

# 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

---

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