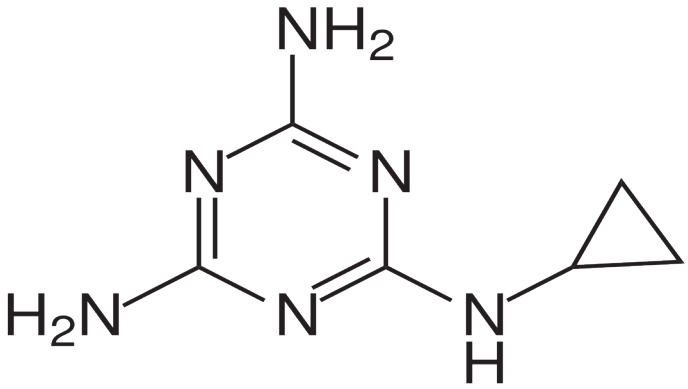
Analysis of Cyromazine in Poultry Feed Using a QuEChERS Approach



UCT Part Numbers

ECMSSA50CT-MP 6 g anhydrous $MgSO_4$ and 1.5 g Na Acetate	**EEC18156** 500 mg endcapped C18, 6 mL cartridge

Introduction:

This summary outlines a QuEChERS procedure for the analysis of the insecticide cyromazine (Trigard or Larvadex) in poultry feed by LC-MS/MS. Processing time is significantly faster than EPA method AG-555 and uses less solvent. Modifications include adding glacial acetic acid to the acetonitrile to increase extraction efficiency.

Procedure:

1. Sample Preparation

a) Homogenize 2 grams of poultry feed and add to a 50 mL centrifuge tube
b) Add 10 mLs of acetonitrile/acetic acid (75:25)
c) Sonicate at 50/60 Hz for 15 minutes
d) Add the contents of **ECMSSA50CT-MP** pouch and shake for 1 minute
e) Centrifuge at 3400 rpm for 10 minutes
f) Transfer 1 mL of supernatant to a calibrated test tube and add 9 mL of water: acetonitrile (95:5) with 0.1% acetic acid

2. Sample Cleanup

a) Add the 10 mLs from 1) f) above to a **EEC18156** cartridge and elute dropwise
b) Filter eluant using a 0.45 µm Teflon filter (Millipore, Billerica, MA) or equivalent
c) Transfer 2 mL of eluant to an HPLC vial for analysis by LC-MS/MS

3. Analysis LC-MS/MS

a) Waters Alliance 2695 HPLC (Waters) coupled with a micromass Quattro Micro triplequadrupole mass spectrometer (Micromass, Manchester, U.K.) or equivalent

HPLC Conditions	
Guard column	(Alltima, C18, 5 µm, 2.1 x 7.5 mm, Deerfield, IL) or equivalent
Analytical column	(Alltima, C18, 5 µm, 2.1 x 250 mm, Waters) or equivalent
Flow rate	0.2 mL/minute
Injection volume	25 µL

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Gradient Program		
Time (min)	**(A) acetonitrile with 0.1% formic acid**	**(B) water with 0.1% formic acid**
0-2	5%	0%
2-5	10%	5%
5-5.5	90%	10%
5.5-8	5%	90%
8-10	5%	90%
10-12	5%	0%

Mass Spectrometer:

a) Positive ion mode electrospray ionization

b) Monitor the ion transition of the parent ion (m/z 167) to the product ion (m/z 85) in multiple reaction monitoring (MRM)

Mass Spectrometry Conditions for Cyromazine Quantitation	
capillary voltage	3.1 kV
cone voltage	65 V
collision energy	21-24 V
source temperature	120° C
desolvation temperature	350° C
cone gas flow	135 L/h
desolvation gas flow rate	750 L/h
collision gas	Argon
parent ion	(m/z) 167
product ion	(m/z) 85

References:

[1] *Summarized with permission from Xia, Kang, Atkins, Jack et al, "Analysis of Cyromazine in Poultry Feed Using the QuEChERS Method | Coupled with LC-MS/MS" J. Agric. Food Chem, DOI:10.1021/jf9034282

[2] Listing of instrument manufacturers does not constitute endorsement by UCT

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