

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	
	geno grinder = 5 minutes, 1500 RPM, 2 balls		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	
	geno grinder = 1 minute, 1500 RPM shake for 30 minutes		geno grinder = 1 minute, 1500 RPM shake for 30 minutes		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		Add contents of AOAC salt packet cat# 26237		Add contents of AOAC salt packet cat# 26237	
	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 

