Application Note 10361

Analysis of Multi-Residue Pesticides Present in Ayurvedic Churna by GC-MS/MS

Manoj Surwade[1], Sunil T Kumar[1], Aarti Karkhanis[1], Manish Kumar[1], Soma Dasgupta[1], Hans-Joachim Huebschmann[2],
[1]Thermo Fisher Scientific, Mumbai, India; [2]Thermo Fisher Scientific, Singapore

Keywords: Traditional herbal medicine, fast liquid/liquid extraction, QuEChERS, timed-SRM, retention time synchronization, MRM, ion ratio confirmation, TraceFinder data processing

Introduction

Ayurveda is a Sanskrit term, made up of the words "ayus" and "veda. "meaning life and science; together translating to 'science of life'. A blend of several herbs and spices make up the powdered mixture known as "churna". Depending on its intended use for medicinal, beauty, or culinary purpose, the recipe varies. Avipittakara "churna" is a traditional Ayurvedic formula used widely and almost daily to control vitiated pitta dosha, remove heat in the digestive system, control indigestion, constipation, vomiting and anorexia. A major analytical challenge for these types of samples is mainly addition of multiple herbs with sugar and the natural color of herbs[1].

The dried leaves result in highly complex extracts from the sample preparation due to the rich content of active ingredients, essential oils and the typical high boiling natural polymer compounds. Due to the use of pesticides in the fresh herbs, the "churna" may contain residual pesticides. Analysis of pesticide residues is thus important and governmentally regulated[2]. Strict quality parameters have been mented to preserve the quality and efficacy of these "churnas".

Sample Preparation

In brief, the QuEChERS sample preparation (see Figure 1) involved the extraction of 15 g of a powder sample of Avipittakara "churna" with 15 mL acetonitrile (containing 1% acetic acid) in the presence of 3 g magnesium sulfate, 1.5 g sodium acetate and 1 g NaCl. The supernatant (1 mL) was collected after centrifugation, and dispersive cleanup was performed using 200 mg PSA and 10 mg GCB. The extract was centrifuged at 10 000 rpm for 5 min, and 3 µL of supernatant was

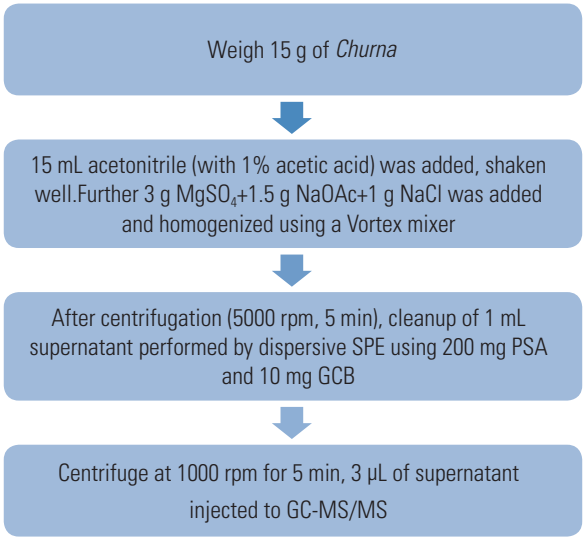

Figure 1. Sample preparation for extraction of pesticides from ayurvedic churnas

Thermo SCIENTIFIC

injected via autosampler for analysis. For recovery and validation studies 15 g of the "churna" was fortified with appropriate quantities of the pesticide standard mixture.

Calibration

Stock standard solutions of each pesticide compound were prepared by weighing 10±0.1 mg, dissolving in 10 mL acetonitrile and storing the solution in amber-colored glass vials at -20°C . A total of ten intermediate mixtures (each containing 15-20 compounds) of 10 mg/L concentration were prepared by diluting an adequate quantity of each compound in acetonitrile. A working standard solution (1 mg/L) was prepared by mixing an adequate quantity of intermediate standard solution and dilution with acetonitrile and storing the solution at -20°C . The calibration standards at 2.5, 5, 10, 25 and 50 µg/L were freshly prepared for measurement of the calibration curves. The calibration graphs (five points) for all the compounds were obtained by plotting the individual peak areas against the concentration of the corresponding calibration standards.

Instrument and Method Setup

The analytical method comprises the sample handling using the Thermo Scientific™ TriPlus™ RSH liquid auto sampler, the Thermo Scientific™ TRACE™ 1300 Series gas chromatograph equipped with a temperature programmable PTV injector, and the Thermo Scientific™ TSQ 8000™ triple quadrupole GC-MS/MS system. The instrument method parameters are summarized in Table 1.

TABLE 1. Instrument method parameters.

TRACE™ 1310 Gas Chromatograph Parameters	
Carrier gas	Helium
Injector	PTV
Mode	splitless
Splitless time	3 min, split flow: 30 mL/min
PTV program	87 °C , 0.3 min (injection) 14.5 °C/min to 285 °C (transfer) 285 °C , 2.5 min (transfer) 14.5 °C/min to 290 °C (cleaning) 290 °C , 20 min (cleaning)
Column	Thermo Scientific TraceGOLD™ TG-5 SilMS, 30 m x 0.25 mm x 0.25 µm (p/n 10177894)
Column flow	1.2 mL/min, constant flow
Oven program	70 °C , 2 min 10 °C/min to 200 °C 200 °C , 1 min 10 °C/min to 28 °C 285 °C , 8.5 min
Injection	3 µL by TriPlus RSH Autosampler

The Thermo Scientific™ TraceFinder™ software was used for method setup and data processing. The TraceFinder software provides a compound database of pesticides compoundsof more than 800 compounds with all required analytical details such as retention times and the optimized SRM transitions for data acquisition and processing. These software features were employed to create the processing method for the screening a large pesticides compound list [2].

For all pesticide compounds two SRM transitions were chosen for the overall MRM acquisition method. The first transition was used for quantitation, the second transition for confirmation by checking the ion intensity ratio by the TraceFinder software during data processing. Retention times had been synchronized between data processing of standards with the acquisition method for the timed-SRM protocol (see Figure 2) in order to lock all compound retention times for robustness independent on the impact of the matrix carried by real life sample.

TSQ-8000 MS/MS Parameters	
Ion source temperature	230 °C
Interface temperature	285 °C
Acquisition mode	EI, 70 eV
MRM detection	Timed SRM mode (see Figure 1)
Acquisition rate	500 ms
MRM parameter	See Table 1

The timed-SRM acquisition method used with the TSQ 8000 MS avoids the laborious and time-consuming process of segment creation and method maintenance.The scan times are automatically calculated based upon the specified cycle time so that uniform cycle times are obtained for each mass transition, thus reducing the extensive optimization process for scan times and data points across a peak. The dwell times for data acquisition are maximized independently for the number of compounds in the MRM method. Table 2 lists the MRM parameters for the compounds analyzed in this method.

The data processing and reporting was done using the quantitation and reporting suite. The software allows retention time locking by synchronization between the data processing and the acquisition setup for all compounds in the method.

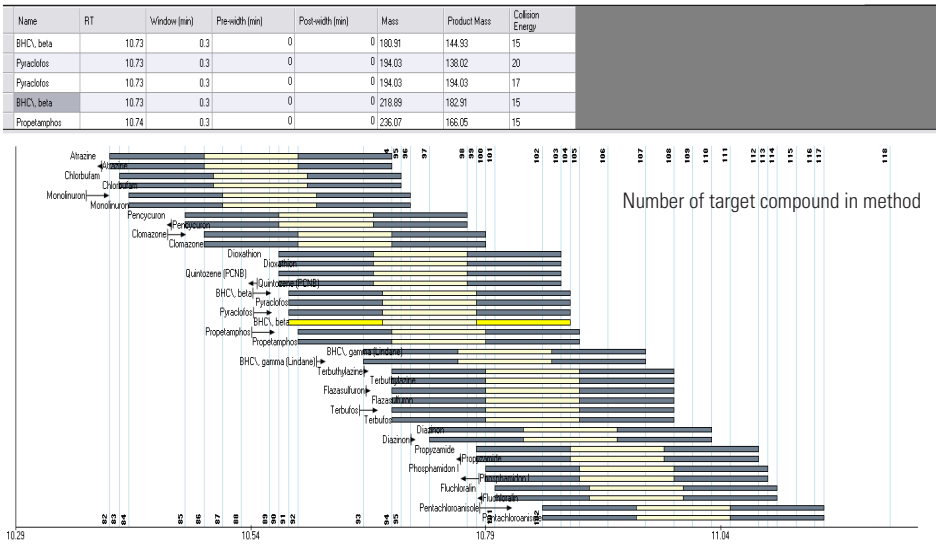

FIGURE 2. Principle of the timed-SRM acquisition setup of the TSQ 8000. The white center parts show the peak width centered to the compound retention time, the grey areas before and after the peak the full SRM acquisition window of 0.3 min.

Results

The multi-residue pesticide analysis of Ayurvedic churnas for routine target analytes detection and quantitationis described using liquid-liquid extraction and GC-MS/MS detection with the TSQ 8000 GC-MS/MS system. All standards and samples were processed using TraceFinder software with high speed and throughput.

All compounds included into this method had very good calibration correlation coefficients of > 0.99 for the concentration range of 2.5 to 50 ng/g, as shown Figure 3. The obtained recoveries were high within 70-120% with < 20% associated RSDs.

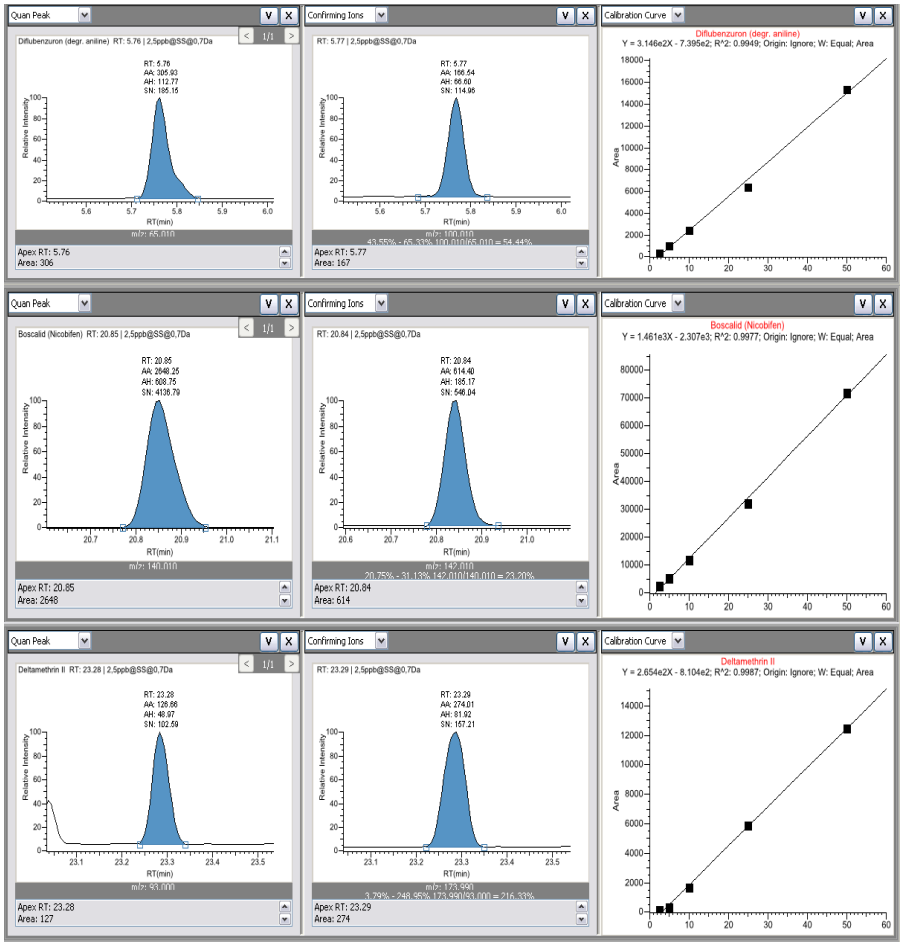

FIGURE 3. Selected pesticide chromatograms at 2.5 ng/g and their calibration curves.

Sample Analysis

Approximately 200 pesticide compounds were included in a routine screening method with an approximately 28 min total run time. The method setup as described above was applied for analyzing samples bought from the regional market.The results from analysis of market samples are presented in Figure 4.

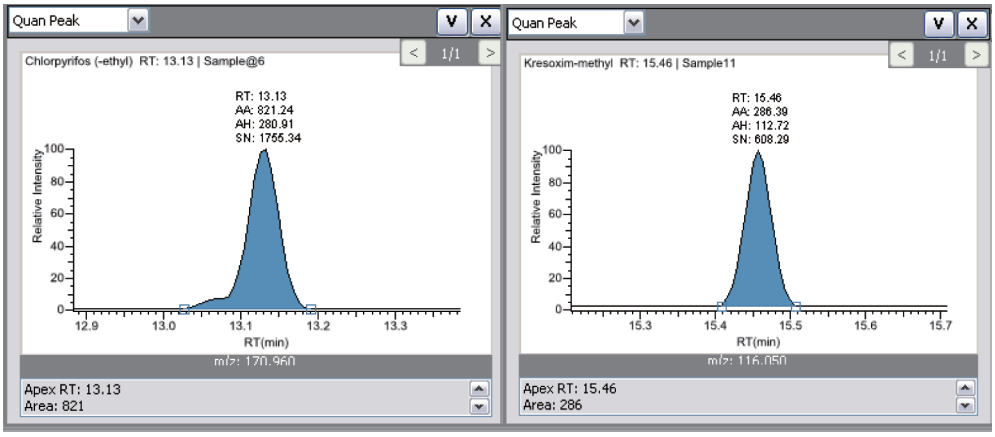

FIGURE 4. Traces of Chlorpyrifos ethyl and Kresoxim methyl were detected at 2.3 and 2.7 µg/kg respectively in regional market samples.

Conclusion

A rapid and sensitive quantitative method for a large number of compounds is always a major goal for analytical laboratories involved in pesticide analysis. Within 28 minutes, 200 pesticides were screened and quantitatively determined using the described pesticide analysis method. The QuEChERS sample preparation method provided high recoveries and good reproducibility. The generic TRACE TR-5MS column coupled with TRACEGuard provided good chromatographic resolution of the pesticides studied. The triple quadrupole mass analyzer TSQ 8000 GC-MS/MS system with TraceFinder™ software was used for data processing to reduce the processing time, thereby resulting in a high throughput method space missing. Linearity, specificity, recovery, and repeatability of the method were established with minimal sample preparation time. The TSQ 8000 system provided very high selectivity for the sensitive detection and reliable quantitation of the pesticides even from these samples with a high matrix load from the short QuEChERS sample preparation.

This method can be utilized for detection and confirmation of trace amounts of pesticides in difficult matrices such as herbal churnas. The method has potential to detect trace level compounds at concentration as low as 2.5 ng/g. As per the available guidelines, the concentration of the detected pesticides (0.0023 and 0.0027 mg/kg) were below the required limits of the Unani Guidelines [3].

References

[1] Narayanaswamy, V., Origin and Development of Ayurveda (A Brief History), Anc. Sci. Life, Jul-Sep 1(1) (1981) 1–7.

[2] The Pesticides Compound Database, Thermo Fisher Scientific, Austin, TX, USA, 2013.

[3] Lohar, D.R., Protocol for testing guideline for Ayurvedic, Siddha and Unani medicines Chapter 2.5.1, Government of India, Department of AYUSH, Ministry of Health & Family Welfare, Pharmacopoeial Laboratory for Indian Medicines, Ghaziabad, see http://www.plimism.nic.in/Protocol_For_Testing.pdf

Table 2. MRM parameter for the pesticide compounds analyzed.

Nr.	Compound Name	RT [min]	Quantitation *m/z*	CE [V]	Confirmation *m/z*	CE [V]	R^2
1.	Diflubenzuron (degr. i-cyanat	5.24	153.02 > 90.01	20	153.02 > 125.01	20	0.9969
2.	Diflubenzuron (degr. aniline)	5.75	127.01 > 65.01	30	127.01 > 100.01	30	0.9949
3.	Methamidophos	5.87	141.00 > 95.00	10	141.00 > 126.00	5	0.9930
4.	Dichlorphos (DDVP)	5.94	184.95 > 92.98	17	219.95 > 184.95	10	0.9960
5.	Dichlobenil	6.82	135.97 > 99.98	10	170.96 > 135.97	15	0.9960
6.	Mevinphos	7.39	127.03 > 109.02	10	192.04 > 127.03	12	0.9964
7.	Acephate	7.50	136.01 > 42.00	10	136.01 > 94.01	15	0.9904
8.	Dichloraniline, 3,5-	7.61	160.98 > 89.99	25	160.98 > 98.99	25	0.9989
9.	Molinate (Ordram)	8.58	126.07 > 55.03	10	187.10 > 126.07	10	0.9941
10.	TEPP	8.60	263.06 > 179.04	15	263.06 > 235.06	5	0.9946
11.	Omethoate	9.00	110.01 > 79.01	15	156.02 > 110.01	10	0.9969
12.	Fenobucarb	9.11	121.07 > 77.05	15	150.09 > 121.07	10	0.9977

Nr.	Compound Name	RT [min]	Quantitation *m/z*	CE [V]	Confirmation *m/z*	CE [V]	R^2
13.	Propoxur	9.13	110.06 > 64.03	10	152.08 > 110.06	10	0.9981
14.	Propachlor	9.16	176.06 > 120.04	10	196.07 > 120.04	10	0.9980
15.	Ethoprophos	9.38	158.00 > 80.90	15	158.00 > 114.00	5	0.9949
16.	Trifluralin	9.58	264.09 > 160.05	15	306.10 > 264.09	15	0.9944
17.	Chlorpropham	9.62	213.00 > 127.00	5	213.00 > 171.00	5	0.9981
18.	Benfluralin	9.63	292.10 > 160.05	21	292.10 > 264.09	10	0.9923
19.	Sulfotep	9.70	322.02 > 202.01	15	322.02 > 294.02	10	0.9943
20.	Bendiocarb	9.72	166.06 > 151.06	15	166.06 > 166.06	15	0.9996
21.	Monocrotophos	9.80	127.03 > 95.03	20	127.03 > 109.03	25	0.9971
22.	Methabenzthiazuron	9.82	164.05 > 136.04	12	164.05 > 164.05	10	0.9974
23.	BHC, alpha	10.15	180.91 > 144.93	15	218.89 > 182.91	15	0.9970
24.	Metamitron	10.36	202.09 > 174.07	5	202.09 > 186.08	10	0.9969
25.	Atrazine	10.54	215.09 > 173.08	10	215.09 > 200.09	10	0.9945
26.	Pencycuron	10.62	125.05 > 89.04	12	180.07 > 125.05	12	0.9914
27.	Dioxathion	10.72	125.00 > 97.00	15	125.00 > 141.00	15	0.9936
28.	BHC, beta	10.73	180.91 > 144.93	15	218.89 > 182.91	15	0.9933
29.	Propetamphos	10.74	236.07 > 166.05	15	236.07 > 194.06	5	0.9918
30.	BHC, gamma (Lindane)	10.81	180.91 > 144.93	15	218.89 > 180.91	5	0.9939
31.	Terbuthylazine	10.84	214.10 > 132.06	10	229.11 > 173.08	10	0.9935
32.	Diazinon	10.88	137.05 > 84.03	10	304.10 > 179.06	15	0.9987
33.	Propyzamide	10.93	173.01 > 145.01	15	175.02 > 147.01	15	0.9939
34.	Fluchloralin	10.95	264.04 > 206.03	10	306.05 > 264.04	10	0.9967
35.	Pyroquilon	11.07	173.08 > 130.06	20	173.08 > 145.07	20	0.9974
36.	Pyrimethanil	11.11	198.11 > 158.09	30	198.11 > 183.10	15	0.9953
37.	Tefluthrin	11.16	177.02 > 127.02	20	197.03 > 141.02	15	0.9991
38.	Etrimfos	11.29	292.06 > 153.03	10	292.06 > 181.04	10	0.9935
39.	Pirimicarb	11.50	166.10 > 96.06	10	238.14 > 166.10	15	0.9937
40.	BHC, delta	11.54	180.91 > 144.93	15	204.07 > 91.03	15	0.9949
41.	Iprobenfos	11.54	204.07 > 122.04	15	218.89 > 182.91	15	0.9997
42.	Formothion	11.74	126.00 > 93.00	8	172.00 > 93.00	5	0.9982
43.	Phosphamidon II	11.83	227.05 > 127.03	15	264.06 > 193.04	15	0.9977
44.	Dichlofenthion	11.90	222.98 > 204.98	10	278.97 > 222.98	15	0.9946
45.	Dimethachlor	11.94	197.08 > 148.06	10	199.08 > 148.06	10	0.9992
46.	Dimethenamid	11.95	230.06 > 154.04	10	232.06 > 154.04	10	0.9953
47.	Propazine	12.02	214.09 > 172.08	12	214.09 > 214.09	10	0.9970
48.	Propanil	12.06	217.01 > 161.00	10	219.01 > 163.00	10	0.9934
49.	Malaoxon	12.07	127.02 > 99.02	10	127.02 > 109.02	20	0.9978
50.	Chlorpyrifos-methyl	12.08	124.96 > 78.97	10	285.91 > 92.97	20	0.9945
51.	Metribuzin	12.13	198.08 > 82.03	20	198.08 > 110.05	20	0.9997
52.	Spiroxamine I	12.15	100.09 > 58.05	15	100.09 > 72.06	15	0.9909
53.	Vinclozolin	12.16	212.00 > 172.00	15	285.00 > 212.00	15	0.9957
54.	Carbofuran, 3-Hydroxy	12.21	137.06 > 81.03	18	180.08 > 137.06	15	0.9974
55.	Parathion-methyl	12.22	263.00 > 109.00	15	263.00 > 246.00	15	0.9966
56.	Alachlor	12.23	161.07 > 146.06	12	188.08 > 160.07	10	0.9997
57.	Tolclofos-methyl	12.25	264.96 > 92.99	20	264.96 > 249.96	15	0.9932
58.	Propisochlor	12.31	162.08 > 144.07	10	223.11 > 147.07	10	0.9983
59.	Metalaxyl	12.37	249.13 > 190.10	10	249.13 > 249.13	5	0.9911
60.	Carbaryl	12.41	144.06 > 115.05	20	144.06 > 116.05	20	0.9919
61.	Fuberidazol	12.41	183.80 > 156.10	10	183.80 > 183.10	20	0.9902
62.	Fenchlorfos (Ronnel)	12.47	284.91 > 269.92	13	286.91 > 271.91	20	0.9994
63.	Prosulfocarb	12.63	100.00 > 72.00	10	128.00 > 43.10	5	0.9938
64.	Pirimiphos-methyl	12.66	290.09 > 233.07	10	305.10 > 290.09	15	0.9911
65.	Spiroxamine II	12.75	100.09 > 58.05	15	100.09 > 72.06	15	0.9916
66.	Ethofumesate	12.80	207.08 > 161.06	10	277.02 > 109.01	8	0.9907
67.	Fenitrothion Confirming 1	12.80	277.02 > 260.02	10	286.11 > 207.08	12	0.9997
68.	Methiocarb	12.84	168.06 > 109.04	15	168.06 > 153.06	15	0.9971
69.	Malathion	12.92	127.01 > 99.01	10	173.02 > 127.01	10	0.9951
70.	Dichlofluanid	12.95	223.97 > 122.99	15	225.97 > 122.99	15	0.9971
71.	Phorate sulfone	13.01	153.00 > 125.00	5	199.00 > 143.00	10	0.9942

Nr.	Compound Name	RT [min]	Quantitation *m/z*	CE [V]	Confirmation *m/z*	CE [V]	R^2
72.	Dipropetryn	13.02	241.90 > 149.80	20	254.90 > 180.30	20	0.9906
73.	Chlorpyrifos (-ethyl)	13.12	198.96 > 170.96	15	313.93 > 285.94	12	0.9995
74.	Fenthionoxon	13.22	277.80 > 109.10	25	329.60 > 298.90	10	0.9927
75.	Chlorthal-dimethyl (DCPA)	13.24	300.91 > 300.91	15	331.90 > 300.91	15	0.9986
76.	Flufenacet	13.26	211.04 > 123.02	10	211.04 > 183.03	10	0.9959
77.	Endosulfan I (alpha)	13.43	240.89 > 205.91	20	264.88 > 192.91	22	0.9942
78.	Imazethapyr	13.49	201.9 > 133.00	15	252.00 > 145.90	20	0.9944
79.	Butralin	13.50	266.14 > 190.10	15	266.14 > 220.11	15	0.9996
80.	Pirimiphos (-ethyl)	13.54	304.12 > 168.06	15	333.13 > 318.12	15	0.9992
81.	Pendimethalin	13.86	252.12 > 162.08	12	252.12 > 191.09	12	0.9912
82.	Fipronil	13.87	212.97 > 177.98	16	366.95 > 212.97	25	0.9938
83.	Cyprodinil	13.91	224.13 > 208.12	20	225.13 > 210.12	18	0.9959
84.	Metazachlor	13.92	133.05 > 117.04	20	209.07 > 132.05	12	0.9939
85.	Penconazole	14.01	248.06 > 157.04	25	248.06 > 192.04	15	0.9977
86.	Tolylfluanid	14.05	137.05 > 91.03	20	238.09 > 137.05	15	0.9922
87.	Chlorfenvinphos-Z	14.05	266.98 > 158.99	15	322.97 > 266.98	15	0.9904
88.	Allethrin	14.06	123.08 > 81.05	10	136.08 > 93.06	10	0.9923
89.	Mecarbam	14.09	226.04 > 198.03	5	329.05 > 160.03	10	0.9979
90.	Phenthoate	14.18	146.01 > 118.01	10	274.03 > 246.02	10	0.9951
91.	Mephosfolan	14.20	196.02 > 140.02	15	196.02 > 168.02	10	0.9973
92.	Quinalphos	14.21	146.03 > 118.02	15	157.03 > 129.02	13	0.9943
93.	Triflumizole	14.31	179.04 > 144.04	15	206.05 > 179.04	15	0.9925
94.	Procymidone	14.31	283.02 > 96.01	15	283.02 > 255.02	10	0.9983
95.	Bromophos-ethyl	14.50	358.89 > 302.91	20	358.89 > 330.90	10	0.9985
96.	Methidathion	14.60	124.98 > 98.99	22	144.98 > 84.99	10	0.9945
97.	Chlordane, alpha (cis)	14.62	372.81 > 265.87	18	374.81 > 267.87	15	0.9967
98.	DDE, o,p	14.63	245.95 > 175.97	25	317.94 > 245.95	20	0.9946
99.	Sulfallate	14.68	188.02 > 132.02	22	188.02 > 160.02	16	0.9945
100.	Paclobutrazol	14.72	236.10 > 125.06	15	236.10 > 167.07	15	0.9926
101.	Disulfoton sulfone	14.74	213.01 > 125.01	10	213.01 > 153.01	5	0.9912
102.	Picoxystrobin	14.77	303.09 > 157.04	20	335.09 > 303.09	10	0.9937
103.	Endosulfan II (beta)	14.88	271.88 > 236.89	18	338.85 > 265.88	15	0.9973
104.	Mepanipyrim	14.89	222.11 > 207.10	15	223.11 > 208.10	15	0.9965
105.	Chlordane, gamma (trans)	14.89	372.81 > 265.87	18	374.81 > 267.87	15	0.9991
106.	Flutriafol	14.97	123.04 > 75.03	15	219.07 > 123.04	15	0.9915
107.	Napropamide	15.00	128.07 > 72.04	10	271.16 > 128.07	5	0.9972
108.	Flutolanil	15.03	173.06 > 145.05	15	173.06 > 173.06	15	0.9988
109.	Pretilachlor	15.13	162.09 > 147.08	15	216.05 > 174.04	20	0.9935
110.	Hexaconazole, confirming 1	15.13	231.06 > 175.04	10	262.14 > 202.11	15	0.9962
111.	Isoprothiolane	15.14	290.06 > 118.03	15	290.06 > 204.05	15	0.9961
112.	Profenofos	15.21	138.98 > 96.98	8	338.94 > 268.95	20	0.9939
113.	Oxadiazon	15.26	258.05 > 175.04	10	304.06 > 260.05	10	0.9927
114.	DDE, p,p	15.32	245.95 > 175.97	25	317.94 > 245.95	20	0.9964
115.	Myclobutanil	15.40	179.07 > 125.05	15	179.07 > 152.06	15	0.9912
116.	Buprofezin	15.43	172.09 > 57.03	10	249.13 > 193.10	10	0.9906
117.	Kresoxim-methyl	15.44	206.09 > 116.05	15	206.09 > 131.06	15	0.9921
118.	DDT, o,p'	15.47	234.94 > 164.96	15	234.97 > 164.98	20	0.9935
119.	DDT, o,p', confirming 1	15.47	236.94 > 164.96	20	236.97 > 164.98	20	0.9963
120.	Aramite-1	15.48	185.06 > 63.02	15	319.10 > 185.06	15	0.9959
121.	Aramite-2	15.69	185.06 > 63.02	15	319.10 > 185.06	15	0.9971
122.	Carpropamid	15.78	139.00 > 103.10	10	222.00 > 125.00	18	0.9982
123.	Cyproconazole	15.79	222.09 > 125.05	20	224.09 > 127.05	20	0.9989
124.	Nitrofen	15.85	201.99 > 138.99	21	282.98 > 252.98	15	0.9997
125.	Chlorobenzilate	15.98	251.02 > 139.01	20	253.03 > 141.01	15	0.9978
126.	Oxadiargyl	15.99	149.90 > 122.90	15	285.00 > 255.00	14	0.9963
127.	Fenthion sulfoxide	16.05	279.01 > 153.01	15	294.02 > 279.01	8	0.9958
128.	Diniconazole	16.11	268.06 > 232.05	15	270.06 > 234.05	15	0.9949
129.	Ethion	16.12	230.99 > 202.99	15	383.99 > 230.99	10	0.9973
130.	Oxadixyl	16.16	132.06 > 117.05	15	163.07 > 132.06	10	0.9985

Nr.	Compound Name	RT [min]	Quantitation *m/z*	CE [V]	Confirmation *m/z*	CE [V]	R^2
131.	DDT, p,p'	16.20	234.94 > 164.96	20	234.94 > 164.96	20	0.9979
132.	DDD, p,p'	16.20	234.97 > 164.98	20	236.97 > 164.98	20	0.9959
133.	Chlorthiophos1	16.20	324.96 > 268.97	15	324.96 > 296.97	10	0.9969
134.	Imiprothrin	16.36	123.00 > 81.00	5	324.90 > 269.20	14	0.9967
135.	Mepronil	16.45	269.14 > 119.06	10	269.14 > 210.11	10	0.9945
136.	Triazophos	16.46	161.03 > 134.03	10	257.05 > 162.03	10	0.9936
137.	Ofurace	16.58	186.05 > 158.05	10	232.07 > 186.05	10	0.9973
138.	Carfentrazone-ethyl	16.59	330.03 > 310.03	20	340.03 > 312.03	10	0.9919
139.	Benalaxyl	16.63	234.12 > 174.09	10	266.14 > 148.08	10	0.9951
140.	Trifloxystrobin	16.65	116.04 > 89.03	15	190.06 > 130.04	10	0.9962
141.	Propiconazole, peak 1	16.77	259.02 > 69.01	20	259.02 > 173.02	20	0.9989
142.	Edifenphos	16.78	173.01 > 109.01	15	310.03 > 173.01	10	0.9904
143.	Quinoxyfen	16.84	272.00 > 237.00	20	307.00 > 272.00	10	0.9982
144.	Endosulfan sulfate	16.85	271.88 > 236.89	15	273.88 > 238.89	15	0.9929
145.	Clodinafop-propargyl	16.87	349.05 > 238.04	15	349.05 > 266.04	15	0.9991
146.	Flupicolide	16.90	208.80 > 182.00	20	261.00 > 175.00	24	0.9988
147.	Hexazinone	17.02	171.00 > 71.00	10	171.00 > 85.00	10	0.9998
148.	Propargite	17.16	135.06 > 107.05	15	350.16 > 201.09	10	0.9991
149.	Diflufenican	17.21	266.05 > 246.05	10	394.07 > 266.05	10	0.9981
150.	Triphenylphosphate (TPP)	17.26	325.07 > 169.04	25	326.07 > 325.07	10	0.9995
151.	Iprodione	17.65	187.02 > 124.01	20	187.02 > 159.02	40	0.9979
152.	Bifenthrin	17.77	181.05 > 153.05	6	181.05 > 166.05	15	0.9922
153.	Picolinafen	17.90	376.08 > 238.05	15	376.08 > 239.05	15	0.9981
154.	Bromopropylate	17.91	184.98 > 156.98	20	342.96 > 184.98	20	0.9967
155.	Fenoxycarb	17.93	186.08 > 186.08	10	255.11 > 186.08	10	0.9933
156.	Fenpropathrin	18.01	181.09 > 152.07	23	265.13 > 210.10	15	0.9956
157.	Fenamidone	18.10	238.08 > 237.08	20	268.09 > 180.06	20	0.9994
158.	Tebufenpyrad	18.11	276.13 > 171.08	15	333.16 > 276.13	10	0.9997
159.	Fenazaquin	18.23	145.08 > 117.07	15	160.09 > 117.07	20	0.9951
160.	Imazalil	18.25	173.03 > 145.02	20	215.04 > 173.03	15	0.9954
161.	Furathiocarb	18.27	163.07 > 107.04	10	325.13 > 194.08	10	0.9989
162.	Flurtamone	18.38	199.06 > 157.05	20	333.10 > 120.04	15	0.9945
163.	Tetradifon	18.46	226.93 > 198.94	18	353.88 > 158.95	15	0.9973
164.	Phosalone	18.54	181.99 > 111.00	15	181.99 > 138.00	10	0.9985
165.	Triticonazole	18.57	217.09 > 182.07	10	235.10 > 217.09	10	0.9945
166.	Pyriproxyfen	18.68	136.06 > 78.03	15	136.06 > 96.04	15	0.9941
167.	Cyhalofop butyl	18.70	256.10 > 120.05	10	256.10 > 256.10	10	0.9969
168.	Tralkoxydim	18.80	137.00 > 57.20	10	181.04 > 152.03	23	0.9995
169.	Cyhalothrin, lambda	18.80	197.04 > 141.03	15	234.90 > 217.20	15	0.9997
170.	Lactofen	18.83	344.04 > 223.02	15	344.04 > 300.03	15	0.9975
171.	Benfuracarb	19.03	164.08 > 149.07	10	190.09 > 144.07	10	0.9975
172.	Pyrazophos	19.05	221.05 > 193.04	10	232.05 > 204.05	10	0.9930
173.	Fenarimol	19.15	139.01 > 111.01	15	219.02 > 107.01	15	0.9993
174.	Azinphos-ethyl	19.20	132.01 > 77.01	20	160.02 > 132.01	5	0.9944
175.	Fenoxaprop-P	19.41	288.03 > 260.03	10	361.04 > 288.03	10	0.9998
176.	Bitertanol1	19.59	170.09 > 115.06	25	170.09 > 141.07	20	0.9993
177.	Permethrin, peak 1	19.68	183.04 > 165.03	15	183.04 > 168.03	15	0.9973
178.	Bitertanol2	19.71	170.09 > 115.06	25	170.09 > 141.07	20	0.9993
179.	Permethrin, peak 2	19.81	183.04 > 165.03	15	183.04 > 168.03	15	0.9909
180.	Prochloraz	19.88	180.01 > 138.01	15	310.03 > 268.02	10	0.9932
181.	Cafenstrole	20.21	100.04 > 72.03	15	188.08 > 119.05	15	0.9991
182.	Cyfluthrin, peak 1	20.26	163.02 > 91.01	12	163.02 > 127.02	10	0.9915
183.	Fenbuconazole	20.34	129.04 > 102.03	15	198.07 > 129.04	10	0.9996
184.	Cypermethrin I	20.65	163.03 > 127.02	10	181.03 > 152.03	25	0.9996
185.	Boscalid (Nicobifen)	20.84	342.03 > 140.01	15	344.03 > 142.01	15	0.9977
186.	Flucythrinate, peak 1	20.85	199.07 > 107.04	22	199.07 > 157.06	10	0.9958
187.	Quizalofop-Ethyl	20.92	299.07 > 255.06	20	372.09 > 299.07	15	0.9969
188.	Etofenprox	21.08	163.09 > 107.06	16	163.09 > 135.07	10	0.9987
189.	Flucythrinate, peak 2	21.12	199.07 > 107.04	22	199.07 > 157.06	10	0.9989

Nr.	Compound Name	RT [min]	Quantitation *m/z*	CE [V]	Confirmation *m/z*	CE [V]	R^2
190.	Fenvalerate, peak 1	21.94	167.05 > 125.04	10	419.13 > 225.07	10	0.9978
191.	Fluvalinate, peak 1	22.09	250.06 > 200.05	20	252.06 > 200.05	20	0.9973
192.	Pyraclostrobin	22.17	132.03 > 77.02	15	325.08 > 132.03	20	0.9936
193.	Fluvalinate, peak 2	22.20	250.06 > 200.05	20	252.06 > 200.05	20	0.9977
194.	Fenvalerate, peak 2	22.28	167.05 > 125.04	10	419.13 > 225.07	10	0.9996
195.	Difenoconazole, peak 1	22.76	323.05 > 265.04	15	325.05 > 267.04	20	0.9995
196.	Indoxacarb	22.95	203.03 > 106.01	20	203.03 > 134.02	20	0.9996
197.	Deltamethrin II	23.28	252.99 > 93.00	18	252.99 > 173.99	18	0.9987
198.	Azoxystrobin	23.63	344.10 > 329.10	20	388.11 > 345.10	15	0.9991
199.	Dimethomorph-1	23.91	301.10 > 165.05	10	387.12 > 301.10	12	0.9992
200.	Dimethomorph-2	24.60	301.10 > 165.05	10	387.12 > 301.10	12	0.9990

Thermo Fisher Scientific, San Jose, CA USA is ISO 9001:2008 Certified.

Africa-Other +27 11 570 1840
Australia +61 3 9757 4300
Austria +43 1 333 50 34 0
Belgium +32 53 73 42 41
Canada +1 800 530 8447
China +86 10 8419 3588
Denmark +45 70 23 62 60
Europe-Other +43 1 333 50 34 0
Finland/Norway/Sweden +46 8 556 468 00
France +33 1 60 92 48 00
Germany +49 6103 408 1014
India +91 22 6742 9434
Italy +39 02 950 591
Japan +81 45 453 9100
Latin America +1 561 688 8700
Middle East +43 1 333 50 34 0
Netherlands +31 76 579 55 55
New Zealand +64 9 980 6700
Russia/CIS +43 1 333 50 34 0
South Africa +27 11 570 1840
Spain +34 914 845 965
Switzerland +41 61 716 77 00
UK +44 1442 233555
USA +1 800 532 4752

AN10361

Thermo SCIENTIFIC
Part of Thermo Fisher Scientific