Analysis of Cyromazine in Poultry Feed Using a QuEChERS Approach

UCT Products**:**

ECMSSA50CT-MP (6 g anhydrous $MgSO_4$ and 1.5 g Na Acetate)

EEC18156 (500 mg endcapped C18, 6 mL cartridge)

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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

- Waters Alliance 2695 HPLC (Waters) coupled with a micromass Quattro Micro triple-quadrupole 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
  Mobile phase: (A) acetonitrile with 0.1% formic acid and (B) water with 0.1% formic acid
  Gradient:
  - 0-2 min, 5%A
  - 2-5 min from 5 to 10% A
  - 5-5.5 min from 10 to 90% A
  - 5.5-8 min 90 to 5% A
  - 8-10 min, from 90 to 5% A
  - 10-12 min, 5% A

  Flow rate 0.2 mL/minute
  Injection volume: 25 µL

- ***Mass Spectrometer***
  - Positive ion mode electrospray ionization
  - 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

*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

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