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The Analysis of 85 Pesticides Commonly Found in Cannabis and Cannabis-Related Products by LC-MS and GC-MS

NACRW

NORTH AMERICAN CHEMICAL RESIDUE WORKSHOP

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Poster #37

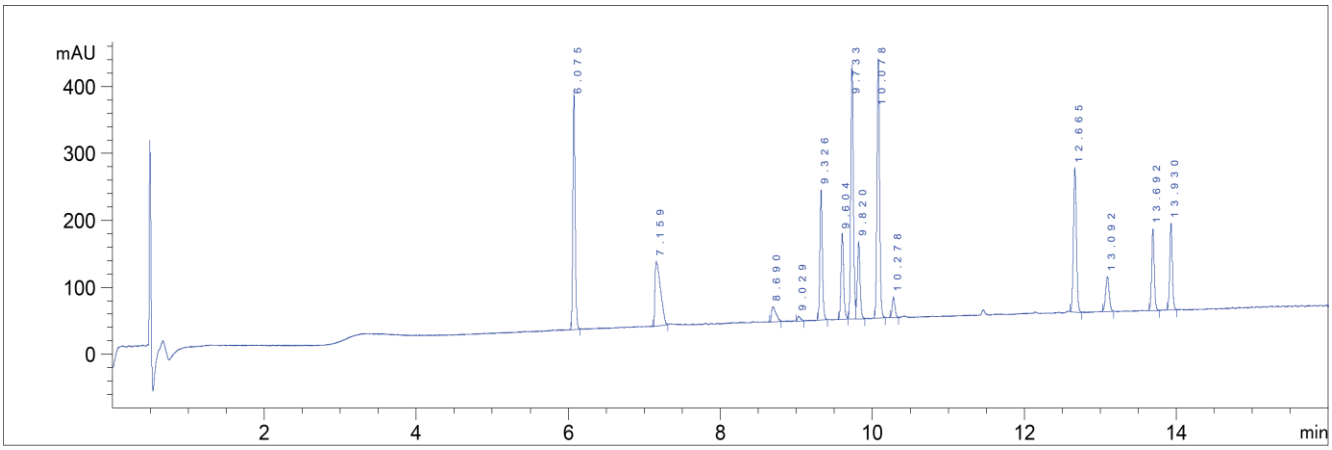
Abstract

Residual pesticides in cannabis and cannabis products can pose a major health risk to consumers in both recreational and medicinal applications. The main source of these chemicals comes during the growing process where chemicals are applied to the plants to control insects, mold or fungus growth. Some of those pesticides, such as organophosphates, are known for their toxic effects to both humans and wildlife.

As a result, regulators and standardization bodies such as ASTM and AOAC have published different test methods to analyze for residual pesticides in cannabis and cannabis-related products.

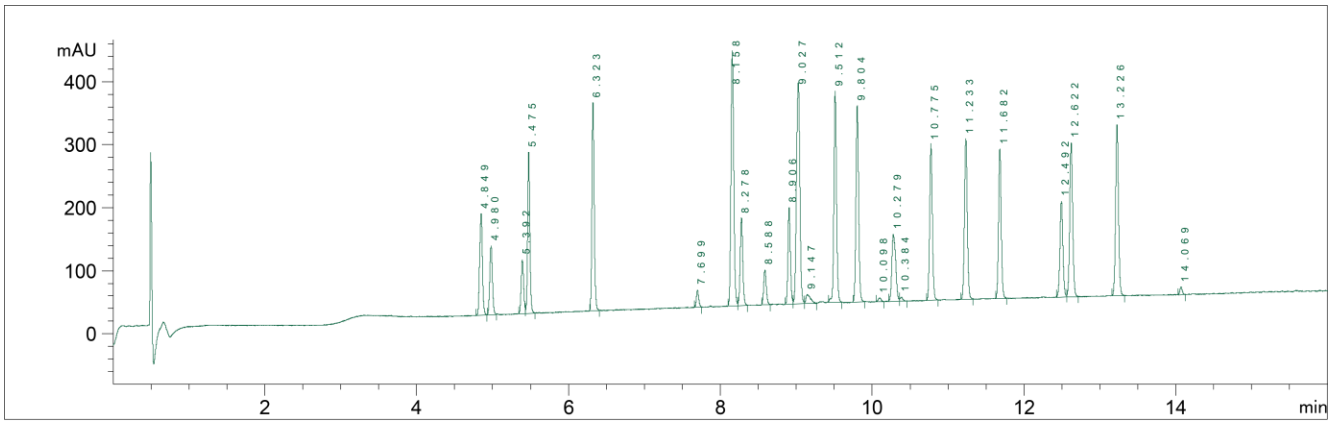
In order to make this screening as efficient as possible, AccuStandard used the target lists from different agencies, and was able to identify and quantify over 80 pesticides commonly used during cannabis cultivation. The analysis was conducted using multiple Certified Reference Materials (CRMs) prepared by AccuStandard and formulated based on the compatibility and the stability of those chemicals. Using both GC-MS and LC-MS we were able to offer a comprehensive and robust analysis for those compounds, most of which are in the target list of compounds in ASTM test method D8399 and AOAC SMPR 2018.011. The results illustrate the proper methods for handling the CRM samples and the appropriate analytical technique used to evaluate every compound. The conclusion is that using both analytical techniques combined with well-characterized CRMs represents a significant benefit for an accurate and reliable analysis of these harmful chemicals.

LC-MS Chromatograms



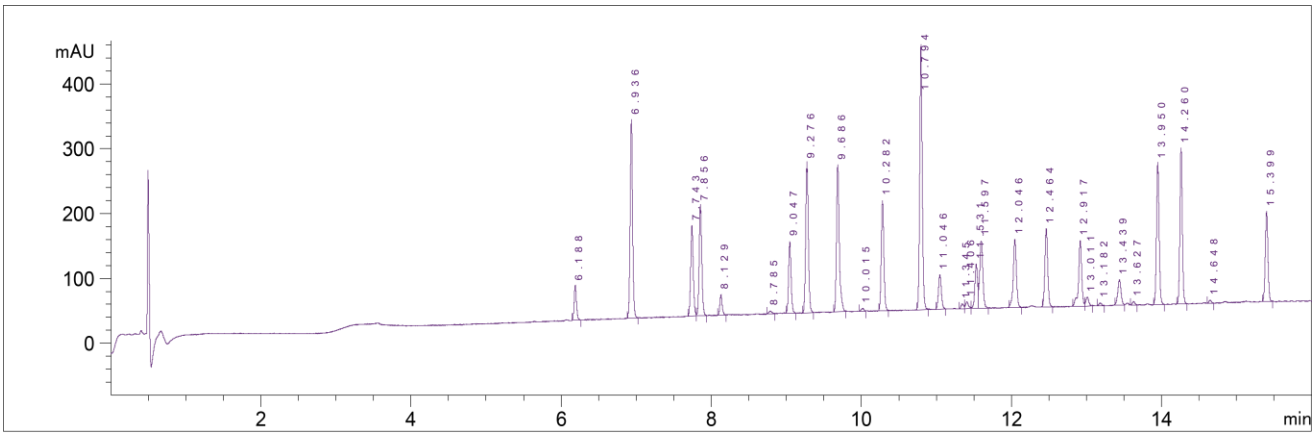
D-8399-01

Peak #	Component
6.075	Imidacloprid (138261-41-3)
7.159	Imazalil (35554-44-0)
8.69	Spinosad (168316-95-8)
9.029	Spinosad (168316-95-8)
9.326	Spirotetramat (203313-25-1)
9.604	Myclobutanil (88671-89-0)
9.733	Azoxystrobin (131860-33-8)
9.82	Tebuconazol (107534-96-3)
10.078	Bifenazate (149877-41-8)
10.278	Malathion (121-75-5)
12.665	Etoxazole (153233-91-1)
13.092	Abamectin (71751-41-2)
13.692	Permethrin (52645-53-1)
13.93	Permethrin (52645-53-1)



D-8399-02

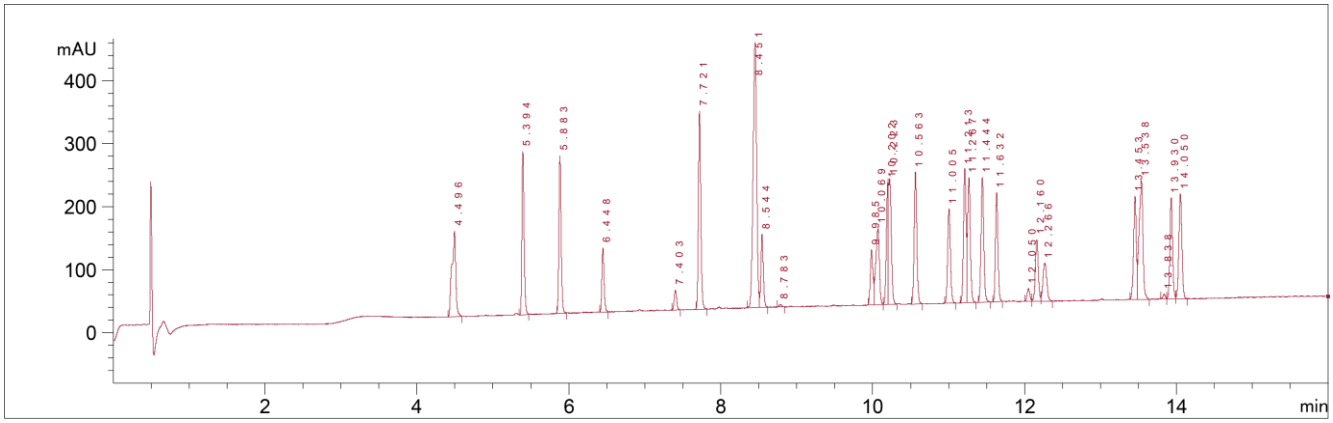
Peak #	Component
4.849	Oxamyl (23135-22-0)
4.98	Methomyl (16752-77-5)
5.392	Flonicamid (158062-67-0)
5.475	Thiamethoxam (153719-23-4)
6.323	Acetamiprid (135410-20-7)
8.158	Carbaryl (63-25-2)
8.278	Metalaxyl (57837-19-1)
8.588	Clofentezine (74115-24-5)
8.906	Dimethomorph (110488-70-5)
9.027	Chlorantraniliprole (500008-45-7)
9.027	Dimethomorph (110488-70-5)
9.147	Spinetoram (935545-74-7)
9.471	Spinetoram (935545-74-7)
9.512	Fludioxonil (131341-86-1)
9.804	Boscalid (188425-85-6)
10.279	Tilt (60207-90-1)
10.775	Kresoxim-methyl (143390-89-0)
11.233	Clofentezine (74115-24-5)
11.682	Trifloxystrobin (141517-21-7)
12.492	Hexythiazox (78587-05-0)
12.622	Fenpyroximate (111812-58-9)
13.226	Pyridaben (96489-71-3)



D-8399-03

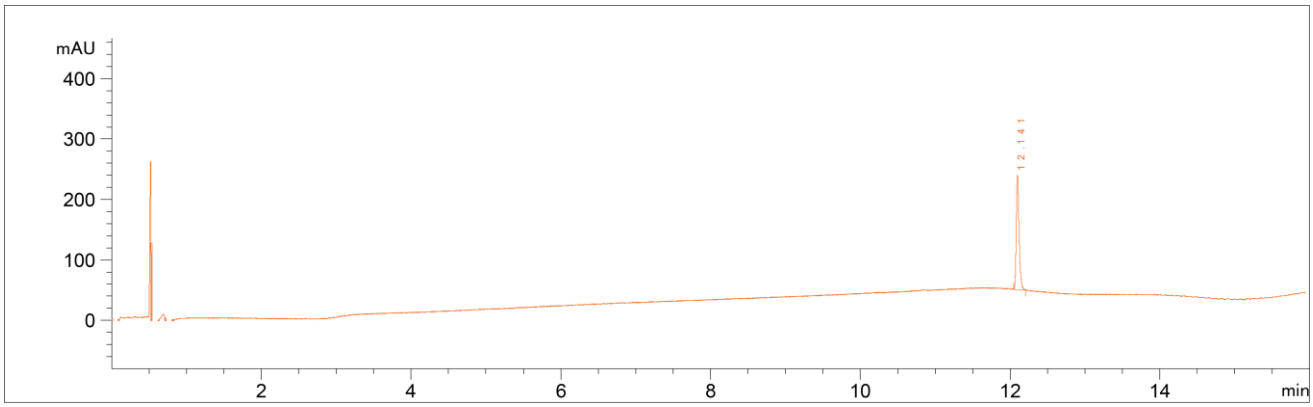
Peak #	Component
0.593	Alar (1596-84-5)*
3.621	Acephate (30560-19-1)*
6.188	Dimethoate (60-51-5)
6.936	Aldicarb (116-06-3)
6.936	Thiacloprid (111988-49-9)
7.588	Meltatox (31717-87-0)*
7.743	Baygon (114-26-1)
7.856	Carbofuran (1563-66-2)
7.856	Spiroxamine (118134-30-8)
8.129	Azadirachtin (11141-17-6)
8.785	Naled (300-76-5)
9.047	Paclobutrazol (76738-62-0)
9.276	Methiocarb (2032-65-7)
9.686	Ethoprop (13194-48-4)
9.686	Imidan (732-11-6)
10.282	Fenoxycarb (72490-01-8)
10.794	Fipronil (120068-37-3)
10.794	Benzovindiflupyr (1072957-71-1)
11.046	Diazinon (333-41-5)
11.531	Pyrethrins (8003-34-7)
11.597	Prallethrin (23031-36-9)
12.046	Piperonyl butoxide (51-03-6)
12.464	Dursban (2921-88-2)
12.917	Pyrethrins (8003-34-7)
13.439	Pyrethrins (8003-34-7)
13.95	Etofenprox (80844-07-1)
14.26	Bifenthrin (82657-04-3)
15.399	Acequinocyl (57960-19-7)

* Compounds detected on MS



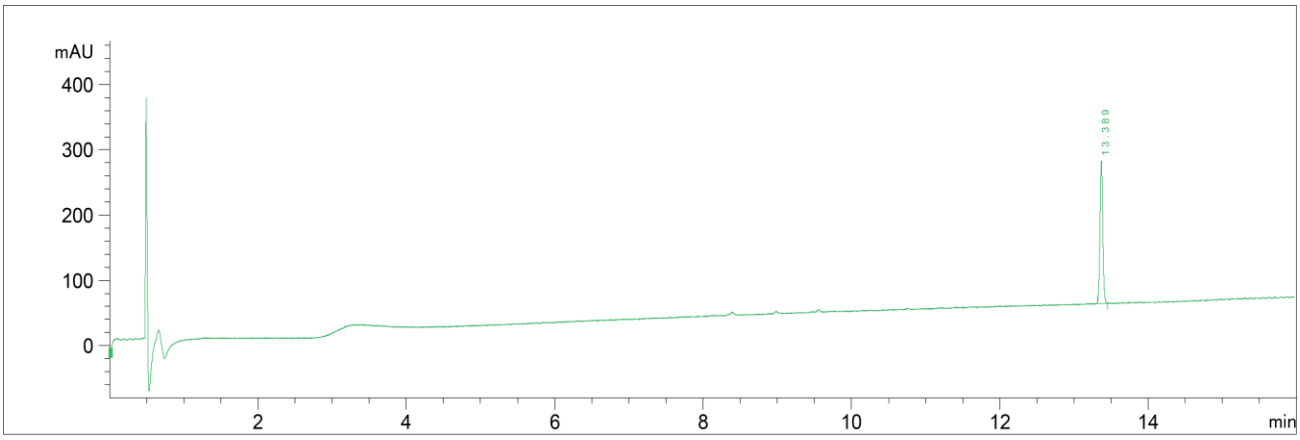
D-8399-04

Peak #	Component
4.496	Dinotefuran (165252-70-0)
5.394	Pirimicarb (23103-98-2)
5.883	Clothianidin (210880-92-5)
6.448	Mevinphos (7786-34-7)
7.403	Dichlorvos (62-73-7)
7.721	Thiophanate-methyl (23564-05-8)
8.451	Cyantraniliprole (736994-63-1)
8.451	Cyprodinil (121552-61-2)
8.544	Fensulfothion (115-90-2)
9.985	Fluopyram (658066-35-4)
10.069	Buprofezin (69327-76-0)
10.202	Iprodione (36734-19-7)
10.223	Tetrachlorvinphos Z (22248-79-9)
10.563	Tebufenozide (112410-23-8)
11.005	Fenthion (55-38-9)
11.213	Pyraclostrobin (175013-18-0)
11.267	Coumaphos (56-72-4)
11.444	Teflubenzuron (83121-18-0)
11.632	Novaluron (116714-46-6)
12.05	Tetramethrin (7696-12-0)
12.16	Tetramethrin (7696-12-0)
12.266	Allethrin (584-79-2)
13.453	Resmethrin (10453-86-8)
13.538	Deltamethrin (52918-63-5)
13.538	Fenvalerate (51630-58-1)
13.93	Sumithrin (26002-80-2)
14.05	Methoprene (40596-69-8)



P-960S-CN

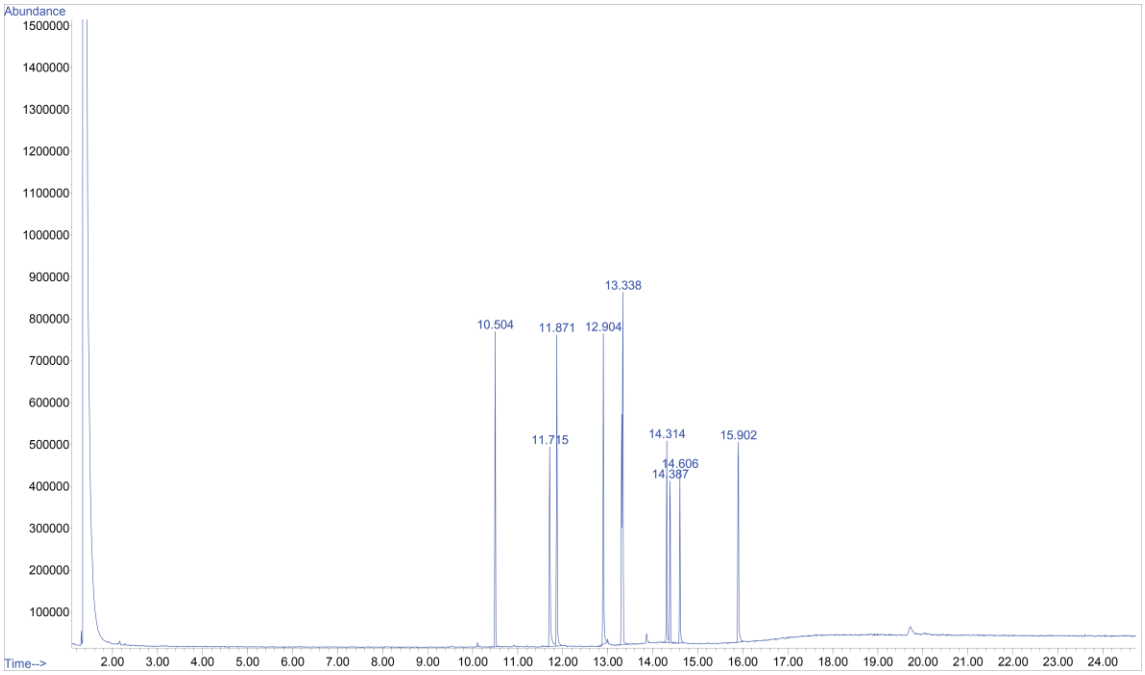
Peak #	Component
12.141	Spiromesifen (283594-90-1)



P-938S-CN

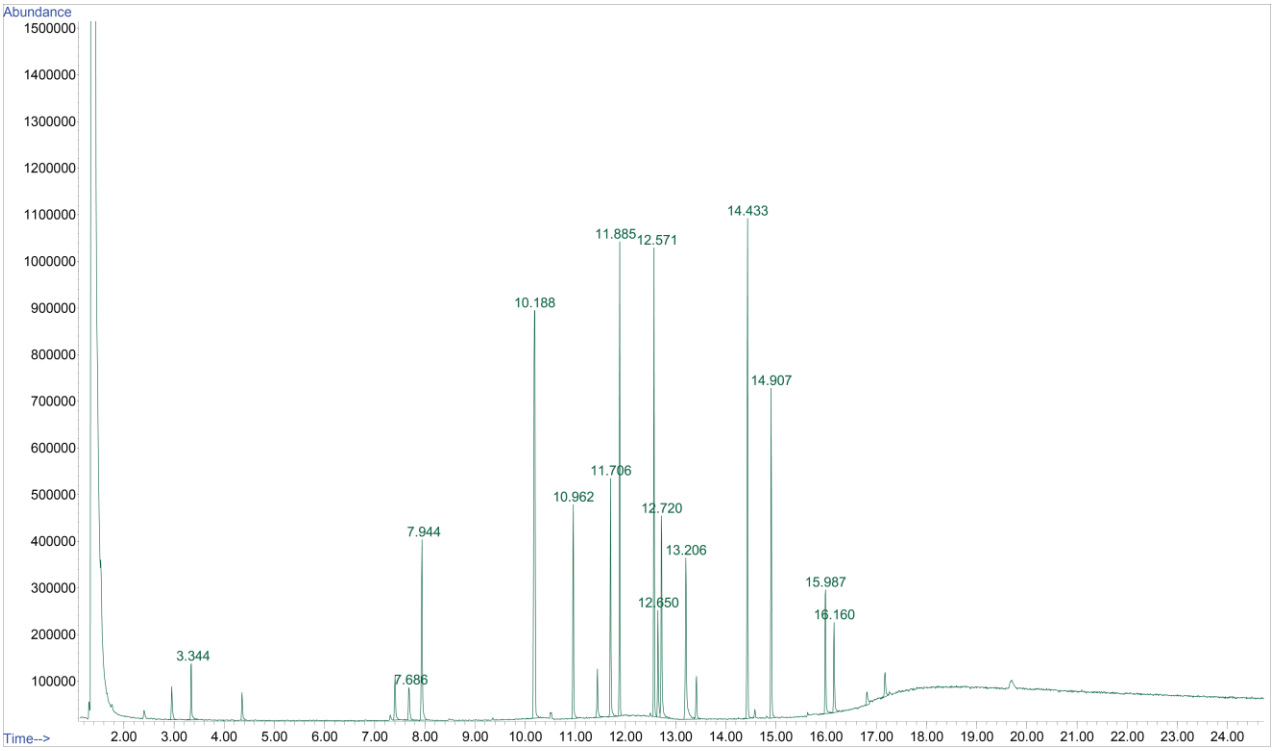
Peak #	Component
13.389	Spirodiclofen (148477-71-8)

GC-MS Chromatograms



D-8399-01

Peak #	Component
10.504	Malathion (121-75-5)
11.715	Imazalil (35554-44-0)
11.871	Myclobutanil (88671-89-0)
12.904	Tebuconazol (107534-96-3)
13.338	Etoxazole (153233-91-1)
13.338	Bifenazate (149877-41-8)
14.314	Permethrin (52645-53-1)
14.387	Permethrin (52645-53-1)
14.606	Spirotetramat (203313-25-1)
15.902	Azoxystrobin (131860-33-8)
LCMS	Abamectin (71751-41-2)
LCMS	Imidacloprid (138261-41-3)
LCMS	Spinosad (168316-95-8)



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Peak #	Component
3.344	Methomyl (16752-77-5)
7.686	Oxamyl (23135-22-0)
10.188	Carbaryl (63-25-2)
10.188	Metalaxyl (57837-19-1)
11.706	Fludioxonil (131341-86-1)
11.885	Kresoxim-methyl (143390-89-0)
12.571	Trifloxystrobin (141517-21-7)
12.65	Tilt (60207-90-1)
12.72	Tilt (60207-90-1)
13.206	Acetamiprid (135410-20-7)
14.433	Pyridaben (96489-71-3)
14.907	Boscalid (188425-85-6)
15.987	Dimethomorph (110488-70-5)
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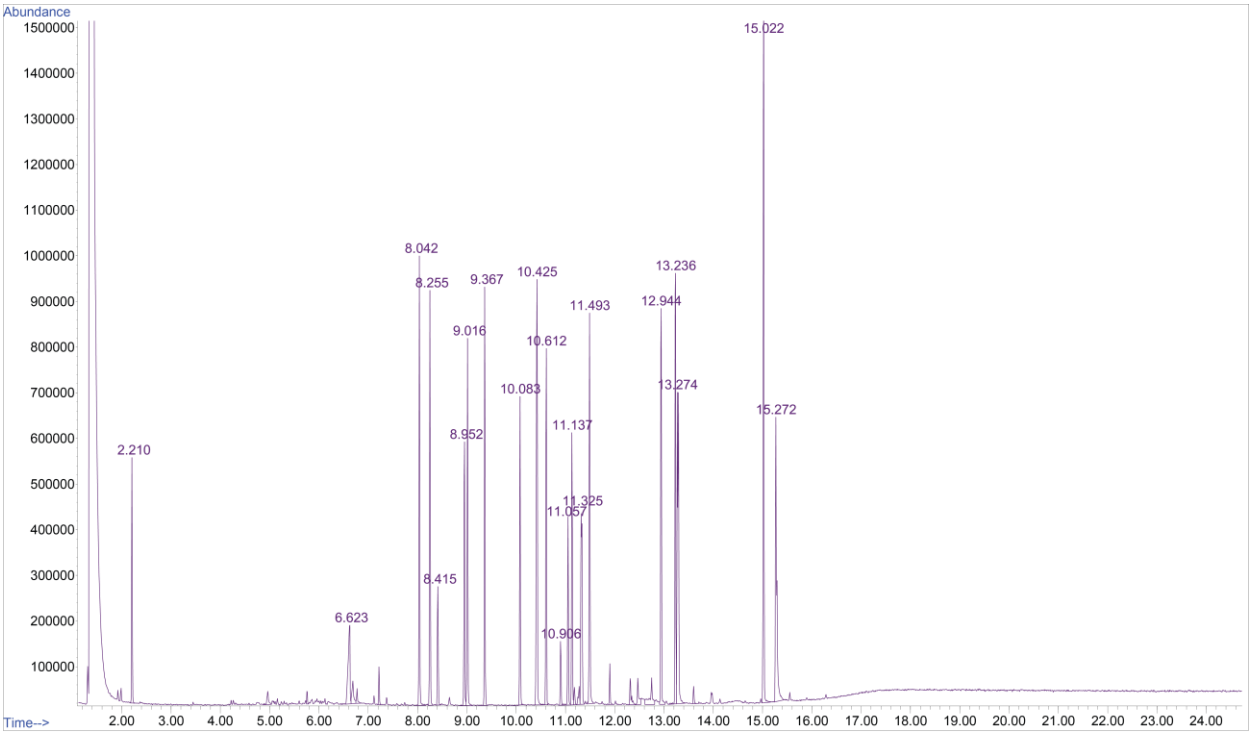
LCMS	Spinetoram (935545-74-7)
LCMS	Thiamethoxam (153719-23-4)



D-8399-03

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6.623	Acephate (30560-19-1)
8.042	Baygon (114-26-1)
8.255	Ethoprop (13194-48-4)
8.415	Naled (300-76-5)
8.952	Dimethoate (60-51-5)
9.016	Carbofuran (1563-66-2)
9.367	Diazinon (333-41-5)
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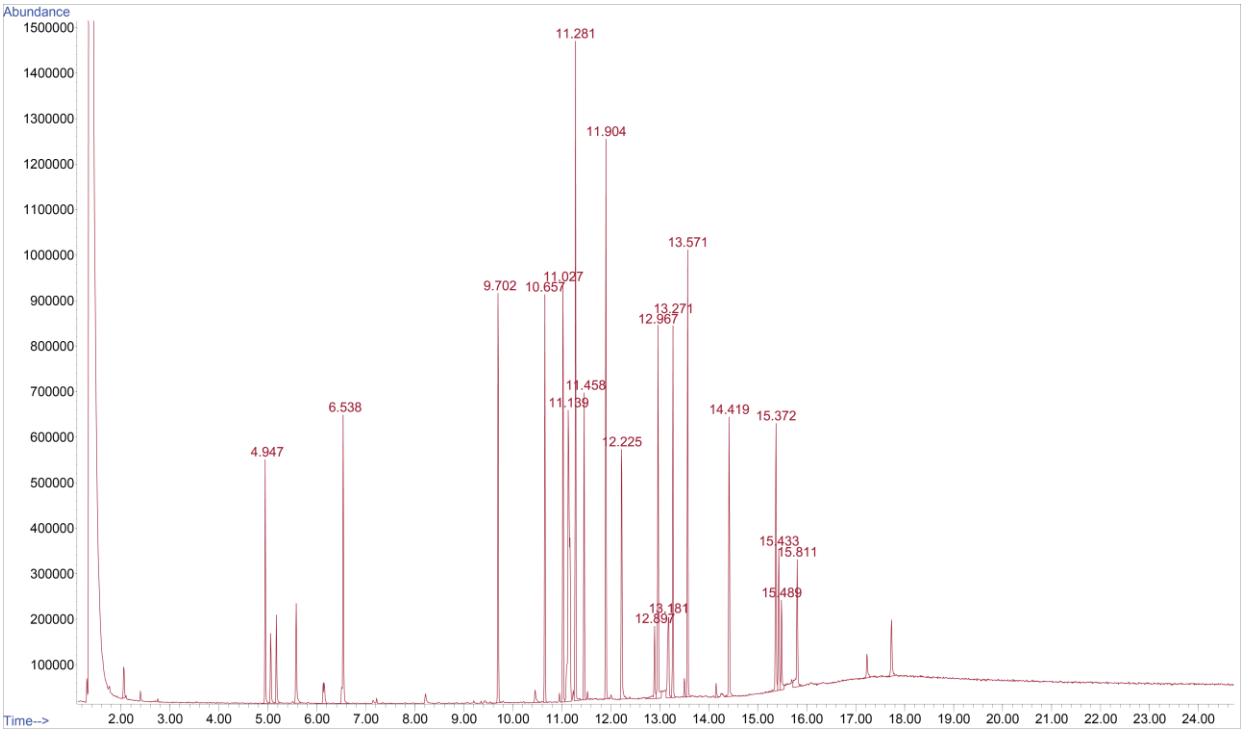

D-8399-04

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10.657	Fenthion (55-38-9)
11.027	Cyprodinil (121552-61-2)
11.139	Allethrin (584-79-2)
11.281	Methoprene (40596-69-8)
11.458	Tetrachlorvinphos Z (22248-79-9)
11.904	Buprofezin (69327-76-0)
12.225	Fensulfothion (115-90-2)
12.897	Resmethrin (10453-86-8)
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13.181	Iprodione (36734-19-7)
13.271	Tetramethrin (7696-12-0)
13.571	Sumithrin (26002-80-2)
14.419	Coumaphos (56-72-4)
15.372	Fenvalerate (51630-58-1)
15.433	Pyraclostrobin (175013-18-0)
15.489	Fenvalerate (51630-58-1)
15.811	Deltamethrin (52918-63-5)
LCMS	Clothianidin (210880-92-5)
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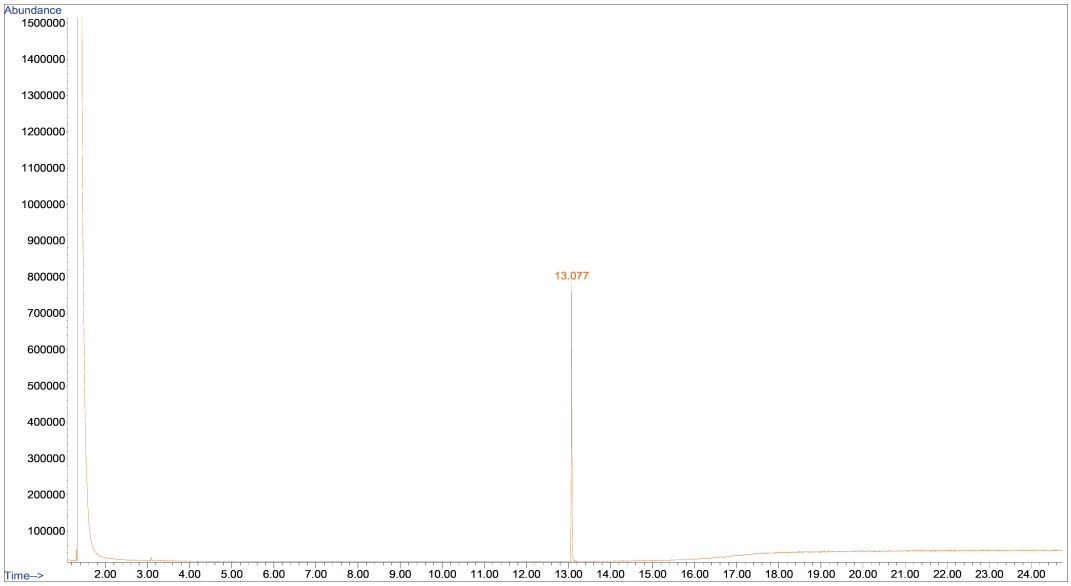
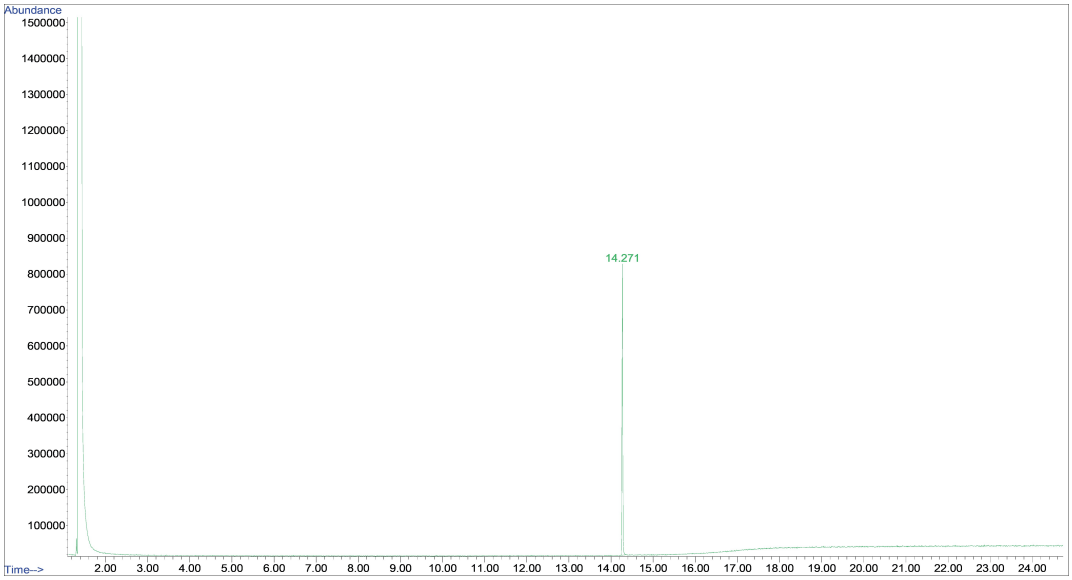


P-960S-CN

Peak #	Component
13.077	Spiromesifen (283594-90-1)



P-938S-CN

Peak #	Component
14.271	Spirodiclofen (148477-71-8)

Conclusion

As more states legalize cannabis for both medicinal and recreational uses, cannabis testing labs face growing pressure to test samples for more pesticides efficiently and accurately to ensure the safety of consumers. During this work we were able to demonstrate the effectiveness of using appropriate and accessible analytical methodologies, in addition to well-characterized Certified Reference Materials (CRMs), in the identification and quantification of residual pesticides in cannabis. Using a combination of both LC-MS and GC-MS techniques offered a significant advantage for the selection of suitable methods to reliably and accurately analyze all targeted compounds in this study.

About this work

This work was conducted using 6 different Certified Reference Materials prepared by AccuStandard. These CRMs are:

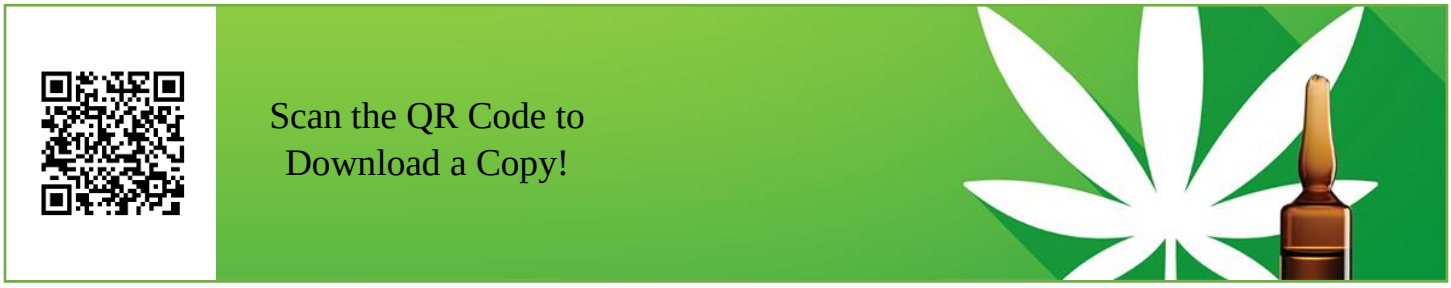
- D-8399-01 (12 components in Acetonitrile at 100 µg/mL)
- D-8399-02 (19 components in Acetonitrile at 100 µg/mL)
- D-8399-03 (26 components in Acetonitrile at 100 µg/mL)
- D-8399-04 (26 components in Acetonitrile at 100 µg/mL)
- P-960S-CN (Spiromesifen in Acetonitrile at 100 µg/mL)
- P-938S-CN (Spirodiclofen in Acetonitrile at 100 µg/mL)

The 6 CRMs listed above are also available as a set: **D-8399-SET**

All CRMs were designed based on compound compatibility and stability studies conducted by AccuStandard. Some compounds such as Spirodiclofen and Spiromesifen were prepared independently from the multiple component mixes due to their unstable nature. Mixes were sonicated for 45 minutes and acclimated to room temperature prior to analyzing to ensure solubility and improve each component's response. The concentration was verified against another independently made standard.

Our analysis was performed using both GC-MS and LC-MS techniques to present the most reliable identification and quantification for all 85 compounds targeted in this work. For instance compounds such as Abamectin (71751-41-2), Imidacloprid (138261-41-3) and Chlorantraniliprole (500008-45-7) couldn't be detected using the GC-MS analysis while it was easily identified via LC-MS. Additionally, compounds such as Alar (1596-84-5), Acephate (30560-19-1) and Meltatox (31717-87-0) were only identified on the Mass Spectrometer detector in the LC.

❖ All standards used in this work are available as part of AccuStandard's comprehensive product line of Certified Reference Materials (CRMs) for Cannabis testing which includes CRMs for the analysis of Heavy Metals, Residual Solvents, Terpenes and Cannabinoids. Visit AccuStandard.com/cannabis-standards to view our selection of Cannabis CRMs.



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