

DIN EN 16215:2020-05 (E)

Animal feeding stuffs: Methods of sampling and analysis - Determination of dioxins and dioxin-like PCBs by GC/HRMS and of indicator PCBs by GC/HRMS

Contents		Page
European foreword		4
Introduction		5
1	Scope	6
2	Normative references	8
3	Terms and definitions	8
4	Principle	9
5	Reagents	10
5.1	General	10
5.2	Dioxins, furans, non-ortho PCBs, mono-ortho PCBs and non-dioxin-like PCBs and their labelled analogues	10
6	Apparatus	10
7	Sampling	10
8	Preparation of test sample	11
9	Procedure	11
9.1	General	11
9.2	Animal feed stuff sample and oil/fat sample	11
10	Extraction	11
10.1	General	11
10.2	Module BI Extraction using automated Pressurized Fluid Extraction (PFE) system	12
10.3	Module BII: Manual extraction procedure	13
11	Clean-up	14
11.1	General	14
11.2	Module CI: Automated clean-up	16
11.3	Module CII: Manual sample clean-up, removal of fat and group separation	19
11.4	Additional clean-up-steps for fraction A and fraction B of module CII	22
11.5	Module CIII: Removal of matrix, manual sample clean-up and group separation	24
12	Module D: Gas chromatograph-high resolution mass spectrometer (GC-HRMS)	29
12.1	General	29
12.2	Reagents and materials	29
12.3	Procedure	29
13	Calculation and expression of results	31
13.1	General	31
13.2	Calibration by Isotope Dilution	31
13.3	Linearity	31
13.4	Calibration criteria	31
13.5	Identification and confirmation	31
13.6	Calculation	32

13.7	Toxic Equivalent (TEQ) values	33
13.8	Recovery	36
14	Precision	36
15	Test report	38
Annex A(informative)İSÜFÜFÓFSÜFSÜ		39
A.1	Large volume injection (100 µl) - GC-HRMS Calibration mixtures dioxins and non- ortho PCBs	39
A.2	Sample preparation procedure	40
A.3	PTV injection conditions using 100 µl injection	40
Annex B(informative)İSÜFÜFFFÜSÜFÜFSFF solutions		41
B.1	CAS Registry numbers for dioxins, furans, non-ortho PCBs, mono-ortho PCBs and non-dioxin-like PCBs and their labelled analogues	41
B.2	Standard solutions	42
B.3	GC-standard solutions	45
Annex C(informative)Example of automated procedure		48
Annex D(informative)FSÜÛSSFÆÑ		50
Annex E(informative)ÒSFÜFÜFÚS-up after fractionation using a small multi- layer silica column .		54
E.1	Preparation of the silica mixtures	54
E.2	Preparation of the small multi-layer column	54
Bibliography		55