

DIN EN 16162:2012-06 (E)

Animal feeding stuffs - Determination of decoquinatone by HPLC with fluorescence detection

Contents		Page
Foreword		4
Introduction		5
1 Scope		6
2 Normative references		6
3 Principle		6
4 Reagents		6
5 Apparatus		8
6 Sampling		8
7 Sample preparation		8
8 Procedure		8
8.1 General		8
8.2 Extraction of feeds (decoquinatone content between 10 mg/kg to 500 mg/kg)		8
8.3 Extraction of complementary feeds, premixtures and feed additives (decoquinatone content higher than 500 mg/kg)		9
8.4 Extraction of trace feeds (decoquinatone content lower than 10 mg/kg)		9
8.5 Quality control spiked feeds		9
8.5.1 Blank Feed to spike at 30 mg/kg		9
8.5.2 Blank feed to spike at 9 mg/kg		9
8.6 HPLC parameters		9
8.7 Standards' injections and calibration curve		10
8.8 Sample extracts		10
8.9 Confirmation procedure		10
9 Calculations		10
10 Precision		11
10.1 Limit of Detection and Limit of Quantification		11
10.2 Interlaboratory test		11
10.3 Repeatability		11
10.4 Reproducibility		11
11 Test report		12
Annex A (informative) Results inter-laboratory study		13
Annex B (informative) Additional recovery results		17
B.1 Recoveries from the familiarization phase		17
B.2 Pre-trial; recovery test on a milk-replacer		18
B.3 Recoveries from the single-laboratory validation		18
Annex C (informative) Additional results for robustness purposes		19

C.1	Robustness in terms of excitation wavelength, during the final collaborative study	19
C.2	Comparison between ground and unground samples during the final collaborative study	19
C.3	Robustness from one laboratory during the final collaborative study	20
Annex D (informative) HPLC parameters and chromatogram examples		22
D.1	General	22
D.2	Chromatogram example: Calibration point at 0,6 µg/ml	22
D.3	Chromatogram example: Commercial Lamb feed containing around 50 mg/kg of decoquinat ²²	23
D.4	Chromatogram example: same commercial Lamb feed, as in D.3, containing around 50 mg/kg of decoquinat ²²	23
Bibliography		24