

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 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
	<a href="#">Add contents of AOAC salt packet cat# 26237 or cat# 26238 (salts only, no tubes)</a>		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	
	<a href="#">Use Universal dSPE tube, cat# 26245 for 6 mL</a>		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	
	<a href="#">Centrifuge the dispersive-SPE tubes at &gt;1500 rcf for 5 minutes</a>		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
	<a href="#">syringe filter if not using filter vials (cat# 25893) (recommended based on particulates and maintaining instrument performance)</a>		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

<u>stock solutions -IS</u>	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><b>Item # 570858</b>  <b>OR Cannabis Pesticide Std #1</b>                      600µg/mL in Acetonitrile                      24 month shelf life</p> <p>Abamectin            71751-41-2                      Spinosad             168316-95-8</p>	<p><b>Item # 570860</b>  <b>OR Cannabis Pesticide Std #3</b>                      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><b>Item # 570862</b>  <b>OR Cannabis Pesticide Std #5</b>                      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 &amp; trans) 52645-53-1                      Prallethrin          23031-36-9                      Pyrethrins          8003-34-7</p>
<p><b>Item # 570859</b>  <b>OR Cannabis Pesticide Std #2</b>                      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><b>Item # 570861</b>  <b>OR Cannabis Pesticide Std #4</b>                      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                      Paclobutrazol      76738-62-0                      Propiconazole      60207-90-1                      Propoxur            114-26-1                      Tebuconazole      107534-96-3</p>	<p><b>Item #570863</b>  <b>OR Cannabis Pesticide Std #6</b>                      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>