

# Extraction of Pesticides from Cannabis Flower and Edibles



## Abstract

Cannabis and its products are on the road to being legalized throughout the United States. Because of the risk of pesticide exposure through inhaled and consumed cannabis products, the pesticides in the plant material and its products must be monitored. As matrices, cannabis and its related products have proven to be difficult to extract, and more traditional pesticide extraction methods, like QuEChERS, have been shown to perform poorly with these products. Thus, a better extraction method is needed. In this application note, in collaboration with SCIEX, the EDGE<sup>®</sup> was utilized to extract the Oregon pesticide list from cannabis flower and the edibles: cookies, chips, chocolate, hard candy, and granola bar. The average recoveries obtained were between 60% and 101%, and the average matrix effects were found to be between 70% and 130%. Thus, the EDGE is an excellent choice for laboratories extracting pesticides from cannabis and its products.

## Introduction

The use of cannabis and its products is growing more popular. With its continued legalization for recreational and medicinal use, it is subject to regulation, including monitoring its pesticide content. The presence of pesticides in cannabis is a concern because cannabis products are often inhaled or eaten and with their consumption, they can bioaccumulate, causing potentially deleterious effects on human health. Because of these concerns, the list of pesticides monitored gets longer each year, and there is a need for good methods to extract and quantitate these compounds. Traditionally, the QuEChERS method has been used to extract pesticides from plants and food matrices, but cannabis and its products have shown time and again that

cannabis is a difficult matrix to work with. Because of this, a simple, efficient method that produces high recoveries and repeatable results is still needed.

The EDGE is an extraction system that utilizes automation, heat, and solvent to extract samples quickly and effectively. It was used in a collaboration with SCIEX to extract pesticides from the Oregon List from cannabis edibles, including hard candy, chips, chocolate, cookies, and granola, and the cannabis flower. The average recoveries obtained for most pesticides in all the matrices were between 60% and 101%. The matrix effects were also assessed and found to be in the range of 70% to 130%. The EDGE offers cannabis laboratories an automated option for high recoveries with good matrix effects.

## Materials and Methods

### Extraction

Cannabis flower and cannabis cookies, chips, chocolate, hard candy, and granola bar were milled using a small coffee grinder. A portion of 300 mg of each matrix was weighed directly into a Q-Cup<sup>®</sup> containing a S1 Q-Disc<sup>®</sup> stack. The S1 Q-Disc stack is a sandwich of three filters including a glass-fiber filter surrounded by two cellulose filters. The samples were spiked with 150 ng of the Restek Oregon Pesticide Standards 1-6 (Catalogue numbers 32586, 32587, 32588, 32589, 32590, and 32591). A Q-Screen<sup>®</sup> was placed on top of each sample after spiking. The samples were loaded into an EDGE rack containing glass collection vials to collect the resulting extract.

The samples were extracted using the indicated EDGE method. Five replicates of each sample type were extracted.

## EDGE Method for Pesticides from Cannabis Flower and Edibles

Q-Disc: S1 Q-Disc stack (C9+G1+C9 sandwich)

### Cycle 1

Extraction Solvent: Acetonitrile with 1.0% Formic Acid (v/v)  
Top Add: 25 mL  
Bottom Add: 0 mL  
Rinse: 5 mL  
Temperature: 40 °C  
Hold Time: 02:00 (mm:ss)

### Wash 1

Wash Solvent: Acetonitrile with 1.0% Formic Acid (v/v)  
Wash Volume: 15 mL  
Temperature: 40 °C  
Hold Time: 00:30 (mm:ss)

### Wash 2

Wash Solvent: Acetonitrile with 1.0% Formic Acid (v/v)  
Wash Volume: 15 mL  
Temperature: - - -  
Hold Time: - :- -

## Post-Extraction

The extracts were placed in a -20 °C freezer for two hours as part of the winterization process. The lipids present in the sample created a flocculant, and the remaining extract was poured into another vial for analysis.

## Analysis

For recovery analysis, the extracts were directly injected. Electrospray ionization (ESI) was used for all analytes except for Chlorfenapyr for which atmospheric pressure chemical ionization (ACPI) was used. Analytes were separated using a Phenomenex Luna Omega Polar C18 column (150 x 4.6 mm, 3 µm). The injection volume was 1 µL. For ESI the mobile phases were A: water with 5 mM ammonium formate and 0.1% formic acid and B: methanol with 5 mM ammonium formate and 0.1% formic acid. For ACPI the mobile phases were: A: water and B: acetonitrile. The samples were analyzed using previously determined MRM transitions for each pesticide on a SCIEX Triple Quad™ 7500 LC-MS/MS.

## Results

The recoveries were determined using solvent-matched calibration curves. The recoveries with their respective standard deviations can be found in **Table 1**. The average recoveries for

all matrices were mostly within the range 60% to 101%, which is generally the ideal range for recoveries. A notable outlier was spinosad, which had lower recovery in chips and hard candy. The standard deviations of the recoveries for almost all compounds were below 15%, which is also ideal.

The exception to this was daminozide, a more difficult, polar pesticide. It had the greatest variability of the pesticides analyzed. Methyl parathion and imidacloprid also had slightly higher standard deviations. Among matrices, cannabis flower and chips showed the greatest variability in recovery.

The matrix effects were also assessed by comparing the extract of each blank matrix spiked with the pesticides at the initial spiking conditions to neat standard. The matrix effects with their standard deviations are shown in **Table 2**. The matrix effects were found to be in the range 70% to 130%. The flower matrix showed the most variability in its matrix effects, with signal enhancement and suppression extremes. Propiconazole, spiromesifen, and acequinocyl were found to be suppressed in the flower matrix. The cookie, chocolate, and chip matrices had the least amount of variability, with a tighter distribution overall.

## Conclusion

With the expanding cannabis industry within the United States, testing laboratories are monitoring cannabis and its products for pesticide content. Traditional pesticide extraction methods, like QuEChERS, have shown to work poorly with cannabis plant and cannabis edibles. Therefore, new and better extraction methods are needed. Within this work, in a partnership with SCIEX, the EDGE was used to extract pesticides from the Oregon List from cannabis flower and the edibles cookies, chips, chocolate, hard candy, and granola bar. The determined recoveries found for all pesticides, except spinosad, in all matrices were between 60% and 101%, which is well within the desired range, and all the recoveries, except for that of daminozide, methyl parathion and imidacloprid, had standard deviations below 15%. The matrix effects for each matrix were found to be 70% to 130%. Thus, these data indicate that the EDGE is an excellent choice for the extraction of pesticides from cannabis material and edibles.

We would like to thank our collaborators at SCIEX for extracting these samples on the EDGE and analyzing the results.

**Table 1.** Recoveries for Each Matrix

Compound	Flower		Cookie		Chips		Chocolate		Hard Candy		Granola Bar	
	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV
Acephate	86.14	3.99	88.71	5.01	77.30	2.99	82.63	4.80	85.17	3.53	87.79	9.52
Acequinocyl	71.08	2.10	76.58	2.02	63.14	1.73	73.29	1.17	75.88	0.95	81.63	1.28
Acetamidrid	85.78	5.05	87.70	2.35	71.78	2.80	82.18	2.42	87.22	3.38	89.15	2.79
Aldicarb	66.85	5.25	78.36	6.99	60.61	8.53	67.31	3.80	59.13	5.30	72.86	6.56
Avermectin	80.08	3.53	92.35	3.02	81.26	2.75	85.39	3.92	89.23	4.13	92.48	5.63
Azoxystrobin	82.43	4.02	87.73	5.62	73.83	3.27	80.73	8.05	85.71	5.56	88.03	2.01
Bifenazate	82.30	1.32	91.15	5.99	70.08	3.02	80.61	4.31	77.76	8.52	87.05	6.19
Bifenthrin	75.84	2.46	83.33	5.35	69.83	2.77	79.19	3.24	83.55	2.00	86.04	3.64
Boscalid	87.57	7.55	92.34	8.22	71.04	7.20	79.94	9.73	87.57	2.86	86.73	12.85
Carbaryl	78.28	10.21	90.40	4.10	74.33	2.61	82.74	2.43	84.29	2.17	86.72	7.78
Carbofuran	85.91	3.27	94.96	3.53	78.92	7.23	88.79	4.90	88.66	8.19	95.27	3.14
Chlorantraniliprole	82.09	8.78	87.62	11.45	77.07	5.10	84.71	7.88	85.26	5.61	84.06	7.15
Chlorefenapyr	81.33	6.96	84.41	2.69	81.09	5.71	80.66	3.58	87.98	6.21	86.77	3.29
Chlorpyrifos	88.80	2.42	87.05	3.27	75.58	3.03	84.56	1.92	87.72	2.38	89.11	3.06
Clofentazine	87.89	3.92	91.98	3.27	78.04	6.93	88.78	2.30	87.88	5.05	94.66	5.02
Cyantraniliprole	83.04	10.36	112.85	12.02	99.83	2.19	98.30	7.25	105.32	7.25	103.65	5.38
Cyfluthrin	75.96	5.70	91.80	3.75	72.50	6.76	82.49	5.64	81.51	4.54	92.57	4.67
Cypermethrin	99.17	2.30	100.65	3.52	85.04	5.24	96.40	0.99	96.90	5.24	100.88	3.71
Daminozide	107.57	30.26	89.56	33.21	70.45	20.83	84.91	37.90	78.21	29.19	97.39	27.43
Diazinon	83.76	1.61	87.07	1.80	74.72	3.49	84.31	2.94	90.69	3.13	87.51	4.66
Dichlorvos	77.92	2.17	89.80	6.38	73.32	4.10	83.91	5.91	81.03	6.79	87.05	1.85
Dimethoate	84.25	6.03	94.24	4.63	79.32	5.80	83.43	6.52	90.15	7.56	95.14	6.99
Ethoprophos	88.86	5.07	91.90	3.97	76.77	3.92	86.83	2.55	90.99	2.50	90.10	4.14
Etofenprox	62.44	2.51	84.09	3.29	71.27	3.03	80.14	2.82	81.13	1.66	85.10	2.44
Etoxazole	83.41	2.66	87.15	2.20	72.61	3.41	82.12	2.60	86.29	3.79	86.03	2.62
Fenoxycarb	80.92	8.33	88.21	4.71	73.62	9.25	84.52	5.58	90.28	3.98	96.00	1.78
Fenproximate	107.80	2.60	91.93	3.13	75.40	8.68	82.81	3.29	86.90	6.43	89.83	6.06
Fipronil	82.01	1.63	84.80	3.19	73.61	3.80	81.13	3.43	80.69	4.81	81.03	8.59
Fonicamid	80.19	11.18	88.44	5.12	71.29	2.89	78.74	7.40	81.35	8.10	83.94	5.22
Fludioxonil	78.02	5.17	86.26	6.86	71.55	3.13	79.83	3.40	81.57	6.67	85.57	4.60
Hexythiazox	76.49	1.82	87.67	3.74	70.70	4.97	83.31	2.17	82.29	9.84	81.01	7.46
Imazalil	79.49	5.21	80.66	5.34	61.73	9.45	75.45	5.13	69.40	4.16	78.39	5.66
Imidacloprid	79.48	17.83	87.63	6.66	70.92	5.99	76.32	5.97	83.31	8.68	81.10	15.60
Kresoxim-methyl	85.10	2.99	90.29	5.21	75.75	6.04	85.19	6.41	87.36	4.91	92.38	3.74
Malathion	78.54	4.11	85.76	4.93	69.86	1.92	74.74	7.08	78.12	4.21	84.31	4.75
Metalaxyl	84.59	3.73	86.13	2.41	72.16	8.57	79.64	6.13	83.48	6.23	82.19	5.02
Methiocarb	80.07	10.50	91.21	6.01	74.71	2.92	85.93	2.35	86.22	4.65	91.42	1.68
Methomyl	78.94	2.39	88.75	2.46	71.46	6.06	76.81	5.39	76.34	3.77	82.64	3.87
Methyl Parathion	70.23	13.21	70.76	15.85	63.51	7.53	73.48	15.84	75.04	2.32	78.74	10.86
MGK-264	84.93	3.34	88.69	7.05	72.77	5.07	89.01	4.01	84.24	9.77	86.87	5.82
Myclobutanil	86.12	4.86	91.74	6.42	79.27	11.23	82.48	6.48	84.20	8.75	84.89	12.15
Naled	83.38	3.78	87.50	5.30	77.68	4.27	80.96	11.17	83.64	6.49	85.34	6.28

**Table 1.** Recoveries for Each Matrix (continued)

Compound	Flower		Cookie		Chips		Chocolate		Hard Candy		Granola Bar	
	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV
Oxyaml	81.00	1.67	87.06	2.13	68.64	0.83	75.20	4.96	75.72	4.31	82.17	6.27
Paclobutrazole	85.36	4.32	90.64	2.96	80.97	7.26	77.28	9.09	83.67	14.06	87.85	4.82
Permethrin trans	95.22	3.63	83.00	5.88	70.42	4.37	83.11	4.35	82.65	3.23	84.20	2.92
Phosmet	75.52	2.78	87.68	3.42	72.09	5.59	73.88	4.58	82.01	3.64	79.84	6.89
Piperonyl Butoxide	92.99	2.99	87.46	2.09	72.36	1.85	80.36	3.05	84.78	3.63	84.04	1.50
Prallethrin	74.13	3.07	113.83	8.00	73.16	5.70	79.79	8.38	86.38	4.66	82.38	4.42
Propiconazole	65.12	10.82	87.74	7.82	72.79	4.95	84.56	5.91	85.28	7.16	90.68	3.53
Propoxur	78.68	4.60	90.53	4.10	73.69	2.19	79.64	3.08	79.34	3.54	83.71	6.89
Pyrethrins	80.38	3.77	88.31	4.33	72.55	7.84	80.90	12.89	82.86	3.41	87.46	4.10
Pyridaben	76.69	1.91	82.11	2.56	68.82	2.22	77.95	1.85	79.98	1.55	82.33	1.93
Spinosad	82.44	5.74	76.56	3.84	57.45	3.22	71.13	3.65	53.98	2.65	71.70	2.95
Spiromesifen	69.73	3.45	82.23	3.37	66.51	3.63	75.82	2.53	73.74	3.75	80.18	1.32
Spirotetramat	84.46	6.98	92.92	6.79	82.29	5.47	91.55	3.80	90.31	9.75	92.30	3.66
Spiroxamine	85.90	3.64	85.61	4.30	71.95	4.82	79.04	2.70	79.89	2.53	86.77	3.07
Tebuconazole	69.34	8.14	81.98	8.18	71.30	5.17	73.49	7.20	83.67	7.28	85.57	4.94
Thiacloprid	83.92	6.06	93.29	3.67	79.20	3.44	87.78	6.41	91.28	3.25	90.92	4.89
Thiamethoxam	81.61	6.49	86.28	5.11	71.22	6.09	82.50	7.70	80.81	7.48	83.49	8.01
Trifloxystrobin	80.05	5.42	84.97	5.72	76.15	5.03	80.50	5.81	86.63	1.76	87.48	2.99

**Table 2.** Matrix Effects for Each Matrix

Compound	Flower		Cookie		Chips		Chocolate		Hard Candy		Granola Bar	
	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV
Acephate	95.64	7.46	99.10	6.34	95.90	4.91	98.54	4.58	93.61	1.08	90.15	8.68
Acequinocyl	73.26	12.61	98.60	4.91	94.20	4.18	99.95	16.44	97.63	1.25	88.25	13.22
Acetamiprid	92.90	2.05	95.29	8.65	95.47	8.56	98.91	2.90	95.35	5.92	91.43	11.14
Aldicarb	77.81	8.68	81.47	10.97	82.47	9.49	85.85	9.90	84.76	5.80	85.62	6.31
Avermectin	86.47	19.67	101.53	12.85	103.73	20.02	96.16	19.31	102.52	9.12	95.81	17.32
Azoxystrobin	86.93	11.15	96.32	5.64	96.65	5.86	97.14	8.71	98.64	2.10	89.89	8.59
Bifenazate	94.36	14.59	99.74	5.69	101.34	6.66	100.99	9.04	88.08	8.44	101.23	8.24
Bifenthrin	80.42	8.93	101.00	5.00	96.99	3.05	99.32	11.51	98.53	3.54	90.14	14.22
Boscalid	96.79	7.76	106.63	5.00	100.98	11.11	109.83	6.87	99.34	13.70	103.96	12.92
Carbaryl	92.67	9.19	95.45	9.63	90.21	9.01	89.80	4.98	88.27	2.09	87.38	13.92
Carbofuran	90.58	5.36	89.32	7.20	100.89	6.84	98.02	6.59	94.61	6.59	87.52	10.30
Chlorantraniliprole	91.11	10.49	107.01	7.75	106.29	10.49	104.79	10.25	104.83	12.37	94.30	9.45
Chlorefenapyr	92.11	5.37	100.72	7.34	95.09	2.09	93.80	5.86	93.93	2.93	86.91	11.40
Chlorpyrifos	94.02	8.49	97.69	4.11	99.00	5.73	102.13	8.86	99.89	3.39	100.22	12.50
Clofentezine	90.70	5.84	104.23	8.45	102.04	7.97	100.65	5.13	100.11	4.53	105.73	12.34
Cyantraniliprole	95.37	14.80	98.47	20.84	96.83	14.91	94.18	14.82	101.66	13.80	99.17	18.02
Cyfluthrin	74.46	3.01	98.97	5.64	98.68	11.15	99.75	12.80	97.18	3.21	93.76	13.99
Cypermethrin	87.80	15.82	100.03	3.56	100.22	6.58	99.14	16.24	93.85	3.44	91.84	11.26
Daminozide	95.74	8.20	97.85	8.03	96.60	8.31	97.57	5.77	91.00	11.99	107.98	12.33
Diazinon	89.43	9.28	95.67	6.10	93.66	2.74	100.10	9.77	96.48	4.62	97.43	13.15

**Table 2.** Matrix Effects for Each Matrix (Continued)

Compound	Flower		Cookie		Chips		Chocolate		Hard Candy		Granola Bar	
	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV	Average	STDEV
Dichlorvos	90.86	5.52	98.99	6.64	93.82	6.88	97.29	7.00	99.32	1.33	96.68	9.36
Dimethoate	105.64	4.38	106.02	7.30	104.62	8.14	109.56	4.32	108.89	3.67	102.77	12.38
Ethoprophos	89.61	9.69	98.14	7.33	97.86	2.01	101.08	4.26	91.42	7.63	92.94	13.54
Etofenprox	68.11	5.58	97.00	6.18	95.68	5.22	97.35	17.71	96.59	3.90	90.54	11.48
Etoazole	86.99	12.80	99.47	5.47	95.84	5.76	99.24	14.05	99.63	0.59	97.04	13.90
Fenoxycarb	86.24	10.04	96.94	10.10	99.31	15.10	101.39	9.91	90.84	12.96	99.57	21.81
Fenproximate	76.36	14.15	89.54	9.36	98.32	10.72	91.65	17.34	96.04	4.94	93.05	15.07
Fipronil	113.12	10.64	102.11	8.21	101.85	6.45	106.31	12.15	96.28	3.71	95.00	11.58
Flonicamid	93.09	7.63	99.59	4.39	98.94	7.36	105.47	5.51	100.52	5.21	98.27	14.54
Fludioxonil	85.41	4.60	95.28	4.97	94.05	0.86	92.82	7.07	94.03	3.51	92.10	10.82
Hexythiazox	83.84	8.27	102.70	8.52	105.13	6.11	106.23	11.90	102.91	5.49	99.85	13.60
Imazalil	100.46	7.26	106.86	4.04	101.07	7.57	101.93	7.37	92.78	6.06	92.33	15.06
Imidacloprid	87.01	4.87	93.77	8.61	98.24	8.08	101.82	13.03	94.55	7.52	94.00	21.94
Kresoxim-methyl	82.71	8.03	98.45	5.12	96.65	3.76	97.89	6.82	95.98	2.01	91.91	14.59
Malathion	86.52	11.44	98.56	7.95	93.18	8.01	93.51	9.05	98.53	2.28	92.35	12.90
Metaxyl	87.80	11.21	100.69	3.77	99.23	8.96	94.99	4.23	86.22	9.50	94.14	9.92
Methiocarb	83.80	9.04	92.51	9.94	92.54	3.28	95.97	10.33	90.90	5.91	89.86	10.60
Methomyl	86.59	3.14	89.48	4.80	88.23	4.20	88.46	4.03	83.86	7.27	78.63	14.40
Methyl Parathion	87.05	3.38	99.12	6.33	96.73	5.29	93.73	3.26	89.67	0.91	86.84	12.91
MGK-264	85.22	6.74	102.16	9.93	95.08	7.15	94.90	5.54	91.65	7.51	86.70	12.17
Myclobutanil	85.56	7.14	97.86	8.51	102.42	15.80	97.27	9.24	98.20	10.51	92.33	11.96
Naled	85.02	7.08	93.00	10.83	97.85	6.86	98.82	9.04	92.90	3.66	92.17	11.66
Oxyaml	92.98	5.29	93.92	8.36	96.29	6.94	93.34	4.73	90.19	3.17	91.22	13.58
Paclobutrazole	80.26	9.76	91.76	10.43	96.50	8.21	94.51	10.50	95.27	8.87	95.05	18.50
Permethrin	106.56	18.41	102.58	8.25	95.66	4.08	97.58	18.02	96.16	3.71	91.17	11.10
Phosmet	86.20	12.46	95.81	10.70	88.18	2.78	96.33	11.17	101.71	6.54	85.85	10.20
Piperonyl Butoxide	88.86	6.91	102.17	4.76	100.36	5.26	101.12	7.28	101.16	2.23	97.37	13.02
Prallethrin	91.02	5.95	120.04	3.22	99.79	6.74	100.86	7.33	104.36	6.77	94.74	15.34
Propiconazole	71.96	13.53	102.27	8.68	99.36	6.38	105.55	15.26	101.49	5.39	109.52	12.19
Propoxur	84.66	7.17	90.33	7.44	88.59	2.17	4.08	10.28	86.53	4.57	82.79	13.81
Pyrethrins	86.62	7.56	103.26	7.56	97.94	6.79	97.98	10.71	98.91	2.38	95.20	16.96
Pyridaben	81.10	14.11	102.05	6.49	101.78	6.18	102.25	16.09	103.29	2.35	97.89	12.97
Spinosad	93.43	8.48	104.72	7.17	96.13	5.31	103.51	7.96	95.22	1.67	92.66	13.15
Spiromesifen	73.46	10.64	92.35	8.53	92.66	7.10	95.09	17.26	96.67	3.53	90.46	12.78
Spirotetramat	90.86	8.20	100.20	9.93	100.66	3.88	113.34	8.95	100.83	5.55	97.97	11.87
Spiroxamine	96.64	6.21	103.24	7.45	98.93	4.43	102.16	3.06	98.24	4.26	92.41	15.49
Tebuconazole	84.23	8.27	100.63	4.09	99.36	4.84	102.40	4.15	99.16	5.85	96.09	12.51
Thiacloprid	98.23	3.51	104.65	9.67	101.91	6.39	104.00	3.33	95.47	9.54	95.81	17.09
Thiamethoxam	101.54	5.15	102.92	9.61	99.74	6.44	106.19	4.83	98.17	6.58	93.29	9.84
Trifloxystrobin	88.36	7.75	97.50	4.93	97.84	5.03	99.82	8.23	97.44	6.56	94.51	12.47



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