

PROTOCOL	cannabis
	dry commodity -plant material

Julie Kowalski, Restek Corporation
julie.kowalski@restek.com

	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 shake for 30 minutes		SPEX Geno/Grinder = 1 minute, 1500 RPM shake for 30 minutes		SPEX Geno/Grinder = 1 minute, 1500 RPM 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	

information about certified reference standards on next page 

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)

<p>Item # 570858 OR Cannabis Pesticide Std #1 600µg/mL in Acetonitrile 24 month shelf life</p> <p>Abamectin 71751-41-2 Spinosad 168316-95-8</p>	<p>Item # 570860 OR Cannabis Pesticide Std #3 600µg/mL in Acetonitrile 18 month shelf life</p> <p>Aldicarb 116-06-3 Fipronil 120068-37-3 Flonicamid 158062-67-0 Hexythiazox 78587-05-0 Methiocarb 2032-65-7 Methomyl 16752-77-5 Oxamyl 23135-22-0 Pyridaben 96489-71-3 Thiacloprid 111988-49-9 Thiamethoxam 153719-23-4</p>	<p>Item # 570862 OR Cannabis Pesticide Std #5 600µg/mL in Acetonitrile 18 month shelf life</p> <p>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</p>
<p>Item # 570859 OR Cannabis Pesticide Std #2 600µg/mL in Acetonitrile 18 month shelf life</p> <p>(E)-Fenpyroximate 134098-61-6 Acequinocyl 57960-19-7 Acetamiprid 135410-20-7 Azoxystrobin 131860-33-8 Bifenazate 149877-41-8 Boscalid 188425-85-6 Chlorfenapyr 122453-73-0 Etoxazole 153233-91-1 Fludioxonil 131341-86-1 Imidacloprid 138261-41-3 Kresoxim methyl 143390-89-0 Metalaxyl 57837-19-1 MGK-264 113-48-4 Piperonyl butoxide 51-03-6 Spiromesifen 283594-90-1 Spirotetramat 203313-25-1 Spiroxamine 118134-30-8 Trifloxystrobin 141517-21-7</p>	<p>Item # 570861 OR Cannabis Pesticide Std #4 600µg/mL in Acetonitrile 18 month shelf life</p> <p>Carbaryl 63-25-2 Carbofuran 1563-66-2 Chlorantraniliprole 500008-45-7 Clofentezine 74115-24-5 Daminozide 1596-84-5 Fenoxycarb 79127-80-3 Imazalil 35554-44-0 Myclobutanil 88671-89-0 Pacllobutrazol 76738-62-0 Propiconazole 60207-90-1 Propoxur 114-26-1 Tebuconazole 107534-96-3</p>	<p>Item #570863 OR Cannabis Pesticide Std #6 600µg/mL in Acetonitrile 12 month shelf life</p> <p>Acephate 30560-19-1 Chlorpyrifos 2921-88-2 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 Naled 300-76-5 Phosmet 732-11-6</p>