Analysis of Pesticides, Mycotoxins, and Cannabinoids in Cannabis Gummies

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Abstract
The state of California demands the analysis of pesticides, mycotoxins, and cannabinoids in all types of cannabis-derived goods [1]. For that reason, reliable workflows for the easy determination of these analytes in diverse matrices are highly desired. Gummies are a very popular cannabis edible, and their composition makes them a highly complex matrix. In this work, we describe a complete workflow for the analysis of California pesticides, mycotoxins, and cannabinoids in gummies using a single extraction procedure.

Introduction

Edibles infused with cannabis or cannabidiol (CBD) are growing in popularity with cannabis consumers. In terms of testing, potency analysis of edibles is mandatory in all states where lab testing is a requisite. In addition, in the state of California, determination of contaminants, such as pesticides and mycotoxins, in edibles and other cannabis-derived goods is also a requirement. Due to the broad variety of edibles available in the market, different analytical strategies should be pursued in order to obtain reliable analytical data for all analytes of interest in all matrices. Among the various types of cannabis edibles, gummies present unique challenges for cannabis testing labs.

Gummies are a type of sticky matrix typically made of ingredients such as sugar, starch, pectin, and gelatin. Due to their composition, sample preparation strategies for potency testing usually include a sample solubilization step in solvents like water or DMSO or, alternatively, large volumes of solvents like methanol may be used. Following sample homogenization, cannabinoids are quantified via HPLC-UV by injecting diluted extract either directly or following a salting-out step using QuEChERS salts. As for the analysis of contaminants like pesticides and mycotoxins in gummy matrix, publicly available information is quite scarce, with only one report showing semiquantitative data for 35 pesticides using QuEChERS [2].

In this work, we provide a robust workflow for the quantitative determination of the California list of pesticides, mycotoxins, and cannabinoids in gummy samples. The optimized sample preparation methodology involves sample solubilization followed by an extraction step using acidified acetonitrile, and a salting-out step using EN QuEChERS salts. For the analysis of cannabinoids and LC-amenable contaminants, a simple dilution was conducted prior to injection. Whereas, for the GC-amenable pesticides, the use of a dSPE sorbent mix including primary/secondary amine (PSA), graphitized carbon black (GCB), and magnesium sulfate was necessary before analysis. Overall, the proposed workflow for the analysis of pesticides, mycotoxins, and cannabinoids in cannabis gummies provides satisfactory results in terms of linearity, accuracy, precision, and limits of quantitation (LOQs).

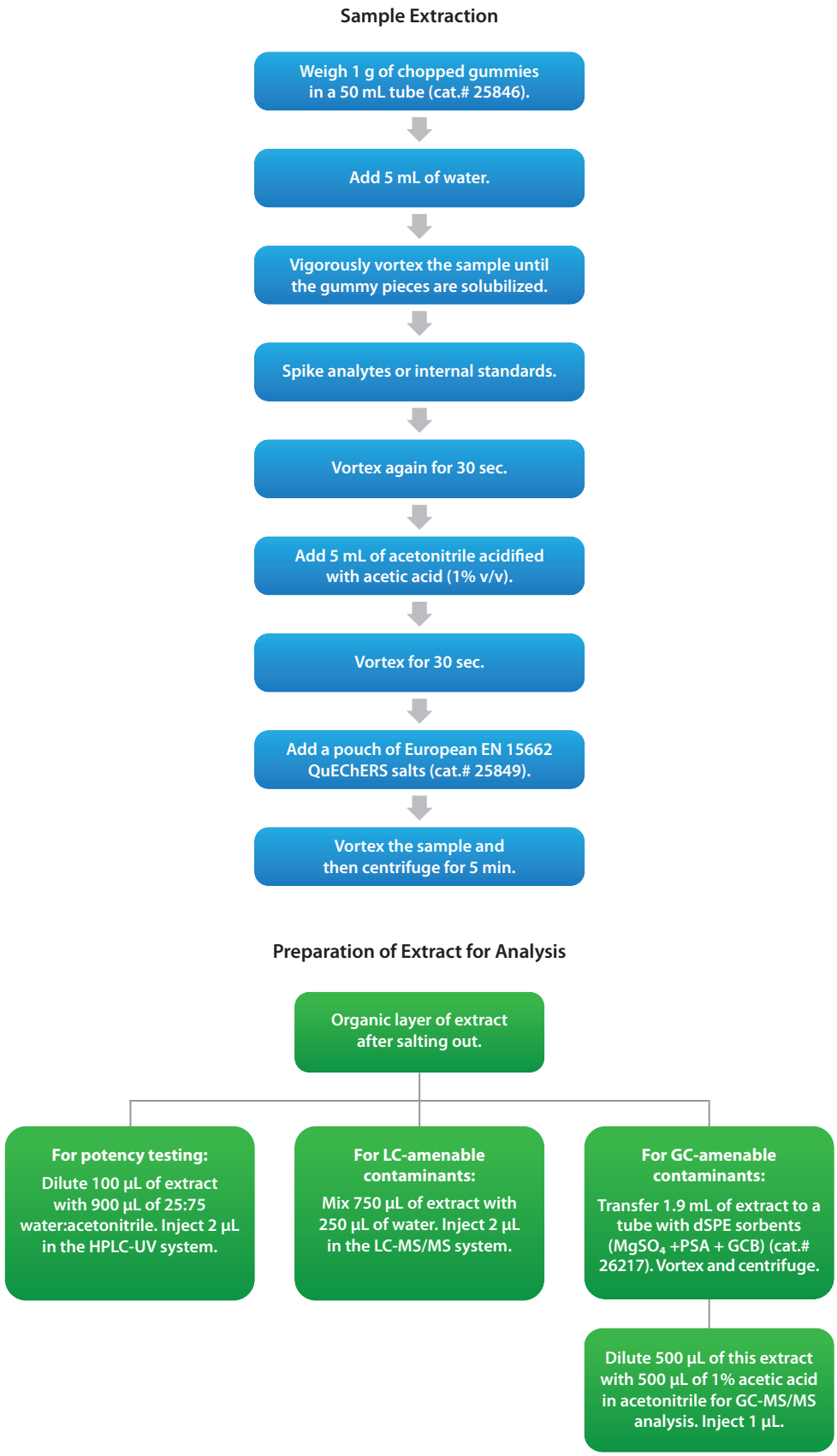
Experimental

Sample Preparation

After preliminary testing of various experimental parameters, the following workflow was implemented (Figure 1). Chopped gummies (1 g) were weighed into a 50 mL tube (cat.# 25846), and 5 mL of water was added. Then, the samples were vortexed vigorously until the gummy pieces were solubilized. The sample solution was then fortified with target analytes and/or internal standards (internal standards were used only for contaminant analysis, not for potency testing) as appropriate and vortexed again for 30 seconds. Next, 5 mL of acetonitrile acidified with acetic acid (1% v:v) was added, and the samples were vortexed another 30 seconds. A pouch of European EN 15662 Q-sep QuEChERS extraction salts (cat.# 25849) was added, and the sample was vortexed again and then centrifuged for 5 minutes.

For potency testing, 100 µL of the sample extract was mixed with 900 µL of 25:75 water:acetonitrile, and 2 µL were analyzed by LC-UV. For the LC-amenable contaminants, 750 µL of the sample extract was mixed with 250 µL of water, and 2 µL were analyzed by LC-MS/MS. For the GC-amenable contaminants, 1.9 mL of extract was transferred to a Q-sep QuEChERS dSPE tube containing pre-weighed PSA, GCB, and magnesium sulfate sorbents (cat.# 26217). After vortexing and centrifuging, 500 µL of extract was mixed with 500 µL of 1% acetic acid in acetonitrile, and 1 µL was analyzed by GC-MS/MS.

Figure 1: Sample preparation procedure for the analysis of pesticides, mycotoxins, and cannabinoids in cannabis gummies.



Quantitation

For the analysis of pesticides and mycotoxins, calibration solutions were prepared by spiking analytes and internal standards in aliquots of extract obtained from blank gummy samples (a pooled extract was obtained by mixing extracts from various blank gummy samples). Table I shows the volume of target analyte spiking solution and internal standard mix solution that was added to each aliquot (final calibration solution volume = 3 mL). To construct calibration curves for the GC-amenable pesticides, 1.9 mL of the 3 mL calibration solutions were subjected to dSPE cleanup as described in the sample preparation section. Method accuracy and precision were evaluated by spiking homogenized gummies (after adding water and vortexing) at 10, 50, 100, and 500 ng/g in quadruplicate (Table II), and performing the full sample preparation workflow described in the sample preparation section.

For the analysis of cannabinoids, calibration solutions were prepared at 2, 5, 10, 20, 50, 100, and 200 ppm in 75:25 acetonitrile:water. Recovery of the cannabinoids was assessed by spiking gummy samples (1 g) solubilized in water at 0.2 and 0.5 mg/g (n=2), and then extracting them as previously described.

Table I: Preparation of calibrators for pesticides and mycotoxins analysis using aliquots of extracts collected from blank gummy samples (final volume of each calibration solution = 3 mL).

Desired Analyte Conc. in Matrix (ng/g)	Analyte Conc. in Final Extract Assuming 100% Recovery from Matrix (ng/mL)	µL of Target Analyte Solution Spiked into Blank Extract	Analyte Conc. in Spiking Solution (ng/mL)	µL of 5000 ng/mL Internal Standard Mix Added to Blank Extract
5	1	30	100	24
20	4	12	1000	24
50	10	30	1000	24
75	15	45	1000	24
150	30	90	1000	24
200	40	24	5000	24
400	80	48	5000	24
700	140	84	5000	24

Table II: Fortification of mycotoxins and pesticides in gummies (1 g of sample homogenized with 5 mL of water) at different concentration levels.

Conc. in Matrix (ng/g)	µL of Target Analyte Solution Spiked into Homogenized Samples in Water	Analyte Conc. in Spiking Solution (ng/mL)	µL of 5000 ng/mL Internal Standards Mix Added to Homogenized Samples in Water
10	10	1000	40
50	50	1000	40
100	100	1000	40
500	100	5000	40

Instrument Conditions

Instrumentation and conditions for the analysis of pesticides, mycotoxins, and cannabinoids in cannabis gummies are presented in Tables III, IV, and V. Ion transitions for LC-amenable and GC-amenable contaminants are presented in Tables VI and VII, respectively. Cannabinoid retention times are shown in Table VIII.

Table III: LC-MS/MS Conditions (Pesticides and Mycotoxins).

Column	Raptor ARC-18 2.7 µm, 100 mm x 2.1 mm (cat.# 9314A12)			
Guard Column	Raptor ARC-18 EXP guard column cartridge 2.7 µm, 5 x 2.1 mm (cat.# 9314A0252)			
Mobile Phase A	Water, 2 mM ammonium formate, 0.1% formic acid			
Mobile Phase B	Methanol, 2 mM ammonium formate, 0.1% formic acid			
Time Program	**Time (min)**	**%B**	**Time (min)**	**%B**
	0	5	10.5	100
	1.5	65	10.6	5
	8.5	95	12.0	5
	9.5	100	—	—
Flow	0.5 mL/min			
Column Temp.	40 °C			
Autosampler Temp.	10 °C			
Inj. Volume	2 µL			
Instrument	Shimadzu LCMS-8060			

Table IV: GC-MS/MS Conditions (Pesticides).

Instrument	Thermo Trace 1310-TSQ 8000
Column	Rxi-5ms, 30 m x 0.25 mm ID x 0.25 µm, (cat.# 13423)
Injection Mode	Splitless
Inj. Vol.	1 µL
Liner	Topaz 4.0 mm ID single taper inlet liner w/ wool (cat.# 23447)
Inj. Temp.	250 °C
Split Flow	14.0 mL/min
Splitless Time	0.50 min
Purge Flow	5 mL/min
Oven	90 °C (hold 1 min) to 310 °C at 25 °C/min C (hold 10 min)
Carrier Gas	He, constant flow
Flow Rate	1.40 mL/min
Detector	MS/MS
Method Type	Acquisition - timed
Ionization Mode	EI
Transfer Line Temp.	290 °C
Source Temp.	330 °C

Table V: HPLC-UV Conditions (Potency Using a Solvent Savings Method [3]).

Instrument	Waters ACQUITY
Column	Raptor ARC-18 2.7 µm, 150 mm x 2.1 mm (cat.# 9314A62)
Guard column	Raptor ARC-18 EXP guard column cartridge 2.7 µm, 5 x 2.1 mm (cat.# 9314A0252)
Inj. Vol.	2 µL
Mobile phase A	Water, 5 mM ammonium formate, 0.1% formic acid
Mobile phase B	Acetonitrile, 0.1% formic acid
Gradient	Isocratic, 75% B
Flow	0.4 mL/min
Column Temp.	30 °C
Autosampler Temp.	10 °C
Wavelength	228 nm

Table VI: LC-MS/MS Transitions.

Name	Retention Time (min)	Precursor Ion	Product Ion 1	Product Ion 2
Daminozide-D6	0.7	167.0	149.3	49.3
Daminozide	0.7	161.1	44.1	143.2
Acephate	1.7	184.0	143.1	95.1
Oxamyl	2.0	237.1	72.1	90.1
Flonicamid	2.1	230.1	203.1	174.1
Methomyl	2.1	163.1	88.1	106.1
Thiamethoxam	2.1	292.0	211.1	181.1
Imidacloprid	2.3	256.1	209.1	175.1
Mevinphos	2.4	225.1	127.1	193.2
Acetamiprid	2.4	223.0	126.1	56.1
Dimethoathe-D6	2.4	236.1	205.1	-
Dimethoate	2.4	230.0	199.1	125.1
Thiacloprid	2.5	253.0	126.0	90.1
Aflatoxin G2	2.5	331.2	189.3	115.2
Aflatoxin G1	2.5	329.2	243.2	215.3
Aldicarb	2.6	116.0	89.2	70.2
Aflatoxin B2	2.6	315.3	287.2	243.3
Dichlorvos	2.7	220.9	109.1	79.2
Dichlorvos-D6	2.7	227.0	115.1	-
Aflatoxin B1	2.7	313.2	241.2	128.2
Imazalil	2.7	297.0	159.0	201.0
Carbofuran	2.7	222.1	123.1	165.2
Propoxur	2.7	210.1	111.1	93.1
Carbaryl-D7	2.8	209.2	152.2	-
Carbaryl	2.8	202.1	145.1	127.1
Diuron-D6	3.0	239.1	78.2	-
Atrazine-D5	3.0	221.2	179.1	-
Naled	3.1	397.8	127.1	109.1
Metalaxyl	3.1	280.2	220.2	192.2
Spiroxamine	3.1	298.3	144.2	100.2
Chlorantraniliprole	3.2	483.9	452.9	285.9
Phosmet	3.2	318.0	160.1	77.2
Azoxystrobin	3.3	404.0	372.1	344.1
Linuron-D6	3.3	255.1	160.1	-
Fludioxonil*	3.4	247.0	180.0	126.0
Methiocarb	3.4	226.1	169.1	121.1
Dimethomorph	3.5	388.2	301.2	165.3
Boscalid	3.5	342.9	307.1	140.1
Paclobutrazol	3.6	294.3	70.1	125.1
Malathion	3.6	331.0	127.2	285.2
Myclobutanil	3.7	289.1	70.1	125.1

Name	Retention Time (min)	Precursor Ion	Product Ion 1	Product Ion 2
Bifenazate	3.7	301.0	198.1	170.2
Ochratoxin A	3.8	404.2	239.1	358.3
Fenhexamid	3.9	302.1	97.1	55.2
Spirotetramat	4.0	374.2	302.1	216.1
Ethoprophos	4.1	243.1	131.1	97.1
Fipronil*	4.1	436.8	331.8	251.9
Fenoxycarb	4.2	302.1	88.1	116.1
Kresoxim methyl	4.4	314.2	267.2	222.2
Tebuconazole	4.4	308.1	70.1	125.1
Diazinon-D10	4.6	315.2	170.2	-
Spinosad (spinosyn A)	4.6	732.4	142.2	98.1
Diazinon	4.6	305.1	169.2	153.2
Coumaphos	4.7	363.1	227.1	307.1
Pyridaben	4.7	365.1	309.2	147.2
Propiconazole	4.7	342.0	159.0	69.2
Clofentezine	4.8	303.0	138.1	102.1
Spinosad (spinosyn D)	5.0	746.5	142.3	98.4
Spinetoram (spinosyn J)	5.1	748.5	142.3	98.3
Trifloxystrobin	5.3	409.2	186.1	145.1
Prallethrin	5.3	301.2	123.2	105.2
Pyrethrin II	5.5	373.1	161.1	133.2
Spinetoram (spinosyn L)	5.6	760.5	142.2	98.1
Piperonyl butoxide	6.0	356.3	177.2	119.2
Chlorpyrifos	6.1	349.9	198.0	97.1
Hexythiazox	6.2	353.1	228.1	168.1
Etoxazole	6.6	360.2	141.1	304.2
Spiromesifen	6.7	273.2	255.2	187.2
Pyrethrin I	6.9	329.2	161.2	105.2
Cyfluthrin (qualifier)	6.9	453.1	193.2	-
Cyfluthrin	6.9	451.1	191.2	-
Cypermethrin	7.1	433.1	191.0	416.0
(E)-Fenpyroximate	7.1	422.2	366.1	138.1
Permethrin-*trans*	7.6	408.3	183.2	355.1
Permethrin-*cis*	7.9	408.3	183.2	355.1
Avermectin B1a	7.9	890.5	305.4	567.4
Etofenprox	8.0	394.3	177.2	359.3
Bifenthrin	8.2	440.0	181.2	166.2
Acequinocyl precursor ion 1	9.4	402.3	343.2	189.0
Acequinocyl precursor ion 2	9.4	386.0	344.2	189.1

*Analyzed in negative mode.

Table VII: GC-MS/MS transitions.

Name	Retention Time (min)	Ion Polarity	Precursor Ion	Product Ion
Atrazine-D5 (IS) (Quan)	6.82	Positive	220.0	58.0
Atrazine-D5 (IS) (Qual)	6.82	Positive	205.0	127.0
Diazinon-D10 (Quan)	7.01	Positive	183.0	139.0
Diazinon-D10 (Qual)	7.01	Positive	183.0	168.0
Quintozene (PCNB) (Quan)	7.03	Positive	294.9	236.9
Quintozene (PCNB) (Qual)	7.03	Positive	236.8	118.9
Methyl parathion (Quan)	7.50	Positive	263.0	109.0
Methyl parathion (Qual)	7.50	Positive	263.0	79.0
Captan (Quan)	8.37	Positive	184.0	149.1
Captan (Qual)	8.37	Positive	184.0	134.1
trans-Chlordane (Quan)	8.41	Positive	271.9	237.0
trans-Chlordane (Qual)	8.41	Positive	372.9	265.9
cis-Chlordane (Quan)	8.53	Positive	372.9	265.9
cis-Chlordane (Qual)	8.53	Positive	271.9	237.0
Chlorfenapyr (Quan)	8.80	Positive	247.1	227.1
Chlorfenapyr (Qual)	8.80	Positive	59.1	31.1
Cyfluthrin (Quan)	10.61	Positive	226.0	206.0
Cyfluthrin (Qual)	10.61	Positive	163.0	127.0
Cypermethrin (Quan)	10.87	Positive	163.0	127.1
Cypermethrin (Qual)	10.87	Positive	181.1	152.1

Table VIII: Cannabinoid Retention Times.

Compound	Retention Time (min)
Cannabidiolic acid (CBDA)	2.142
Cannabigerol (CBG)	2.405
Cannabidiol (CBD)	2.535
Cannabinol (CBN)	3.776
Delta-9-tetrahydrocannabinol (Delta-9-THC)	4.753
Tetrahydrocannabinolic acid (THCA)	6.279

Results and Discussion

Method Optimization

The diversity of cannabis matrices and differences in the chemical characteristics of the target analytes requires multiple strategies to ensure accurate results. Analysis of pesticides, mycotoxins, and cannabinoids in cannabis gummies, as required by the state of California, needed a completely different set of experimental conditions from the ones used for brownies in our previous technical article [4]. First, we observed that dealing with pulverized gummies (grinding was conducted using dry ice and a food processor) can be extremely challenging as the matrix becomes very sticky once it gets to room temperature. For that reason, chopping gummies in small pieces was found to be a much better alternative for ease of handling when weighing the desired amount of sample.

The next challenge was finding the best approach to dissolve the matrix prior to extraction in order to obtain reliable data. Since the sample was chopped in small pieces of random size, having a homogeneous matrix was crucial to guarantee satisfactory method reproducibility. First, we evaluated the use of solvents such as acetonitrile and methanol to dissolve the gummy pieces, and to extract analytes of interest as in a simple solvent extraction. However, we found that it was very difficult to solubilize the matrix under those conditions. We also evaluated the use of DMSO for this purpose. Although all gummy pieces were easily dissolved in this solvent, daminozide was not detected in any of the extracts due to ionization suppression caused by the small percentage of DMSO that remained in the injected samples. After these tests, we concluded that hydrating the sample with 5 mL of water followed by vigorous vortexing was the best way to obtain a homogeneous sample. Subsequently, to extract all the contaminants from the matrix, 5 mL of acetonitrile acidified at 1% with acetic acid was added to the dissolved sample.

To separate the organic layer from the aqueous layer, three different Q-sep QuEChERS extraction salts were compared: AOAC (cat.# 25851), unbuffered (cat.# 25847), and EN salts (cat.# 25849). EN salts resulted in the best performance with all the compounds showing recoveries above 83% except for daminozide and its deuterated analogue, which displayed recoveries of around 25%. As for the cleanup step, the effect of four different Q-sep QuEChERS dSPE sorbent mixes (cat.#s 26215, 26216, 26217, 26242) was assessed for all the LC-MS-amenable pesticides. It was confirmed that all the mixes that contained 25 mg of PSA (cat.#s 26215, 26216, 26217) led to significant losses of daminozide and ochratoxin A with only 40% of the original amount present in solution being recovered. The dSPE mix that contained MgSO4 and C18 (cat.# 26242) did not cause significant losses of pesticides; however, we decided to evaluate the suitability of using the organic extract without any further cleanup step for the quantification of LC-amenable analytes.

To assess the feasibility of analyzing the extracts directly, experiments to investigate absolute matrix effects in gummy extracts without any cleanup step were conducted using the methodology proposed by Matuszweski et al. [5]. Extracts obtained from blank samples were spiked at 5, 15, and 50 ppb final concentrations, and their responses were compared to neat solvent spiked at the same concentration levels using LC-MS/MS. At 5 ppb, 12 pesticides showed matrix effects greater than 120%, and at 15 ppb and 50 ppb, only daminozide showed significant enhancement (Table IX). Based on this and on the poor recovery of daminozide, the use of daminozide-d6 as the internal standard was crucial to obtaining reliable data.

In regard to the recoveries of GC-amenable pesticides, data corresponding to the evaluation of three dSPE sorbent mixes (cat.#s 26215, 26216, and 26217) showed that, in all the cases, recoveries were above 96% when comparing the response of the cleaned extract vs. the response of the original extract. Considering the high content of sugar and pigments present in gummies extracts, the dSPE sorbent containing PSA, GCB, and magnesium sulfate (cat.# 26217) was chosen for the final sample preparation procedure.

Method Verification

Table X presents results corresponding to limits of quantitation, linearity, accuracy, and precision for the California list of pesticides and mycotoxins determined in gummy matrix. For all the contaminants analyzed via LC-MS/MS, calibration curves were plotted using analyte/internal standard response ratios and a weighing factor of 1/x. For the GC-amenable analytes, only the calibration curve of PCNB was plotted using the analyte/internal standard ratio with diazinon-d10 being chosen as internal standard. Quantification for the rest of the GC-amenable compounds was carried out with external calibration curves (area vs. spiked concentration) because this provided better results than when the internal standard was used. RSDs values below 24% were obtained for all the analytes at all the concentration levels tested. Accuracy values were within 75–118%, and coefficients of determination (R2) were all above 0.99.

Finally, the results for cannabinoids analysis demonstrated that the extract collected for contaminants determination is also suitable for potency testing. Table XI presents data corresponding to the calibration curves prepared in solvent that were used for the quantitation of each cannabinoid. As shown in Table XII, gummy samples spiked with six cannabinoids at 0.2 mg/g exhibited recoveries ranging from 99 to 107%, whereas samples spiked at 0.5 mg/g showed recoveries from 99 to 106%. Representative chromatograms are presented in Figures 2–4.

Table IX: Absolute Matrix Effects (ME) for Pesticides and Mycotoxins in Cannabis Gummies.

	ME at 5 ppb (%)	RSD	ME at 15 ppb (%)	RSD	ME at 50 ppb (%)	RSD
Daminozide	216	3	251	7	185	3
Acephate	95	6	85	1	89	6
Oxamyl	105	5	96	2	98	5
Flonicamid	91	32	90	22	97	17
Methomyl	104	4	92	3	99	4
Thiamethoxam	105	5	92	5	96	5
Imidacloprid	114	9	87	9	100	9
Mevinphos	102	6	93	3	93	6
Acetamiprid	100	2	88	0	91	2
Dimethoate	97	3	92	3	93	3
Thiacloprid	109	7	93	1	93	7
Aflatoxin G2	102	6	86	0	93	6
Aflatoxin G1	106	7	93	1	91	7
Aldicarb	81	22	85	14	90	22
Aflatoxin B2	113	11	79	9	95	11
Dichlorvos	126	15	90	6	92	15
Aflatoxin B1	104	9	93	5	95	9
Imazalil	92	1	96	3	98	1
Carbofuran	106	3	98	1	102	3
Propoxur	104	3	96	4	96	3
Carbaryl	103	4	99	8	93	4
Naled	101	4	94	2	84	4
Metalaxyl	105	3	97	2	97	3
Spiroxamine	105	4	94	1	97	4
Chlorantraniliprole	114	6	88	5	100	6
Phosmet	108	10	100	6	92	10
Azoxystrobin	104	1	94	1	94	1
Fludioxonil	101	7	90	11	94	7
Methiocarb	102	5	94	3	95	5
Dimethomorph	108	15	93	0	93	15
Boscalid	119	11	94	4	85	11
Paclobutrazol	110	10	90	2	92	10
Malathion	101	3	92	4	95	3
Myclobutanil	92	11	93	0	95	11
Bifenazate	105	2	98	4	99	2

	ME at 5 ppb (%)	RSD	ME at 15 ppb (%)	RSD	ME at 50 ppb (%)	RSD
Ochratoxin A	111	10	111	0	98	10
Fenhexamid	135	6	102	0	91	6
Spirotetramat	107	7	87	5	95	7
Ethoprophos	104	5	97	2	96	5
Fipronil	99	12	93	9	96	12
Fenoxycarb	108	9	97	1	93	9
Kresoxim methyl	93	21	108	3	90	21
Tebuconazole	102	6	95	3	97	6
Spinosyn A	100	5	98	2	95	5
Diazinon	107	2	95	1	96	2
Coumaphos	114	7	94	3	97	7
Pyridaben	139	18	103	15	94	18
Propiconazole	106	2	97	1	95	2
Clofentezine	126	14	87	3	92	14
Spinosyn D	104	8	93	8	97	8
Spinosyn J	110	6	92	6	101	6
Trifloxystrobin	103	2	95	2	101	2
Prallethrin	101	17	103	0	98	17
Pyrethrin II	92	23	82	5	101	23
Spinosyn L	106	6	95	0	98	6
Piperonyl butoxide	103	2	97	0	96	2
Chlorpyrifos	105	10	93	0	91	10
Hexythiazox	121	15	96	0	86	15
Etoxazole	102	1	95	1	95	1
Spiromesifen	112	6	95	5	100	6
Pyrethrin I	131	13	97	4	95	13
Cyfluthrin	-	-	-	-	80	8
Cypermethrin	-	-	95	16	96	16
(E)-Fenpyroximate	107	6	95	4	94	6
Permethrin-*trans*	136	22	103	4	94	22
Permethrin-*cis*	113	12	98	8	101	12
Avermectin B1a	122	17	96	0	87	17
Etofenprox	110	4	100	2	101	4
Bifenthrin	135	5	98	6	85	5
Acequinocyl	129	5	95	5	78	5

Table X: LOQ, Linearity, Accuracy, and Precision for Pesticides and Mycotoxins in Cannabis Gummies.

Contaminant	Action level (ng/g)	LOQ (ng/g)	R2	10 ng/g (n=4)		50 ng/g (n=4)		100 ng/g (n=4)		500 ng/g (n=4)	
				Accuracy (%)	Precision (RSD)	Accuracy (%)	Precision (RSD)	Accuracy (%)	Precision (RSD)	Accuracy (%)	Precision (RSD)
Daminozide*	<LOD	20	0.9999	-	-	114	6	114	4	116	4
Acephate	5000	5	0.9996	100	4	93	3	90	2	88	2
Oxamyl	200	5	0.999	108	1	102	3	105	4	99	3
Flonicamid	2000	50	0.999	-	-	117	6	105	11	101	4
Methomyl	100	20	0.9989	-	-	102	3	103	3	100	1
Thiamethoxam	4500	10	0.9988	112	23	108	5	108	6	100	3
Imidacloprid	3000	10	0.9985	105	17	107	3	109	4	102	5
Mevinphos (I and II)*	<LOD	20	0.9981	-	-	101	4	104	7	101	3
Acetamiprid	5000	10	0.9968	100	8	108	6	109	5	105	1
Dimethoate*	<LOD	5	0.9994	109	15	104	3	101	5	99	2
Thiacloprid*	<LOD	20	0.9981	-	-	100	1	103	4	103	3
Aflatoxin G2	20[#]	5	0.9957	112	17	101	6	97	2	-	-
Aflatoxin G1	20[#]	5	0.9984	114	9	98	1	100	4	-	-
Aldicarb*	<LOD	20	0.9971	-	-	91	17	104	8	97	4
Aflatoxin B2	20[#]	5	0.9973	97	23	107	6	94	7	-	-
Dichlorvos*	<LOD	10	0.9984	98	17	103	4	97	18	106	4
Aflatoxin B1	20[#]	5	0.9978	113	5	101	6	96	5	-	-
Imazalil*	<LOD	5	0.9977	97	19	109	5	107	4	105	3
Carbofuran*	<LOD	5	0.9973	93	7	108	1	109	5	99	4
Propoxur*	<LOD	5	0.9977	108	6	108	2	107	4	102	3
Carbaryl	500	5	0.9988	95	14	109	6	108	5	102	2
Naled	500	5	0.9968	98	4	112	8	110	3	101	5
Metalaxyl	15,000	5	0.9988	101	5	105	4	106	5	99	3
Spiroxamine*	<LOD	5	0.9977	104	6	106	2	105	2	101	3
Chlorantraniliprole	40,000	20	0.9971	-	-	93	7	104	6	104	5
Phosmet	200	5	0.9992	109	14	105	3	104	3	100	4
Azoxystrobin	40,000	5	0.9992	100	3	104	1	105	4	102	3
Fludioxonil	30,000	20	0.9949	-	-	109	13	97	6	101	6
Methiocarb*	<LOD	5	0.9988	116	16	105	5	107	5	101	4
Dimethomorph (I and II)	20,000	10	0.999	75	14	101	6	93	8	101	5
Boscalid	10,000	10	0.9964	108	15	102	12	102	2	103	3
Paclobutrazol*	<LOD	10	0.9979	99	9	106	1	108	3	100	4
Malathion	5000	10	0.9989	117	12	112	3	106	2	101	3
Myclobutanil	9000	10	0.9986	102	22	102	6	104	3	101	3
Bifenazate	5000	10	0.9994	118	20	110	3	104	10	102	5
Ochratoxin A	20	10	0.9957	99	8	99	24	104	8	-	-
Fenhexamid	10,000	10	0.9969	96	21	109	4	106	3	107	5
Spirotetramat	13,000	10	0.9987	83	18	107	6	106	3	105	3
Ethoprophos*	<LOD	5	0.9985	101	3	106	4	104	1	102	3
Fipronil*	<LOD	20	0.998	-	-	96	5	104	8	103	4
Fenoxycarb*	<LOD	10	0.9967	115	5	106	4	107	2	102	3
Kresoxym-methyl	1000	10	0.9993	112	20	104	10	102	4	103	3
Tebuconazole	2000	5	0.999	100	3	110	1	105	3	101	4
Spinosad- spinosyn A (71 %)[a]	3000[¥]	7.1	0.9988	117	2	110	3	108	2	102	1
Diazinon	200	5	0.9997	104	1	102	1	104	2	101	1

Continued

Table X *(cont.)*

Contaminant	Action level (ng/g)	LOQ (ng/g)	R2	10 ng/g (n=4)		50 ng/g (n=4)		100 ng/g (n=4)		500 ng/g (n=4)	
				Accuracy (%)	Precision (RSD)	Accuracy (%)	Precision (RSD)	Accuracy (%)	Precision (RSD)	Accuracy (%)	Precision (RSD)
Coumaphos*	<LOD	10	0.9991	107	11	109	5	108	2	103	2
Pyridaben	3000	50	0.9994	-	-	93	12	113	6	103	1
Propiconazole	20,000	5	0.9991	96	6	108	4	104	5	103	3
Clofentezine	500	20	0.9978	-	-	102	5	105	3	106	4
Spinosad - spinosyn D (29%)[b]	3000[¥]	2.9	0.9993	106	8	101	2	103	5	104	4
Spinetoram - spinosyn J (80%)[c]	3000[$]	4	0.9995	104	7	102	3	108	4	102	2
Trifloxystrobin	30,000	5	0.9996	107	3	106	3	106	3	104	2
Prallethrin	400	10	0.9967	107	21	99	16	111	6	101	3
Pyrethrin II (34%)[f]	1000[£]	17	0.9977	-	-	94	11	112	4	106	6
Spinetoram - spinosyn L (20%)[d]	3000[$]	2	0.9993	107	10	109	4	107	3	102	1
Piperonyl Butoxide	8000	5	0.9998	110	5	99	2	94	3	95	5
Chlorpyrifos*	<LOD	20	0.9995	-	-	103	9	106	4	104	5
Hexythiazox	2000	10	0.9975	104	15	102	3	107	4	106	5
Etoxazole	1500	5	0.9995	103	4	104	2	103	1	100	1
Spiromesifen	12,000	5	0.9992	98	8	109	7	111	2	102	2
Pyrethrin I (54%)[e]	1000[£]	11	0.9977	-	-	98	5	105	12	104	5
Cyfluthrin	1000	50	0.999	-	-	93	11	102	23	115	10
Cypermethrin	1000	50	0.9961	-	-	115	11	98	18	104	6
(E)-Fenpyroximate	2000	5	0.9995	102	9	109	3	109	2	105	2
Permethrin-*trans* (59%)[h]	20,000[c]	12	0.9994	-	-	99	8	104	5	101	3
Permethrin-*cis* (41%)[g]	20,000[c]	8	0.9996	95	5	104	5	103	4	101	3
Avermectin B1a	300	50	0.9988	-	-	114	3	108	2	105	2
Etofenprox*	<LOD	5	0.9994	104	7	107	1	106	2	103	1
Bifenthrin	500	5	0.999	99	5	103	2	108	6	104	2
Acequinocyl	4000	10	0.9997	104	7	109	3	108	3	106	2
Quintozene (PCNB) (GC)	200	10	0.9966	110	14	102	5	101	1	97	3
Methyl parathion (GC)*	<LOD	5	0.9934	89	10	89	5	87	3	89	6
Captan (GC)	5000	10	0.9924	110	14	96	10	94	18	92	7
Chlordane (GC)*	<LOD	20	0.9913	-	-	105	9	93	8	85	10
Chlorfenapyr (GC)*	<LOD	10	0.9924	97	9	90	19	89	6	85	13
Cyfluthrin (GC)	1000	5	0.9935	107	10	92	18	91	7	89	11
Cypermethrin (GC)	1000	5	0.9938	95	9	83	17	97	15	93	9

*Category I pesticides, LOQ ≤100 ng/g
[a] Spinosad- spinosyn A: Conc. 1: 7 ng/g; Conc. 2: 35.5 ng/g; Conc. 3: 71 ng/g; Conc. 4: 355 ng/g
[b] Spinosad - spinosyn D: Conc. 1: 3 ng/g; conc. 2: 14.5 ng/g; Conc. 3: 29 ng/g; Conc. 4: 145 ng/g
[c] Spinetoram - spinosyn J: Conc. 1: 8 ng/g; Conc. 2: 40 ng/g; Conc. 3: 80 ng/g; Conc. 4: 400 ng/g
[d] Spinetoram - spinosyn L: Conc. 1: 2 ng/g; Conc. 2: 10 ng/g; Conc. 3: 20 ng/g; Conc. 4: 100 ng/g
[e] Pyrethrin I: Conc. 1: 5 ng/g; Conc. 2: 27 ng/g; Conc. 3: 54 ng/g; Conc. 4: 270 ng/g
[f] Pyrethrin II: Conc. 1: 3 ng/g; Conc. 2: 17 ng/g; Conc. 3: 34 ng/g; Conc. 4: 170 ng/g
[g] Permethrin-*cis*: Conc. 1: 4 ng/g; Conc. 2: 20.5 ng/g; Conc. 3: 41 ng/g; Conc. 4: 205 ng/g
[h] Permethrin-*trans*: Conc. 1: 6 ng/g; Conc. 2: 29.5 ng/g; Conc. 3: 59 ng/g; Conc. 4: 295 ng/g
[#] Total of aflatoxin B1, B2, G1, and G2 should not exceed 20 ng/g.
[¥] Total spinosad should not exceed 3000 ng/g.
[$] Total spinetoram should not exceed 3000 ng/g.
[£] Total pyrethrins should not exceed 1000 ng/g.
[c] Total permethrins should not exceed 20,000 ng/g.

Table XI: Linearity for Cannabinoids in Cannabis Gummies.

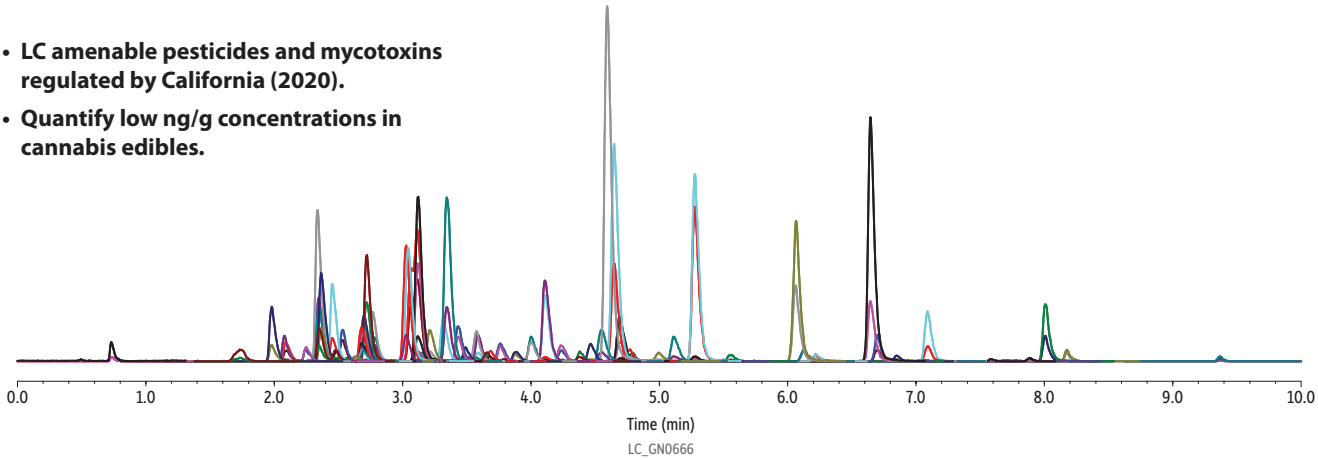
Cannabinoids	Retention time	R^2	Equation
Cannabidiolic acid (CBDA)	2.142	0.9993	y = 2.08e+004x + 1.03e+003
Cannabigerol (CBG)	2.405	0.9981	y = 1.16e+004x + 1.85e+003
Cannabidiol (CBD)	2.535	0.9972	y = 1.17e+004x + 1.42e+003
Cannabinol (CBN)	3.776	0.9980	y = 2.70e+004x + 6.14e+004
Delta-9-tetrahydrocannabinol (Delta-9-THC)	4.753	0.9970	y = 1.06e+004x + 7.29e+003
Tetrahydrocannabinolic acid (THCA)	6.279	0.9986	y = 1.78e+004x - 1.28e+003

Table XII: Accuracy and Precision for Cannabinoids in Cannabis Gummies.

Cannabinoid/Spike Level	Diluted Extract Concentration (ppm)		Average (ppm)	SD	RSD (%)	Undiluted Extract Conc. (ppm)	Estimated Sample Concentration (mg/g)	Accuracy (%)	Percent Error
Gummy spiked at 0.2 mg/g	Replicate 1	Replicate 2							
Cannabidiolic acid (CBDA)	4.2	4.4	4.3	0.1	3	43	0.2	107	7
Cannabigerol (CBG)	4.0	3.9	4.0	0.1	2	40	0.2	99	1
Cannabidiol (CBD)	4.1	4.2	4.1	0.1	2	41	0.2	103	3
Cannabinol (CBN)	4.0	4.1	4.1	0.1	3	41	0.2	101	1
Delta-9 tetrahydrocannabinol (Delta 9 THC)	4.0	4.2	4.1	0.1	3	41	0.2	103	3
Tetrahydrocannabinolic acid (THCA)	4.1	4.3	4.2	0.1	3	42	0.2	105	5
Gummy spiked at 0.5 mg/g									
Cannabidiolic acid (CBDA)	10.4	10.7	10.6	0.2	2	106	0.5	106	6
Cannabigerol (CBG)	9.5	10.3	9.9	0.5	5	99	0.5	99	1
Cannabidiol (CBD)	9.8	10.6	10.2	0.5	5	102	0.5	102	2
Cannabinol (CBN)	9.8	10.3	10.0	0.4	4	100	0.5	100	0
Delta-9-tetrahydrocannabinol (Delta-9-THC)	9.9	10.3	10.1	0.3	3	101	0.5	101	1
Tetrahydrocannabinolic acid (THCA)	10.3	9.9	10.1	0.2	2	101	0.5	101	1

Figure 2: LC-MS/MS chromatogram of an extract obtained from blank gummy spiked with pesticides and mycotoxins at 100 ng/g.

- **LC amenable pesticides and mycotoxins regulated by California (2020).**
- **Quantify low ng/g concentrations in cannabis edibles.**



Peaks	t_R (min)	Precursor Ion	Product Ion 1	Product Ion 2	Polarity
1. Daminozide-d6	0.7	167.0	149.3	49.3	+
2. Daminozide	0.7	161.1	44.1	143.2	+
3. Acephate	1.7	184.0	143.1	95.1	+
4. Oxamyl	2.0	237.1	72.1	90.1	+
5. Flonicamid	2.1	230.1	203.1	174.1	+
6. Methomyl	2.1	163.1	88.1	106.1	+
7. Thiamethoxam	2.1	292.0	211.1	181.1	+
8. Imidacloprid	2.3	256.1	209.1	175.1	+
9. Mevinphos	2.4	225.1	127.1	193.2	+
10. Acetamiprid	2.4	223.0	126.1	56.1	+
11. Dimethoathe-d6	2.4	236.1	205.1	-	+
12. Dimethoate	2.4	230.0	199.1	125.1	+
13. Thiacloprid	2.5	253.0	126.0	90.1	+
14. Aflatoxin G2	2.5	331.2	189.3	115.2	+
15. Aflatoxin G1	2.5	329.2	243.2	215.3	+
16. Aldicarb	2.6	116.0	89.2	70.2	+
17. Aflatoxin B2	2.6	315.3	287.2	243.3	+
18. Dichlorvos	2.7	220.9	109.1	79.2	+
19. Dichlorvos-d6	2.7	227.0	115.1	-	+
20. Aflatoxin B1	2.7	313.2	241.2	128.2	+
21. Imazalil	2.7	297.0	159.0	201.0	+
22. Carbofuran	2.7	222.1	123.1	165.2	+
23. Propoxur	2.7	210.1	111.1	93.1	+
24. Carbaryl-d7	2.8	209.2	152.2	-	+
25. Carbaryl	2.8	202.1	145.1	127.1	+
26. Diuron-d6	3.0	239.1	78.2	-	+
27. Atrazine-d5	3.0	221.2	179.1	-	+
28. Naled	3.1	397.8	127.1	109.1	+
29. Metalaxyl	3.1	280.2	220.2	192.2	+
30. Spiroxamine	3.1	298.3	144.2	100.2	+
31. Chlorantraniliprole	3.2	483.9	452.9	285.9	+
32. Phosmet	3.2	318.0	160.1	77.2	+
33. Azoxystrobin	3.3	404.0	372.1	344.1	+
34. Linuron-d6	3.3	255.1	160.1	-	+
35. Fludioxonil	3.4	247.0	180.0	126.0	-
36. Methiocarb	3.4	226.1	169.1	121.1	+
37. Dimethomorph	3.5	388.2	301.2	165.3	+
38. Boscalid	3.5	342.9	307.1	140.1	+
39. Paclobutrazol	3.6	294.3	70.1	125.1	+
40. Malathion	3.6	331.0	127.2	285.2	+
41. Myclobutanil	3.7	289.1	70.1	125.1	+
42. Bifenazate	3.7	301.0	198.1	170.2	+
43. Ochratoxin A	3.8	404.2	239.1	358.3	+
44. Fenhexamid	3.9	302.1	97.1	55.2	+
45. Spirotetramat	4.0	374.2	302.1	216.1	+
46. Ethoprophos	4.1	243.1	131.1	97.1	+
47. Fipronil	4.1	436.8	331.8	251.9	-
48. Fenoxycarb	4.2	302.1	88.1	116.1	+
49. Kresoxim-methyl	4.4	314.2	267.2	222.2	+
50. Tebuconazole	4.4	308.1	70.1	125.1	+
51. Diazinon-d10	4.6	315.2	170.2	-	+
52. Spinosyn A (Spinosad)	4.6	732.4	142.2	98.1	+
53. Diazinon	4.6	305.1	169.2	153.2	+
54. Coumaphos	4.7	363.1	227.1	307.1	+
55. Pyridaben	4.7	365.1	309.2	147.2	+
56. Propiconazole	4.7	342.0	159.0	69.2	+
57. Clofentezine	4.8	303.0	138.1	102.1	+
58. Spinosyn D (Spinosad)	5.0	746.5	142.3	98.4	+
59. Spinosyn J (Spinetoram)	5.1	748.5	142.3	98.3	+
60. Trifloxystrobin	5.3	409.2	186.1	145.1	+
61. Prallethrin	5.3	301.2	123.2	105.2	+
62. Pyrethrin II	5.5	373.1	161.1	133.2	+
63. Spinosyn L (Spinetoram)	5.6	760.5	142.2	98.1	+
64. Piperonyl butoxide	6.0	356.3	177.2	119.2	+
65. Chlorpyrifos	6.1	349.9	198.0	97.1	+
66. Hexythiazox	6.2	353.1	228.1	168.1	+
67. Etoxazole	6.6	360.2	141.1	304.2	+
68. Spiromesifen	6.7	273.2	255.2	187.2	+
69. Pyrethrin I	6.9	329.2	161.2	105.2	+
70. Cyfluthrin (qualifier)	6.9	453.1	193.2	-	+
71. Cyfluthrin	6.9	451.1	191.2	-	+
72. Cypermethrin	7.1	433.1	191.0	416.0	+
73. (E)-Fenpyroximate	7.1	422.2	366.1	138.1	+
74. *trans*-Permethrin	7.6	408.3	183.2	355.1	+
75. *cis*-Permethrin	7.9	408.3	183.2	355.1	+
76. Avermectin B1a	7.9	890.5	305.4	567.4	+
77. Etofenprox	8.0	394.3	177.2	359.3	+
78. Bifenthrin	8.2	440.0	181.2	166.2	+
79. Acequinocyl (precursor ion 1)	9.4	402.3	343.2	189.0	+
80. Acequinocyl (precursor ion 2)	9.4	386.0	344.2	189.1	+

Column Raptor ARC-18 (cat.# 9314A12)
Dimensions: 100 mm x 2.1 mm ID
Particle Size: 2.7 µm
Pore Size: 90 Å
Guard Column: Raptor ARC-18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9314A0252)
Temp.: 40 °C
Sample California pesticide standard #1 (cat.# 34124); California pesticide standard #2 (cat.# 34125); California pesticide standard #3 (cat.# 34126); California pesticide standard #4 (cat.# 34127); California pesticide standard #5 (cat.# 34128); California pesticide standard #6 (cat.# 34129); Dimethoate-d6 (cat.# 31988); Dichlorvos-d6 (cat.# 31987); Carbaryl-d7 (cat.# 31985); Diazinon-d10 (cat.# 31986); Atrazine-d5 (cat.# 31984); Diuron-d6 (cat.# 31989); Liuron-d6 (cat.# 31990); Aflatoxins standard (cat.# 34121); Ochratoxin A (cat.# 34122); Compounds not present in these mixes were obtained separately.
Diluent: 75:25 Acetonitrile:water
Conc.: 3.75-15 ng/mL (Expected concentration range in extract of gummy initially spiked at 100 ng/g.)
Inj. Vol.: 2 µL
Mobile Phase
A: Water, 2 mM ammonium formate, 0.1% formic acid
B: Methanol, 2 mM ammonium formate, 0.1% formic acid

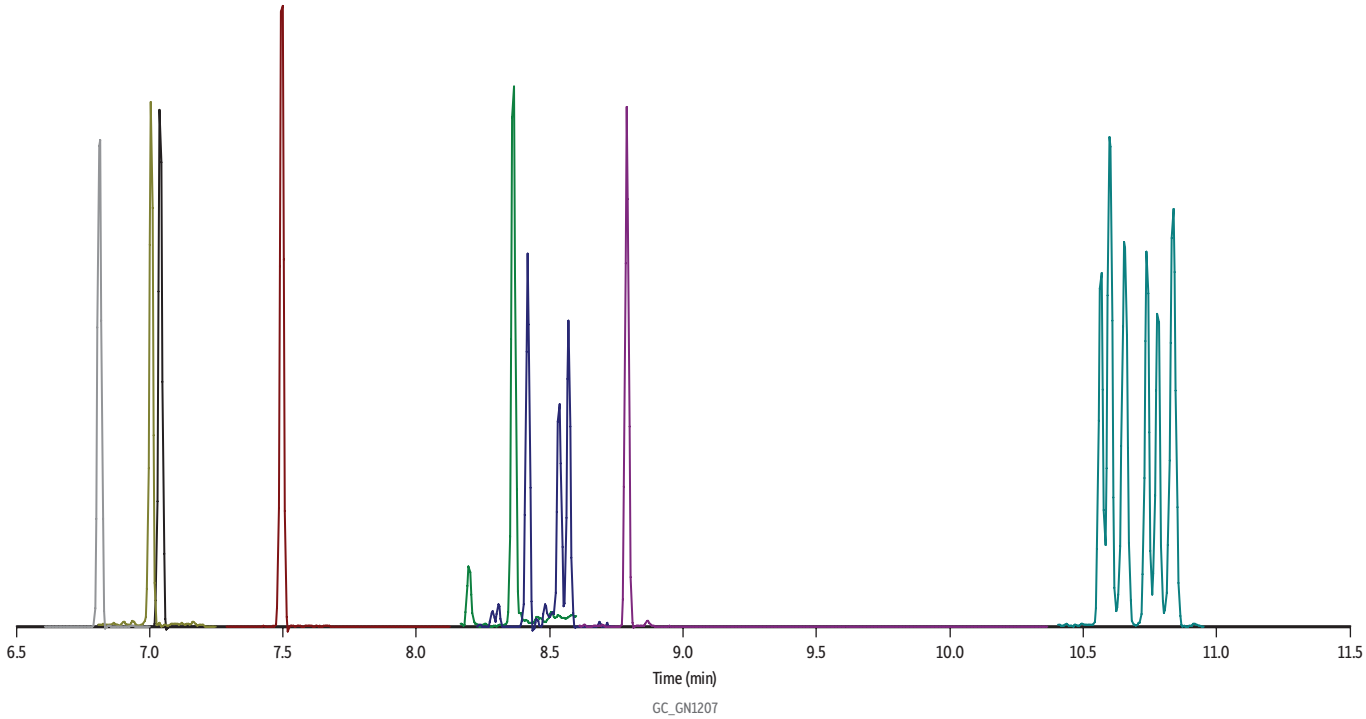
Time (min)	Flow (mL/min)	%A	%B
0.00	0.5	95	5
1.5	0.5	35	65
8.5	0.5	5	95
9.5	0.5	0	100
10.5	0.5	0	100
10.6	0.5	95	5
12.0	0.5	95	5

Detector MS/MS
Ion Mode: ESI+/ESI-
Mode: MRM
Instrument UHPLC
Notes Gummies were manually chopped into small pieces, and 1 g of sample was weighed in a 50 mL polypropylene tube. The sample was mixed with 5 mL of water and then vigorously vortexed until all gummy pieces were fully solubilized. The sample was fortified with pesticides and mycotoxins at 100 ng/g. A mix of internal standards was added at 200 ng/g. The spiked sample was further vortexed for 30 sec. 5 mL of acetonitrile acidified with 1% acetic acid was added to the sample, and this was followed by a 30 sec vortex agitation. Then, a pouch of European EN 15662 QuEChERS extraction salts (cat.# 25849) was added to the sample. The sample was vortexed for 30 sec and then centrifuged for 5 min. 750 µL of organic extract was mixed with 250 µL of water. 2 µL of final extract was injected into the LC-MS/MS system.

Notes Want even better performance when analyzing metal-sensitive compounds? Check out Inert LC columns at www.restek.com/inert

Figure 3: GC-MS/MS chromatogram of an extract obtained from blank gummy spiked with pesticides and mycotoxins at 100 ng/g.

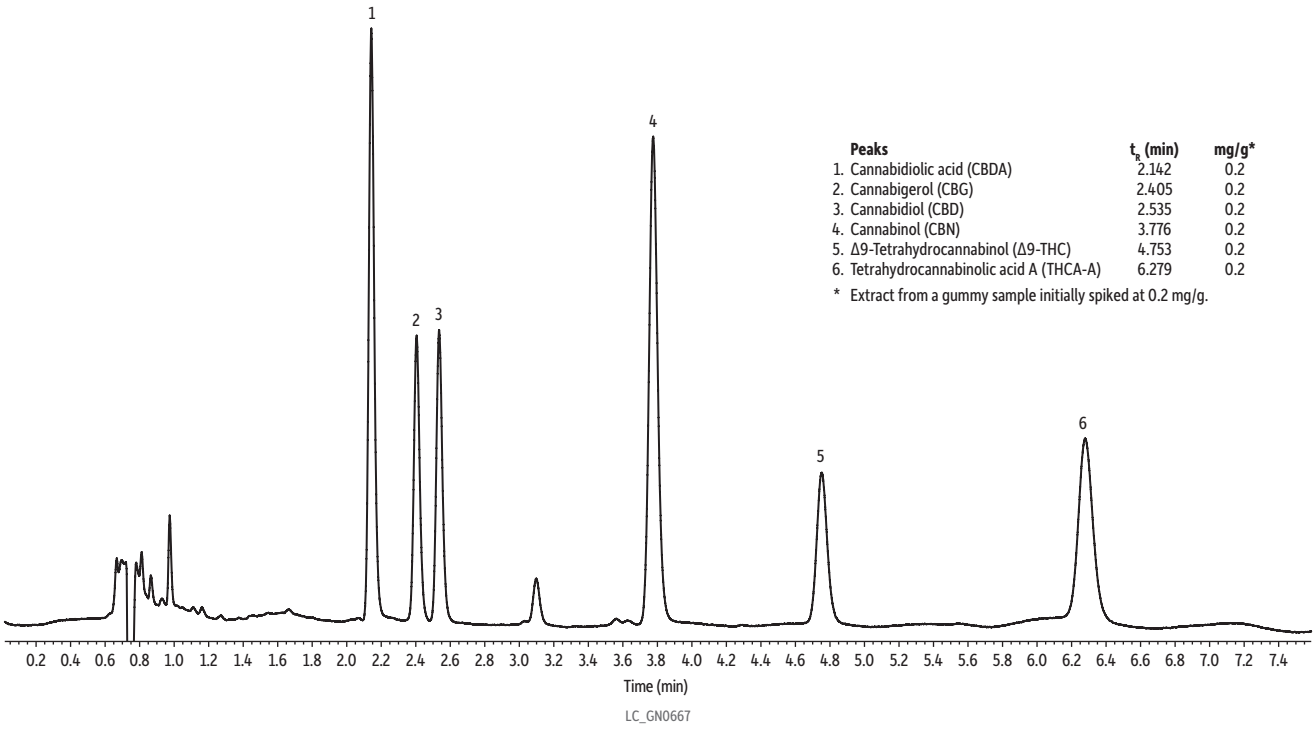
- **GC amenable pesticides regulated by California (2020).**
- **Quantify pesticides in cannabis edibles at low ng/g concentrations.**



Peaks	t_R (min)	Polarity	Precursor Ion	Product Ion	Transition Type
1. Atrazine-d5	6.82	Positive	220.0	58.0	Quantifier
2. Atrazine-d5	6.82	Positive	205.0	127.0	Qualifier
3. Diazinon-d10 (diethyl-d10)	7.01	Positive	183.0	139.0	Quantifier
4. Diazinon-d10 (diethyl-d10)	7.01	Positive	183.0	168.0	Qualifier
5. Quintozene	7.03	Positive	294.9	236.9	Quantifier
6. Quintozene	7.03	Positive	236.8	118.9	Qualifier
7. Methyl parathion	7.50	Positive	263.0	109.0	Quantifier
8. Methyl parathion	7.50	Positive	263.0	79.0	Qualifier
9. Captan	8.37	Positive	184.0	149.1	Quantifier
10. Captan	8.37	Positive	184.0	134.1	Qualifier
11. *trans*-Chlordane	8.41	Positive	271.9	237.0	Quantifier
12. *trans*-Chlordane	8.41	Positive	372.9	265.9	Qualifier
13. *cis*-Chlordane	8.53	Positive	372.9	265.9	Quantifier
14. *cis*-Chlordane	8.53	Positive	271.9	237.0	Qualifier
15. Chlorfenapyr	8.80	Positive	247.1	227.1	Quantifier
16. Chlorfenapyr	8.80	Positive	59.1	31.1	Qualifier
17. Cyfluthrin	10.61	Positive	226.0	206.0	Quantifier
18. Cyfluthrin	10.61	Positive	163.0	127.0	Qualifier
19. Cypermethrin	10.87	Positive	163.0	127.1	Quantifier
20. Cypermethrin	10.87	Positive	181.1	152.1	Qualifier

Column	Rxi-5ms, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13423)
Sample	California pesticide standard #1 (cat.# 34124) California pesticide standard #2 (cat.# 34125) California pesticide standard #3 (cat.# 34126) California pesticide standard #4 (cat.# 34127) California pesticide standard #5 (cat.# 34128) California pesticide standard #6 (cat.# 34129) Atrazine-d5 (cat.# 31984) Diazinon-d10 (cat.# 31986)
Diluent:	Acetonitrile
Conc.:	2.5-10 ng/mL Expected concentration range in extract after extracting from gummy fortified at 100 ng/g (final extract was diluted in half with acetonitrile).
Injection	
Inj. Vol.:	1 µL splitless
Liner:	Topaz 4.0 mm ID single taper inlet liner w/wool (cat.# 23447)
Inj. Temp.:	250 °C
Purge Flow:	5 mL/min
Oven	
Oven Temp.:	90 °C (hold 1 min) to 310 °C at 25 °C/min (hold 10 min)
Carrier Gas	He, constant flow
Flow Rate:	1.4 mL/min
Detector	MS/MS
Transfer Line Temp.:	290 °C
Analyzer Type:	Quadrupole
Source Temp.:	330 °C
Electron Energy:	70 eV
Tune Type:	PFTBA
Ionization Mode:	EI
Instrument	Thermo Scientific TSQ 8000 Triple Quadrupole GC-MS
Notes	Gummies were manually chopped into small pieces, and 1 g of sample was weighed in a 50 mL polypropylene tube. The sample was mixed with 5 mL of water and then vigorously vortexed until all gummy pieces were fully solubilized. The sample was fortified with pesticides and mycotoxins at 100 ng/g. A mix of internal standards was added at 200 ng/g. The spiked sample was further vortexed for 30 sec. 5 mL of acetonitrile acidified with 1% acetic acid was added to the sample, and this was followed by 30 sec vortex agitation. Then, a pouch of European EN 15662 QuEChERS extraction salts (cat.# 25849) was added to the sample. The sample was vortexed for 30 sec and then centrifuged for 5 min. 1.9 mL of supernatant was transferred to a Q-sep QuEChERS dSPE tube containing pre-weighed magnesium sulfate, PSA, and GCB (cat.# 26217). After vortexing and centrifuging, 500 µL of extract was mixed with 500 µL of acidified acetonitrile. 1 µL of final extract was injected into the GC-MS/MS system.

Figure 4: HPLC-UV chromatogram of an extract obtained from blank gummy spiked with six cannabinoids at 0.2 mg/g.



Peaks	t_R (min)	mg/g*
1. Cannabidiolic acid (CBDA)	2.142	0.2
2. Cannabigerol (CBG)	2.405	0.2
3. Cannabidiol (CBD)	2.535	0.2
4. Cannabinol (CBN)	3.776	0.2
5. Δ9-Tetrahydrocannabinol (Δ9-THC)	4.753	0.2
6. Tetrahydrocannabinolic acid A (THCA-A)	6.279	0.2

* Extract from a gummy sample initially spiked at 0.2 mg/g.

Column	Raptor ARC-18 (cat.# 9314A62)
Dimensions:	150 mm x 2.1 mm ID
Particle Size:	2.7 µm
Pore Size:	90 Å
Guard Column:	Raptor ARC-18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9314A0252)
Temp.:	30 °C
Sample	Cannabinoids standard (cat.# 34014) Cannabigerol (cat.# 34091) d9-Tetrahydrocannabinol (cat.# 34067) d9-Tetrahydrocannabinolic acid A (cat.# 34111)
Diluent:	75:25 Acetonitrile:water
Conc.:	Expected concentration of 4 ppm in final extract from gummy initially spiked at 0.2 mg/g.
Inj. Vol.:	2 µL

Mobile Phase	
A:	Water, 5 mM ammonium formate, 0.1% formic acid
B:	Acetonitrile, 0.1% formic acid

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	25	75
10.00	0.4	25	75

Detector	UV/Vis @ 228 nm
Instrument	UHPLC
Notes	Gummies were manually chopped into small pieces, and 1 g of sample was weighed in a 50 mL polypropylene tube. The sample was mixed with 5 mL of water and then vigorously vortexed until all gummy pieces were fully solubilized. The sample was fortified with cannabinoids at 0.2 mg/g. The spiked sample was further vortexed for 30 sec. 5 mL of acetonitrile acidified with 1% acetic acid was added to the sample, and this was followed by 30 sec vortex agitation. Then, a pouch of European EN 15662 QuEChERS extraction salts (cat.# 25849) was added to the sample. The sample was vortexed for 30 sec and then centrifuged for 5 min. 100 µL of organic extract was mixed with 900 µL of 75:25 acetonitrile:water. 2 µL of final extract was injected into the HPLC-UV system.

Conclusion

An easy and effective workflow for the analysis of pesticides, mycotoxins, and cannabinoids in cannabis gummies was developed. Sample preparation conditions involved matrix homogenization of gummy pieces with water, extraction of analytes using acidified acetonitrile followed by a salting-out step using Q-sep QuEChERS extraction salts; extract dilution (for LC-MS/MS amenable contaminants and cannabinoids); and dSPE cleanup using magnesium sulfate, PSA, and GCB (for GC-MS/MS amenable pesticides). Satisfactory results in terms of LOQ, linearity, accuracy, and precision were obtained for all the target contaminants. In addition, our data demonstrated that the proposed methodology is suitable for potency testing with accuracy values ranging from 99 to 107% for the six cannabinoids listed in the cannabis regulations of the state of California. Overall, the presented workflow streamlines work for cannabis testing labs by enabling the satisfactory quantitation of multiple analyte classes in gummy samples using a single extract.

References

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[5] B. K. Matuszewski, M. L. Constanzer, C. M. Chavez-Eng, Strategies for the assessment of matrix effect in quantitative bioanalytical methods based on HPLC–MS/MS, Anal. Chem. 75 (2003) 3019–3030. https://pubs.acs.org/doi/10.1021/ac020361s

Raptor ARC-18 LC Columns (USP L1)

- Ideal for high-throughput LC-MS/MS applications with minimal sample preparation.
- Well-balanced retention profile for better detection and integration of large, multiclass analyte lists.
- Sterically protected to endure low-pH mobile phases without sacrificing retention or peak quality.
- Part of Restek's Raptor LC column line featuring 1.8, 2.7, and 5 µm SPP core-shell silica.

Stationary Phase Category: C18, octadecylsilane (L1)
Ligand Type: Sterically protected C18
Particle: 1.8 µm, 2.7 µm, or 5 µm superficially porous particle (SPP or "core-shell" particle) silica
Pore Size: 90 Å
Carbon Load: 7% (1.8 µm), 7% (2.7 µm), 5% (5 µm)
End-Cap: no
Surface Area: 125 m^2/g (1.8 µm), 130 m^2/g (2.7 µm), or 100 m^2/g (5 µm)
Recommended Usage:
pH Range: 1.0–8.0
Maximum Temperature: 80 °C
Maximum Pressure: 1034 bar/15,000 psi* (1.8 µm), 600 bar/8700 psi (2.7 µm); 400 bar/5800 psi (5 µm)
* For maximum lifetime, recommended maximum pressure for 1.8 µm particles is 830 bar/12,000 psi.

Properties:
- Well-balanced retention profile.
- Sterically protected and acid resistant to resist harsh, low-pH mobile phases.
- Ideal for use with sensitive detectors like mass spec.

Switch to an ARC-18 column when:
- You are analyzing large, multiclass lists by LC-MS/MS.
- Strongly acidic (pH 1–3) mobile phases are required.

ID	Length	qty.	cat.#
2.7 µm Particles Raptor ARC-18			
2.1 mm	30 mm	ea.	9314A32
	50 mm	ea.	9314A52
	100 mm	ea.	9314A12
	150 mm	ea.	9314A62
3.0 mm	30 mm	ea.	9314A3E
	50 mm	ea.	9314A5E
	100 mm	ea.	9314A1E
	150 mm	ea.	9314A6E
4.6 mm	30 mm	ea.	9314A35
	50 mm	ea.	9314A55
	100 mm	ea.	9314A15
	150 mm	ea.	9314A65

ordering notes

Certificates of analysis for new Restek LC columns are now provided electronically. To view and download, visit www.restek.com/documentation then enter your cat.# and serial #.

Want even better performance when analyzing mycotoxins? Check out Inert LC columns at **www.restek.com/inert**

Raptor Inert ARC-18 HPLC Columns

- Inert LC column technology reduces nonspecific binding of chelating analytes, enabling sensitive analysis and smooth integration of peaks.
- Ideal for the analysis of metal-sensitive compounds, such as organophosphorus pesticides.
- Increased response and analyte recovery, allowing lower detection limits.
- Improved peak shape without additional passivation or mobile phase additives.
- Part of Restek's Raptor ARC-18 column line featuring 2.7 µm SPP core-shell silica.

ID	Length	qty.	cat.#
2.7 µm Particles Raptor Inert ARC-18			
2.1 mm	50 mm	ea.	9314A52-T
	100 mm	ea.	9314A12-T
3.0 mm	50 mm	ea.	9314A5E-T
	100 mm	ea.	9314A1E-T

Raptor EXP Guard Column Cartridges

- Free-Turn architecture lets you change cartridges by hand without breaking inlet/outlet fluid connections—no tools needed.
- Patented titanium hybrid ferrules can be installed repeatedly without compromising high-pressure seal.
- Auto-adjusting design provides ZDV (zero dead volume) connection to any 10-32 female port.
- Guard column cartridges require EXP direct connect holder (cat.# 25808).
- Pair with EXP hand-tight fitting (cat.# 25937–25938) for tool-free installation.

Description	Particle Size	Length	ID	qty.	cat.#
Raptor ARC-18 EXP Guard Column Cartridge	2.7 µm	5 mm	2.1 mm	3-pk.	9314A0252

Maximum cartridge pressure: 1034 bar/15,000 psi* (UHPLC); 600 bar/8700 psi (2.7 µm); 400 bar/5800 psi (5 µm).
* For maximum lifetime, recommended maximum pressure for UHPLC particles is 830 bar/12,000 psi.
Intellectual Property: optimizetech.com/patents

Rxi-5ms Columns (fused silica)

low-polarity phase; Crossbond diphenyl dimethyl polysiloxane

- Ideal for pesticides in food.
- General-purpose columns that can be used for phenols, residual solvents, drugs of abuse, pesticides, semivolatiles, PCB congeners (e.g., Aroclor mixes), and solvent impurities.
- Tested and guaranteed for ultra-low bleed; improved signal-to-noise ratio for better sensitivity and mass spectral integrity.
- Temperature range: -60 °C to 330/350 °C.
- Equivalent to USP G27 and G36 phases.

ID	df	Length	Temp. Limits	qty.	Similar to Part #	cat.#
Rxi-5ms						
0.25 mm	0.25 µm	30 m	-60 to 330/350 °C	ea.	Agilent 19091S-433UI; Phenomenex 7HG-G032-11	13423
	0.25 µm	30 m	-60 to 330/350 °C	6-pk.		13423-600

Topaz 4.0 mm ID Single Taper Inlet Liner w/ Wool

for Thermo TRACE 1300/1310, 1600/1610 GCs equipped with SSL inlets

Description	Length	ID	OD	Deactiva-tion	Material	Packing	qty	Similar to Part #	cat.#
Single Taper									
4.0 mm ID Single Taper Liner w/ Wool	78.5 mm	4.0 mm	6.5 mm	Premium	Borosilicate Glass	Quartz Wool	5-pk.	Thermo Fisher Scientific 453A1925-UI	23447

California Pesticide Standards

(6 separate mixes)

- Meet specific cannabis analysis needs of California set forth by the Bureau of Cannabis Control for regulated category I and II residual pesticide reporting—and of states with similar regulations/programs.
- Ideal for creating multipoint (5-point minimum suggested) calibration curves for GC-MS/MS and LC-MS/MS.
- Verified composition and stability.
- 66 compounds in 6 x 1 mL ampuls at 100 µg/mL.
- Prepared stock product eliminates the need for in-house standards preparation.

Each ampul sold separately.

Cat.# 34124: California Pesticide Standard #1 (12 components)
Acephate (30560-19-1)
Chlorpyrifos (2921-88-2)
Coumaphos (56-72-4)
Diazinon (333-41-5)
Dichlorvos (DDVP) (62-73-7)
Dimethoate (60-51-5)
Ethoprophos (13194-48-4)
Malathion (121-75-5)
Methyl parathion (298-00-0)
Mevinphos (7786-34-7)
Naled (300-76-5)
Phosmet (732-11-6)

Cat.# 34125: California Pesticide Standard #2 (11 components)
Abamectin (71751-41-2)
Acequinocyl (57960-19-7)
Bifenthrin (82657-04-3)
Cyfluthrin (68359-37-5)
Cypermethrin (52315-07-8)
Etofenprox (80844-07-1)
Permethrin (*cis* & *trans*) (52645-53-1)
Prallethrin (23031-36-9)
Pyrethrins (8003-34-7)
Spinetoram (J&L) (935545-74-7)*
Spinosad (A&D) (168316-95-8)**

Cat.# 34126: California Pesticide Standard #3 (9 components)
Aldicarb (116-06-3)
Bifenazate (149877-41-8)
Carbaryl (Sevin) (63-25-2)
Carbofuran (1563-66-2)
Fenoxycarb (72490-01-8)
Methiocarb (2032-65-7)
Methomyl (16752-77-5)
Oxamyl (23135-22-0)
Propoxur (Baygon) (114-26-1)

Cat.# 34127: California Pesticide Standard #4 (9 components)
Boscalid (188425-85-6)
Captan (133-06-2)
Chlorantraniliprole (500008-45-7)
Daminozide (1596-84-5)
Dimethomorph (110488-70-5)
Fenhexamid (126833-17-8)
Flonicamid (158062-67-0)
Hexythiazox (78587-05-0)
Pyridaben (96489-71-3)

Cat.# 34128: California Pesticide Standard #5 (10 components)
Azoxystrobin (131860-33-8)
Chlorfenapyr (122453-73-0)
(E)-Fenpyroximate (134098-61-6)
Kresoxim methyl (143390-89-0)
Metalaxyl (57837-19-1)
Piperonyl butoxide (51-03-6)
Spiromesifen (283594-90-1)
Spirotetramat (203313-25-1)
Spiroxamine (118134-30-8)
Trifloxystrobin (141517-21-7)

Cat.# 34129: California Pesticide Standard #6 (15 components)
Acetamiprid (135410-20-7)
Chlordane (57-74-9)
Clofentezine (74115-24-5)
Etoxazole (153233-91-1)
Fipronil (120068-37-3)
Fludioxonil (131341-86-1)
Imazalil (35554-44-0)
Imidacloprid (138261-41-3)
Myclobutanil (88671-89-0)
Paclobutrazol (76738-62-0)
Pentachloronitrobenzene (Quintozene) (82-68-8)
Propiconazole (Tilt) (60207-90-1)
Tebuconazole (107534-96-3)
Thiacloprid (111988-49-9)
Thiamethoxam (153719-23-4)

both the Spinetoram J and L isomers (187166-40-1 and 187166-15-0); however, CAS# 935545-74-7 is displayed on the certificate as this is the neat material dissolved in the solution. CAS# 935545-74-7 is a blend of Spinetoram J and L and the ratio of each material isomer are displayed on your certificate of analysis.

**This reference material contains Spinosad A and D isomers (131929-60-7 and 131929-63-0); however, CAS# 168316-95-8 is displayed on the certificate as this is the neat material dissolved in the solution. CAS# 168316-95-8 is a blend of Spinosad A and D and the ratio of each material isomer are displayed on your certificate of analysis.

Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
California Pesticide Standard #1							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	14 months	On Ice	–20 °C or colder	ea.	34124
California Pesticide Standard #2							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	24 months	Ambient	–20 °C or colder	ea.	34125
California Pesticide Standard #3							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	24 months	Ambient	–20 °C or colder	ea.	34126
California Pesticide Standard #4							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	24 months	Ambient	0 °C or colder	ea.	34127
California Pesticide Standard #5							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	24 months	Ambient	–20 °C or colder	ea.	34128
California Pesticide Standard #6							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	24 months	Ambient	–20 °C or colder	ea.	34129

Aflatoxins (B1, B2, G1, G2) Standard

(4 components)

Ideal for mycotoxin analyses in cannabis and food testing labs.

Aflatoxin B1 (1162-65-8) Aflatoxin B2 (7220-81-7) Aflatoxin G1 (1165-39-5) Aflatoxin G2 (7241-98-7)

Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	Data pack available?	qty.	cat.#
10 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	36 months	On Ice	0 °C or colder	No	ea.	34121

Ochratoxin A Standard

Ideal for mycotoxin analyses in cannabis and food testing labs.

Ochratoxin A (303-47-9)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	Data pack available?	qty.	cat.#
Ochratoxin A	303-47-9	10 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	36 months	On Ice	0 °C or colder	No	ea.	34122

Cannabinoids Standard

(3 components)

U.S. DEA-exempted formulation—no additional customer permits or licensing are required to purchase within the U.S.

Cannabidiol (CBD) (13956-29-1) Cannabinol (CBN) (521-35-7) d9-Tetrahydrocannabinol (d9- THC) (1972-08-3)

Conc. in Solvent	CRM?	DEA Status	Canadian Test Kit Registration	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Cannabinoids Standard									
1000 µg/mL each in P&T methanol, 1 mL/ampul	Yes	Exempt	T.K.# 71-048	6 months	24 months	On Ice	–20 °C or colder	ea.	34014

Cannabigerol (CBG) Standard

Excluded from U.S. DEA Controlled Substances Act (CSA) regulatory controls—no customer permits or licensing required to purchase within the U.S.

Cannabigerol (CBG) (25654-31-3)

Description	CAS #	Conc. in Solvent	CRM?	DEA Status	Canadian Test Kit Registration	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Cannabigerol (CBG)	25654-31-3	1000 µg/mL in P&T methanol, 1 mL/ampul	Yes	Not Controlled	T.K.# 71-052	6 months	36 months	On Ice	10 °C or colder	ea.	34091

d9-Tetrahydrocannabinol (d9-THC) Standard

U.S. DEA-exempted formulation—no additional customer permits or licensing are required to purchase within the U.S.

d9-tetrahydrocannabinol (d9-THC) (1972-08-3)

Description	CAS #	Conc. in Solvent	CRM?	DEA Status	Canadian Test Kit Registration	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
d9-Tetrahydrocannabinol (d9-THC)	1972-08-3	1000 µg/mL in methanol, 1 mL/ampul	Yes	Exempt	T.K.# 71-049	6 months	24 months	On Ice	10 °C or colder	ea.	34067

d9-Tetrahydrocannabinolic Acid A (THCA-A) Standard

U.S. DEA-exempted formulation—no additional customer permits or licensing are required to purchase within the U.S.

d9-tetrahydrocannabinolic acid A (THCA-A) (23978-85-0)

Description	CAS #	Conc. in Solvent	CRM?	DEA Status	Canadian Test Kit Registration	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	Data pack available?	qty.	cat.#
d9-Tetrahydrocannabinolic acid A (THCA-A)	23978-85-0	1000 µg/mL in acetonitrile, 1 mL/ampul	Yes	Exempt	C.T.K.# 002-011	6 months	36 months	On Ice	–20 °C or colder	No	ea.	34111

Atrazine-d5 Standard

Isotopically labeled to provide the best approach for pesticide residue quantification.

Atrazine-d5 (163165-75-1)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Atrazine-d5	163165-75-1	100 µg/mL in acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	10 °C or colder	ea.	31984

Dimethoate-d6 Standard

Isotopically labeled to provide the best approach for pesticide residue quantification.

Ddimethoate-d6 (1219794-81-6)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Dimethoate-d6	1219794-81-6	100 µg/mL in acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	10 °C or colder	ea.	31988

Dichlorvos-d6 Standard

Isotopically labeled to provide the best approach for pesticide residue quantification.

Dichlorvos-d6 (203645-53-8)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Dichlorvos-d6	203645-53-8	100 µg/mL in acetone, 1 mL/ampul	Yes	3 months	12 months	Ambient	10 °C or colder	ea.	31987

Carbaryl-d7 Standard

Isotopically labeled to provide the best approach for pesticide residue quantification.

Carbaryl-d7 (362049-56-7)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Carbaryl-d7	362049-56-7	100 µg/mL in acetonitrile, 1 mL/ampul	Yes	6 months	31 months	Ambient	10 °C or colder	ea.	31985

Diazinon-d10 Standard

Isotopically labeled to provide the best approach for pesticide residue quantification.

Diazinon-d10 (diethyl-d10) (100155-47-3)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Diazinon-d10 (diethyl-d10)	100155-47-3	100 µg/mL in acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	10 °C or colder	ea.	31986

Diuron-d6 Standard

Isotopically labeled to provide the best approach for pesticide residue quantification.

Diuron-d6 (1007536-67-5)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Diuron-d6	1007536-67-5	100 µg/mL in acetonitrile, 1 mL/ampul	Yes	6 months	31 months	Ambient	10 °C or colder	ea.	31989

Linuron-d6 Standard

Isotopically labeled to provide the best approach for pesticide residue quantification.

Linuron-d6 (1219804-76-8)

Description	CAS #	Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Linuron-d6	1219804-76-8	100 µg/mL in acetonitrile, 1 mL/ampul	Yes	6 months	31 months	Ambient	10 °C or colder	ea.	31990

Q-sep QuEChERS Extraction Salts

- Free-flowing salts transfer easily and completely.
- Easy-open packets eliminate the need for a second empty tube for salt transfer.
- Convenient slim packets fit perfectly into tubes to prevent spills.
- Ready-to-use tubes, no glassware required.
- Pre-weighed, ultra-pure extraction salts.
- Ideal for original unbuffered, AOAC (2007.01), and European (EN 15662) QuEChERS methods.

Description	Material	Method	qty.	cat.#
Q-sep QuEChERS Extraction Salt Packets Only	4 g $MgSO_4$, 1 g NaCl, 1 g TSCD, 0.5 g DHS	European EN 15662	50 packets	25849

DHS – disodium hydrogen citrate sesquihydrate; $MgSO_4$ – magnesium sulfate; NaCl – sodium chloride; NaOAc – sodium acetate; TSCD – trisodium citrate dihydrate

Q-sep QuEChERS dSPE Tubes for Extract Cleanup

Fast, Simple Sample Prep for Multiresidue Pesticide Analysis

26217

- Packaged in foil subpacks of 10 for enhanced protection and storage stability.
- Ready-to-use tubes, no glassware required.
- Pre-weighed, ultra-pure sorbents.
- Support original unbuffered, AOAC (2007.01), European (EN 15662), and mini-multiresidue QuEChERS methods.

Description	Material	Method	Type	Volume	qty.	Similar to Part #	cat.#
Pigmented fruits and vegetables (e.g., strawberries, sweet potatoes, and tomatoes)							
Q-sep QuEChERS dSPE Tubes	150 mg $MgSO_4$, 25 mg PSA, 2.5 mg GCB	Mini-multiresidue, EN 15662	2 mL Micro-Centrifuge Tubes Prefilled with dSPE Materials for Cleanup (1 mL Extract)	2 mL	100 tubes		26217

Note: No entry in the Method column refers to dSPE formulations not specifically included in one of the cited references. These products can be used to accommodate the various needs of specific matrices not directly met by the cited references.

Empty Centrifuge Tubes, Polypropylene

Description	qty.	cat.#
Empty 50 mL Centrifuge Tube, Polypropylene w/Cap	50-pk.	25846
	500-pk.	28290

RESTEK
Pure Chromatography

Questions? Contact us or your local Restek representative (www.restek.com/contact-us).

www.restek.com

Lit. Cat.# FFAN3481B-UNV