Determination of Carbendazim in Orange Juice Using QuEChERS with LC/MS/MS Detection



UCT Part Numbers

ECQUEU750CT-MP Pouch contains: 4000 mg MgSO₄, 1000 mg NaCl, 500 mg Na citrate dibasic sesquihydrate and 1000 mg Na citrate tribasic dihydrate

CUMPSC18CT 2 mL centrifuge tube contains: 150 mg MgSO₄, 50 mg PSA, 50 mg endcapped C18

Introduction:

The planar fungicide carbendazim (CASRN 10605-21-7) can be used to control mold on citrus crops but is not approved for use in the US or on imported products. Concentrations in citrus products can be rapidly and accurately determined using a QuEChERS extraction with dSPE cleanup. LOD and LOQ for this method are 0.4 and 1.4 ng/mL, respectively.

Procedure:

1. Extraction

- a) Add 10 mL of orange juice to a 50 mL centrifuge tube.
- b) Add 10 mL acetonitrile then vortex.
- c) Add the contents of ECQUEU750CT-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. Dispersive Cleanup

- a) Add 1 mL of supernatant to CUMPSC18CT tube.
- b) Shake sample(s) for 1 min.
- c) Centrifuge at 10,000 rpm for 5 min.
- d) Transfer 0.5 mL to 2 mL autosampler vial.
- e) Add 25 µL 1 ppm TPP, vortex.
- f) Samples are ready for analysis.

3. LC/MS//MS Analysis





LC Conditions								
Instrumentation		Thermo Accela HPLC with autosampler						
Column		Guard column: Restek C18, 2.1 x 20 mm 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 water B: 0.1 formic acid in methanol						
Flow Rate		200 μL/min						
LC Gradient Program								
Time (min)		% Mobile Phase A	% Mobile Phase B					
0		50	50					
3		0	100					
8		0 100						
9		50	50					
14		50	50					
		MS Condit	tions					
Instrumentation		Thermo TSQ Vantage MS						
lon source		Heated ESI						
lon polarity		ESI ⁺						
Spray voltage		3000 V						
Sheath gas pressure		N ₂ @ 40 psi						
Auxiliary gas pressure		N ₂ @ 10 psi						
lon transfer capillary temperature		350 °C						
Scan type		SRM (0-10 min)						
CID conditions		Ar @ 1.5 mTorr						





SRM Transitions								
Compound	Parent	Product ion 1	CE	Product ion 2	CE	S-Lens	Dwell time (s)	
Carbendazim	192.093	132.080	29	160.080	17	81	0.20	
TPP (IS)	327.093	77.020	37	152.070	33	98	0.10	

Matrix Matched Calibration Curve

Dynamic linearity range is from 2 to 500 ng/mL with R²=0.9992

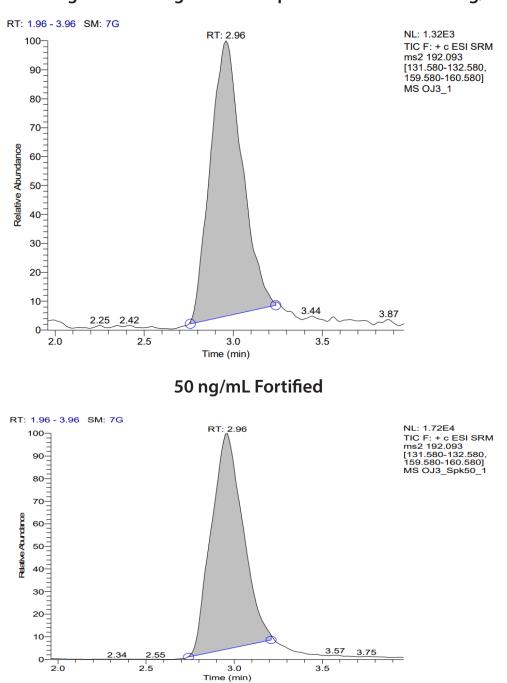
Carbendazim Y = -0.00137422+0.00434038*X R^2 = 0.9992 W: 1/X 2.2 2.0-1.8-1.6 1.4 Area Ratio 1.2 1.0 0.8-0.6-0.4 0.2-0.0-200 500 550 50 100 150 250 300 350 400 450 n ng/mL

Accuracy and Precision of Carbendazim Data from Spiked Orange Juice Sample

Fortified (ng/mL)	Recovery %	RSD % n=4		
10	96.6	4.5		
50	100.2	3.4		
250	103.7	2.1		







Chromatogram of Orange Juice Sample and Fortified at 50 ng/mL

DCN-212020-230

UCT, LLC • 2731 Bartram Road • Bristol, PA 19007 800.385.3153 • 215.781.9255 <u>www.unitedchem.com</u> Email: <u>methods@unitedchem.com</u> ©UCT, LLC 2023 • All rights reserved





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