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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)

Qualité de l'eau — Détermination des alcanes polychlorés à chaîne courte (SCCP) dans l'eau — Méthode par chromatographie gazeuse-spectrométrie de masse (CG-SM) avec ionisation chimique négative (ICN)

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Foreword

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ISO 12010 was prepared by Technical Committee 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 water — Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard 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 to this International Standard 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 49 % and 67 %, including approximately 6 300 of approximately 8 000 congeners.

This method is applicable to the determination of the sum of SCCPs in unfiltered surface water, ground water, drinking water and waste water using gas chromatography-mass spectrometry with electron capture negative ionization (GC-ECNI-MS).

The method can be applied to samples containing 0,1 µg/l to 10 µg/l. Depending on the waste water matrix, the lowest detectable concentration is estimated to be $>$0,1 µg/l.

2 Normative references

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

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

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/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 49 % 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.

SCCPs in whole water samples are fortified with an internal standard and extracted using liquid-liquid extraction with an organic solvent. The sample enrichment procedure is followed by a clean-up procedure 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 selection of the mass fragments is specific for the variety of technical mixtures as well as for their chlorine content and C-number distribution patterns. Alternative mass fragment combinations for qualification are also given in this International Standard.

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 multiple linear regression, measuring solutions of different technical mixtures fortified with an internal standard.

The calibration requires a minimum of three different composed standard mixtures, each of which resembles the C-number distribution pattern and chlorine content of different technical mixtures. This is to reflect the variety of chlorine contents and C-number distribution patterns of technical SCCP mixtures and SCCP levels found in environmental samples, which cannot be described by a single defined standard substance.

The method allows a quantification of the sum of SCCPs expected to be within an expanded measurement uncertainty of less than 50 %.

4 Interferences

Non-specific matrix interferences, as well as interferences from other environmental situations, are dealt with using the given clean-up procedure. Following the entire procedure, including the concentration factor of approximately 5 000, the following pollutants have been tested and found not to cause interferences below the following concentrations.

Potential interfering compounds	Highest concentration level at which no interferences higher than the limit of detection are detected
Aroclor 1262[a]	0,5 µg/l
Aroclor 1242[a]	0,5 µg/l
Aroclor 1221[a]	1 µg/l
Campheclor	0,2 µg/l
Halowax 1014[a]	1 µg/l
Halowax 1051[a]	1 µg/l
Technical chlordane	0,5 µg/l
MCCP (medium-chain chlorinated *n*-alkanes) 42 %	0,2 µg/l
MCCP (medium-chain chlorinated *n*-alkanes) 52 %	0,2 µg/l
MCCP (medium-chain chlorinated *n*-alkanes) 57 %	0,2 µg/l

[a] Product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

If the clean-up procedure is repeated, interferences can be further reduced.

5 Reagents and standards

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).

NOTE Check blanks regularly over the entire procedure to ensure they are suitable and establish proper analytical control.

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

Use 2,2,4-trimethylpentane (C_8H_{18}, isooctane) for conditioning of the glass bottles (6.1).

For preparation of the stock solutions, use dilutions in propanone (acetone), C_3H_6O.

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

For the first elution step of the filtrated suspended matter, use methanol (CH_3OH).

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 a defined carbon chain length and with different defined chlorine contents (see Table 1, first two columns). Alternatively, use commercially available ready-mixed solutions as described in Table 1.

Prepare the solutions Hordalub 17[1)] -s1, SCCP 51,5 % -s1, Hordalub 80[1)] -s1, Cereclor 60[1)] -s1, Hordalub 500[1)] -s1, and Cereclor 70[1)] -s1 according to Table 1. The suffix "-s1" denotes synthetically mixed standard solutions, which resemble the technical mixtures.

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

Store the prepared solutions in a refrigerator at a temperature of 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 49 % and 67 % are suitable as internal standards, e.g.

— 1,1,1,3,10,11-hexachloroundecane, with e.g. 0,1 µg/ml;

— 1,1,1,3,11,13,13,13-octachlorotridecane, with e.g. 0,1 µg/ml;

— 1,2,5,5,6,9,10-heptachlorodecane, with e.g. 0,01 µg/ml.

NOTE 1 The different individual SCCP congeners used as internal standard substances probably contribute to the sum of SCCPs in environmental samples. 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 to use different concentrations.

If validated, other individual SCCP congeners can be used as the internal standard if the congener shows the same properties over the entire analytical process as the SCCPs being determined.

The solutions can be stored in a refrigerator at a temperature of 2 °C to 6 °C.

5.4 Copper powder, mesh size <63 µm. Copper powder is used in the clean-up procedure to remove sulfur and sulfur-containing matrix components.

5.5 Hydrochloric acid, 2 mol/l. Used for copper activation in the clean-up column.

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

5.6 Activated magnesium silicate,[2] MgO/3,75 SiO_2/(x) H_2O, for column chromatography.

Activated magnesium silicate is used in the clean-up procedure to separate organohalogenic compounds like polychlorinated biphenyls and naphthalenes.

Use activated magnesium silicate with the following characteristics: particle size 0,15 mm to 0,25 mm, of which 80 % >0,15 mm; surface area, determined according to the BET method, 170 m^2/g to 300 m^2/g; pH 9 to pH 10.

Activate the magnesium silicate by heating, for example, 200 g in a shallow dish at 140 °C for at least 4 h. Allow the activated magnesium silicate to cool to room temperature in a desiccator. Activated magnesium silicate can be stored in a closed bottle at room temperature for up to 1 month.

Table 1 — Reference substances stock solutions

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)**	Hordalub 17 -s1	SCCP 51,5 % -s1	Hordalub 80 -s1	Cereclor 60 -s1	Hordalub 500 -s1	Cereclor 70 -s1
			Chlorine content calculated, %					
			49,0	51,5	56,0	59,0	62,0	66,7
			Composition, ng/ml					
C_{10}	44,82	3,22	500					
C_{10}	50,18	3,97	500	500	500			
C_{10}	55,00	4,79		500	500			
C_{10}	60,09	5,86				1 000	900	
C_{10}	65,02	7,16				500	300	2 000
C_{11}	45,50	3,63	1 200					
C_{11}	50,21	4,37	2 600	2 500	500	700		
C_{11}	55,20	5,31		1 000	2 000	1 300	400	
C_{11}	60,53	6,55			1 900	1 200	2 500	
C_{11}	65,25	7,94					2 500	3 200
C_{12}	45,32	3,93	1 000					
C_{12}	50,18	4,76	2 400	2 500	500			
C_{12}	55,00	5,74		1 500	2 500	2 000	1 000	
C_{12}	65,08	8,59			200	1 500	1 700	
C_{12}	69,98	10,62						3 100
C_{13}	44,90	4,19		500				
C_{13}	50,23	5,16	1 800	1 000				
C_{13}	55,03	6,22			1 000	400		
C_{13}	59,98	7,56			400	1 300	700	
C_{13}	65,18	9,34				100		1 700
Sum of SCCPs, ng/ml			**10 000**	**10 000**	**10 000**	**10 000**	**10 000**	**10 000**

5.7 Sodium sulfate, Na_2SO_4, anhydrous, granular.

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

2) Florisil is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

5.9 Nitrogen, N_2, purity ≥99,996 % volume fraction, for drying of the sorbent packing material and for concentrating solutions.

5.10 Calibration working solutions. Use a minimum of three different composed standard mixtures according to Table 1, Hordalub 17 -s1, Hordalub 80 -s1, and Cereclor 70 -s1. Prepare a minimum of nine calibration solutions (see bold figures in Table 2) with concentrations that correspond the detection capability of the mass spectrometer. Combine and dilute the solutions (5.2) and the internal standard solution (5.3) with *n*-heptane to produce solutions for the calibration range, e.g. as shown in Table 2.

The solutions may be stored in a refrigerator for up to 4 weeks. Check the concentration of the calibration solutions against an independently prepared standard prior to use.

Table 2 — Calibration working solutions

Mixture	**Hordalub 17 -s1** µg/ml	**SCCP 51,5 -s1** µg/ml	**Hordalub 80 -s1** µg/ml	**Cereclor 60 -s1** µg/ml	**Hordalub 500 -s1** µg/ml	**Cereclor 70 -s1** µg/ml	**Internal standard: e.g. 1,1,1,10,11,13-hexachloro-decane** 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**
0,15				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**
0,6				0,6			0,1
0,6					0,6		0,1
0,6						**0,6**	**0,1**
1,0	**1,0**						**0,1**
1,0		1,0					0,1
1,0			**1,0**				**0,1**
1,0				1,0			0,1
1,0					1,0		0,1
1,0						**1,0**	**0,1**

5.11 Quality control multi-component stock solution of SCCP for assuring calibration procedure. Quality control check solutions should be prepared to check the calibration independently. Prepare a minimum of three different solutions; see Table 3 or Annex A. These mixtures are also commercially available (e.g. in cyclohexane or *n*-hexane).

The solutions can be stored in a refrigerator for up to 4 weeks.

5.12 Test solution for checking linearity of internal standards. Prepare solutions of the internal standard used at concentrations of 0,1 µg/ml, 0,5 µg/ml, and 1 µg/ml.

6 Apparatus

Glassware and equipment which may come into contact with water samples or their extracts should be free from interfering compounds.

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

6.1 Flat-bottomed glass bottles, conical shoulder, 1 000 ml capacity, for collecting water samples, preferably with glass stoppers.

The sample bottle shall enable direct extraction of the sample.

Before use, to condition it, rinse the dry sample bottle with e.g. 2 ml of isooctane (5.1), invert it and allow the solvent to drain and evaporate from it.

Table 3 — Calibration assurance solutions

***n*-Alkane chain length**	**Chlorine content of the individual C-number mixtures according to the manufacturer**, %	**SCCP 51,5 -s2**	**SCCP 55,5 -s2**	**SCCP 63 -s2**	**Hordalub 17 -s2**	**Hordalub 80 -s2**	**Hordalub 500 -s2**	**Cereclor 60 -s2**
		Calculated mean chlorine content, %						
		51,45	55,77	63,22	49,07	55,91	61,87	59,07
		Composition, ng/ml						
C_{10}	44,82				50			
C_{10}	50,18	50			50			
C_{10}	55	50	100			100		
C_{10}	60,09			50			90	150
C_{10}	65,02			50			20	
C_{11}	45,5	200	200		100			
C_{11}	50,21				280	50		
C_{11}	55,2	150				250		200
C_{11}	60,53		150	200		140	350	120
C_{11}	65,25			300			200	
C_{12}	45,32	150	100		100			
C_{12}	50,18	150	50	50	240	50		
C_{12}	55					250	100	200
C_{12}	65,08	100	200	100		20	170	150
C_{12}	69,98			50				
C_{13}	44,9	50						
C_{13}	50,23	100	50		180			
C_{13}	55,03		100			100		
C_{13}	59,98		50	100		40	70	170
C_{13}	65,18			100				
Sum of SCCPs, ng/ml		**1 000**	**1 000**	**1 000**	**1 000**	**1 000**	**1 000**	**990**

6.2 Evaporation device, e.g. rotary evaporator or nitrogen evaporating system.

6.3 Separator, for example micro-separator or other suitable device for phase separation.

6.4 Vials, compatible with the GC-autosampler (e.g. with a capacity of 1,5 ml).

6.5 Chromatographic column, internal diameter (ID) 10 mm (empty) for clean-up.

6.6 Gas chromatograph, temperature-programmable, with all required accessories, including gases, capillary column, split/splitless injector and mass spectrometer detector with negative-ion chemical ionization option and appropriate reactant gas (CH_4).

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

6.8 Disposable glass Pasteur pipettes, e.g. 150 mm or 250 mm.

6.9 Syringes, 2 µl, 5 µl, 10 µl and 50 µl.

6.10 Analytical column. Fused silica column with non-polar low-bleed separating phase (see Annex C for examples); e.g. ID <0,25 mm, length 15 m and film thickness 0,1 µm.

6.11 Glass fibre filter.

6.12 Vacuum filtration device, volume 1 l.

6.13 Shaking device or magnetic stirrer device (with a magnetic stir bar).

7 Sampling and sample pretreatment

Take samples as specified in ISO 5667-1 and ISO 5667-3. To collect water samples, use conditioned glass bottles (6.1). Do not fill the sample bottle completely (e.g. fill to the shoulder) in order to allow the addition of the extracting solvent.

Samples are extracted without filtering the sample and suspended solids are not removed prior to analysis.

Weigh, to the nearest gram, the sample bottle with its contents and cap, and record the mass for subsequent use. Thoroughly shake the bottle to homogenize the water sample. Add the internal standard solution (5.3) to achieve a concentration of, for example, 0,1 µg/l in the water sample. Record the mass, in micrograms, of internal standard added. Shake the bottle thoroughly.

8 Procedure

8.1 Extraction with liquid-liquid extraction method

Add 10 ml of extraction solvent, *n*-heptane (5.1), to the bottle and shake it or stir (6.13) thoroughly for about 2 h to carry out the extraction directly in the sample bottle. Allow the phases to separate and use the separator (6.3) to collect the organic extract in a separate tube. If an emulsion forms, break it by centrifuging and/or by adding sodium sulfate (5.7) to the tube. Discard the remaining water to waste. Transfer the solvent from the tube to the evaporating device (6.2) or, using a gentle stream of nitrogen (5.9), carefully evaporate the solvent (at a temperature of 40 °C) to about 1 ml. Weigh, to the nearest gram, the empty sample bottle and cap. Calculate the volume of water extracted and the concentration of internal standard in the water.

Proceed as in 8.3.

8.2 Extraction of samples with higher content of suspended matter

If the content of suspended matter is higher than approximately 200 mg/l, filter the sample through a glass fibre filter (6.11) and collect the filtrate in the bottle (6.1). Add 10 ml of methanol to the filter (without vacuum) to extract the suspended matter separately. Allow to soak for 5 min, then use vacuum to add methanol to the sample filtrate collected.

Add 10 ml *n*-heptane (without vacuum) to the filter, allowing it to soak for another 5 min, then use weak vacuum to add *n*-heptane also to the sample filtrate collected.

Proceed as in 8.1.

8.3 Extract clean-up

Before using the clean-up procedure with real samples, the analyst shall demonstrate that the fraction collected contains more than 80 % of the SCCP by performing recovery tests according to ISO/TS 13530. This shall apply to internal standard substances as well as to the calibration working solutions Hordalub 17 -s1 and Cereclor -s1 (5.10) at concentrations of 1 µg/ml in *n*-heptane. Test the recovery of the internal standard according to 8.5.5.

Place a glass-wool plug in a 10 mm ID chromatography column (6.5). Pack the column from bottom to top in the following sequence.

Add 2,5 g copper powder (5.4), then activate it by rinsing the column with 10 ml hydrochloric acid (5.5). Proceed to wash the copper powder with 25 ml deionized water followed by 20 ml propanone (5.1). Finally, rinse the column three times with 2 ml of *n*-heptane.

Add 3 g of activated magnesium silicate (5.6) and 1 g granular anhydrous sodium sulfate (5.7) to the column.

Gently tap the column to allow the adsorbents to settle.

Condition the prepared column with 10 ml of *n*-heptane (5.1). Do not allow the meniscus of the solvent to go below the level of the sodium sulfate, at any time. Discard the eluate and check the column for channelling. If a channelling effect is observed, discard the column and prepare another.

Add the concentrated extract (8.1) to the conditioned column, elute the column and discard the first eluate of approximately 1 ml. Do not allow the meniscus of the extract to go below the level of the sodium sulfate.

The following elution should be tested and optimized by each laboratory undertaking SCCP determinations. SCCPs are expected to elute in fraction d of Table 4.

Concentrate the eluate of fraction d (or an alternative fraction depending on optimization) to, for example, 0,2 ml ± 0,1 ml and transfer to a sample vial (6.4) for injection into the GC-MS line.

NOTE Use 2 g of basic aluminium oxide (Al_2O_3) 90, activity basic super I, particle size 0,063 mm to 0,2 mm, for a further reduction of non-specific background. The aluminium oxide should display maximum activity; avoid storage or contact with humid air. A combination of aluminium oxide with the other chromatographic phases, as described above, is possible, because SCCPs are eluted from aluminium oxide in the same liquid fraction as activated magnesium silicate. The elution fractions (see Table 4) have to be newly optimized.

Table 4 — Elutions to be tested and optimized

Step	Volume of elution solvent		Elution solvent composition, volume fractions	Eluate
a	5×	2 ml	*n*-heptane + propanone (5.1) (98 % + 2 %)	Discard.
b	1×	2 ml	*n*-heptane + propanone (5.1) (85 % + 15 %)	Discard.
c	1×	2 ml	*n*-heptane + propanone (5.1) (85 % + 15 %)	Collect and concentrate to 0,2 ml only for optimization. Discard if less than 10 % of the internal standard peak area is obtained.
d	2×	2 ml	*n*-heptane + propanone (5.1) (50 % + 50 %)	Collect and concentrate to 0,2 ml.
e	1×	1 ml	*n*-heptane + propanone (5.1) (50 % + 50 %)	Collect and concentrate to 0,2 ml only for optimization. Discard if less than 10 % of the internal standard peak area is obtained.

8.4 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, establish the operating conditions and verify performance of the GC-ECNI-MS system by analysing calibration standards. Use as a minimum the calibration solutions Hordalub 17 -s1 and Cereclor 70 -s1 to optimize the GC-MS system. Check the GC-ECNI-MS system performance regularly, e.g. every 10 to 20 samples.

The measurement is performed in the selected ion mode with four selected mass ion fragments, i.e. m/z 327, m/z 375, m/z 409 and m/z 423. For an explanation of this selection, see Reference [3].

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 G is given in Table 5.

Table 5 — Typical retention time ranges

m/z Value	Approximate retention time range min	Approximate maximum retention time range[a] min
327	4,0 to 5,0	4,2 to 4,5
375	4,5 to 5,5	4,6 to 5,0
409	4,7 to 5,4	4,8 to 5,2
423	4,5 to 6,0	4,9 to 5,2

[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 of integration of a real sample is given in Annex G.

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 6).

Table 6 — m/z values of internal standard substances

Internal standard substance	m/z for quantification	m/z for qualification
1,1,1,3,10,11-Hexachloroundecane	364	362
1,1,1,3,11,13,13,13-Octachlorotridecane	460	458
1,2,5,5,6,9,10-Heptachlorodecane	348	346

Examples of chromatograms of the mixtures are given in Annex D.

8.5 Calibration

8.5.1 General

Short-chain polychlorinated *n*-alkanes with 49 % to 67 % chlorine content are mixtures containing approximately 6 300 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 independently of chlorine content. See Annex B and Reference [3].

While modern mass spectrometric software frequently does not enable multiple linear regression techniques to be carried out, commercial software is available that does.

8.5.2 Basic calibration

Analyse the calibration working solutions (5.10) and integrate the responses as described in 8.4. Calibration is carried out by multiple linear regression using Equation (1):

$$\rho_{\Sigma \mathrm{SCCPs}} = b_1 \frac{A_1}{A_{\mathrm{IS}}} + b_2 \frac{A_2}{A_{\mathrm{IS}}} \tag{1}$$

where

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

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

A_1, A_2 are the peak areas of the analyte, e.g. *m*/*z* 327, *m*/*z* 423;

A_{IS} is the peak area of the internal standard, e.g. *m*/*z* 364.

The regression coefficients determined (see Table 7) are used for quantification of unknown concentrations in samples. A graphical presentation of the three-dimensional calibration area is given in Figure B.3.

The graphical presentation of sum concentrations of SCCPs calculated against target is a suitable means for two-dimensional graphical assessment of the goodness of fit. A typical example is given in Annex E, using the 18 calibration solutions in Table 2.

The calibration should be checked with at least two independent quality control mixtures (5.11) of known concentrations of the sum of SCCPs. Any variation in the expected values should not exceed specified levels. Typical examples are shown in Annex F.

The graphical presentation of the goodness of fit of the basic calibration (target concentrations of the sum of SCCP vs. calculated values, see Annex E) is the basis for verification of the limit of quantification and the limit of detection of the basic calibration according to ISO/TS 13530.

8.5.3 Identification and quantification with mass fragment combinations

Values based on mass ion fragment combinations *m*/*z* 327 and *m*/*z* 423 are used to quantify the concentration, as this combination produces more precise results.

Identification criteria are as follows:

a) the chromatographic hump, i.e. the major portion of the SCCPs for the mass ion fragment monitored represented by an unresolved complex mixture of peaks, should be situated in the *m*/*z* specific retention time range of the different SCCP standard solutions (see Table 5);

b) the shape of the chromatographic hump, i.e. the major portion of the SCCPs for the mass ion fragment monitored represented by an unresolved complex mixture of peaks, should resemble the SCCP standard solutions (see Annex D);

c) the calculated result based on *m*/*z* 327 and *m*/*z* 409 should not differ by more than ±50 % of the result based on *m*/*z* 327 and *m*/*z* 423;

d) the calculated result based on *m*/*z* 375 and *m*/*z* 423 should not differ by more than ±60 % of the result based on *m*/*z* 327 and *m*/*z* 423.

If criteria a) and b) are fulfilled, one of the criteria c) or d) is sufficient for an identification.

Table 7 — Typical regression coefficients for the sum of SCCPs with 49 % to 67 % chlorine content based on internal standardization

	Regression coefficient with associated standard deviation for the mass ion fragment combination *m*/*z* 327 and *m*/*z* 423	Regression coefficient with associated standard deviation for the mass ion fragment combination *m*/*z* 375 and *m*/*z* 423	Regression coefficient with associated standard deviation for the mass ion fragment combination *m*/*z* 327 and *m*/*z* 409	Degrees of freedom
Concentration range 0,15 µg/ml to 1,0 µg/ml with nine calibration solutions (see Table 2)	*m*/*z* 327: 0,227 9 (0,023 7) *m*/*z* 423: 0,228 9 (0,034 5)	*m*/*z* 375: 0,286 6 (0,029 1) *m*/*z* 423: 0,175 8 (0,037 1)	*m*/*z* 327: 0,221 4 (0,031 5) *m*/*z* 409: 0,131 5 (0,026 6)	7
Standard deviation of the predicted concentration	0,11	0,11	0,145	
Correlation coefficient	0,989	0,989	0,982	
Concentration range 0,15 µg/ml to 1,0 µg/ml with 18 calibration solutions (see Table 2)	*m*/*z* 327: 0,242 2 (0,016 4) *m*/*z* 423: 0,220 9 (0,030 4)	*m*/*z* 375: 0,267 3 (0,018 0) *m*/*z* 423: 0,174 4 (0,032 4)	*m*/*z* 327: 0,231 0 (0,033 5) *m*/*z* 409: 0,080 6 (0,024 6)	16
Standard deviation of the predicted concentration	0,11	0,11	0,17	
Correlation coefficient	0,989	0,989	0,970	

8.5.4 Calculation of the results

Calculate the results according to Equation (2) using the regression coefficients determined by the calibration (see 8.5.2).

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

where

$\rho_{\Sigma \mathrm{SCCPs}}$ is the concentration of the sum of SCCPs in the water sample, in micrograms per litre;

$\rho_{\mathrm{IS,s}}$ is the concentration of the internal standard in the water sample, in micrograms per litre;

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

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

A_1, A_2 are the peak areas of the analyte, e.g. *m*/*z* 327, *m*/*z* 423;

A_{IS} is the peak area of the internal standard, e.g. *m*/*z* 364.

Precision data are listed in Annex H.

8.5.5 Quality checks for internal standardization

Perform a linearity check according to ISO 8466-1 with the solutions, e.g. 5.12.

Determine recovery rates of the internal standard after optimizing the clean-up procedure. Contrary to what is indicated in 8.3, adjust the final volume to 1 ml. Then calculate the recovery rates of the internal standards from:

$$R = \frac{A_{\mathrm{IS,\,cal}}}{A_{\mathrm{IS,\,s}}} \times 100 \left[\%\right] \tag{3}$$

where

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

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

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

9 Expression of results

Report the results as the sum of SCCPs (with chlorine content between 49 % to 67 %), in micrograms per litre, 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 (ISO 12010:2012);

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

c) sample storage and pre-treatment;

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

e) any deviations from this procedure as well as the selection of alternative mass fragments for quantification.

Annex A
(informative)

Additional quality control check solutions

Other quality control check solutions may also be used in addition to those described in 5.11.

Table A.1 — Further solutions for quality assurance

***n*-Alkane chain length**	**Chlorine content of the individual C-number mixtures according to Ehrenstorfer**, %	**Sediment**	**Perch**	**Catfish**	**Zebra mussels**	**C_{10}/C_{11} mostly**
		-s2	-s2	-s2	-s2	-s2
		Calculated mean chlorine content, %				
		61,82	61,02	61,13	57,99	50,45
		Composition, ng/ml				
C_{10}	44,82					100
C_{10}	50,18				50	200
C_{10}	55				50	200
C_{10}	60,09	30	50	100		
C_{10}	65,02	30	110	120	30	
C_{11}	45,5				20	100
C_{11}	50,21				30	200
C_{11}	55,2	30	60	100	150	100
C_{11}	60,53	150	100	200	210	
C_{11}	65,25	140	300	120	50	
C_{12}	45,32					100
C_{12}	50,18	40	80	50	120	
C_{12}	55	160	200	100	100	
C_{12}	65,08	190	90	100	80	
C_{12}	69,98	50		50	40	
C_{13}	44,9					
C_{13}	50,23					
C_{13}	55,03	60		30	40	
C_{13}	59,98	60	10	30	30	
C_{13}	65,18	60				
Sum of SCCPs, ng/ml		**1 000**	**1 000**	**1 000**	**1 000**	**1 000**

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

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

where

ρ is the concentration of the analyte;

b_0, b_1 are the regression coefficients;

y peak area or response of the analyte.

It is also possible to use the inverse function, i.e.

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

The concentration is now a function of the response of the analyte. The difference between Equations (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 y axis. This difference is not relevant or significant.

The type of calibration function [expressed in Equation (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

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

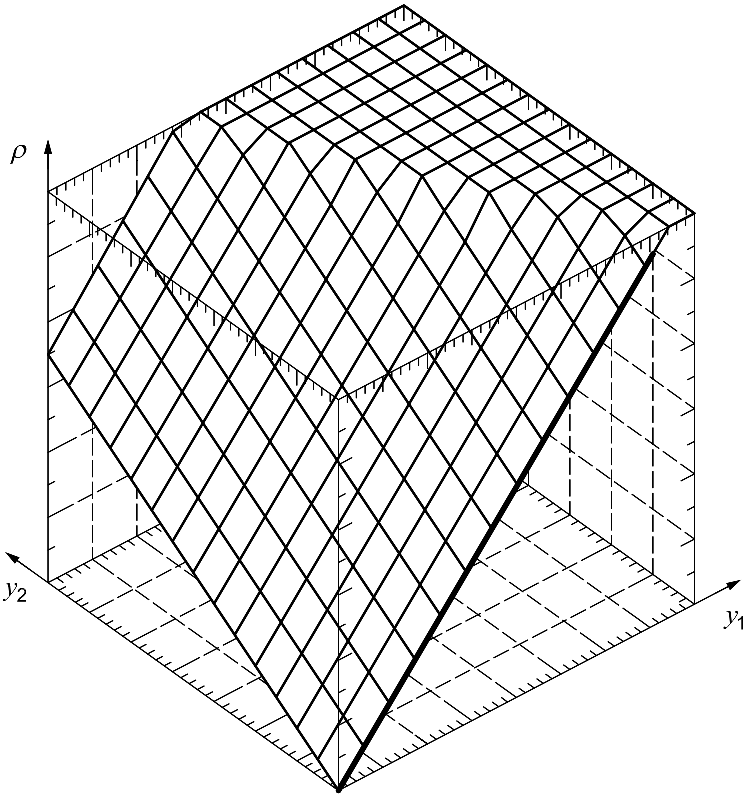
where

$\bar{\rho}_i$ 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 RMSEP reflects the deviation between known concentration values and calculated concentration values.



Key

y_1 response 1

y_2 response 2

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 [Equation (B.1)], the inverse multiple linear regression attempts to model the relationship between two or more explanatory variables, 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.

$$\rho = b_0 + b_1 y_1 + b_2 y_2 \quad \text{(B.4)}$$

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 RMSEP (root-mean-square error of prediction), given by

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

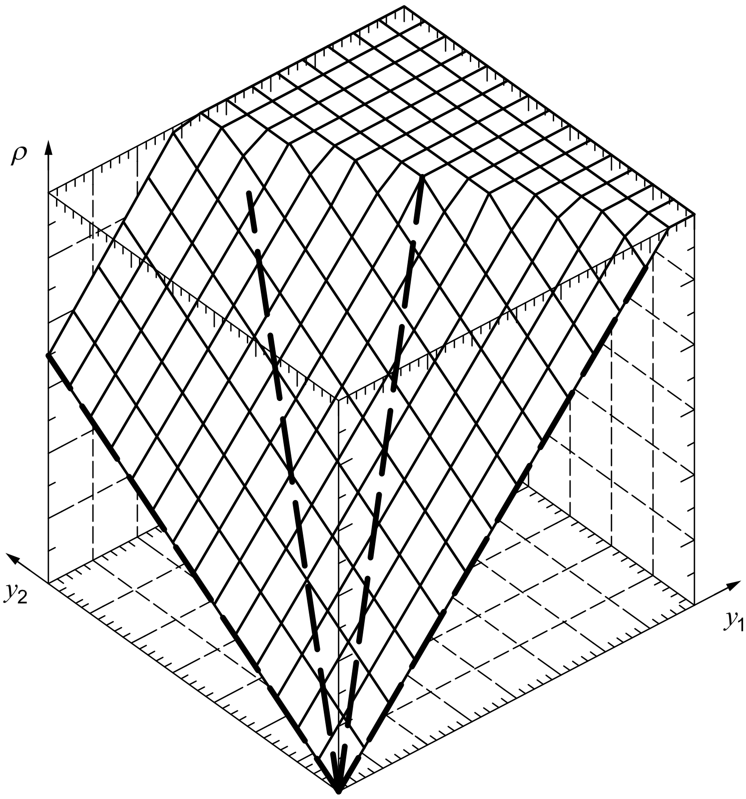
where

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

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

ν 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

y_1 response 1

y_2 response 2

ρ concentration

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

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

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 (Reference [1]). A calibration by multiple regression is a way of using the information of the peak areas of two mass fragments.

SCCP congeners with smaller chlorine contents contribute to smaller mass fragments, e.g. *m/z* 327. In addition, SCCP congeners with higher chlorine contents contribute to higher mass fragments, e.g. *m/z* 423. A description of the sum of SCCPs is made possible by weighing the sum of the two selected mass fragments. See Reference [3] for the selection procedure and the validation experiments.

A typical regression area by the selected mass fragments *m/z* 327 and *m/z* 423 is shown in Figure B.3. The calibration solutions described in Table 2 as well as the quality assurance solutions of Tables 3 and A.1 are demonstrated as points in the area. Some of the data points located in the calibration area are labelled and attributed to the calibration solutions and the quality assurance solutions used.

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

where

$\rho_{\Sigma\text{SCCPs}}$ is the concentration of the sum of SCCPs, in micrograms per litre;

b_1, b_2 are the regression coefficients;

A_1, A_2 are the peak areas of the analyte, e.g. *m/z* 327, *m/z* 423;

A_{IS} is the peak area of the internal standard, e.g. *m/z* 364.

With the help of, for example, Excel[3)]the calculation with the LINEST function[4)] is easily possible and an example of a working sheet is given in:

http://standards.iso.org/iso/12010/

The recovery presentation belonging to the same data is shown in Figure E.1.

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

In ECNI-MS 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 $[\text{M-Cl}]^-$, $[\text{M-HCl}]^-$ and $[\text{M+Cl}]^-$. 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 $[\text{M-Cl}]^-$, *m/z* 344 $[\text{M-HCl}]^-$ and *m/z* 415 $[\text{M+Cl}]^-$ along with smaller amounts of *m/z* 309 $[\text{M-Cl-HCl}]^-$ or $[\text{M-HCl-Cl}]^-$, *m/z* 308 $[\text{M-HCl-HCl}]^-$, and *m/z* 272 $[\text{M-HCl-HCl-HCl}]^-$.

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

Some elemental compositions of the selected mass ions are:

For the mass ion 327:

M+Cl $[C_{11}H_{20}{}^{35}Cl_5]^-$, M-Cl $[C_{11}H_{18}{}^{35}Cl_4{}^{37}Cl]^-$

M-Cl-HCl $[C_{11}H_{16}{}^{35}Cl_3{}^{37}Cl_2]^-$, M-HCl $[{}^{12}C_{10}{}^{13}C_{12}H_{17}{}^{35}Cl_4{}^{37}Cl]^-$

M+Cl $[C_{13}H_{25}{}^{35}Cl_1{}^{37}Cl_3]^-$,

For the mass ion 423:

M+Cl $[C_{13}H_{22}{}^{35}Cl_7]^-$, M+Cl $[C_{10}H_{15}{}^{35}Cl_4{}^{37}Cl_4]^-$

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.

4) RGP in the German version of Excel.

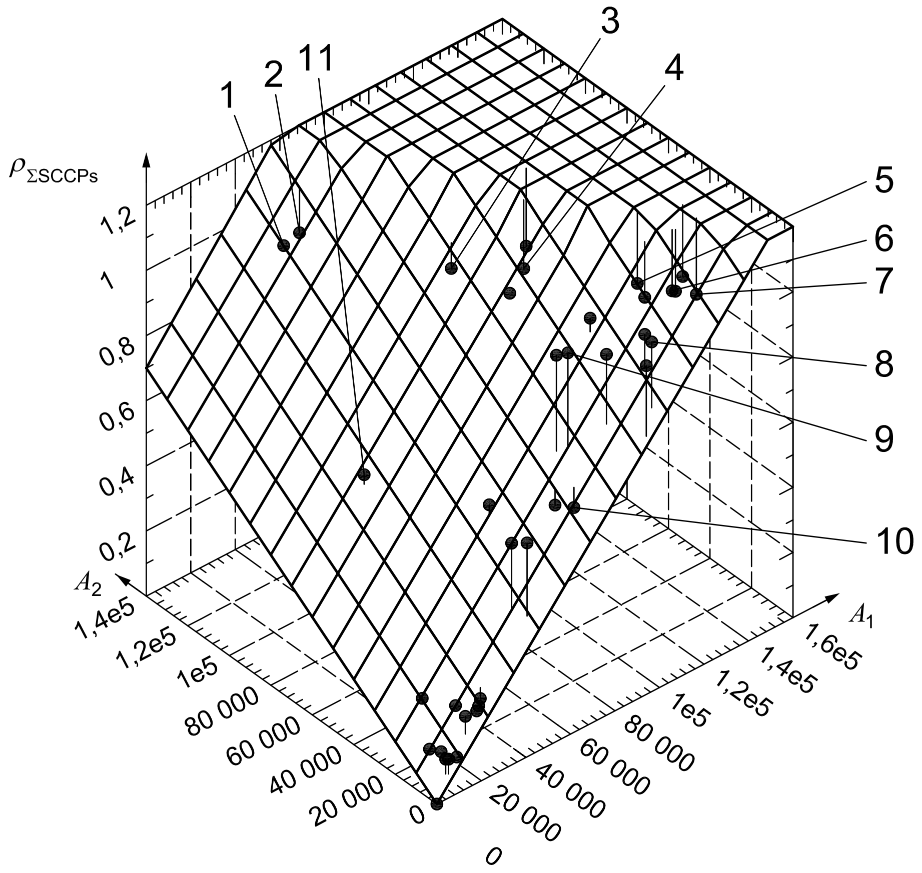
M-Cl $[C_{10}H_{13}{}^{35}Cl_3{}^{37}Cl_5]^-$, M-Cl $[C_{13}H_{20}{}^{35}Cl_6{}^{37}Cl]^-$

M-Cl-HCl $[C_{10}H_{11}{}^{35}Cl_2{}^{37}Cl_6]^-$, M-Cl-HCl $[C_{13}H_{18}{}^{35}Cl_5{}^{37}Cl_2]^-$

M-HCl $[{}^{12}C_{12}{}^{13}C_1H_{19}{}^{35}Cl_6{}^{37}Cl]^-$, M-HCl $[{}^{12}C_9{}^{13}C_1H_{12}{}^{35}Cl_3{}^{37}Cl_5]^-$

Why these particular mass ions are the most suitable for quantification is difficult to explain by mass spectrometry. 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 G.1 and G.2), it can be 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) belong to a variety of single compounds.

The selection for quantification was carried out using a specific empirical approach (see Reference [3]).



Key

1	Cereclor 70 -s2, 1 µg/ml	8	perch -s2, 1 µg/ml
2	Cereclor 70 -s1, 1 µg/ml	9	Hordalub 17 -s2, 1 µg/ml
3	Sediment -s2, 1 µg/ml	10	Hordalub 500 -s1, 0,6 µg/ml
4	Cereclor 60 -s2, 1 µg/ml	11	Cereclor 70 -s1, 0,6 µg/ml
5	Hordalub 80 -s2, 1 µg/ml	A_1	relative peak area *m*/*z* 327
6	Hordalub 500 -s1, 1 µg/ml	A_2	relative peak area *m*/*z* 423
7	zebra mussels -s2, 1 µg/ml	$\rho_{\Sigma SCCPs}$	sum of SCCPs, µg/ml

Figure B.3 — Regression area of the calibration solutions of Table 2 and quality assurance solutions of Tables 3 and A.1

Annex C
(informative)

Typical GC-MS conditions

C.1 Example 1

Injection:	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 → 280°C, 2 min → 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:	HB5 MS	
Temperature programme:	80 °C, 2 min → 40 °C/min to 300 °C → 300°C, 3 min → 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 → 325 °C, 9 min	

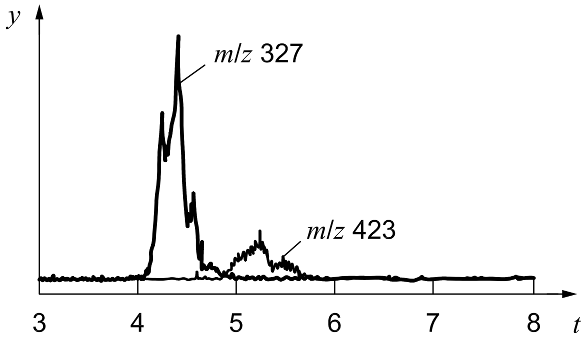
C.4 Example 4

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

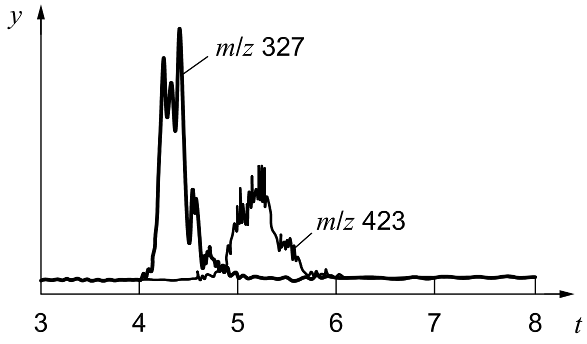
	inner diameter:	0,25 mm
Column material:	slightly polar, e.g. 5 % diphenyl- / 95 % dimethylpolysiloxane (DB5-MS)	
Temperature programme:	60 °C, 2 min → 45 °C/min to 340 °C, 10 min	

Annex D
(informative)

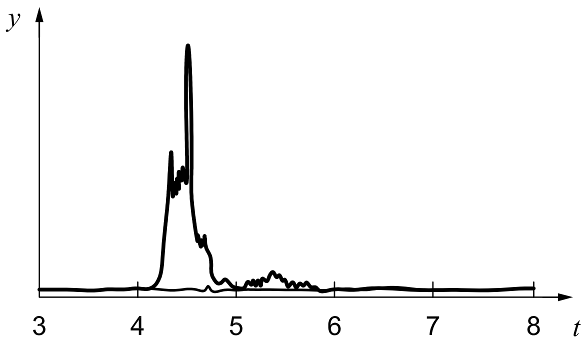
Typical chromatograms of standard solutions 1 μg/ml



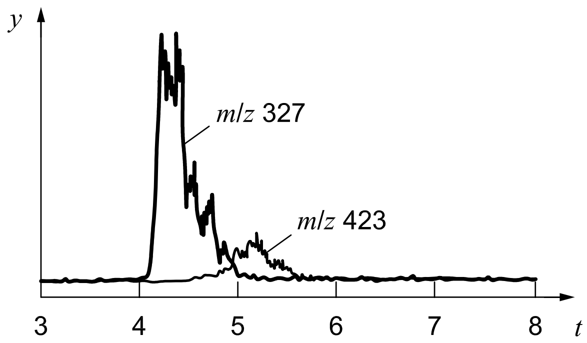
a) Hordalub 17 -s1



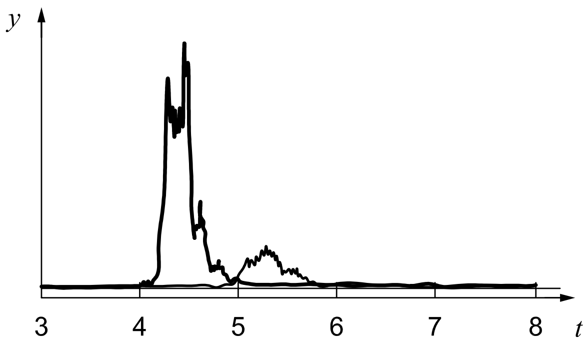
b) Cereclor 60 -s1



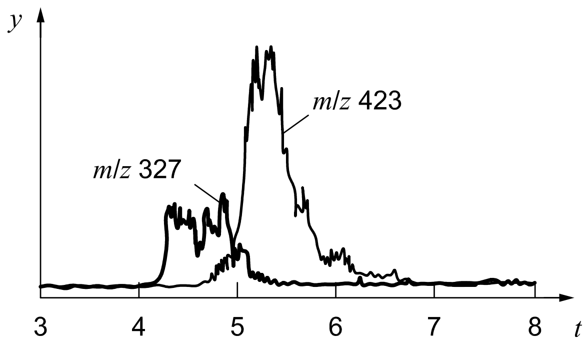
c) SCCP 51,5 % -s1



d) Hordalub 500 -s1



e) Hordalub 80 -s1



f) Cereclor 70 -s1

Key

y response

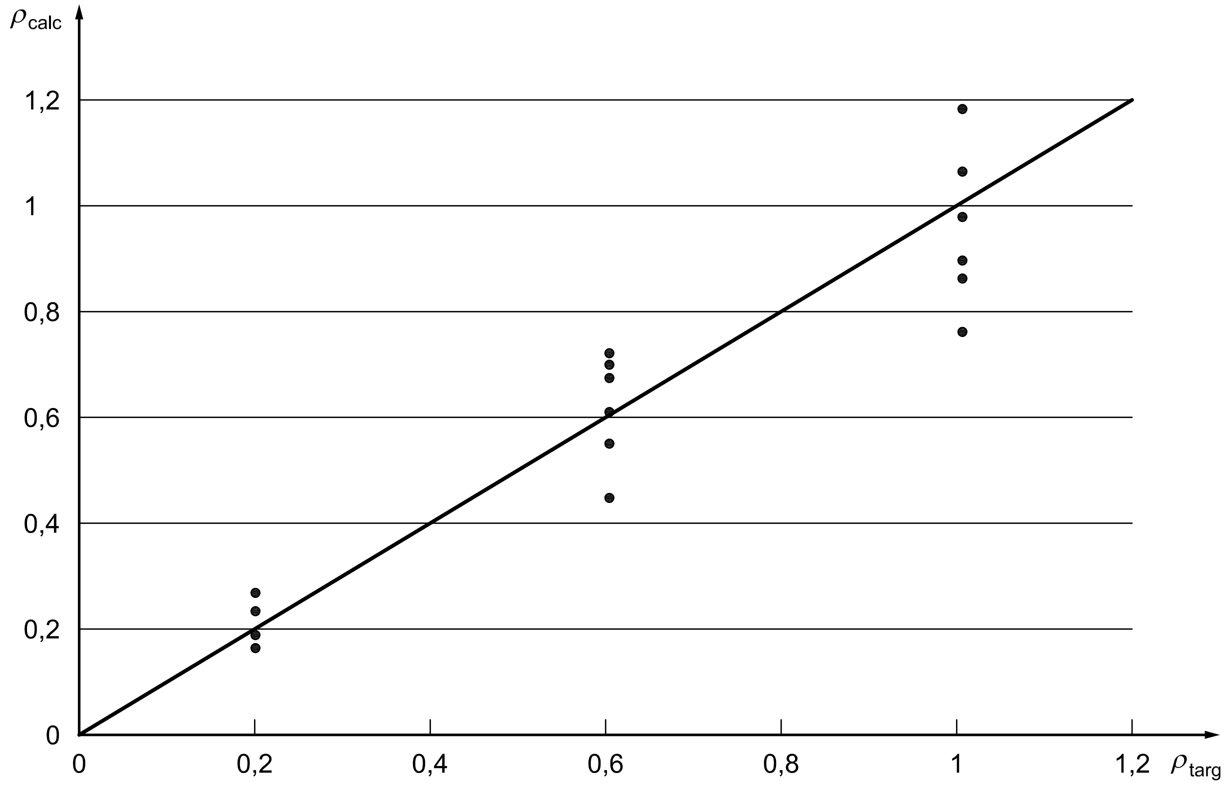
t time

Figure D.1 — Typical chromatograms of standard solutions 1 μg/ml

Annex E
(informative)

Presentation of goodness of fit

An example for a presentation of goodness of fit (calculated and target concentrations), with the calibration working solutions in 5.10, after basic calibration is given in Figure E.1. (In this example, one of the concentration levels was 0,2 µg/ml, not 0,15 µg/ml as described in Table 2.)



Key

ρ_{calc} calculated concentration, µg/ml

ρ_{targ} target concentration, µg/ml

Figure E.1 — Example for a recovery with the calibration working solution (5.10)

Annex F
(informative)

Example for recoveries of quality assurance solutions

Table F.1 — Experimental results with sum of SCCPs in solutions of Table 3

Values in micrograms per millilitre

m/*z* combination	SCCP 51,5 -s2	SCCP 55,5 -s2	SCCP 63 -s2	Hordalub 17 -s2	Hordalub 80 -s2	Hordalub 500 -s2	Cereclor 60 -s2
423 and 327	0,928	0,966	1,316	0,711	1,227	1,322	1,185
375 and 423	0,953	1,140	1,335	0,599	1,099	1,007	1,489
327 and 409	0,934	0,971	1,326	0,636	1,149	1,482	1,192

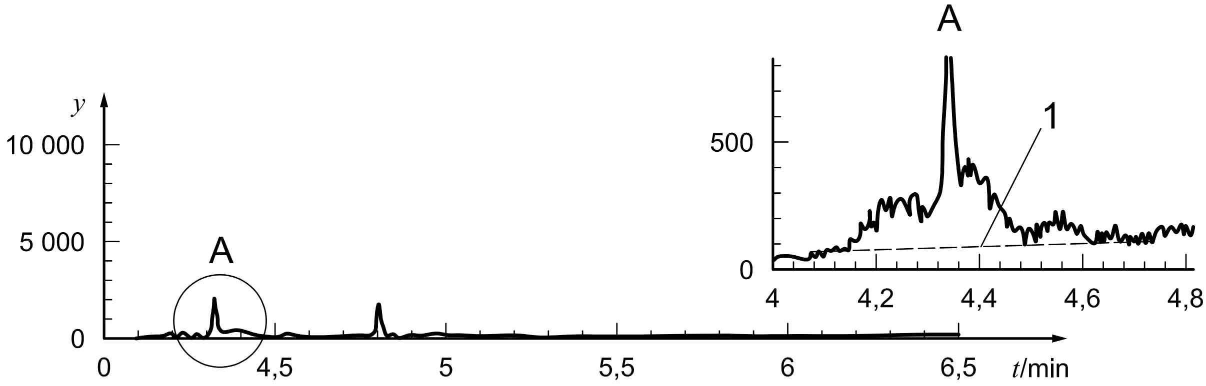
Table F.2 — Experimental results with sum of SCCPs in solutions of Table A.1

Values in micrograms per millilitre

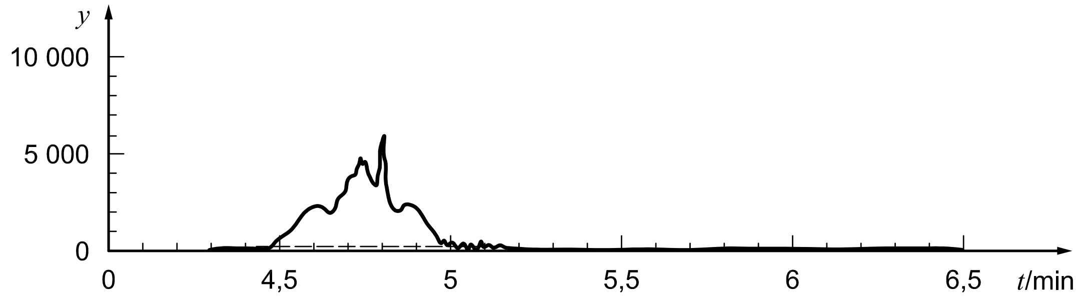
m/*z* combination	Sediment -s2	Perch -s2	Catfish -s2	Zebra mussels -s2	C_{10}/C_{11} mostly -s2
423 and 327	1,091	0,890	1,211	1,253	0,777
375 and 423	1,597	1,206	1,082	1,113	0,097
327 and 409	1,128	1,075	1,369	1,383	0,763

Annex G
(informative)

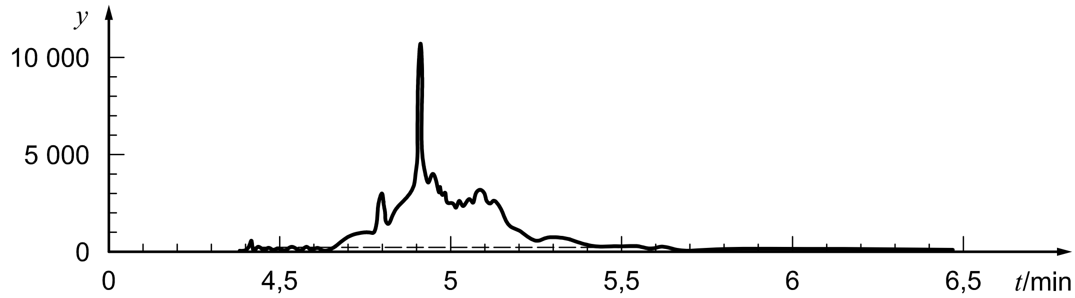
Chromatogram of a real water sample with a sum of SCCPs concentration of 2,59 µg/l



a) *m*/*z* **327**

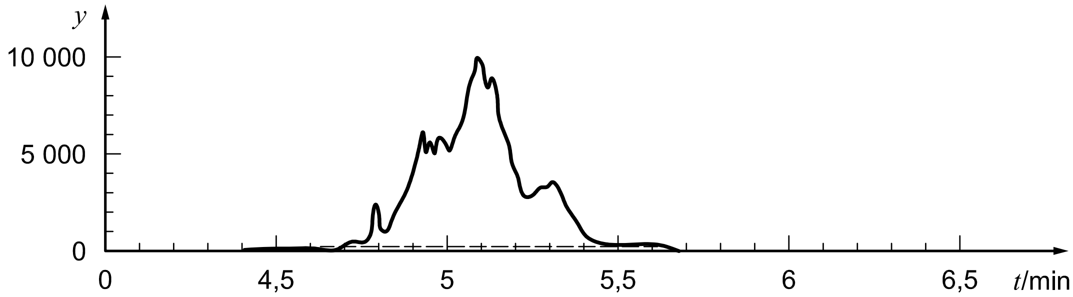


b) *m*/*z* **375**



c) *m*/*z* **409**

Figure G.1 (*continued*)



d) *m*/*z* **423**

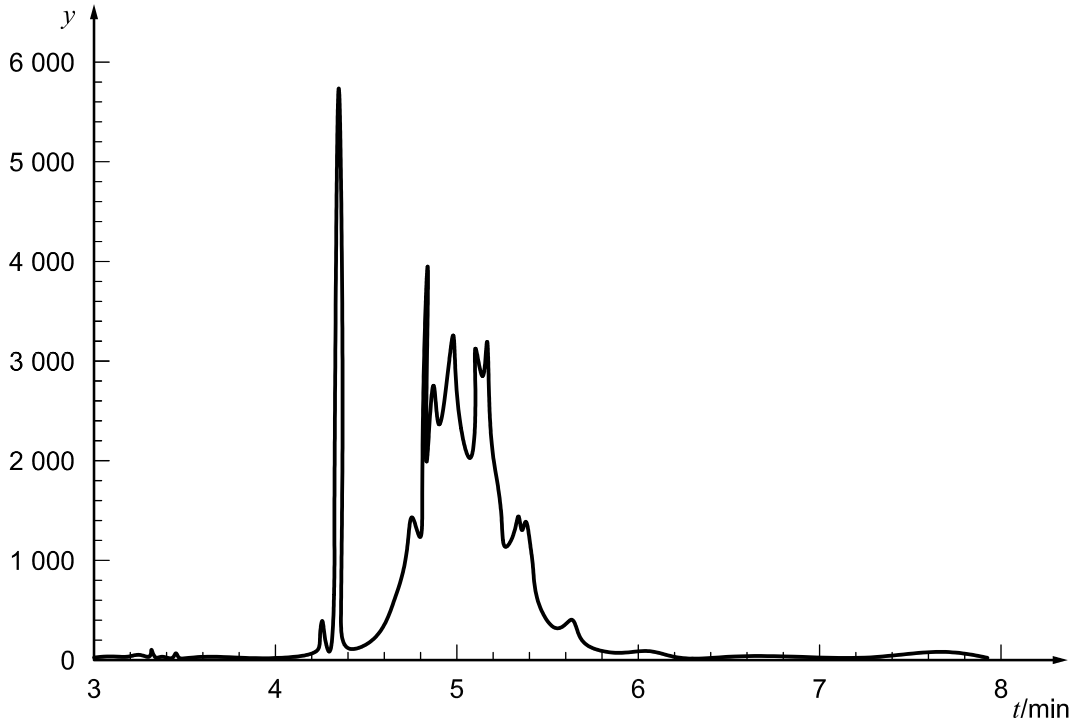
Key

y abundance

t time

1 integration baseline

Figure G.1 — Chromatogram of a real water sample with integration baselines



Key

y abundance

t time

Figure G.2 — Chromatogram of 1,1,1,3,10,11-hexachloroundecane (retention time, 4,18 min) as the internal standard in the real water sample in Figure G.1

Annex H
(informative)

Precision data

Sample	Matrix	Parameter	l	n	o %	$\bar{\bar{x}}$ µg/l	X µg/l	η %	s_R µg/l	$C_{V,R}$ %	s_r µg/l	$C_{V,r}$ %
A	surface water	SCCP	10	19	0	0,341	0,4	85,2	0,105 6	31,0	0,041 6	12,2
B	surface water	SCCP	12	24	0	0,786	0,6	130,9	0,268 4	34,2	0,136 7	17,4
C	waste water	SCCP	11	21	0	0,729	0,6	121,6	0,203 1	27,8	0,093 7	12,8
						µg/ml	µg/ml		µg/ml		µg/ml	
D	standard solution	SCCP	16	16	0	0,970	1,0	97,0	0,1150	11,9	—	—

l is the number of laboratories after outlier rejection

n is the number of individual test results after outlier rejection

o is the percentage of outliers

$\bar{\bar{x}}$ is the overall mean of results (without outliers)

X is the assigned value

η is the recovery rate of the analyte

s_R is the reproducibility standard deviation

$C_{V,R}$ is the coefficient of variation of reproducibility

s_r is the repeatability standard deviation

$C_{V,r}$ is the coefficient of variation of repeatability

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