DIN EN 16162:2012-06 (E)

Animal feeding stuffs - Determination of decoquinate 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 (decoquinate content between 10 mg/kg to 500 mg/kg) 8
8.3 Extraction of complementary feeds, premixtures and feed additives (decoquinate content higher than 500 mg/kg) 9
8.4 Extraction of trace feeds (decoquinate 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 decoquinate22 D.4 Chromatogram example: same commercial Lamb feed, as in D.3, containing around 50 mg/kg of decoquinate 23

Bibliography 24