

PROTOCOL	cannabis
	dry commodity -plant material

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	SAMPLES					
	SPIKED	Recovery check - 200ppb (dry)	NO SPIKE	No spike	Process Blank	Blk
Homogenize/hydration/ solvent extraction	pre-mix material		pre-mix material		NA	
	weigh 1.5 ± 0.01 g homogenized sample		weigh 1.5 ± 0.01 g homogenized sample		NA	
	SPEX Geno/Grinder = 5 minutes, 1500 RPM, 2 balls		SPEX Geno/Grinder = 5 minutes, 1500 RPM, 2 balls		NA	
	add 15 mL high purity water		add 15 mL high purity water		add 15 mL high purity water	
	75 µL [10 ppm] 6 compound deuterated pest mix (gives 50ppb injection conc)		75 µL [10 ppm] 6 compound deuterated pest mix		75 µL [10 ppm] 6 compound deuterated pest mix	
	60 µL [5 ppm] Pesticide standard (gives 20ppb inj conc)		NO SPIKE		NO SPIKE	
	add 15 mL 1% acetic acid in acetonitrile, v/v		add 15 mL 1% acetic acid in acetonitrile, v/v		add 15 mL 1% acetic acid in acetonitrile, v/v	
	SPEX Geno/Grinder= 1 minute, 1500 RPM		SPEX Geno/Grinder = 1 minute, 1500 RPM		SPEX Geno/Grinder = 1 minute, 1500 RPM	
	shake for 30 minutes		shake for 30 minutes		shake for 30 minutes	
PULL OFF 1ML FOR POLAR PESTICIDES TESTING (chlormequat)						
Salting Out Partitioning Step	SPIKED	Recovery check	NO SPIKE	No spike	Process Blank	Blk
	Add contents of AOAC salt packet cat# 26237 or cat# 26238 (salts only, no tubes)		Add contents of AOAC salt packet cat# 26237 or cat# 26238 (salts only, no tubes)		Add contents of AOAC salt packet cat# 26237 or cat# 26238 (salts only, no tubes)	
	hand shake 1 minute		hand shake 1 minute		hand shake 1 minute	
	Centrifuge the tubes at >1500 rcf for 5 minutes		Centrifuge the tubes at >1500 rcf for 5 minutes		Centrifuge the tubes at >1500 rcf for 5 minutes	
Dispersive solid phase extraction (dSPE) cleanup	SPIKED	Recovery check	NO SPIKE	No spike	Process Blank	Blk
	remove and retain top acetonitrile layer		remove and retain top acetonitrile layer		remove and retain top acetonitrile layer	
	Use Universal dSPE tube, cat# 26245 for 6 mL		Use Universal dSPE tube, cat# 26245 for 6 mL (process two dSPE tubes for enough volume to make matrix-matched curve)	2x	Use Universal dSPE tube, cat# 26243 to process 1 mL and cat# 26245 for 6 mL	
	Hand shake vigorously for 2 minutes		Hand shake vigorously for 2 minutes		Hand shake vigorously for 2 minutes	
	Centrifuge the dispersive-SPE tubes at >1500 rcf for 5 minutes		Centrifuge the dispersive-SPE tubes at >1500 rcf for 5 minutes		Centrifuge the dispersive-SPE tubes at >1500 rcf for 5 minutes	
	pull off supernatant		pull off supernatant		pull off supernatant	
Prepare to Analyze	SPIKED	Recovery check	NO SPIKE	No spike	Process Blank	Blk
	syringe filter if not using filter vials (cat# 25893) (recommended based on particulates and maintaining instrument performance)		syringe filter if not using filter vials (cat# 25893) (recommended based on particulates and maintaining instrument performance)		syringe filter if not using filter vials (cat# 25893) (recommended based on particulates and maintaining instrument performance)	
	place 0.5 or 1.0 mL in a vial so formic acid and Instrument IS can be added (see below)		combine supernatant from two dSPE tubes so formic acid and Instrument IS can be added (see below) = expect 8-10 mLs		place 0.5 or 1.0 mL in a vial so formic acid and Instrument IS can be added (see below)	
	formic acid adjustment : Add 10µL 5% formic acid in acetonitrile PER 1 mL of sample		formic acid adjustment : Add 10µL 5% formic acid in acetonitrile PER 1 mL of sample (scale for total volume needed for matrix-matched calibration curve)		formic acid adjustment : Add 10µL 5% formic acid in acetonitrile PER 1 mL of sample	
	Instrument IS: Add 5µL [10 ppm (µg/mL)] carbaryl-d7, 0.1% formic acid in acetonitrile PER 1 mL of sample		Instrument IS : Add 5µL [10 ppm (µg/mL)] carbaryl-d7, 0.1% formic acid in acetonitrile PER 1 mL of sample (scale for total volume needed for matrix-matched calibration curve)		Instrument IS: Add 5µL [10 ppm (µg/mL)] carbaryl-d7, 0.1% formic acid in acetonitrile PER 1 mL of sample	
	vortex few seconds to mix well		vortex few seconds to mix well		vortex few seconds to mix well	
	RECOVERY CHECK SAMPLE: place small volume in autosampler vial (filter vial uses 500 µL)		INCURRED SAMPLE: place small volume in autosampler vial (filter vial uses 500 µL)		PROCESS BLANK: place small volume in autosampler vial (filter vial uses 500 µL)	
	NA		use remaining volume to make matrix-matched calibration curve		NA	

Stock Solutions

stock solutions -IS	cat#	concentration	volume
atrazine-d5	31984	100µg/mL each in acetonitrile	1mL
carbaryl-d7	31985	100µg/mL each in acetonitrile	1mL
diazinon-d10	31986	100µg/mL each in acetonitrile	1mL
dichlorvos-d6	31987	100µg/mL each in acetonitrile	1mL
dimethoate-d6	31988	100µg/mL each in acetonitrile	1mL
diuron-d6	31989	100µg/mL each in acetonitrile	1mL
linuron-d6	31990	100µg/mL each in acetonitrile	1mL

stock solutions -Oregon Cannabis Pesticides "OR6"	cat#	concentration	volume
OR Cannabis Pesticide Std #1	570858	600µg/mL each in acetonitrile	1mL
OR Cannabis Pesticide Std #2	570859	600µg/mL each in acetonitrile	1mL
OR Cannabis Pesticide Std #3	570860	600µg/mL each in acetonitrile	1mL
OR Cannabis Pesticide Std #4	570861	600µg/mL each in acetonitrile	1mL
OR Cannabis Pesticide Std #5	570862	600µg/mL each in acetonitrile	1mL
OR Cannabis Pesticide Std #6	570863	600µg/mL each in acetonitrile	1mL

Oregon Cannabis Pesticide Standards (call to inquire 800.356.1688)					
Item # 570858		Item # 570860		Item # 570862	
OR Cannabis Pesticide Std #1		OR Cannabis Pesticide Std #3		OR Cannabis Pesticide Std #5	
600µg/mL in Acetonitrile		600µg/mL in Acetonitrile		600µg/mL in Acetonitrile	
24 month shelf life		18 month shelf life		18 month shelf life	
Abamectin	71751-41-2	Aldicarb	116-06-3	Bifenthrin	82657-04-3
Spinosad	168316-95-8	Fipronil	120068-37-3	Cyfluthrin	68359-37-5
		Flonicamid	158062-67-0	Cypermethrin	52315-07-8
		Hexythiazox	78587-05-0	Etofenprox	80844-07-1
Item # 570859		Methiocarb	2032-65-7	Permethrin (cis & trans)	52645-53-1
OR Cannabis Pesticide Std #2		Methomyl	16752-77-5	Prallethrin	23031-36-9
600µg/mL in Acetonitrile		Oxamyl	23135-22-0	Pyrethrins	8003-34-7
18 month shelf life		Pyridaben	96489-71-3		
(E)-Fenpyroximate	134098-61-6	Thiacloprid	111988-49-9		
Acequinocyl	57960-19-7	Thiamethoxam	153719-23-4	Item #570863	
Acetamiprid	135410-20-7			OR Cannabis Pesticide Std #6	
Azoxystrobin	131860-33-8	Item # 570861		600µg/mL in Acetonitrile	
Bifenazate	149877-41-8	OR Cannabis Pesticide Std #4		12 month shelf life	
Boscalid	188425-85-6	600µg/mL in Acetonitrile			
Chlorfenapyr	122453-73-0	18 month shelf life		Acephate	30560-19-1
Etoxazole	153233-91-1			Chlorpyrifos	2921-88-2
Fludioxonil	131341-86-1	Carbaryl	63-25-2	Diazinon	333-41-5
Imidacloprid	138261-41-3	Carbofuran	1563-66-2	Dichlorvos (DDVP)	62-73-7
Kresoxim methyl	143390-89-0	Chlorantraniliprole	500008-45-7	Dimethoate	60-51-5
Metalaxyl	57837-19-1	Clofentezine	74115-24-5	Ethoprophos	13194-48-4
MGK-264	113-48-4	Daminozide	1596-84-5	Malathion	121-75-5
Piperonyl butoxide	51-03-6	Fenoxycarb	79127-80-3	Methyl parathion	298-00-0
Spiromesifen	283594-90-1	Imazalil	35554-44-0	Naled	300-76-5
Spirotetramat	203313-25-1	Myclobutanil	88671-89-0	Phosmet	732-11-6
Spiroxamine	118134-30-8	Paclobutrazol	76738-62-0		
Trifloxystrobin	141517-21-7	Propiconazole	60207-90-1		
		Propoxur	114-26-1		
		Tebuconazole	107534-96-3		

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