

Analysis of Amprolium in Eggs Using Bond Elut Plexa SPE and LC/MS/MS

Authors

Dan Li

China Institute of Veterinary
Drug Control, Beijing, China

Congcong Zhang, Cuiling Wu,
Juan An, and Xia Yang
Agilent Technologies, Inc.

Abstract

This study developed and validated a method for the quantitative analysis of amprolium in eggs. We used solid phase extraction (SPE), Agilent Bond Elut Plexa cleanup, then Agilent 6470 LC/MS/MS analysis. The method provided a reliable solution with good recoveries and reproducibility that fulfilled the MRL requirement of Chinese, US, and Japanese regulations.

Experimental

Instrument method

The samples were run on an Agilent 1260 Infinity UHPLC system coupled to an Agilent G6470 triple quadrupole LC/MS system equipped with an Agilent Jet Stream Electrospray ion source. Agilent MassHunter workstation software was used for data acquisition and analysis.

Sample extraction

The procedure is shown in Figure 1.

HPLC conditions

Parameter	Value															
Column	Agilent InfinityLab Poroshell 120 HILIC-Z, 100 × 3.00 mm, 2.7 μm (p/n 685975-324)															
Flow Rate	0.4 mL/min															
Mobile Phase	A) Water with 0.1% formic acid and 5 mM ammonium acetate B) Acetonitrile															
Gradient	<table border="1"> <thead> <tr> <th>Time (min)</th> <th>%B</th> <th>Flow Rate (mL/min)</th> </tr> </thead> <tbody> <tr> <td>0</td> <td>85</td> <td>0.4</td> </tr> <tr> <td>2.0</td> <td>85</td> <td>0.4</td> </tr> <tr> <td>4.0</td> <td>60</td> <td>0.4</td> </tr> <tr> <td>6.0</td> <td>60</td> <td>0.4</td> </tr> </tbody> </table>	Time (min)	%B	Flow Rate (mL/min)	0	85	0.4	2.0	85	0.4	4.0	60	0.4	6.0	60	0.4
Time (min)	%B	Flow Rate (mL/min)														
0	85	0.4														
2.0	85	0.4														
4.0	60	0.4														
6.0	60	0.4														
Post Time	4 min															

MS conditions

Parameter	Value
Gas Temperature	250 °C
Gas Flow	7 L/min
Nebulizer	35 psi
Sheath Gas Heater	325 °C
Sheath Gas Flow	11 L/min
Capillary	3500 V (POS)
Nozzle	300 V
Data Acquisition	MRM as in Table 1.

Table 1. Target analytes MRM conditions.

Analyte	Precursor Ion (m/z)	Product Ion (m/z)	Fragmentor (V)	CE (V)
Amprolium	243.1	150.1	135	10
		94.1	135	5

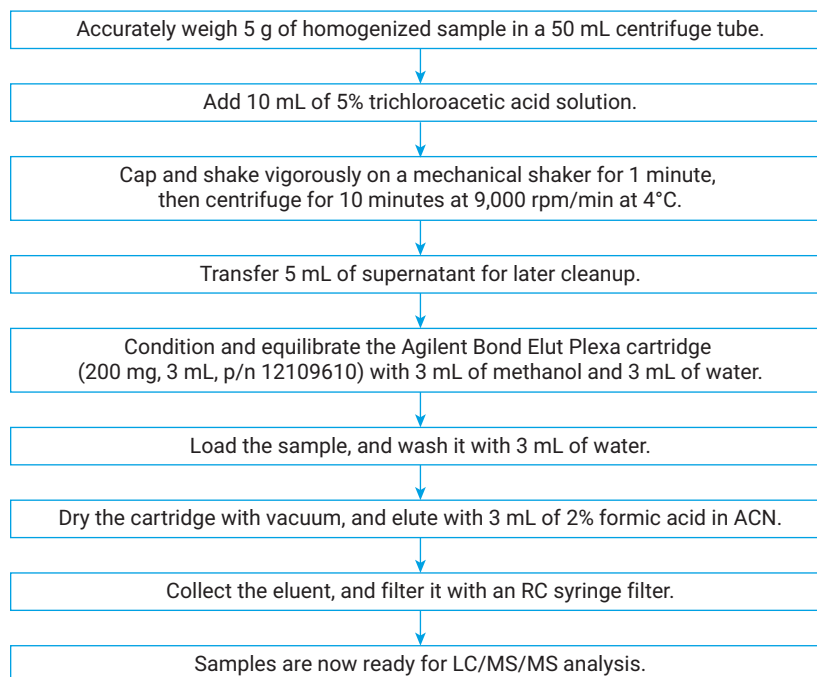


Figure 1. Sample preparation workflow chart.

Results and discussion

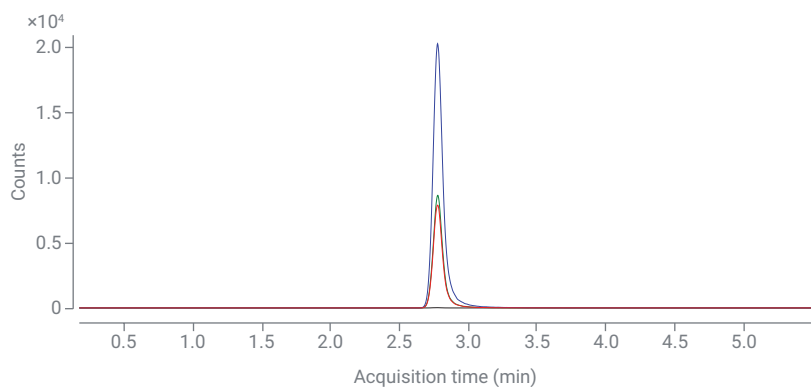


Figure 2. MRM chromatogram of amprolium in neat solution at 250 ng/mL (blue), postspiked in SPE cleaned egg matrix at 250 ng/mL (green), prespiked in egg at 100 ng/g (red), blank egg sample (black).

Table 2. Method recovery and RSDs.

Analyte	Spiking Level (ng/g)	Recoveries (%)			Average Recovery (%)	RSD% (n = 3)
Amprolium	10	71.6	66.4	74.4	70.8	5.7
	20	83.3	79.9	82.7	82.0	2.2
	100	91.3	89.5	95.7	92.2	3.5

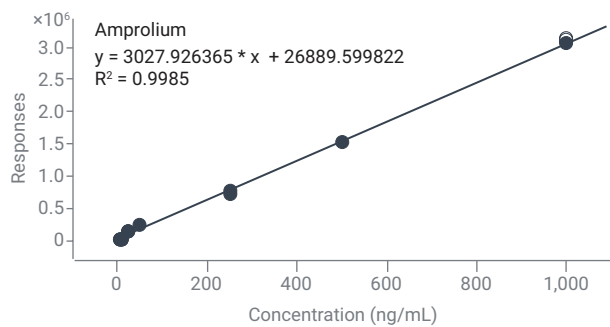


Figure 3. Calibration curves for egg samples.

Conclusion

A simple method using Agilent Bond Elut Plexa is established for the fast and reliable analysis of amprolium in eggs using LC/MS/MS. The matrix effect is 40%, limit of quantitation is 10 ng/g, and the limit of detection is 1 ng/g. The method provides excellent analyte recovery, reproducibility, and a simplified workflow.

www.agilent.com/chem

DE.2886574074

This information is subject to change without notice.

© Agilent Technologies, Inc. 2020
Printed in the USA, June 24, 2020
5994-1945EN

