

DIN EN 16204:2012-08 (E)

Foodstuffs - Determination of lipophilic algal toxins (okadaic acid group toxins, yesso toxins, azaspiracids, pectenotoxins) in shellfish and shellfish products by LC-MS/MS

Contents		Page
Foreword		3
Introduction		4
1 Scope		5
2 Normative references		5
3 Principle		5
4 Reagents		5
5 Apparatus		7
6 Procedure		8
6.1 Preparation of samples		8
6.1.1 General		8
6.1.2 Raw samples		8
6.1.3 Cooked samples		8
6.2 Homogenization and extraction		8
6.3 Hydrolysis		8
7 HPLC-MS/MS analysis		9
7.1 General		9
7.2 HPLC operating conditions (chromatography under acidic conditions)		9
7.3 HPLC operating conditions (chromatography under basic conditions)		10
7.4 Mass spectrometric operating conditions		10
7.5 Calibration curve		10
7.6 Determination of algal toxins in sample test solutions		11
7.7 Quality control measures for sequences		11
8 Calculation		12
8.1 Peak identification		12
8.2 Quantitative determination by means of external calibration and matrix correction		12
8.3 Description of matrix correction		13
8.4 Calculation of the total toxicity		14
9 Precision		14
10 Test report		14
Annex A (informative) Precision data		15
A.1 Details on the inter-laboratory study		15
A.2 Recovery		28
Annex B (informative) Examples for suitable MS detection conditions		29
B.1 Examples suitable for SCIEX API 4000 or API 4000 Q-Trap		29

B.2	Examples suitable for Waters (Micromass) TSQ Ultima	31
B.3	Examples suitable for Thermo Fisher TSQ Quantum Ultra	33
B.4	Examples suitable for Agilent 6410 or 6460 QQQ	35
	Annex C (informative) Typical chromatogram	37
	Bibliography	38