200 µL of each of the internal stock solutions and diluting to a total volume of 20.0 mL with acetonitrile. Final volumes may be changed provided the proportions remain the same.

10.2.6 Prepare matrix-matched calibration standards in 15 mL centrifuge tubes by spiking 1.00 g ± 0.05 g of dried and ground cannabis known to be free of pesticides with the appropriate spiking solution, or equivalent, as described in Table 3. The spiked cannabis is extracted alongside unknown cannabis samples by following the procedure detailed in Section 11.

10.2.7 After completing the extractions detailed in Section 11, inject each CAL to obtain the chromatograms, monitoring the SRM transitions of each analyte and its internal standard. Calibration software is used to conduct quantitation of the target analytes with SRM transitions of each analyte used for quantitation and confirmation.

10.2.8 The calibration software manual should be consulted to use the software properly. The quantitative method uses peak area ratios of analyte/internal standard versus the analyte concentration in units of ng/g (ng of pesticide per g of dried cannabis). Regressions (that is, linear or quadratic depending on the instrument used) may be generated using the data system software. Regression type used shall be validated by each laboratory. Forcing the regression line through the origin is not recommended. Each CAL used to generate the regression shall have a calculated concentration ≤25 % bias (≤40 % for CAL 1) from the nominal concentration and may be rejected if this criteria is not met.

10.2.9 Linear calibration may be used if the coefficient of determination, r^2, is ≥0.99. A weighting of 1/x or $1/x^2$ is recommended to give more emphasis to the lower concentrations. Regression type used shall be validated by each laboratory. A minimum of five (5) points is considered acceptable for each analyte. If the low or high CAL point are rejected, the reporting range shall be modified to reflect this change (Note 4). The linearity of group-calibrated analytes shall be evaluated on the group calibration rather than the calibration of the individual components comprising the group.

10.2.10 Quadratic calibration may be used if the coefficient of determination, r^2, is ≥0.99. A weighting of 1/x or $1/x^2$ is recommended to give more emphasis to the lower concentrations. Regression type used shall be validated by each laboratory. A minimum of five (5) points is considered acceptable for each analyte. If the low or high CAL point is rejected, the reporting range shall be modified to reflect this change (Note 4).

NOTE 4—Certain jurisdictions or regulations may prohibit the rejection of calibration points and those regulations will take precedence.

10.2.11 The retention time window of the SRM transitions shall be within ±5 % of the retention time of the analyte in a mid-point CAL sample. If this is not the case, re-examine the CAL samples to determine if there was a shift in retention time during the analysis. If a retention time shift occurred, the sample shall be re-injected. If the retention time is still incorrect in the sample, refer to the peak as an unknown.

10.2.12 ICS samples shall be injected at the beginning, middle, and end of the run, including ICS samples injected a minimum of every 10 samples. The concentration of the ICS samples, at concentrations above the LOQ for each analyte, shall have a bias ≤30 % of the nominal concentration for a given analyte. If this is not the case, any unknown samples displaying a signal for the biased analyte injected between accepted ICS injections shall be re-analyzed. The ICS with the area count nearest the unknown sample shall be used to evaluate this criterion.

10.3 *Method Blanks:*

10.3.1 A blank sample shall be injected at least once in the run. Any peak in the blank sample at the retention time and SRM transitions of the internal standards shall have a peak area ≤4 0% of the average of the internal standard peak areas of the CAL samples.

10.3.2 A blank-0 sample shall be injected at least once in the run. Any peak in the blank-0 sample at the retention time and SRM transition of the analyte shall have a concentration ≤40 % of the LOQ for that analyte.

TABLE 2 Spiking Solution Preparation for Matrix-matched Calibration

NOTE 1—Final volumes may be changed provided the proportions remain the same.

Spiking Solution	Solution Used	Volume of Solution (µL)	Volume of ACN (µL)	Final Volume (µL)	Conc. (µg/mL)
Spike 1	MCS-1	10	990	1000	0.083
Spike 2	MCS-1	20	980	1000	0.17
Spike 3	MCS-1	40	960	1000	0.33
Spike 4	MCS-2	50	950	1000	0.42
Spike 5	MCS-1	100	900	1000	0.83
Spike 6	MCS-2	150	850	1000	1.25
Spike 7	MCS-1	200	800	1000	1.67
Spike 8	MCS-1	400	600	1000	3.33
Spike 9	MCS-2	500	500	1000	4.17
Spike 10	MCS-1	1000	0	1000	8.33

TABLE 3 Analyte Concentrations in Matrix-matched Calibration Curve

NOTE 1—Final volumes may be changed provided the proportions remain the same.

Cal Level	Spiking Solution Used	Spiking Solution Conc. (µg/mL)	Volume Used (µL)	Pesticides (µg/mL)
Blank	Spike 0	0.00	60	0.000
CAL 5	Spike 1	0.08	60	0.005
CAL 10	Spike 2	0.17	60	0.010
CAL 20	Spike 3	0.33	60	0.020
CAL 50	Spike 5	0.83	60	0.050
CAL 100	Spike 7	1.67	60	0.100
CAL 200	Spike 8	3.33	60	0.200
CAL 500	Spike 10	8.33	60	0.500
ICS 25	Spike 4	0.42	60	0.025
ICS 75	Spike 6	1.25	60	0.075
ICS 250	Spike 9	4.17	60	0.250

10.3.3 A blank-S sample shall be injected a minimum of every 10 samples. Any peak in the blank-S sample at the retention time and SRM transition of the internal standards or analytes shall have an area count ≤40 % of CAL-1.

10.4 If a laboratory has not performed the test before or if there has been a major change in the measurement system, for example: a new analyst or new equipment, perform a precision and bias study to demonstrate laboratory capability.

10.4.1 Analyze at least four (4) replicates of the ICS samples. The sample shall be taken through the complete analytical test method. Calculate mean (average) concentration and % RSD and compare to the concentration of the ICS samples. The calculated concentration of the analytes in the ICS samples (with concentrations above the LOQ) shall have a calculated percent deviation ≤30 % and an RSD ≤30 %.

10.4.2 This study shall be repeated until a single operator precision and RSD are within the acceptance criteria.

11. Conditioning and Instrument Parameters

11.1 Analyze using a tandem mass spectrometer (MS/MS) coupled to a high performance liquid chromatography (HPLC) system.

11.2 Introduce sample using an autosampler and achieve analyte separation on an appropriate reverse phase column (Note 5). Equilibrate the instrument by injecting a minimum of three blank-S samples. See Tables 4-6 for additional instrument parameters. Parameters in Tables 4-6 and RF lens parameter in Table 7 are examples only and may be different in name, number and setting for various instruments. Parameters should be optimized for specific LC-MS systems.

TABLE 4 HPLC Conditions

Parameter	Setting
Column	C18 2.2 µm, 120 Å, 2.1 mm x 100 mm
Guard Column	Tri-mode (WCX, WAX, RP) 5 µm, 2.1 mm x 10 mm
Mobile Phase A	3 mM Ammonium formate in water + 0.1 % formic acid
Mobile Phase B	3 mM Ammonium formate in methanol + 0.02 % formic acid
Flow Rate (mL/min)	0.5
Run Time (min)	16
Column Temp. (°C)	30
Switch Valve Times (min)	0-0.3 min to waste, 0.3-11.5 min to the mass spectrometer, 11.5-16 min to waste
Injection Volume (µL)	1.0
Needle Wash	ACN:MeOH:Water:formic acid (40:40:20:1, v:v:v:v)
AS Temperature (°C)	5 °C

TABLE 5 HPLC Gradient Conditions

Time (min)	Flow (mL/min)	%B
0.000	0.5	20
5.500	0.5	75
10.500	0.5	98
13.000	0.5	98
13.100	0.5	20
16.000	0.5	20

TABLE 6 MS Ion Parameters

Parameter	Setting
Scan Type	MRM
Ion Source	Heated Electrospray
Polarity	Positive/Negative
Ion Spray Voltage (V)	4000/3000
Sheath Gas (arbitrary units)	70
Aux Gas (arbitrary units)	25
Sweep Gas (arbitrary units)	2
Ion Transfer Tube Temperature (°C)	310
Vaporizer Temperature (°C)	150
RF Lens	Analyte Specific
Collision Gas (mTorr)	2.0
Cycle Time (s)	0.4

NOTE 5—A C18, 2.2 µm, 120 Å, 2.1 mm × 100 mm HPLC column fitted with a tri-mode (WCX, WAX, RP) 5 µm, 2.1 mm × 10 mm guard column was used with the gradient described in Table 5 to develop this test method.

11.3 Equilibrate the instrument by injecting a minimum of two blank solution samples. Blank solutions consist of acetonitrile:acetic acid, (100:1, v:v) diluted 2-fold in a 20:80 mixture of mobile phase A and mobile phase B, or equivalent.

11.4 Table 6 illustrates the Mass Spectrometer Parameters.

11.5 Table 7 illustrates the required SRM transitions used for pesticides. Bold entries indicate transitions used for quantitation, while non-bold entries indicate transitions used for confirmation.

12. Procedure

12.1 Record all sample information in conformance within the requirements of the existing lab management practices as defined within your quality management system (QMS).

TABLE 7 SRM Transitions

NOTE 1—Bold entries indicate transitions used for quantitation, while non-bold entries indicate transitions used for confirmation.

NOTE 2—Retention times will vary with column and mobile phase used.

NOTE 3—Collision energy may be optimized for specific instruments used.

Compound	RT (min)	RT Window (min)	Polarity	Precursor (m/z)	Product (m/z)	CE (V)	RF Lens (V)	IS Used
Daminozide	0.61	0.5	Positive	161.1	**44.0** 61.1 128.9 142.9	**21** 12 6 10	36	IS-1
Daminozide-d_4 (IS-1)	0.61	0.5	Positive	165.1	**44.0** 45.1 62.1 147.1	**20** 22 13 12	36	NA
Acephate	0.91	0.5	Positive	184.0	124.8 **143.0**	18 **9**	30	IS-2
Dinotefuran	1.40	0.5	Positive	203.0	**113.1** 129.1	**10** 12	30	IS-2
Oxamyl (+NH4)	1.42	0.5	Positive	237.1	**72.1** 90.1	**12** 9	30	IS-2
Methomyl	1.81	0.5	Positive	162.9	**88.0** 106.1	**9** 10	30	IS-2
Flonicamid	2.07	0.5	Positive	230.1	174.0 **203.1**	17 **16**	41	IS-2
Thiamethoxam	2.20	0.5	Positive	292.0	181.0 **211.1**	23 **12**	52	IS-2
Mevinphos Iso 1	2.55	0.3	Positive	225.0	**127.0** 193.0	**10** 8	31	IS-2
Pirimicarb	2.70	0.5	Positive	239.1	**72.1** 182.1	**21** 16	47	IS-2
Imidacloprid	2.80	0.5	Positive	256.1	175.1 **209.1**	19 **16**	41	IS-2
Dimethoate	3.08	0.5	Positive	230.0	124.9 **199.0**	21 **11**	30	IS-2
Clothianidin	3.31	0.5	Positive	250.0	112.9 **131.9**	27 **17**	34	IS-2
Acetamiprid	3.32	0.5	Positive	223.1	55.9 **125.9**	16 **20**	47	IS-2
Mevinphos Iso 2	3.23	0.3	Positive	225.0	**127.0** 193.0	**17** 8	31	IS-2
Aldicarb (+NH4)	3.6	0.5	Positive	208.1	89.0 **116.0**	17 **10**	68	IS-2
Propoxur	4.6	2	Positive	210.1	**111.0** 168.1	**14** 8	30	IS-2
Dodemorph	4.8	2	Positive	282.2	98.1 **116.1**	27 **21**	52	IS-2
Thiacloprid	4.1	0.5	Positive	253.1	90.1 **125.9**	36 **21**	56	IS-2
Dichlorvos	4.2	0.5	Positive	220.9	**109.0** 145.0	**18** 14	86	IS-2
Imazalil	4.49	0.8	Positive	297.1	**159.0** 255.1	**22** 18	64	IS-2
Carbofuran	4.36	0.5	Positive	222.1	123.0 **165.1**	21 **12**	32	IS-2

TABLE 7 *Continued*

Compound	RT (min)	RT Window (min)	Polarity	Precursor (m/z)	Product (m/z)	CE (V)	RF Lens (V)	IS Used
Spiroxamine	5.2	2	Positive	298.3	100.2 **144.2**	30 **20**	48	IS-2
Thiophanate Methyl	4.63	0.5	Positive	343.0	**151.0** 268.0	**20** 10	47	IS-2
Metalaxyl	4.7	0.5	Positive	280.1	192.1 **220.1**	20 **15**	50	IS-2
Azoxystrobin	5.5	2	Positive	404.1	344.1 **372.1**	25 **13**	49	IS-2
Carbaryl	4.85	0.5	Positive	202.1	127.0 **145.0**	28 **11**	30	IS-2
Chlorantraniliprole	5.6	2	Positive	481.9	**283.9** 450.9	**13** 17	54	IS-2
Azadirachtin	4.85	0.5	Positive	703.2	**585.2** 685.2	**14** 10	77	IS-2
Cyantraniliprole	5	0.5	Positive	475.0	**285.9** 443.9	**14** 19	70	IS-2
Fensulfothion	5.1	0.5	Positive	309.0	235.0 **280.9**	22 **15**	58	IS-2
Naled	5.2	0.5	Positive	**380.8** 382.8	**127.0** 127.0	**18** 15	72	IS-2
Dimethomorph	5.6	1	Positive	388.0	165.2 **301.0**	35 **25**	76	IS-2
Phosmet	5.75	1	Positive	**318.0** 335.0	**160.0** 160.0	**10** 20	77	IS-2
Ethoprophos	5.8	1	Positive	243.1	96.9 **130.9**	32 **20**	45	IS-2
Iprodione	5.8	1	Positive	330.1	**216.1** 298.1	**35** 22	103	IS-2
Spirotetramat	5.8	1	Positive	374.2	**216.1** 330.2	**34** 16	55	IS-2
Malathion	5.82	1	Positive	331.0	**127.0** 284.9	**12** 10	63	IS-2
Methiocarb	5.83	1	Positive	226.1	121.0 **169.1**	19 **10**	36	IS-2
Paclobutrazol	5.88	1	Positive	294.1	**70.1** 125.0	**22** 33	52	IS-2
Myclobutanil	5.97	1	Positive	289.1	**70.0** 125.0	**18** 31	62	IS-2
Myclobutanil-d_4 (IS-2)	5.97	1	Positive	293.1	**70.0** 129.0	**18** 31	62	NA
Boscalid	6	1	Positive	343.1	272.1 **307.1**	31 **19**	71	IS-2
Bifenazate	6.1	1	Positive	301.2	170.1 **198.1**	21 **8**	34	IS-3
Fluopyram	6.1	1	Positive	397.0	145.0 **208.0**	51 **22**	70	IS-3
Tetrachlorvinphos	6.32	1	Positive	365.0	**127.0** 203.8	**13** 38	73	IS-3
Kresoxim Methyl	6.37	1	Positive	314.1	235.1 **267.1**	16 **7**	30	IS-3