Determination of Pesticides in Red Wine by QuEChERS Cleanup, and LC-MS/MS Detection



UCT Part Numbers

RFV0050CT 50 mL polypropylene centrifuge tube

ECQUUS2-MP Mylar Pouch contains: 4000 mg MgSO₄, 2000 mg NaCl

ECPURMPSMC Quick QuEChERS mini-cartridge contains: 110 mg MgSO₄, 180 mg PSA

Procedure:

1. Extraction

- a) Add 10 mL red wine sample to 50-mL polypropylene centrifuge tubes (RFV0050CT).
- b) Add 10 mL acetonitrile, vortex 30 sec.
- c) Add salts in Mylar pouch (ECQUUS2-MP).
- d) Shake vigorously for 1 min.
- e) Centrifuge at 5000 rpm for 5 min at 20 °C.
- f) Supernatant is ready for cleanup.

2. Quick QuEChERS Cleanup

- a) Load 1 mL of supernatant using disposable syringe.
- b) Pass the supernatant slowly through Quick QuEChERS mini-cartridge (ECPURMPSMC).
- c) Collect 0.5 mL cleaned extract into a 2 mL auto-sampler vial.
- d) Add triphenyl phosphate as internal standard.
- e) Samples are ready for LC-MS/MS analysis.







Cleanup red wine extract with Quick QuEChERS

LC-MS/MS Detection

LC: Thermo Accela 1250 pump with PAL auto-sampler

LC Conditions						
Column	Guard column: Restek C18, 2.1 x 20 mm, 3 μm Column: Sepax HP-C18, 2.1 x 100 mm, 3 μm,120 Å					
Column Temperature	Ambient					
Injection Volume	10 μL at 15° C					
Mobile Phase	A: 0.1% formic acid in Milli-Q-water B: 0.1% formic acid in methanol					
Flow Rate	200 μL/min					
LC Gradient Program						
Time		A%	B%			
0		95	5			
1		95	5			
3		50	50			
8		5	95			
14.2		95	5			
16		95	5			





MS Conditions				
MS/MS	Thermo TSQ Vantage tandem MS			
Ion Source	Heated ESI			
Ion Polarity	ESI⁺			
Spray Voltage	3000 V			
Sheath Gas Pressure	N ₂ @ 40 psi			
Auxiliary Gas Pressure	N ₂ @ 10 psi			
Ion Transfer Capillary Temperature	350 ℃			
Scan Type	SRM (0-16 min)			
CID Conditions	Ar @ 1.5 mTorr			

SRM Transitions									
Analyte	Parent	Product ion 1	CE 1	Product ion 2	CE 2	S-Lens	Dwell time		
Methamidophos	142.044	94.090	14	125.050	16	59	0.15		
Carbendazim	192.093	132.080	29	160.080	17	81	0.10		
Thiabendazole	202.059	131.060	31	175.070	31	103	0.10		
Pyrimethanil	200.116	107.060	23	183.140	22	66	0.10		
Cyprodinil	226.122	77.030	40	93.050	33	88	0.10		
TPP (IS)	327.093	77.020	37	152.070	33	98	0.10		
Diazinone	305.135	153.090	15	169.08	14	89	0.10		
Pyrazophos	374.103	194.060	20	222.130	20	104	0.10		
Chlorpyrifos	349.989	96.890	32	197.940	17	69	0.10		

Accuracy and Precision Data							
	Fortified at 10 ng/mL		Fortified at 50 ng/mL		Fortified at 100 ng/mL		
Compound	Recovery %	RSD % (n=4)	Recovery %	RSD % (n=4)	Recovery %	RSD % (n=4)	
Methamidophos	93.7	3.4	81.6	5.8	84.2	3.5	
Carbendazim	105.7	10.8	100.1	10.6	90.5	7.6	
Thiabendazole	91.2	4.9	87.9	6.8	85.0	4.0	
Pyrimethanil	91.2	2.7	107.0	3.2	102.8	4.9	
Cyprodinil	104.3	3.2	99.9	6.1	100.2	4.9	
Diazinone	104.9	5.6	102.0	6.6	99.2	6.8	
Pyrazophos	99.9	4.0	96.6	5.6	91.3	4.1	
Chlorpyrifos	91.7	4.6	99.5	5.2	97.2	3.8	





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