DIN EN 17683:2023-04 (E)

Animal feeding stuffs - Methods of sampling and analysis - Determination of pyrrolizidine alkaloids in animal feeding stuff by LC-MS/MS

Contents

Page

European foreword 4

Introduction 5

1 Scope 6

2 Normative references 7

3 Terms and definitions 7

4 Principle 7
4.1 General 7
4.2 Reagents 7
4.3 Analytical standards 7
4.4 Chemicals 8
4.5 Solutions 9
4.5.1 Extraction solution 0,05 mol/l aqueous sulphuric acid 9
4.5.2 Aqueous ammoniacal solution (20/80) (v/v) for neutralization of extracts before SPE 9
4.5.3 Examples of HPLC mobile phase 9
4.5.4 PA stock solutions 9
4.5.5 Standard working solution (PA mixture), 1 µg/ml 9

5 Apparatus 10

6 Procedure 11
6.1 General 11
6.2 Sample preparation 11
6.3 Extraction 12
6.4 SPE procedure 12
6.5 Reconstitution of the sample 13
6.6 Quality control samples 13
6.7 Calibration by means of matrix matched standards (MMS) 13

7 HPLC-MS/MS analysis 14
7.1 Liquid chromatographic separation 14
7.2 Mass spectrometric operating conditions 15
7.3 Analysis sequence 15

8 Results 15
8.1 Peak identification 15
8.2 Calibration function 16
8.3 Quantification 16
8.4 Reporting of results 16
8.5 Quality control - Performance criteria 17

9 Precision 17
9.1 General 17
9.2 Repeatability 17
9.3 Reproducibility 18

10 Test report 18

Annex A (informative) Precision data 19

Annex B (informative) Example of LC-MS/MS conditions 52

Annex C (informative) Example LC-MS/MS chromatogram of a pyrrolizidine alkaloid mixture 55

Annex D (informative) Method protocol for the determination of pyrrolizidine alkaloids in animal feeding stuff by LC-MS/MS after reduction of N-oxides using metallic zinc 56

Annex E (informative) List of potentially co-eluting pyrrolizidine alkaloid (PA) isomers 66

Bibliography 67