

Determination of Pesticide Residues in Marijuana and Tea by QuEChERS and LC-MS/MS



UCT Part Numbers

ECPAHFR50CT
50 mL centrifuge tubes

ECQUUS2-MP
Pouch containing 4 g MgSO₄
and 2 g NaCl

CUMPSGG2CT
2 mL dSPE tube with 150 mg
MgSO₄, 50 mg PSA and
50 mg ChloroFiltr®

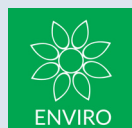
Summary:

Various pesticides can be rapidly determined in dried tea leaves or marijuana using this simple method. Samples are hydrated in water and then extracted by QuEChERS and dSPE cleanup using ChloroFiltr®. Results are determined by LC/MS/MS.

Internal Standard:

Prepare a 10 ppm TPP solution by mixing 20 µL of the 5000 ppm TPP solution with 10 mL of MeCN.

Transfer all standards to amber glass vials and store at -20°C until needed.



Procedure:

1. QuEChERS Extraction

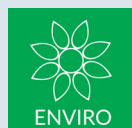
- Weigh 2 g of the homogenized tea or marijuana into a 50 mL centrifuge tube
- Add 10 mL of reagent water to each tube and hydrate the samples for 1 hour using a horizontal shaker
- Add 100 µL of the 10 ppm TPP solution to all samples
- Add 10 mL of acetonitrile (MeCN). Vortex for 1 min
- Add contents of **ECQUUS2-MP** pouch then shake vigorously for 1 min
- Centrifuge at 5000 rpm for 5 min

2. dSPE Cleanup

- Transfer 1 mL of the supernatant to 2 mL dSPE tube **CUMPSGG2CT**
- Shake for 30 seconds
- Centrifuge at 10,000 rpm for 5 min
- Transfer 0.3 mL of the cleaned extract into a 2-mL auto-sampler vial
- Add 0.3 mL of reagent water
- Vortex, then filter using a 0.45 µm syringe filter
- The samples are ready for LC/MS/MS analysis

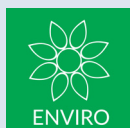
HPLC Parameters		
Instrumentation	Thermo Scientific Dionex Ultimate 3000 LC system coupled to a TSQ Vantage® triple quadrupole mass spectrometer	
HPLC	Thermo Scientific Dionex UltiMate 3000® LC System	
Column	Thermo Scientific, Accucore aQ®, 100 x 2.1 mm, 2.6 µm	
Guard Column	Thermo Scientific, Accucore aQ®, 10 x 2.1 mm, 2.6 µm	
Column Temperature	40 °C	
Column Flow Rate	0.200 mL/min	
Auto-sampler Temperature	10 °C	
Injection Volume	10 µL	
Gradient Program		
Time (min)	Mobile Phase A (%) 0.3 % formic acid and 0.1 % ammonia formate in water	Mobile Phase B (%) 0.1 % formic acid in MeOH
0	99	1
1.5	99	1
3.5	20	80
10	10	90
12	0	100
15	0	100
15.2	99	1
20	99	1

Divert mobile phase to waste from 0 - 0.5 and 15 - 20 min to prevent ion source contamination



MS Parameters	
Polarity	ESI ⁺
Spray voltage V	4000 V
Vaporizer Temperature	300 °C
Ion transfer capillary	200 °C
Sheath gas pressure	50 arbitrary units
Auxiliary gas pressure	25 arbitrary units
Q1 and Q3 peak width	0.2 and 0.7 Da
Collision gas and pressure	Ar at 1.5 mTorr
Scan type	SRM
Cycle time	1 sec
Acquisition method	EZ Method

SRM Transitions							
Name	Rt (min)	Precursor ion	Product ion 1	CE 1	Product ion 2	CE 2	S-lens (V)
Methamidophos	1.24	142.007	124.57	14	111.6	5	60
Carbendazim	6.37	192.093	132.08	29	160.08	17	81
Dicrotophos	6.46	238.009	126.58	17	108.60	33	73
Acetachlor	6.48	269.417	111.86	15	71.69	33	72
Thiabendazole	6.61	202.059	131.06	31	175.07	24	103
DIMP	7.30	181.283	96.60	13	78.62	32	44
Tebuthiuron	7.32	228.946	115.59	26	171.63	17	72
Simazine	7.34	201.400	67.68	33	103.60	24	85
Carbaryl	7.41	201.956	126.63	30	144.63	7	40
Atrazine	7.68	215.957	67.65	35	173.60	16	79
DEET	7.72	191.947	118.63	15	90.66	28	92
Pyrimethanil	8.10	200.116	107.06	23	183.14	22	66
Malathion	8.12	331.011	98.57	23	126.86	12	60
Bifenazate	8.22	300.925	169.82	15	197.62	5	51
Tebuconazole	8.73	308.008	69.66	29	124.56	35	97
Cyprodinil	8.81	226.122	77.03	40	93.05	33	88
TPP (IS)	8.81	327.093	77.02	37	152.07	33	98
Diazinone	8.87	305.135	153.09	15	169.08	14	89
Zoxamide	8.90	335.807	186.50	20	158.51	38	102
Pyrazophos	8.99	374.103	194.06	20	222.13	20	104
Profenofos	9.59	372.300	302.37	19	143.48	35	104
Chlorpyrifos	10.23	349.989	96.89	32	197.94	17	69
Abamectin	11.20	890.486	304.40	18	306.68	15	102
Bifenthrin	12.77	440.039	165.21	39	180.42	11	66



Accuracy and Precision Data Obtained from the Fortified Tea Samples

Analyte	Spiked at 2 ng/mL		Spiked at 10 ng/mL		Spiked at 50 ng/mL	
	Rec %	RSD (n=6)	Rec %	RSD (n=6)	Rec %	RSD (n=6)
Methamidophos	112.5	7.3	100.7	1.5	85.9	10.1
Carbendazim	nd	nd	87.8	14.6	79.5	13.2
Dicrotophos	114.2	3.3	102.3	3.4	93.9	11.0
Acetachlor	108.3	10.4	111.3	4.6	105.5	6.5
Thiabendazole	86.7	6.0	84.0	2.9	73.5	10.8
DIMP	111.7	6.7	109.3	2.7	102.4	7.8
Tebuthiuron	113.3	3.6	108.3	2.4	100.4	8.1
Simazine	110.8	6.0	118.8	2.1	105.6	10.2
Carbaryl	115.8	5.0	122.0	2.1	111.2	10.4
Atrazine	124.2	7.8	117.5	2.6	105.2	9.8
DEET	149.2	12.8	125.5	3.1	106.3	10.1
Pyrimethanil	99.2	5.9	98.0	5.0	90.3	2.6
Malathion	143.3	9.5	125.0	6.7	110.2	4.4
Bifenazate	114.2	12.2	106.8	6.7	98.2	3.8
Tebuconazole	72.5	7.2	80.2	5.4	79.1	4.7
Cyprodinil	90.8	5.4	77.5	2.7	74.1	3.2
Diazinone	108.3	7.5	99.5	1.9	97.7	4.3
Zoxamide	95.0	7.4	92.2	1.3	90.1	3.4
Pyrazophos	90.0	5.0	91.7	2.4	88.3	5.1
Profenofos	96.7	7.8	80.8	4.5	75.0	5.3
Chlorpyrifos	80.0	7.9	82.5	2.7	81.6	5.4
Abamectin	99.2	8.1	89.8	4.0	82.2	9.4
Bifenthrin	89.2	7.5	119.8	7.6	126.5	19.0

nd: < LOQ, not determined

Crude Tea and Marijuana Extracts Before, and After dSPE Cleanup Using 150 mg MgSO₄, 50 mg PSA, and 50 mg ChloroFiltr®



Figure 1a. Tea extracts before (left) and after dSPE cleanup (right)

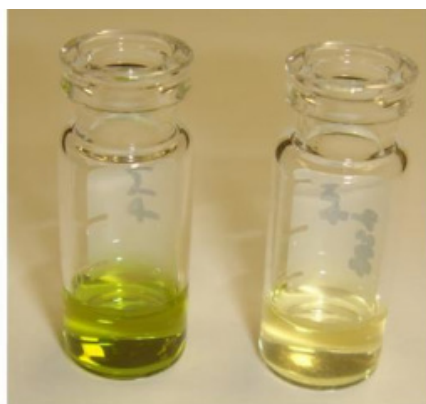


Figure 1b. Marijuana (Sample #4) extracts before (left) and after dSPE cleanup (right)

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