INTERNATIONAL STANDARD

ISO 18635

First edition
2016-04-01

Water quality — Determination of short-chain polychlorinated alkanes (SCCPs) in sediment, sewage sludge and suspended (particulate) matter — Method using gas chromatography-mass spectrometry (GC-MS) and electron capture negative ionization (ECNI)

Qualité de l'eau — Détermination des alcanes polychlorés à chaîne courte dans les sédiments et matières en supsension (particules) — Méthode par chromatographie en phase gazeuse-spectrométrie de masse (CPG-SM) et ionisation chimique négative (ICN)

Reference number
ISO 18635:2016(E)

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Foreword

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The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

The user should be aware that particular problems might require the specifications of additional marginal conditions.

Water quality — Determination of short-chain polychlorinated alkanes (SCCPs) in sediment, sewage sludge and suspended (particulate) matter — Method using gas chromatography-mass spectrometry (GC-MS) and electron capture negative ionization (ECNI)

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This International Standard specifies a method for the quantitative determination of the sum of short-chain polychlorinated *n*-alkanes also known as short-chain polychlorinated paraffins (SCCPs) in the carbon bond range, *n*-C_{10} to *n*-C_{13}, inclusive in mixtures with chlorine mass fractions ("contents") between 50 % and 67 %, including approximately 6 000 of approximately 8 000 congeners.

This method is applicable to the determination of the sum of SCCPs in sediment and suspended (particulate) matter, sewage sludge, and soil using gas chromatography-mass spectrometry with electron capture negative ionization (GC-ECNI-MS).

Depending on matrix and the detection capabilities of the GC-ECNI-MS, the method can be applied to samples containing, e.g. 0,03 µg/g to 3 µg/g sum of SCCPs.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5667-12, *Water quality — Sampling — Part 12: Guidance on sampling of bottom sediments*

ISO 5667-13, *Water quality — Sampling — Part 13: Guidance on sampling of sludges*

ISO 5667-17, *Water quality — Sampling — Part 17: Guidance on sampling of bulk suspended solids*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

ISO 12010, *Water quality — Determination of short-chain polychlorinated alkanes (SCCPs) in water — Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI)*

ISO/TS 13530, *Water quality — Guidance on analytical quality control for chemical and physicochemical water analysis*

3 Principle

Determination of the sum of SCCPs in the carbon bond range, *n*-C_{10} to *n*-C_{13}, inclusive in technical and environmental transposed mixtures with chlorine mass fractions ("contents") between 50 % and 67 %

(e.g. approximately 3 to 10 chlorine atoms per molecule) and independent of the C-number distribution pattern of the congeners. No recognition of the chlorine content is necessary.

The samples are fortified with an internal standard and extracted using pressurized liquid extraction with an organic solvent. The sample extraction procedure is followed by a clean-up procedure by column chromatography and gel permeation chromatography to eliminate interfering compounds. Gas chromatography (GC) is undertaken using a short capillary column within a short retention time range. The detection of selected mass fragments is carried out using mass spectrometry (MS) in selected ion-monitoring mode using electron capture negative ionization mode (ECNI). The mass fragments and the compositions of the calibration solutions used in this International Standard are essential for the analysis of the sum of SCCPs.

The selected ion chromatogram is integrated over the full retention time range of the SCCPs. The quantification of the sum of SCCPs is carried out after establishing a calibration by a multiple linear regression measuring solutions of different SCCP mixtures fortified with an internal standard.

The analysed sum of SCCPs includes the variety of SCCPs with their differing chlorine content and C-number distribution patterns as found in technical mixtures, as well as compositions in the environment. The calibration requires at least three differently composed standard mixtures.

These standard mixtures mimic different mixtures found in the environment. Only the multiple linear regression quantification with these specific mixtures enables the quantification of the variety of observed mixtures of SCCP in the environment and in technical compositions described in the scope and in Reference [6]. It is not possible to use only one standard mixture for that complex task.

4 Interferences

Non-specific matrix interferences, as well as interferences from other environmental situations, are dealt with using the given clean-up procedure. A further reduction of matrix effects can be achieved by reducing the mass spectrometric resolution to, e.g., 0,4 amu, which is often possible with a quadrupole mass spectrometer. The exact masses are 374,9588; 410,9169; and 422,9355 (see Reference [6]).

Applying the entire procedure, a selection of chlorinated pollutants has been tested and found not to cause interferences below the concentrations given in Table 1.

Table 1 — Highest concentration level which causes no interferences higher than the limit of quantification of 0,03 µg/g

Potential interfering compounds	Highest concentration level which causes no interferences higher than the limit of quantification of 0,03 µg/g
Aroclor 1262[a]	0,25 µg/g
Aroclor 1242[a]	2 µg/g
Aroclor 1221[a]	2 µg/g
Campheclor (toxaphene)	0,35 µg/g
Halowax 1014[a]	2 µg/g
Halowax 1051[a]	0,08 µg/g
MCCP (medium-chain chlorinated *n*-alkanes) 42 %	2 µg/g
MCCP (medium-chain chlorinated *n*-alkanes) 52 %	1,2 µg/g
MCCP (medium-chain chlorinated *n*-alkanes) 57 %	2 µg/g

[a] Aroclor 1262, Aroclor 1242, Aroclor 1221, Halowax 1014, and Halowax 1051 are products commercially available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

5 Reagents

Use solvents and reagents of sufficient purity, i.e. with negligibly low concentrations of SCCPs, e.g. lower than the limit of detection of the method.

Check blanks regularly over the entire procedure to establish proper analytical quality control.

5.1 Solvents for extraction, column chromatography, and preparation of stock solutions.

The solvent for extraction is *n*-heptane. Other non-polar solvents, e.g. *n*-hexane (C_6H_{14}), cyclohexane (C_6H_{12}) can be used if the extraction efficiency is comparable with those of *n*-heptane.

For conditioning of the clean-up columns, use mixtures of *n*-heptane and propanone (acetone) as described below.

5.2 Reference SCCP stock solutions.

Use commercially available solutions, e.g. in cyclohexane or *n*-hexane, of the single mixtures of SCCP congeners with defined carbon chain length and with different defined chlorine contents (see Table 2, first two columns). Alternatively, use commercially available ready mixed solutions with the same composition.

Mixtures of synthetic solutions are used to simulate environmentally occurring SCCPs or technical products of SCCPs. For example, the synthetic mixed calibration stock solution "Lake Ontario water" is mixed to resemble a Lake Ontario water as reported in Reference [5]. Its characteristic is a relatively high content of C_{10} to C_{12}, especially C_{12} and a low chlorine content as partly reported in water samples too. The synthetic mixed calibration stock solution "Perch" simulates a C-number distribution found in a perch (see Reference [6]). The standard mixture "Sediment Drevnice" simulates a natural mixture reported about a sediment of the river Drevnice (see Reference [7]) with a high content of C_{13} and a higher chlorine content.

The compositions of the calibration mixtures, as well as of the independent quality assurance solutions, are mandatory to achieve the quantification of the variety of SCCP-mixtures. An example for recoveries of quality assurance solutions is given in Annex H.

Prepare the solutions "Lake Ontario water", "Perch", and "Sediment Drevnice" according to Table 2.

Table 2 — Reference substances stock solutions

Standard solutions, e.g. in *n*-heptane			**Synthetic mixed standard solutions which resemble environmental mixtures**		
n-alkane chain length	Chlorine content (%) of the individual C-number mixtures	Mean number of chlorines in the molecules (calculated)	"Lake Ontario water"	"Perch"	"Sediment Drevnice"
Chlorine content calculated (%)			**50,2**	**60,6**	**65,0**
Composition, ng/ml					
C_{10}	44,82	3,22			
C_{10}	50,18	3,97	1 000		
C_{10}	55,00	4,79	1 000		
C_{10}	60,09	5,86		500	
C_{10}	65,02	7,16		1 100	280
C_{11}	45,50	3,63	1 000		
C_{11}	50,21	4,37	1 000		

Table 2 *(continued)*

Standard solutions, e.g. in *n*-heptane			**Synthetic mixed standard solutions which resemble environmental mixtures**		
n-alkane chain length	Chlorine content (%) of the individual C-number mixtures	Mean number of chlorines in the molecules (calculated)	"Lake Ontario water"	"Perch"	"Sediment Drevnice"
Chlorine content calculated (%)			**50,2**	**60,6**	**65,0**
Composition, ng/ml					
C_{11}	55,20	5,31		600	
C_{11}	60,53	6,55		1 000	500
C_{11}	65,25	7,94		3 000	660
C_{12}	45,32	3,93	2 000		
C_{12}	50,18	4,76	2 000	800	
C_{12}	55,00	5,74	2 000	2 000	
C_{12}	65,08	8,59		900	1 000
C_{12}	69,98	10,62			830
C_{13}	44,90	4,19			
C_{13}	50,23	5,16			
C_{13}	55,03	6,22			
C_{13}	59,98	7,56		100	730
C_{13}	65,18	9,34			6 000
Sum of SCCP (ng/ml)			**10 000**	**10 000**	**10 000**

The chlorine content (third column) of the mixtures is calculated as the weighted mean.

Store the prepared solutions in a refrigerator at 2 °C to 6 °C.

5.3 Internal standard stock solutions from individual congeners.

Use commercially available individual congener standard solutions and prepare a stock solution in propanone (acetone) (5.1) at a concentration of, for example, 1 µg/ml.

— Individual SCCP congeners with chlorine contents of between 50 % and 67 % are suitable as internal standards if the mass trace in mass spectrometric detection is not affected by matrix components, e.g. 1,1,1,3,11,13,13,13-octachlorotridecane at e.g. 0,1 µg/ml.

NOTE 1 The different individual SCCP congeners used as internal standard substances probably contribute in environmental samples to the sum of SCCPs. Nevertheless, the contribution is approximately <1 % which means that the enhancement of the measurement uncertainty is negligible.

NOTE 2 Different individual SCCP congeners can produce different response factors, hence, it can be necessary for different concentrations to be used.

The solutions should be stored in a refrigerator at 2 °C to 6 °C.

5.4 Calibration solutions.

Use the standard mixtures according to Table 2. Prepare a minimum of nine calibration solutions (see Table 3) with concentrations according to the detection capability of the mass spectrometer. Combine and dilute the solutions (5.2) and the internal standard stock solution (5.3) with *n*-heptane to produce solutions for the calibration range, e.g. as shown in Table 3.

Table 3 — Calibration solutions

Mixture	"Lake Ontario" water	"Perch"	"Sediment Drevnice"	Internal standard
				1,1,1,3,11,13,13,13-octachlorotridecane
	µg/ml	µg/ml	µg/ml	µg/ml
Sum of SCCPs, µg/ml				
0,15	0,15			0,1
0,15		0,15		0,1
0,15			0,15	0,1
0,6	0,6			0,1
0,6		0,6		0,1
0,6			0,6	0,1
1	1			0,1
1		1		0,1
1			1	0,1
2	2			0,1
2		2		0,1
2			2	0,1
3	3			0,1
3		3		0,1
3			3	0,1

The solutions can be stored in a refrigerator, for at least four weeks. Check the concentration of the calibration solutions against an independently prepared standard prior to use.

Quality control check solutions can be prepared to check the calibration independently. For that, use the calibration mixtures as given in ISO 12010 (see Annex K). These mixtures are commercially available (e.g. in cyclohexane or *n*-hexane).

5.5 Extraction auxiliary and clean-up materials.

5.5.1 High purity silica sand, grain size 600 µm to 850 µm, blank free from contamination.

5.5.2 Copper powder, grain size <63 µm.

5.5.3 2 mol/l hydrochloric acid.

5.5.4 Al_2O_3, neutral, high activity, (10 % water).

5.5.5 Glass wool.

5.6 Operating gases, for GC-MS of high purity and in accordance with manufacturer's specifications.

5.7 Nitrogen, N_2, purity ≥99,996 % volume fraction for concentrating the solutions.

5.8 Sodium sulfate, anhydrous, Na_2SO_4, powdered.

6 Apparatus

Clean all glassware by rinsing with acetone (propanone) (5.1).

6.1 Wide-necked bottle, 1 000 ml up to 5 000 ml capacity for wet sediment or sludge.

6.2 Freeze drying apparatus.

6.3 Deep freezer.

6.4 Mortar and pestle or a grinding mill.

6.5 Drying ovens, capable of maintaining temperatures in the ranges of 100 °C to 400 °C for baking and storage of clean-up materials, for baking of glassware, and for dry residue determination of samples.

6.6 Sieve shaker with appropriate sieve meshes (aperture size), e.g. 2 mm.

6.7 Desiccator.

6.8 Pressurized liquid extractor (PLE) and filter suited for the device.

6.9 Evaporation device.

For example, rotary evaporator, turboevaporator, or vacuum concentration device.

6.10 Glass columns for chromatographic clean-up.

6.11 GPC clean-up system (with modular design).

6.11.1 Pump, sampling injector, sample rack, fraction collector.

6.11.2 Column, Shodex CLNpakPAE 800 AC®[1)] 8,0 mm × 300 mm, or equivalent.

6.12 Volumetric cylinders, 250 ml and 500 ml.

6.13 Volumetric flasks, 1 ml, 2 ml, 10 ml, and 25 ml.

6.14 Pasteur pipettes, e.g. 2 ml.

6.15 Syringes, 2 µl, 5 µl, 10 µl, and 50 µl, volume precision ±2 %.

6.16 Sample vials.

Amber glass with fluoropolymer-lined screw-cap is most suitable.

6.17 Gas chromatograph, with a splitless injection port coupled to a mass spectrometer (GC-MS) with chemical ionization (ECNI) and appropriate reactant gas (e.g. CH_4).

1) Shodex CLNpakPAE 800 AC is a product commercially available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

6.18 Analytical column.

A fused silica column with non-polar low bleed separating phase (see Annex C for examples), e.g. inner diameter <0,25 mm, length 15 m, and film thickness of 0,1 µm is recommended.

7 Sampling and sample pretreatment

Take samples as specified in ISO 5667-12, ISO 5667-13, or ISO 5667-17 in a bottle (6.1). Transport them in the dark between 2 °C and 8 °C. Store them in the laboratory between 1 °C to 5 °C. Pretreat the samples immediately in the laboratory by homogenizing and freeze-drying. Dependent on the analytical task, grind the samples, e.g. using apparatus (6.4), and sieve them using a sieve shaker (6.6) with the appropriate aperture.

8 Procedure

8.1 Extraction

Place the filter and the sand in the extractor according to the instructions for the extractor (PLE). Transfer a suitable mass, e.g., 5 g of suspended particulate matter or 0,5 g of sewage sludge of the pretreated, dry sample into the prepared extractor cell. Depending on the expected concentration in the sample, add the internal standard solution (5.3). The solvent *n*-heptane allows for a complete extraction with an extraction program given in Annex A.

Concentrate the extract from the extractor (e.g. 20 ml) gently (at a temperature of 40 °C) to approximately 1 ml using a suitable evaporation device (6.9).

Other extraction techniques, e.g. Soxhlet extraction or ultrasonic extraction, may be used after performing comparability exercise with PLE and the given programme.

8.2 Extract clean-up

The following two-step clean-up procedure should be carried out starting from approximately 1 ml concentrated extract in *n*-heptane (see 8.1):

a) column chromatographic clean-up with 2 g activated copper powder (5.5.2) and 2 g Al_2O_3, neutral, high activity (5.5.4);

b) gel chromatographic clean-up (6.11) with Shodex CLNpakPAE 800 AC®[2)] 8,0 mm × 300 mm and 0,5 ml/min propanone (acetone) as the eluent.

Fill the copper powder in a glass column with a glass wool plug. Activate the copper by adding 10 ml of 2 mol/l hydrochloric acid (5.5.3). Allow all of the hydrochloric acid to soak into the copper powder before washing the column, first with 25 ml of water and subsequently, with 20 ml of acetone to remove acid and water from the column. Then, a stopcock may be attached to the bottom of the column for controlling the elution progress. After the solvent level has reached the upper level of the copper powder, wash the copper layer with 3×2 ml *n*-heptane. Then, 2 g of aluminium oxide (10 % water) and about 10 ml of *n*-heptane are added for obtaining a copper/aluminium oxide - sandwich column. The wet column is used for applying the sample extract. The SCCPs are eluted by 10 ml of a mixture of *n*-heptane/acetone (98:2) which is concentrated to approximately 1,2 ml. Never let the chromatography column run dry.

This concentrate is used for the subsequently following GPC clean-up. Inject, e.g., 1 ml of the concentrate and elute in a fraction between 12 ml and 13,5 ml. This fraction is concentrated again to, e.g., 1 ml dried by sodium sulfate (5.8) and transferred into a sample vial for injection into the GC-MS.

2) Shodex CLNpakPAE 800 AC is a product commercially available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

After using a new column for the GPC clean-up, verify the eluent volume for complete elution of the analytes of interest by analysing an appropriate standard solution and/or spiked sample extract. Recoveries of SCCPs should be >50 % and no interfering peak should appear in the gas chromatogram. If necessary, GPC conditions need to be modified to meet these requirements.

NOTE Alternative clean-up procedures, an extended column chromatographic clean-up (see Annex D), and a modified gel chromatographic clean-up (see Annex E) can be used. The interferences quantified in Clause 4 apply only to the conditions described in this subclause.

8.3 Measurement and integration of the chromatogram

Optimize the operating conditions of the GC-ECNI-MS system, e.g. according to the manufacturer's instructions. Examples of the gas chromatographic conditions are given in Annex C.

Prior to analysis, verify the performance of the GC-ECNI-MS system by analysis of calibration standards. Use, as a minimum, the calibration solutions "Lake Ontario water" and "Sediment Drevnice" to optimize the GC-ECNI-MS system.

Check the GC-ECNI-MS system performance regularly, e.g. between every 10 to 20 samples, by independently prepared calibration solutions (Table 3) with a concentration of, e.g. 1 µg/ml sum of SCCP.

The measurement is performed in the selected ion mode with four selected mass ion fragments (mass to charge values, *m/z*), i.e. *m/z* 375, *m/z* 411, and *m/z* 423, and *m/z* 449. For an explanation of this selection, see Reference [4].

The integration of the different *m/z* values should be carried out within different time retention ranges that are established from calibration solutions. An example of the integration ranges dotted in Annex F is given in Table 4.

Table 4 — Typical retention time ranges

m/z value	Approximate retention time range min	Approximate retention time range of the response maximum [a] min
375	4,3 to 4,9	4,4 to 4,7
411	4,5 to 5,0	4,7 to 4,9
423	4,5 to 5,3	4,8 to 5,0
449	4,8 to 5,7	5,0 to 5,5

[a] This represents the major portion of the SCCPs for the mass ion fragment monitored and is represented by an unresolved complex mixture of peaks.

An example for integration of a real sample is given in Annex I.

Use selected ion mode measurements for detecting the internal standard. Integrate the response of the internal standard as a single peak with the following *m/z* values (see Table 5).

Table 5 — *m/z* values of internal standard

Internal standard	*m/z* for quantification	*m/z* for qualification
1,1,1,3,11,13,13,13-Octachlorotridecane	460	458

8.4 Calibration

8.4.1 General

Short-chain polychlorinated *n*-alkanes with 50 % to 67 % chlorine content are mixtures containing approximately 6 000 congeners. SCCP compounds of different chlorine contents exhibit different

response factors in ECNI-MS. Interferences occur in the mass spectra because individual compounds cannot be separated by GC.

Using multiple linear regression techniques quantification can be carried out to a large extent independent of chlorine content (see Annex B and Reference [4]).

While modern mass spectrometric software frequently does not enable multiple linear regression techniques to be carried out, commercial software is available that does. See also the ready to use spreadsheet (http://standards.iso.org/iso/18635/).

8.4.2 Basic calibration

Analyse the calibration solutions (5.4) and integrate the responses as described in 8.3. Calibration is carried out by multiple linear regression using Formula (1).

$$\rho_{\Sigma SCCPs} = b_1 \frac{A_1}{A_{IS}} + b_2 \frac{A_2}{A_{IS}} \quad (1)$$

or in case of calibration only with m/z 411

$$\rho_{\Sigma SCCPs} = b_1 \frac{A_1}{A_{IS}}$$

where

$\rho_{\Sigma SCCPs}$ is the target concentration of the sum of SCCPs in the calibration solution, in micrograms per millilitre, µg/ml;

b_1, b_2 are the regression coefficients, in micrograms per millilitre, µg/ml;

A_1, A_2 are the peak areas of the analyte, e.g. m/z 375, m/z 423;

A_{IS} is the peak area of the internal standard, e.g. m/z 460.

The regression coefficients determined are used for quantification of unknown concentrations in samples. A graphical presentation of the three-dimensional calibration area is given in B.3.

The graphical presentation of calculated against target sum concentrations of SCCPs is a suitable means for two-dimensional graphical assessment of the goodness of fit. A typical example is given in Annex G using 15 calibration solutions in Table 3.

Table 6 — Typical regression coefficients for the sum of SCCP with 50 % to 67 % chlorine content based on internal standardization

	Regression coefficient with associated standard deviation (in brackets) **for the mass ion fragment combination** *m/z* 375 and *m/z* 423	**Regression coefficient with associated standard deviation** (in brackets) **for the mass ion fragment combination** *m/z* 411 and *m/z* 449	**Regression coefficient with associated standard deviation** (in brackets) **for the mass ion fragment** *m/z* 411
Concentration range 0,15 µg/ml to 3,0 µg/ml, e.g. 0,03 µg/g to 0,6 µg/g with 15 calibration solutions, see Table 3	*m/z* 375	*m/z* 411	*m/z* 411
	0,665 3 (0,027 5)	0,958 0 (0,129 6)	0,826 0 (0,096 5)
	m/z 423	*m/z* 449	
	0,146 3 (0,012 13)	−0,559 1 (0,316 7)	
Standard deviation of the predicted concentration	0,216	0,416	0,446
Correlation coefficient	0,986	0,948	0,935

The calibration can be checked with independent quality control mixtures of known concentrations of the sum of SCCPs. Any variation in the expected values should not exceed specified levels in 8.4.3. Typical examples are shown in Annex K (see also ISO 12010).

Verify the limit of quantification and the limit of detection according to ISO/TS 13530. Use the graphical presentation of the goodness of fit (see Annex G) as a start-estimate of the concentration of the limits of quantification and detection. Verify these measures in a suitable matrix.

8.4.3 Identification and quantification with mass fragment combinations

Values based on mass ion fragment combinations, *m/z* 375 and *m/z* 423, are used to quantify the concentration as this combination produces most precise results.

The following are the identification criteria:

a) the chromatographic hump should be situated in the *m/z* specific retention time range of the different SCCP-standard solutions (see Table 6);

b) the shape of the chromatographic hump should resemble the SCCP-standard patterns (see Annex F);

c) the calculated result based on *m/z* 411 (simple linear regression) or on *m/z* 411 and *m/z* 449 (negative results are a hint to MCCP-abundance) should not differ more than ±70 % of the result based on *m/z* 375 and *m/z* 423. Higher deviations can be observed with single SCCP-mixtures containing lower chlorine.

8.4.4 Calculation of the results

Calculate the results according to Formula (2) using the regression coefficients determined by the calibration (see 8.4.2).

$$\omega_{\Sigma\mathrm{SCCPs}} = \left(b_1 \frac{A_1}{A_{\mathrm{IS}}} + b_2 \frac{A_2}{A_{\mathrm{IS}}} \right) \frac{\rho_{\mathrm{IS},s}}{\rho_{\mathrm{IS,cal}}} \qquad (2)$$

where

$\omega_{\Sigma SCCPs}$ is the sum of SCCPs in the sample, in micrograms per gram, µg/g;

$\rho_{IS,s}$ is the concentration of the internal standard in the sample, in micrograms per gram, µg/g;

$\rho_{IS,cal}$ is the concentration of the internal standard in the calibration solutions, in micrograms per millilitre, µg/ml;

b_1, b_2 are the regression coefficients, in micrograms per millilitre, known from Formula (1);

A_1, A_2 are the peak areas of the analyte, e.g. m/z 375, m/z 423;

A_{IS} is the peak area of the internal standard in the sample, e.g. m/z 460.

8.4.5 Quality checks for internal standardization

Perform a linearity check according to ISO 8466-1 with the solutions according to 5.3.

Determine recovery rates of the internal standard after optimizing the clean-up procedure. Adjust the final volume to 1 ml. Then, calculate the recovery rates of the internal standards from Formula (3).

$$R = \frac{A_{IS,S}}{A_{IS,cal}} \times 100[\%] \quad (3)$$

where

$A_{IS,cal}$ is the average peak area of the internal standard in the calibration samples;

$A_{IS,s}$ is the peak area of the internal standard in a sample.

The minimum recovery rate of the internal standard in real samples is 25 %.

Performance data from an interlaboratory trial are provided in Annex J.

9 Expression of results

Report the results as the sum of SCCPs (with chlorine content from 50 % to 67 %), in micrograms per gram, to two significant figures.

10 Test report

This test report shall contain at least the following information:

a) the test method used, together with a reference to this International Standard, i.e. ISO 18635;

b) all information required for the complete identification of the sample;

c) the sample storage and pretreatment;

d) the expression of the results as indicated in Clause 9;

e) any deviations from this procedure.

Annex A
(normative)

Programme for pressurized liquid extraction

Table A.1 — Programme for pressurized liquid extraction

Parameter		Unit
Preheat	1	min
Heat	5	min
Static	10	min
Flush	60	Vol %
Purge	60	s
Cycles	2	
Pressure	103,4	bar
Temperature	100	°C
Solvent A	*n*-heptane, 100	Vol %
Solvent B	0	Vol %

Annex B
(informative)

Explanation of the calibration of the sum of SCCPs with multiple linear regression

B.1 Common calibration with linear regression and inverse calibration

Linear regression is used usually with one independent variable (concentration, ρ) and one dependent variable (response, y).

The common calibration function is as shown in Formula (B.1).

$$y = b_0 + b_1\rho \tag{B.1}$$

where

ρ is the concentration of the analyte;

b_0, b_1 are the regression coefficients;

y is the peak area or response of the analyte.

It is also possible to use the inverse function, i.e. as in Formula (B.2).

$$\rho = b_0 + b_1 y \tag{B.2}$$

The concentration is now a function of the response of the analyte. The difference between Formulae (B.1) and (B.2) is that linear regression now minimizes the error squares in the concentration, ρ axis and not, as before, in peak area, axis y. This difference is not relevant or significant.

The type of calibration function [expressed in Formula (B.2)] can be graphically expressed in two dimensions. This two-dimensional expression line can also be expressed three-dimensionally (see Figure B.1).

The goodness of fit can be expressed by the root mean square error of prediction (RMSEP) given by Formula (B.3).

$$\sqrt{\frac{\sum_i \left(\rho_i - \bar{\rho}_i\right)^2}{v}} \tag{B.3}$$

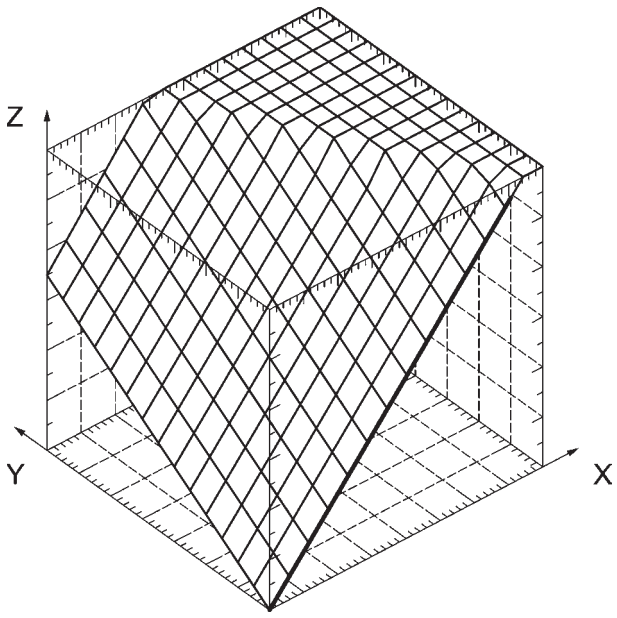
where

ρ_i is the predicted concentration of the analyte;

$\bar{\rho}_i$ is the true concentration of the analyte in the calibration sample;

v is the degrees of freedom.

The RMSEP reflects the deviation between known concentration values and calculated concentration values.



Key

X	y_1	response 1
Y	y_2	response 2
Z	ρ	concentration

Figure B.1 — Common (inverse) calibration function in a three-dimensional space

B.2 Multiple linear regression calibration

Compared to the common inverse linear regression with only one independent variable [Formula (B.1)], by inverse multiple linear regression attempts to model the relationship between two or more explanatory variables is modelled, e.g. peak areas of certain m/z values and the concentration of the analyte. In the case of this International Standard, the calibration is performed with two variables. The concentration is now dependent on two different responses, y_1, y_2.

$$\rho = b_0 + b_{1y1} + b_{2y2} \tag{B.4}$$

where

ρ is the concentration;

b_0, b_1, b_2 are the regression coefficients, determined by the software algorithm;

y_1, y_2 are the peak areas of the analyte.

The goodness of fit can also be expressed by the root mean square error of prediction (RMSEP), given by Formula (B.5).

$$\sqrt{\frac{\sum_i \left(\rho_i - \bar{\rho}_i\right)^2}{\nu}} \tag{B.5}$$

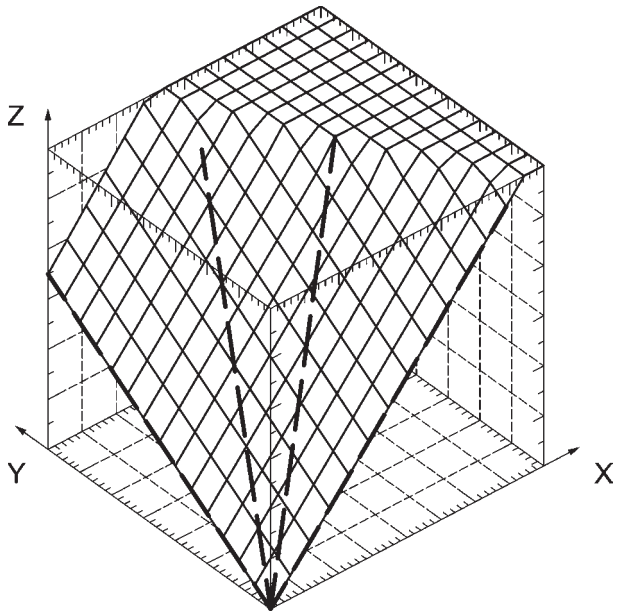
where

$\bar{\rho}_i$ is the predicted concentration of the analyte;

ρ_i is the true concentration of the analyte in the calibration sample;

v is the degrees of freedom.

The calibration function is now expressed not by a line, but by an area demonstrated here by an area and four lines in it.



Key

X	y_1	response 1
Y	y_2	response 2
Z	ρ	concentration

Figure B.2 — Calibration function with two variables y_1, y_2 — The calibration area

A two-dimensional presentation of the goodness of fit can be given as a recovery of predicted concentrations against true concentrations, see also Annex G.

B.3 Multiple regression calibration for the sum of SCCPs

The determination of the sum of SCCPs by ECNI-MS is demanding because very different response factors are given depending on the chlorine content of the compounds (see Reference [1]). A calibration by multiple regression is a way to use the information of the peak areas of two mass fragments.

SCCP congeners with smaller chlorine content contribute to smaller mass fragments, e.g. m/z 375. In addition, SCCP congeners with higher chlorine content contribute to higher mass fragments, e.g. m/z 423. By a weighted sum of the two selected mass fragments, a description of the sum of SCCPs is possible (see Reference [3] for the selection procedure and the validation experiments).

A typical regression area by the selected mass fragments, m/z 375 and m/z 423, is shown in Figure B.3. The calibration solutions described in Table 3 are demonstrated as points in the area. The data points

located in the calibration area are labelled and attributed to the calibration solutions and the quality assurance solutions used.

$$\rho_{\Sigma SCCPs} = b_1 \frac{A_1}{A_{IS}} + b_2 \frac{A_2}{A_{IS}} \quad \text{(B.6)}$$

where

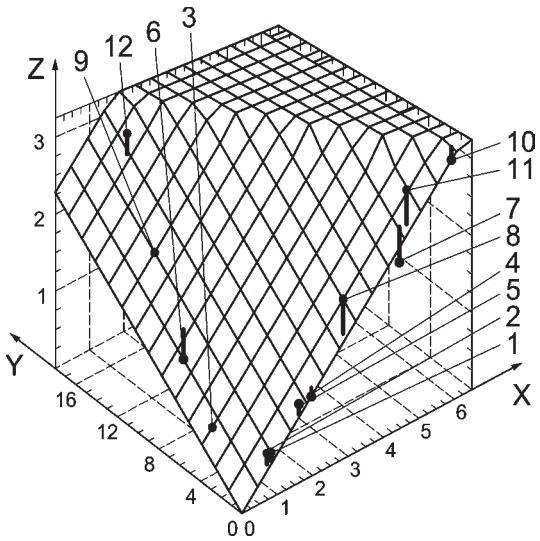
$\rho_{\Sigma SCCPs}$ is the target concentration of the sum of SCCPs in the calibration solution, in micrograms per litre, µg/l;

b_1, b_2 are the regression coefficients;

A_1, A_2 are the peak areas of the analyte, e.g. m/z 375, m/z 423;

A_{IS} is the peak area of the internal standard, e.g. m/z 460.

With the help of, for example, Excel®[3], the calculation with the LINEST function is easily possible and an example of a working sheet is given in http://standards.iso.org/iso/18635/.



Key

1	"Lake Ontario water", 0,6 µg/ml	8	"Perch", 2 µg/ml
2	"Perch", 0,6 µg/ml	9	"Sediment Drevnice", 2 µg/ml
3	"Sediment Drevnice", 0,6 µg/ml	10	"Lake Ontario water", 3 µg/ml
4	"Lake Ontario water", 1 µg/ml	11	"Perch", 3 µg/ml
5	"Perch", 1 µg/ml	12	"Sediment Drevnice", 3 µg/ml
6	"Sediment Drevnice", 1 µg/ml		
7	"Lake Ontario water", 2 µg/ml	X	Relative peak area m/z 375
$\rho_{\Sigma SCCPs}$	Sum of SCCPs, µg/ml	Y	Relative peak area m/z 423
		Z	Concentration, µg/ml

Figure B.3 — Regression area of the calibration solutions of Table 3 and quality assurance solutions of Tables 4 and A.1

3) Excel is the trade name of a product supplied by Microsoft. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

B.4 A mass spectrometric interpretation of the selected mass fragment ions

In ECNI-mass spectrometry of SCCPs, the degree of fragmentation is relatively low when compared with techniques like electron impact and positive chemical ionization (Reference[2]). In ECNI-MS, the predominant *m/z* values are $[M\text{-}Cl]^-$, $[M\text{-}HCl]^-$, and $[M+Cl]^-$. This fact was confirmed by a spectrum of 1,2,5,5,6,9,10-heptachlorodecane. It was measured under the same ionization conditions as described in Annex C, but in full scan mode. No molecular ion was detected. The major ions and their associated chlorine isotopes are *m/z* 345 $[M\text{-}Cl]^-$, *m/z* 344 $[M\text{-}HCl]^-$, and *m/z* 415 $[M+Cl]^-$ along with smaller amounts of *m/z* 309 $[M\text{-}Cl^{-}HCl]^-$ or $[M\text{-}HCl\text{-}Cl]^-$, *m/z* 308 $[M\text{-}HCl\text{-}HCl]^-$, and *m/z* 272 $[M\text{-}HCl\text{-}HCl\text{-}HCl]^-$.

After confirming that fragmentation way in the ECNI-MS used, possible fragments belonging to the selected mass ions for quantification are the following.

Below are some of the elemental compositions of the selected mass fragment ions.

For the mass ion 375:

$M+Cl$ $[C_{12}H_{21}{}^{35}Cl_6]^-$ $M\text{-}HCl$ $[{}^{12}C_{11}{}^{13}C_1H_{18}{}^{35}Cl_5{}^{37}Cl]^-$

$M\text{-}Cl$ $[C_{12}H_{19}{}^{35}Cl_5{}^{37}Cl]^-$

$M\text{-}Cl^{-}HCl$ $[{}^{12}C_{12}H_{17}{}^{35}Cl_4{}^{37}Cl_2]^-$

For the mass ion 423:

$M+Cl$ $[C_{13}H_{22}{}^{35}Cl_7]^-$,	$M+Cl$ $[C_{10}H_{15}{}^{35}Cl_4{}^{37}Cl_4]^-$
$M\text{-}Cl$ $[C_{10}H_{13}{}^{35}Cl_3{}^{37}Cl_5]^-$,	$M\text{-}Cl$ $[C_{13}H_{20}{}^{35}Cl_6{}^{37}Cl]^-$
$M\text{-}Cl^{-}HCl$ $[C_{10}H_{11}{}^{35}Cl_2{}^{37}Cl_6]^-$,	$M\text{-}Cl^{-}HCl$ $[C_{13}H_{18}{}^{35}Cl_5{}^{37}Cl_2]^-$
$M\text{-}HCl$ $[{}^{12}C_{12}{}^{13}C_1H_{19}{}^{35}Cl_6{}^{37}Cl]^-$,	$M\text{-}HCl$ $[{}^{12}C_9{}^{13}C_1H_{12}{}^{35}Cl_3{}^{37}Cl_5]^-$

It is difficult to explain, by mass spectrometric reasons, why these particular mass ions are most suitable for quantification. The different possible elemental compositions illustrate the complex overlapping that occurs when integrating over the full retention range. Using the selected ion chromatograms of the standard mixtures (see figures in Annex I), it is demonstrated that the selected *m/z* values with a retention range of nearly 1 min in a very fast GC heating rate (70 °C/min) and a relatively short column (15 m) should belong to a variety of single compounds.

The selection for quantification was carried out using a described empirical approach (see Reference [4]).

Annex C
(informative)

Examples of typical GC-MS conditions

C.1 Example 1

Injection:	Pressure-pulse splitless	344,7 kPa (50 psi); 1 min splitless time
Injector temperature:	275 °C	
Injection volume:	2 µl	
Transfer line temperature:	280 °C	
Ion source (ECNI)	150 °C	CH_4 (99,995 %); 5 ml/min
Quadrupole	106 °C	
Resolution	Low resolution, 0,9 mass unit	
Flow rate:	1,3 ml/min constant	
Carrier gas:	Helium 99,999 %	
Capillary column:	length:	15 m
	film thickness:	0,1 µm
	inner diameter:	0,25 mm
Column material:	medium polar, e.g. methylphenyl silicone phase DB5 MS	
Temperature programme:	100 °C 2 min → 70 °C/min to 280 °C → 2 min 280°C → 70 °C/min to 320 °C 7 min	

C.2 Example 2

Injection:	Pulse splitless	90 kPa; 1,5 min splitless time
Injector temperature:	300 °C	
Injection volume:	5 µl	
Transfer line temperature:	280 °C	
Ion source (ECNI)	150 °C	CH_4 (99,995 %); 5 ml/min
Quadrupole	106 °C	
Resolution	Low resolution. 0,9 mass unit	
Flow rate:	1,8 ml/min constant	
Carrier gas:	Helium 99,999 %	
Capillary column:	length:	15 m
	film thickness:	0,25 µm
	inner diameter:	0,25 mm
Column material:	HP5 MS	
Temperature programme:	80 °C 2 min → 40 °C/min to 300 °C → 3 min 300°C → 70 °C/min to 320 °C 7 min	

C.3 Example 3

Injection:	Pulse splitless	150 kPa (21,8 psi); 1,25 min splitless time
Injector temperature:	275 °C	
Injection volume:	2 µl	
Transfer line temperature:	280 °C	
Ion source (ECNI)	150 °C	CH_4 (99,995 %); 5 ml/min
Quadrupole	150 °C	
Resolution	Low resolution, 0,9 mass unit	
Flow rate:	1,6 ml/min constant	
Carrier gas:	Helium 99,999 %	
Capillary column:	length:	15 m
	film thickness:	0,1 µm
	inner diameter:	0,25 mm
Column material:	DB5-MS	
Temperature programme:	120 °C 2 min → 50 °C/min to 325 °C → 9 min 325 °C	

C.4 Example 4

Injection:	splitless	
Injector temperature:	260 °C	
Injection volume:	2 µl	
Transfer line temperature:	320 °C	
Ion source (NCI)	160 °C	CH_4 (99,995 %); 1,7 ml/min
Quadrupole	—	
Resolution	0,8 mass unit	
Flow rate:	1,5 ml/min constant	
Carrier gas:	Helium 99,999 %	
Capillary column:	length:	15 m
	film thickness:	0,10 µm
	inner diameter:	0,25 mm
Column material:	slightly polar, e.g. 5 % diphenyl-/95 % dimethylpolysiloxane (DB5-MS)	
Temperature programme:	60°C, 2 min → 45 °C/min to 340 °C, 10 min	

Annex D
(normative)

Alternative clean-up with column chromatography

D.1 General

The following alternative clean-up procedure can be used together with an alternative internal standard substance.

***m/z* values of internal standard**

Internal standard	*m/z* for quantification	*m/z* for qualification
1,2,5,5,6,9,10-Heptachlorodecane	348	346

D.2 Extraction

2 g Al_2O_3 (Al_2O_3 90, standardized for column chromatographic adsorption analysis according to Brockmann) and a mixture of 10 g Na_2SO_4, 10 g sand (see 5.5.1) and the dried sample is filled in the extraction cell of the PLE (see 8.1) (PLE solvent: cyclohexane/ethylacetate 1:1). Use a PLE program according to Annex A.

D.3 Column chromatography

Put about 0,5 g Na_2SO_4 on a quartz wool plug into a glass column (length: 20 cm, inner diameter: 1,5 cm).

Wash the column with 3 ml cyclohexane.

Fill in three layers of silica gel (activated: 8 h at 550 °C) with the following reagent and suspended in cyclohexane:

— 3 g silica gel 3 % H_2O;

— 3 g silica gel 40 % H_2O;

— 10 g silica gel 44 % H_2SO_4.

Apply the sample extract, e.g. 0,5 ml.

Elution is done using 25 ml *n*-hexane followed by 25 ml 1:1 *n*-hexane/dichloromethane. Discard the first 30 ml and collect the rest (20 ml).

Concentrate the eluate to a volume of 1 ml and transfer it into a sample vial for measurement.

D.4 Interferences with chlorinated chemicals (technical products)

Table D.1 — Highest concentration level which causes no interferences higher than the limit of quantification 0,03 µg/g

Potential interfering compounds	Highest concentration level which causes no interferences higher that the limit of quantification, 0,03 µg/g
Aroclor 1262[a]	0,5 µg/g
Aroclor 1242[a]	7 µg/g
Aroclor 1221[a]	7 µg/g
Campheclor (toxaphene)	Cannot be removed sufficiently
Halowax 1014[a]	1 µg/g
Halowax 1051[a]	1 µg/g
MCCP (medium-chain chlorinated *n*-alkanes) 42 %	1 µg/g
MCCP (medium-chain chlorinated *n*-alkanes) 52 %	0,5 µg/g
MCCP (medium-chain chlorinated *n*-alkanes) 57 %	1 µg/g

[a] Aroclor 1262, Aroclor 1242, Aroclor 1221, Halowax 1014, and Halowax 1051 are products commercially available. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Annex E
(informative)

Alternative clean-up with gel chromatography

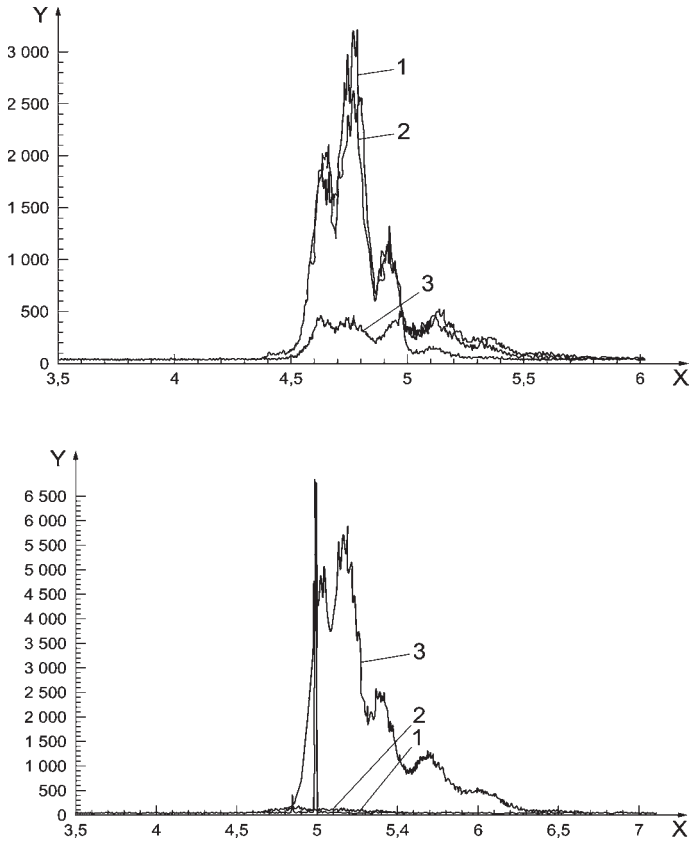
The following gel chromatographic column may be used alternatively. Campheclor (toxaphene) cannot be removed sufficiently. It has to be tested with other interferences occur.

GPC: column: Nucleogel 300 mm × 7,7 mm (MACHEREY-NAGEL),

0,5 ml/min acetone, collect fraction 9,5 ml to 11 ml

Annex F
(informative)

Typical chromatograms of standard solutions 1 µg/ml



Key

1 "Lake Ontario" water

2 "Perch"

3 "Sediment Drevnice", each with 1 µg/ml

X time, min

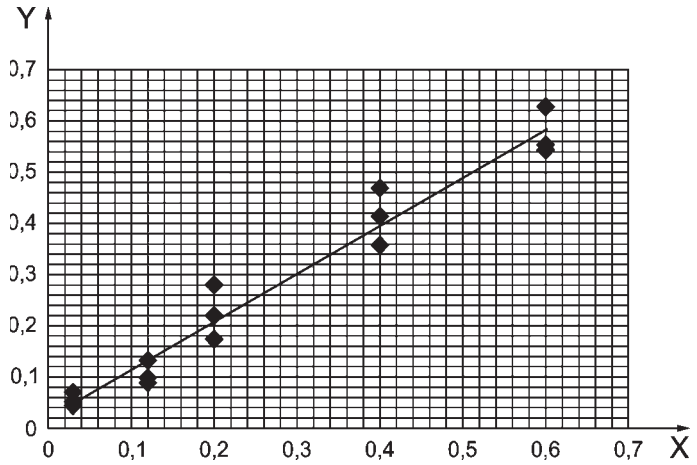
Y abundance

NOTE The single peak is a signal of the internal standard.

Figure F.1 — Chromatograms [selected ion monitoring (SIM)] of the *m/z* value 375 (upper chromatogram) and *m/z* value 423 (lower chromatogram) of the selected calibration standard mixtures

Annex G
(informative)

Presentation of goodness of fit of the calibration



Key

X target sum of SCCP, in µg/g

Y experimental result of the sum of SCCP, in µg/g

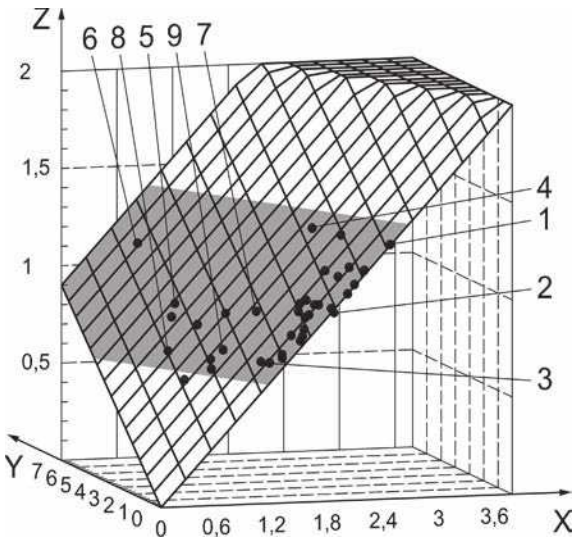
Figure G.1 — Presentation of goodness of fit of the calibration

Annex H
(informative)

Example for recoveries of quality assurance solutions

Table H.1 — The variety of SCCP-mixtures included in the model

	37 SCCP-mixtures for quality check of the model		
Characterizing of the SCCP-mixtures	**Min**	**Max**	**Mean**
% C10	2,80	24,00	12,21
% C11	11,60	55,00	32,72
% C12	16,50	76,70	35,07
% C13	0,00	67,30	19,99
% chlorine content	50,19	66,66	59,06



Key

1 to 9 1 µg/ml ± 0,4 µg/ml, selected SCCP-mixtures, see Table H.2

X relative peak area, m/z 375

Y relative peak area, m/z 423

Z sum of SCCPs, µg/ml

Figure H.1 — Calibration area of the sum of SCCP calculated by multiple linear regression

Table H.2 — Selected, labelled SCCP-mixtures and their location in the calibration area

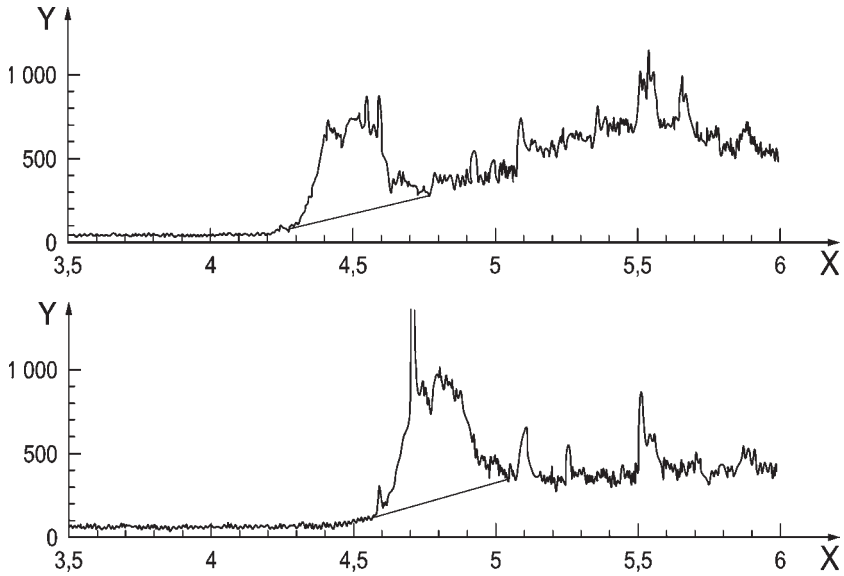
Label	% Chlorine content	% C10	% C11	% C12	% C13	Quantification result, µg/ml (target 1 µg/ml)
1	57,60	10	20	70	0	1,30
2	50,19	20	20	60	0	0,97
3	61,75	12	53	28	7	0,70
4	59,45	7,1	18	35,2	39,7	1,32

Table H.2 *(continued)*

Label	% Chlorine content	% C10	% C11	% C12	% C13	Quantification result, µg/ml (target 1 µg/ml)
5	61,26	7,5	15,9	18,4	58,2	0,90
6	64,96	2,8	11,6	18,3	67,3	1,14
7	62,97	10	50	20	20	0,92
8	60,33	4,7	14,3	16,5	64,5	0,69
9	66,66	20	32	31	17	0,73

Annex I
(informative)

Chromatograms of real samples

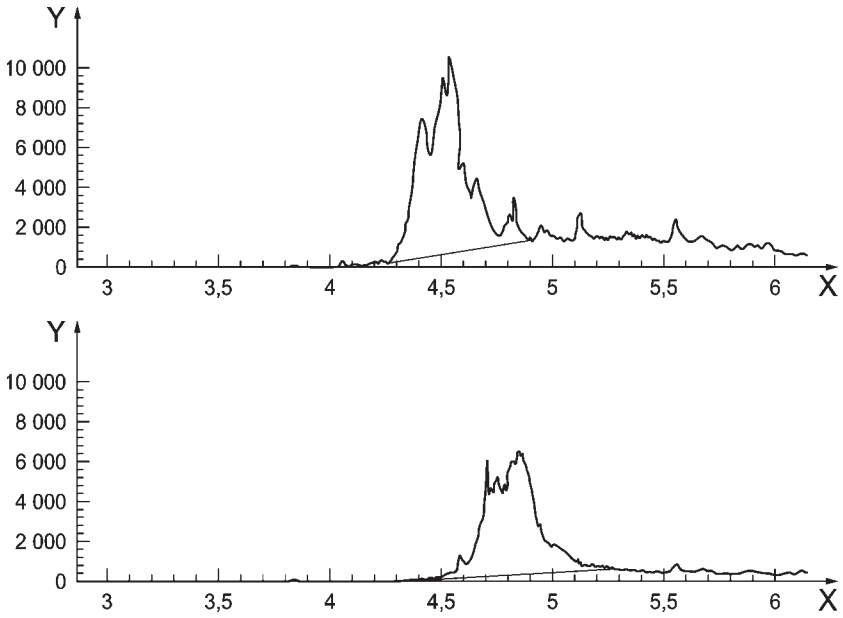


Key

X time, min

Y abundance

Figure I.1 — Sediment sample — Sum of SCCPs concentration of 0,07 µg/g; *m/z* 375 (upper chromatogram); *m/z* 423 (lower chromatogram)

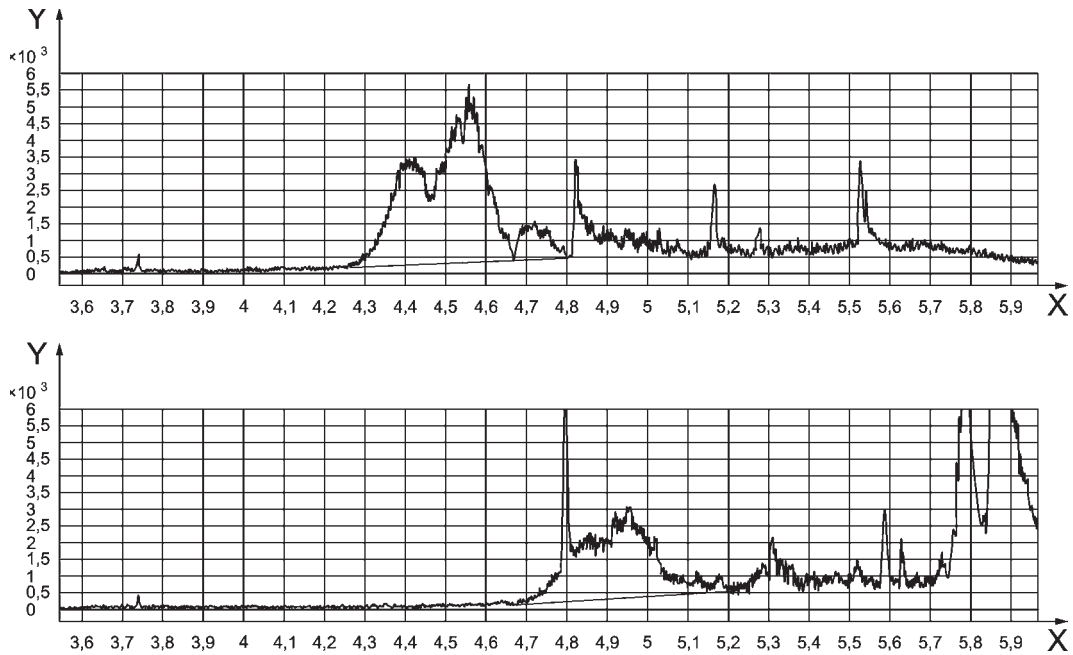


Key

X time, min

Y abundance

Figure I.2 — Sediment sample — Sum of SCCPs concentration of 0,55 µg/g; *m/z* 375 (upper chromatogram); *m/z* 423 (lower chromatogram)



Key

X acquisition time, min

Y abundance

Figure I.3 — Sample of an activated sewage sludge — Sum of SCCPs concentration of 2 µg/g; *m/z* 375 (upper chromatogram); *m/z* 423 (lower chromatogram)

Annex J
(informative)

Performance data

An interlaboratory trial for the validation of ISO 18635 was carried out in spring of 2015. The results are shown in Table J.1.

Table J.1 — Performance data (quantification using *m/z* 375 and *m/z* 423) (in accordance with ISO 5725-2)

Sample	l	n	o	X	$\bar{x}$	η	s_R	$C_{V,R}$	s_r	$C_{V,r}$
			%	µg/g	µg/g	%	µg/g	%	µg/g	%
1) Chinese sediment	8	16	0,0	—	0,58	—	0,24	41,4	0,027	4,7
2) Fortified suspended particulate matter	7	28	12,5	0,2	0,213	106,5	0,069	32,4	0,021	9,9
3) Fortified sewage sludge	8	32	0,0	3	3,635	121,2	1,506	41,4	0,372	10,2
4) SCCP-Mix 63 %	8	8	0,0	1	1,602	160,2	0,148	9,2	—	—

l number of laboratories after outlier rejection
n number of individual test results after outlier rejection
o percentage of outliers
X assigned value
$\bar{x}$ overall mean of results (without outliers)
η recovery rate
s_R reproducibility standard deviation
$C_{V,R}$ coefficient of variation of reproducibility
s_r repeatability standard deviation
$C_{V,r}$ coefficient of variation of repeatability

Annex K
(informative)

Quality control check solutions

Independent quality control check solutions may also be used.

Table K.1 — Further solutions for quality assurance composition, ng/ml

Standard solutions			**Synthetic mixed standard solutions which resemble technical mixtures**		
n-Alkane chain length	Chlorine content (%) of the individual C-number mixtures	Mean number of chlorines in the molecules (calculated)	**SCCP 51,5 -s1**	**Hordalub 80 -s1**	**Cereclor 70 -s1**
Chlorine content calculated (%)			**51,5**	**56,0**	**66,7**
			Composition, ng/ml		
C_{10}	44,82	3,22			
C_{10}	50,18	3,97	500	500	
C_{10}	55,00	4,79	500	500	
C_{10}	60,09	5,86			
C_{10}	65,02	7,16			2 000
C_{11}	45,50	3,63			
C_{11}	50,21	4,37	2 500	500	
C_{11}	55,20	5,31	1 000	2 000	
C_{11}	60,53	6,55		1 900	
C_{11}	65,25	7,94			3 200
C_{12}	45,32	3,93			
C_{12}	50,18	4,76	2 500	500	
C_{12}	55,00	5,74	1 500	2 500	
C_{12}	65,08	8,59		200	
C_{12}	69,98	10,62			3 100
C_{13}	44,90	4,19	500		
C_{13}	50,23	5,16	1 000		
C_{13}	55,03	6,22		1 000	
C_{13}	59,98	7,56		400	
C_{13}	65,18	9,34			1 700
Sum of SCCP (ng/ml)			**10 000**	**10 000**	**10 000**

Use commercially available solutions, e.g. in cyclohexane or *n*-hexane, of the single mixtures of SCCP congeners with defined carbon chain length and with different defined chlorine contents (see Table K.1, first two columns). Alternatively, use commercially available ready-mixed solutions as described in Table K.1.

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