DIN EN 14526:2026-06 (E)

Foodstuffs - Determination of saxitoxin-group toxins in shellfish - HPLC method using pre-column derivatization with peroxide or periodate oxidation

Contents

Page

Introduction 5

1 Scope 6

2 Normative references 7

3 Terms and definitions 7

4 Principle 7

5 Reagents 10

6 Apparatus 14

7 Procedure 15
7.1 Sample preparation 15
7.2 Extraction procedure 16
7.3 Sample purification 16
7.3.1 SPE-C18 clean-up 16
7.3.2 SPE-COOH clean-up (fractionation) 17
7.3.3 Alternative weak cation exchange SPE clean-up [2], [11] 17
7.4 Conversion of GTX6 into NEO and/or C3,4 into GTX1,4 18
7.4.1 General 18
7.4.2 Hydrolysis of SPE-COOH Fraction 1 or 2 18
7.5 Oxidation procedure 19
7.5.1 General 19
7.5.2 Periodate oxidation 19
7.5.3 Peroxide oxidation 20

8 HPLC determination 20

9 Calibration curve 23

10 Identification 23

11 Calculation 23
11.1 General 23
11.2 Calculation method with standard calibration curve 24
11.3 Calculation of GTX1,4 in the presence of GTX2,3 and dcGTX2,3 24
11.3.1 General 24
11.3.2 Method 1 26
11.4 Calculation of NEO in the presence of dcSTX 26
11.4.1 General 26
11.4.2 Method 2 28
11.5 Calculation of NEO in the presence of dcSTX, dcNEO and STX 28
11.5.1 General 28
11.5.2 Method 3 29
11.6 Calculation of dcNEO in the presence of dcSTX 30
11.6.1 General 30
11.6.2 Method 4 31
11.7 Calculation of C3,4 32

11.8 Calculation of GTX6 32
11.9 Conversion to STX 2HCl equivalents 32

12 Quality controls 33
12.1 General 33
12.2 Acceptance criteria series of analysis 33
12.3 Overall recovery 34

13 Verification 34

14 Test report 35

Annex A (informative) Precision data 36

Annex B (informative) Chromatograms 64

Annex C (normative) Alternative calculation methods 68

Bibliography 76