BRITISH STANDARD

BS EN ISO 27108:2013

Water quality — Determination of selected plant treatment agents and biocide products — Method using solid-phase microextraction (SPME) followed by gas chromatography-mass spectrometry (GC-MS)

ICS 13.060.50

BSI British Standards

National foreword

This British Standard is the UK implementation of EN ISO 27108:2013. It is identical to ISO 27108:2010. It supersedes BS ISO 27108:2010 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/3, Water quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

The publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

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EUROPEAN STANDARD

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English Version

Water quality - Determination of selected plant treatment agents and biocide products - Method using solid-phase microextraction (SPME) followed by gas chromatography-mass spectrometry (GC-MS) (ISO 27108:2010)

Qualité de l'eau - Détermination d'agents de traitement et de produits d'usine sélectionnés - Méthode utilisant une micro-extraction en phase solide (MEPS) suivie d'une chromatographie en phase gazeuse-spectrométrie de masse (CG-SM) (ISO 27108:2010)

Wasserbeschaffenheit - Bestimmung ausgewählter Pflanzenschutzmittel und Biozidprodukte - Verfahren mittels Festphasenmikroextraktion (SPME) gefolgt von der Gaschromatographie und Massenspektrometrie (GC-MS) (ISO 27108:2010)

This European Standard was approved by CEN on 12 April 2013.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

The text of ISO 27108:2010 has been prepared by Technical Committee ISO/TC 147 “Water quality” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 27108:2013 by Technical Committee CEN/TC 230 “Water analysis” the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2014, and conflicting national standards shall be withdrawn at the latest by February 2014.

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Endorsement notice

The text of ISO 27108:2010 has been approved by CEN as EN ISO 27108:2013 without any modification.

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Introduction

In recent years, ground water contamination as well as surface water contamination by pesticides has become a matter of public concern. Identification and quantification of pesticides at trace level concentrations often require both high sensitive chromatographic equipment and effective enrichment steps. In the analysis of aqueous samples, sample preparation techniques including solid-phase extraction (SPE) are frequently the most time-consuming steps and in many cases can be effectively replaced by solid-phase microextraction (SPME).

When using this International Standard, it may be necessary in some cases to determine whether and to what extent particular problems could require the specification of additional marginal conditions.

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INTERNATIONAL STANDARD ISO 27108:2010

Water quality — Determination of selected plant treatment agents and biocide products — Method using solid-phase microextraction (SPME) followed by gas chromatography-mass spectrometry (GC-MS)

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 according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method for the determination of the dissolved amount of selected plant treatment agents and biocide products in drinking water, ground water and surface water by solid-phase microextraction (SPME) followed by gas chromatography-mass spectrometry (GC-MS). The limit of determination depends on the matrix, on the specific compound to be analysed and on the sensitivity of the mass spectrometer. For most plant treatment agents and biocides to which this International Standard applies, it is at least 0,05 µg/l. Validation data related to a concentration range between 0,05 µg/l and 0,3 µg/l have been demonstrated in an interlaboratory trial.

This method may be applicable to other compounds not explicitly covered by this International Standard or to other types of water. However, it is necessary to verify the applicability of this method for these special cases.

NOTE Determinations by this International Standard are performed on small sample amounts (e.g. sample volumes between 8 ml and 16 ml).

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 3696, *Water for analytical laboratory use — Specification and test methods*

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: Guidance on the preservation and handling of water samples*

3 Principle

Substances under investigation are extracted from the water sample by solid-phase microextraction (SPME) according to their equilibrium of distribution. The extraction is performed by a chemically modified fused-silica

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fibre, the surface of which is coated with a suitable adsorbent polymer. During extraction, the fibre is immersed in the liquid sample. After completion of the extraction procedure, the fibre is drawn back into the needle, removed from the sample vial, and introduced directly into the GC injector. The analytes are transferred to the GC column by thermal desorption.

The analytes are separated, identified and quantified by means of capillary gas chromatography with mass spectrometric detection (GC-MS) using electron impact (EI) ionisation mode.

Table 1 — Plant treatment agents and biocide products determined by this method

Name	Molecular formula	CAS registry No.	Molar mass	Reference No. in example chromatograms of Figure		
			g/mol	A.1	A.2	A.3
Dichlobenil	$C_7H_3Cl_2N$	1194-65-6	172,0	1	1	1
Desethylatrazine	$C_6H_{10}ClN_5$	6190-65-4	187,6	2	2	3
Desethylterbutylazine	$C_7H_{12}ClN_5$	30125-63-4	201,7	3	3	2
Simazine	$C_7H_{12}ClN_5$	122-34-9	201,7	4	4	7
Atrazine	$C_8H_{14}ClN_5$	1912-24-9	215,7	6	5	5
Lindane	$C_6H_6Cl_6$	58-89-9	290,8	7	6	8
Terbutylazine	$C_9H_{16}ClN_5$	5915-41-3	229,7	8	7	6
Metribuzine	$C_8H_{14}N_4OS$	21087-64-9	214,3	9	8	14
Parathion-methyl	$C_8H_{10}NO_5PS$	298-00-0	263,2	10	9	11
Heptachlor	$C_{10}H_5Cl_7$	76-44-8	373,3	11	10	9
Terbutryn	$C_{10}H_{19}N_5S$	886-50-0	241,4	12	11	12
Aldrin	$C_{12}H_8Cl_6$	309-00-2	364,9	13	12	10
Metolachlor	$C_{15}H_{22}ClNO_2$	51218-45-2	283,8	14	13	13
Parathion-ethyl	$C_{10}H_{14}NO_5PS$	56-38-2	291,3	15	14	15
exo-Heptachlorepoxide	$C_{10}H_5Cl_7O$	1024-57-3	389,3	16	16	16
Pendimethalin	$C_{13}H_{19}N_3O_4$	40487-42-1	281,3	17	15	17
endo-Heptachlorepoxide	$C_{10}H_5Cl_7O$	28044-83-9	389,3	18	17	18
Triclosan	$C_{12}H_7Cl_3O_2$	3380-34-5	289,5	19	18	19
Dieldrin	$C_{12}H_8Cl_6O$	60-57-1	380,9	20	19	20
Carfentrazone-ethyl	$C_{15}H_{14}Cl_2F_3N_3O_3$	128639-02-1	412,2	21	20	21
Diflufenican	$C_{19}H_{11}F_5N_2O_2$	83164-33-4	394,3	22	21	22
Mefenpyr-diethyl	$C_{16}H_{18}Cl_2N_2O_4$	135590-91-9	373,2	23	22	23

4 Interferences

4.1 Interferences during sampling

To avoid interference, collect samples as specified in Clause 7, observing the instructions specified in ISO 5667-1 and ISO 5667-3.

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4.2 Interferences during extraction procedure

Commercially available SPME fibres differ frequently in quality. Variations in the selectivity of the materials also frequently occur from batch to batch, thus possibly causing significant deviations in extraction yield. This does not basically impair their suitability, apart from a resulting higher detection limit of individual substances.

Inadequately conditioned fibres frequently result in lower extraction yields and poorly reproducible results; therefore precondition new fibres according to Clause 8. Also condition used fibres by performing the whole SPME process using at least two sampling vials containing only water (5.1) prior to starting with the first sample of a new sample sequence.

Sensitivity of fibres gradually decreases throughout a sequence of samples. Therefore regular measurements of the reference solution within the sample sequence (see 9.1) are recommended. The fibre is still usable if the method shows required sensitivity for substances under investigation.

Adding sodium chloride to the sample results in a clear improvement of the extraction yield for most substances listed in Table 1. The addition of common salt (near saturation) is therefore recommended. Some substances listed in Table 1 show a reverse effect, which in most cases is weaker. Salt additions of < 20 % of the saturation concentration (e.g. about 0,5 g of NaCl in an 8 ml water sample) cause a deterioration in reproducibility. It is important to keep to exactly the same salt additions for all samples of a calibration sequence and/or sample sequence.

Salt deposits may accumulate in the metal syringe needle of the fibre holder after extended use. Salt deposits always occur when the syringe needle of the fibre holder is immersed in the water sample during extraction. This may damage the fibres and the injector liner. Therefore adjust the immersion depth precisely, and, if necessary, rinse out the SPME syringe needle to dissolve any encrusted salt.

To ensure that the measurements are of high accuracy and precision, keep extraction time constant (e.g. 60 min) within a sample sequence for all samples. It is highly preferable to use an automatic sampler with an SPME option.

For automatic operation, preferably use sampling vials with a thin septum (e.g. 0,9 mm to 1,3 mm thickness) in order to avoid any mechanical problems when piercing the septum of the sample vial with the metal syringe needle.

NOTE This is of particular importance when using automatic sampler systems that move sample vials in a circle, because otherwise damage to the piercing metal syringe needle (including the exposed fibre) can occur during extraction.

Extraction of some of the substances listed in Table 1 using the procedure according to Clause 8 depends on the temperature. As a rule, somewhat higher extraction yields are obtained at lower temperatures. Maintain extraction temperature constant (e.g. 30 °C) within a sample sequence for all samples in order to obtain reproducible extraction yields.

4.3 Interferences during gas chromatography and mass spectrometry procedure

Interferences may be caused, e.g. by the injection system used or by inadequate separation of the analytes. Experienced operators, using the information given in the instrument manuals, may be able to minimise this type of interference. Regular checking of the chromatographic and spectrometric system is required to maintain adequate performance. Required system stability should be checked regularly by the use of a GC standard.

Ascertain the necessary penetration depth for the fibres for thermal desorption in the GC injector. The penetration depth corresponds to the hottest point of the injector and shall be kept constant during a measuring sequence.

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5 Reagents

The reagents shall be free from impurities possibly interfering with the GC-MS analysis.

Use solvents and reagents of sufficient purity, i.e. with negligibly low impurities compared with the concentration of analytes to be determined. As reagents, use, as far as available, “residual grade” or better in order to obtain low blanks. Verify by blank determinations and, if necessary, apply additional cleaning steps.

5.1 Water, complying with the requirements of ISO 3696, grade 1 or equivalent.

5.2 Operating gases for the gas chromatograph-mass spectrometer, of high purity and in accordance with manufacturer's specifications.

5.3 Sodium chloride, NaCl.

5.4 Solvents, e.g. ethyl acetate, $C_4H_8O_2$; acetone (propanone), C_3H_6O; acetonitrile, CH_3CN.

For the preparation of stock solutions of individual reference substances (5.9.2) use the appropriate solvent. However, it is recommended to prepare multi-component stock solutions (5.9.3) using either acetone or ethyl acetate.

5.5 Sodium hydroxide solution, $w(NaOH) = 25$ % mass fraction.

5.6 Hydrochloric acid, $w(HCl) = 25$ % mass fraction or **sulfuric acid**, $w(H_2SO_4) = 12{,}5$ % mass fraction.

5.7 Sodium thiosulfate pentahydrate, $Na_2S_2O_3 \cdot 5\ H_2O$.

5.8 Internal standard, e.g. atrazine-d_5, lindane-d_6 or parathion-ethyl-d_{10}.

As internal standard, choose a substance with similar physicochemical properties (extraction behaviour, retention time) as the substance to be determined. The internal standard should not be present in the sample to be analysed. The choice of a substance may be difficult and it depends on the problem to be resolved; in any case, the suitability should be checked. It is highly recommended to use a deuterium-labelled or ^{13}C-enriched substance listed in Table 1 as an internal standard. It may be advantageous to use more than one internal standard.

Prepare stock solutions of individual internal standard substances in the same way as specified for individual reference substances (5.9.2).

5.9 Reference substances

5.9.1 General

Reference substances (listed in Table 1) of defined concentration suitable for both the preparation of stock solutions and the preparation of spiked aqueous multi-component reference solutions used for calibration of the total procedure (9.2).

5.9.2 Stock solutions of individual reference substances

As an example, place 50 mg of a reference substance into a 100 ml one-mark volumetric flask (6.6), dissolve in an appropriate solvent (5.4) and make up to the mark with the same solvent.

Store stock solutions at temperatures between 1 °C and 5 °C according to ISO 5667-3, protected from light. They are stable for at least 12 months.

NOTE Deep freezing of stock solutions is also possible and commonly applied.

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5.9.3 Multi-component stock solutions of reference substances

As an example, transfer 1 ml of each of the solutions of the individual substances (5.9.2) and the internal standard substances (5.8) into a 100 ml one-mark volumetric flask (6.6) and make up to the mark with ethyl acetate or acetone (5.4).

Store multi-component stock solutions at temperatures between 1 °C and 5 °C, protected from light. They are stable for at least 6 months.

5.9.4 Aqueous multi-component reference solutions used for calibration of the total procedure

Prepare the aqueous reference solution for calibration of the total procedure as follows.

Measure 100 ml of water, e.g. in a one-mark volumetric flask (6.6) and add a magnetic stir bar.

Place the flask on a magnetic stirrer and switch on.

Using a microlitre syringe, measure 10 µl of the multi-component stock solution (5.9.3) and dispense it below the surface of the stirred water. Continue to stir for about 5 min with the volumetric flask covered.

Adjust the agitation speed so that no turbulence funnel is formed.

Prepare reference solutions of higher and lower concentrations in the same way using correspondingly prepared multi-component stock solutions (5.9.3). All aqueous reference solutions suitable for multipoint calibration should contain equal amounts of internal standard.

Do not dilute the spiked aqueous solutions.

Always keep the spike volume constant.

NOTE A small spiking volume (e.g. 10 µl in 100 ml water) is recommended to avoid any interference of the solvent within the fibre adsorption process of the analytes under investigation.

Store reference solutions at temperatures between 1 °C and 5 °C, protected from light. They may not be stable for more than a few days and therefore shall be prepared each working day.

6 Apparatus

Equipment or parts of it which are likely to come into contact with the water sample or its extract shall be free from residues causing interferences. The use of vessels made of glass, stainless steel or polytetrafluoroethylene (PTFE) is recommended.

Usual laboratory equipment and in particular the following.

6.1 Sample flasks, e.g. brown glass, flat bottomed, with glass- or PTFE-coated stoppers, e.g. 100 ml or 250 ml.

6.2 Glass sample bottles (head space vials), with caps (6.3), e.g. 10 ml or 20 ml.

6.3 Crimp caps, with PTFE-coated septa (e.g. magnetic caps with butyl/PTFE septa, 0,9 mm to 1,3 mm).

NOTE Commercially available head space vials usually have a flanged rim suitable for a 3 mm septum. A thinner septum (e.g. 0,9 mm to 1,1 mm) requires suitable vials with a thicker flanged rim. Alternatively, a perforated spacer ring (e.g. made of natural rubber or butyl, 1,3 mm thick) can be placed between septum and crimp cap.

6.4 Crimper and decapper (e.g. manual crimper and manual decapper, 20 mm).

6.5 Graduated measuring cylinders, capacity, e.g. 100 ml or 250 ml, ISO 4788[3] class A.

6.6 One-mark volumetric flasks, capacity, e.g. 10 ml, 25 ml, 50 ml and 100 ml, ISO 1042[2] class A.

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6.7 Single volume pipettes, capacities between 1 ml and 50 ml, ISO 648[1] class A.

6.8 Microlitre syringes, e.g. capacities between 5 µl and 50 µl.

6.9 Magnetic stirrer, including PTFE-coated magnetic stir bar of suitable size.

6.10 Capillary gas chromatograph with mass spectrometric detector (GC-MS) using EI ionisation mode, gas supply in accordance with the respective manufacturer's instructions.

6.11 Non-discriminating GC injector, e.g. splitless mode of a split or splitless injection system or programmable temperature vaporiser (PTV).

6.12 Automatic sampler with SPME option, including SPME syringe and the necessary software.

6.13 SPME fibres, e.g. 10 mm medium polar polyacrylate-phases (PA coating: e.g. 85 µm) or bipolar polydimethylsiloxane/divinylbenzene phases (PDMS/DVB coating: e.g. 65 µm). Other fibres as mentioned above may be applicable as well. However, it is necessary to verify their sensitivity for the substances under investigation (see 9.1).

NOTE Polyacrylate phases (PA 85) have proved to be most sensitive for the substances listed in Table 1.

Preferably use 23-gauge needles (particularly in combination with a septumless GC inlet system). If using a septum type injection system, 24-gauge needles should be used to reduce septum coring.

6.14 Capillary columns, for gas chromatography (examples of chromatographs appear in Annex A). It is advantageous to use non-polar columns (e.g. low-bleed 5 %-phenylsiloxane column).

6.15 Borosilicate glass fibre filter, fibre diameter of 0,75 µm to 1,5 µm, with inorganic binding material.

6.16 Centrifuge, e.g. capable of reaching 2 000 r/min with appropriate centrifuge tubes.

6.17 pH meter, with electrodes.

7 Sampling and sample pretreatment

Collect samples as specified in ISO 5667-1 and ISO 5667-3.

For sampling, use thoroughly cleaned, flat bottomed glass flasks (6.1). Rinse flasks and caps with the water to be sampled.

Fill the bottles completely with the water to be examined.

Dechlorinate water samples containing chlorine by immediately adding sodium thiosulfate pentahydrate (5.7), resulting in a concentration of approximately 100 mg/l.

Treat and analyse the samples as soon as possible after sample collection as specified in ISO 5667-3. Store the samples at temperatures between 1 °C and 5 °C, protected from light.

8 Procedure

8.1 Sample preparation and extraction

Remove any suspended matter by, for example, filtration of the sample through a glass fibre filter (6.15) or centrifugation (6.16) to remove suspended matter.

NOTE Filtration or centrifugation of drinking water samples is not mandatory.

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The pH value of the water sample only requires adjustment if it is below 6 ± 0,2 or above 8 ± 0,2. In this case, adjust to pH 7 ± 0,2 with hydrochloric acid (5.6), sulfuric acid (5.6) or sodium hydroxide solution (5.5).

Add the internal standard (5.8) dissolved in ethyl acetate or acetone (5.4) (e.g. by adding a 10 µl aliquot to a 100 ml sample volume as described in 5.9.4).

For example, measure exactly 8 ml (or 16 ml) of the water sample being examined and pour into a 10 ml (or 20 ml) head space vial (6.2). The measured volume shall be equal for both calibration and sample measurement.

Choose the sample volume so that there is a distance of 15 mm to 25 mm between the level of the liquid and the upper edge of the vial.

CAUTION — This is particularly important in automatic systems which rotate the sample vials, causing rotation of the metal syringe needle (including the exposed fibre) during extraction to avoid any mechanical problems (broken fibre or broken needle). Take note of the information given in 4.2, paragraph 6.

Add sodium chloride (5.3) near saturation point, i.e. 0,3 g per millilitre of sample volume (e.g. 2,4 g NaCl in 8 ml water) and dissolve. Avoid concentrations of NaCl higher than 0,35 g/ml. Close with a septum fitted crimp cap (6.3) using a crimper (6.4). Place the vials in sampling sequence on the automatic sampler with SPME option (6.13).

Use SPME fibres (6.13).

Condition new fibres by heating them up in the “bake-out” station of the SPME automatic sampler or in the injector of the GC. Refer to the manufacturer's instructions for appropriate fibre bake-out times. Proceed with at least two sampling vials containing only water (5.1) within a new sample sequence prior to starting with the first sample. Recalibration is needed when a new fibre is installed.

Adjust the extraction temperature to approximately 30 °C (see 4.2). Maintain extraction temperature constant within a sample sequence for all samples in order to obtain reproducible extraction yields.

Set the agitation speed to a maximum reproducible value (e.g. 250 min^{-1}). In systems using a magnetic stirrer, pierce the septum approx. 3 mm from the middle.

Extraction time should be set to 60 min.

NOTE An extraction time of 60 min produces acceptable sensitivity for all substances listed in Table 1. However, depending on sensitivity requirements, shorter extraction times are possible.

Desorb in the injector for 10 min at 280 °C.

8.2 Gas chromatograph

Check the required system stability regularly. Adjust and optimise instrument parameter settings in accordance with the respective manufacturer's instructions.

For separation, use appropriate capillary columns (6.14) and adjust chromatographic conditions for maximum selectivity (see Annex A for examples).

8.3 Identification of individual compounds by means of GC-MS

Identify the sample component by matching both retention times and relative intensities of the diagnostic ions (Table 2) of sample components and reference substances (5.9).

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The target compound is present (is identified) in the sample if:

a) the relative or the absolute sample component retention time measured in the selected ion current chromatogram matches the relative or absolute retention time of the authentic compound within ± 0,2 % in the chromatogram of the latest calibration standard solution (e.g. aqueous multi-component reference solution; see 5.9.4), measured under identical conditions;

b) three selected diagnostic ions (see Table 2) are present at the substance-specific retention time;

c) relative intensities of all selected diagnostic ions measured in the sample do not deviate by more than ± (0,1I + 10) % from the relative intensities determined in the calibration standard solution, where I is the relative intensity of the diagnostic ion of the individual reference substance.

EXAMPLE

Three selected diagnostic ions have the following relative intensities: 100 %, 50 % and 15 %.

The maximum allowed deviation in the sample is:

a) ± (0,1 I x 100 + 10) % = ± 20 %; relative intensity in the sample shall lie within 80 % and 120 %;

b) ± (0,1 I x 50 + 10) % = ± 15 %; relative intensity in the sample shall lie between 35 % and 65 %;

c) ± (0,1 I x 15 + 10) % = ± 11,5 %; relative intensity in the sample shall lie between 3,5 % and 26,5 %.

NOTE 1 No ion of significant intensity should be present in the mass spectrum after background subtraction with a larger mass than the highest possible mass for a compound to be identified.

NOTE 2 Further guidance on identification is given in ISO 22892[6].

8.4 Blank value measurements

Perform blank determinations using water (5.1) in accordance with 8.1 during a series of analyses, at least once per batch. Blank measurements shall include all steps in the analytical procedure. If blank values are unusually high (over 50 % of the lowest reporting level), every step in the procedure shall be checked in order to find the reason for these high blanks. Ensure that blanks are reduced as much as possible by various procedures, such as elimination of the contamination of the sample by ambient air and solvents, and checking of the analytical instrumentation. If sample concentrations are close to the limit of detection, however, blank values of higher than 50 % of the lowest reported value can be tolerated.

9 Calibration

9.1 General requirements

Correct calibration requires both knowledge of the retention times of the compounds to be analysed (see Table 1) and sufficient recovery from the fibre in use (see 4.2). These shall be determined with spiked aqueous solutions of reference substances under the specified chromatographic conditions. The calibration function for a substance is valid only for the measured concentration range. Additionally, the calibration function depends on the condition of the gas chromatograph, the type and age of fibre actually used and shall be checked regularly.

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Table 2 — Selected ions for identification and quantification in mass spectrometric detection
(informative)

Compound (see Table 1)	Selected ions for identification and quantification [a] *m/z*
Dichlobenil	100, 136, 171, 173
Desethylatrazine	145, 172, 174, 187
Desethylterbutylazine	186, 188, 201
Simazine	173, 186, 201
Atrazine	173, 200, 215
Lindane	109, 181, 183, 219
Terbutylazine	173, 214, 229
Metribuzine	103, 144, 198, 214
Parathion-methyl	109, 125, 263
Heptachlor	237, 272, 274, 337
Terbutryn	170, 185, 226, 241
Aldrin	261, 263, 265, 293
Metolachlor	162, 238, 240
Parathion-ethyl	109, 155, 261, 291
exo-Heptachlorepoxide	353, 355, 357
Pendimethalin	162, 191, 252, 281
endo-Heptachlorepoxide	183, 253, 289
Triclosan	218, 288, 290
Dieldrin	79, 263, 277, 279
Carfentrazone-ethyl	290, 312, 340, 411
Diflufenican	246, 266, 394
Mefenpyr-diethyl	227, 253, 255, 299

[a] Other options may be possible.

Calibrate the total procedure using aqueous reference solutions (preferably multi-component reference solutions) (5.9.4). Ensure that a linear dependence of signal to concentration for each substance under investigation is achieved. Determine the linear working range using at least five measuring points of different concentrations distributed as evenly as possible over the calibration range. Adjust the calibration range to real requirements (e.g. the blank should be less than 50 % of the lower reporting level).

For routine analysis, a recalibration by measurement of two concentration levels is sufficient. Recalibrate regularly within a sample sequence (at least after 10 to 12 samples) using reference solutions.

Table 3 gives an explanation of the subscripts used in the equations and in the following text.

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Table 3 — Definition of subscripts

Subscript	Meaning
e	Calibration step
g	Total procedure
IS	Internal standard
i	Identity of the substance
j	Consecutive figure for pairs of values

9.2 Calibration with internal standard covering the total procedure

The use of an internal standard procedure helps to minimise unavoidable minor errors that may occur throughout the SPME total procedure.

Determination becomes, to a certain degree, independent of matrix effects in the sample. Calibration using an internal standard is relatively independent of a potential decrease of performance of the fibre coating in use as long as recalibration of the linear regression function takes place at regular intervals within routine analysis, e.g. within a sample sequence after a set of 10 samples (see 9.1).

NOTE Deterioration of SPME fibre is an unavoidable process. Under normal operational conditions, sensitivity of fibres gradually decreases throughout a sequence of samples. Fibres can remain in use as long as recalibration shows the necessary sensitivity for all substances under investigation.

Add a known amount of the internal standard (5.8) to the water sample prior to analysis.

The mass concentration of the internal standard, ρ_{IS}, should be equal for both calibration and sample measurement. All aqueous reference solutions suitable for multipoint calibration should contain equal amounts of internal standard.

For calibration covering the total procedure, treat and analyse aqueous multi-component reference solutions (5.9.4) according to Clause 8 (e.g. in each case 8 ml).

Set up a calibration curve from the values $y_{i\mathrm{eg}j}/y_{\mathrm{ISeg}j}$ and $\rho_{i\mathrm{eg}j}/\rho_{\mathrm{ISeg}j}$ and establish a linear regression function using Equation (1):

$$\frac{y_{i\mathrm{eg}}}{y_{\mathrm{ISeg}}} = m_{i\mathrm{ISg}} \frac{\rho_{i\mathrm{eg}}}{\rho_{\mathrm{ISeg}}} + b_{i\mathrm{ISg}} \tag{1}$$

where

$y_{i\mathrm{eg}}$ is the measured response of the substance i during calibration, a variable dependent on $\rho_{i\mathrm{e}}$, whose unit depends on the evaluation, e.g. area unit;

y_{ISeg} is the measured response of the internal standard during calibration, whose unit depends on the evaluation, e.g. area unit;

NOTE All reference solutions contain equal amounts of internal standard.

$\rho_{i\mathrm{eg}}$ is an independent variable, the mass concentration, in micrograms per litre, of substance i in the spiked aqueous reference solution;

ρ_{ISeg} is an independent variable, the mass concentration, in micrograms per litre, of the internal standard;

$m_{i\mathrm{ISg}}$ is the slope of the calibration curve from $y_{i\mathrm{e}}/y_{\mathrm{ISe}}$ as a function of the mass concentration ratio $\rho_{i\mathrm{eg}}/\rho_{\mathrm{ISeg}}$, often called the response factor;

$b_{i\mathrm{ISg}}$ is the ordinate intercept of the calibration curve. The unit depends on the evaluation.

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10 Calculation

Calculate the mass concentration, $\rho_{i\mathrm{g}}$, in micrograms per litre, of substance i in the sample under analysis according to Equation (2).

$$\rho_{i\mathrm{g}} = \frac{(y_{i\mathrm{g}} / y_{\mathrm{ISg}}) - b_{i\mathrm{ISg}}}{m_{i\mathrm{ISg}}} \rho_{\mathrm{ISg}} \quad (2)$$

where

$y_{i\mathrm{g}}$ is the measured value, e.g. in area units, of substance i;

y_{ISg} is the measured response, e.g. in area units, of the internal standard;

ρ_{ISg} is the mass concentration, in micrograms per litre, of the internal standard;

$b_{i\mathrm{ISg}}$, $m_{i\mathrm{ISg}}$ see Equation (1).

11 Expression of results

The analytical results obtained using this International Standard are subject to an uncertainty which needs to be taken into account when interpreting the results. In this International Standard, water samples are spiked with various concentrations of selected plant treatment agents and biocide products for calculating the reproducibility as a percentage and expressed as reproducibility coefficient of variation, $C_{V,R}$. As can be seen from the values of Table C.1, $C_{V,R}$ is between 11,5 % and 35,6 %.

The mass concentration, in micrograms per litre, of the individual compounds listed in Table 1 shall be reported to two significant figures, except when the mass concentration is $< 0{,}1$ µg/l, in which case only one significant figure shall be reported.

EXAMPLES

Atrazine	12 µg/l	Carfentrazone-ethyl	0,23 µg/l
Triclosan	2,8 µg/l	Dichlobenil	0,07 µg/l

12 Test report

The test report shall contain at least the following information:

a) the test method used, together with a reference to this International Standard (ISO 27108:2010);

b) complete identification of the sample;

c) the results expressed in accordance with Clause 11;

d) any details not specified in this International Standard or regarded as optional, as well as any factor which may have affected the results.

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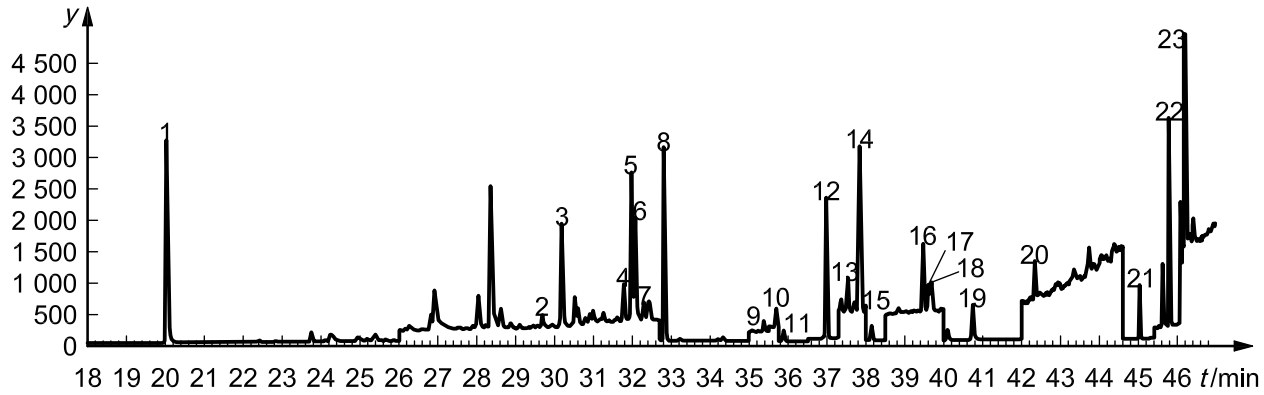
Annex A
(informative)

Examples of gas chromatograms for compounds listed in Table 1



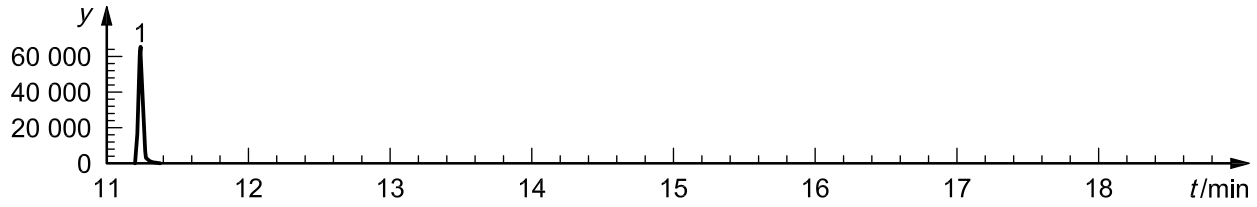
GC equipment:	Agilent 6890[1)], Gerstel MPS 2[1)] (fibre rotation), CTC Cycle-Composer software[1)]
SPME conditions:	according to Clause 8 with sample volume: 8 ml, amount NaCl: 2,4 g, fibre: polyacrylate 85 µm [Supelco PA 85[1)], 1 cm, 23 gauge], agitation speed: 250 min^{-1}, extraction time: 60 min
Injection	Gerstel KAS[1)], 60 °C; 10 °C/s to 280 °C; 10 min; splitless (6 min)
Capillary column:	ZB 5 (Phenomenex)[1)], 30 m × 0,25 mm × 0,25 µm
Range of concentration:	0,25 µg/l to 0,35 µg/l
Carrier gas:	helium (5,0); 0,9 ml/min
GC oven temperature:	60 °C, 2 min; 4 °C/min to 200 °C; 10 °C/min to 300 °C
MS detector:	Agilent 5973 MSD [1)], quadrupole MS, EI 70 eV, SIM, transfer line 280 °C

Key

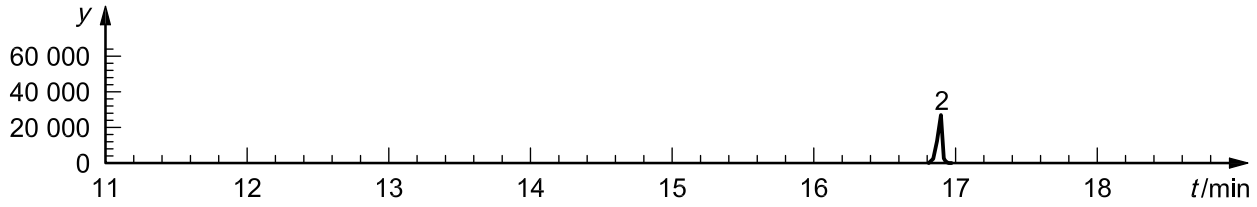
1 dichlobenil
2 desethylatrazine
3 desethylterbutylazine
4 simazine
5 atrazine-d_5
6 atrazine
7 lindane
8 terbutylazine
9 metribuzine
10 parathion-methyl
11 heptachlor
12 terbutryn
13 aldrin
14 metolachlor
15 parathion-ethyl
16 *exo*-heptachlorepoxide
17 pendimethalin
18 *endo*-heptachlorepoxide
19 triclosan
20 dieldrin
21 carfentrazone-ethyl
22 diflufenican
23 mefenpyr-diethyl
t time
y abundance

Figure A.1 — Gas chromatogram 1 of a standard solution (selected ion mode)

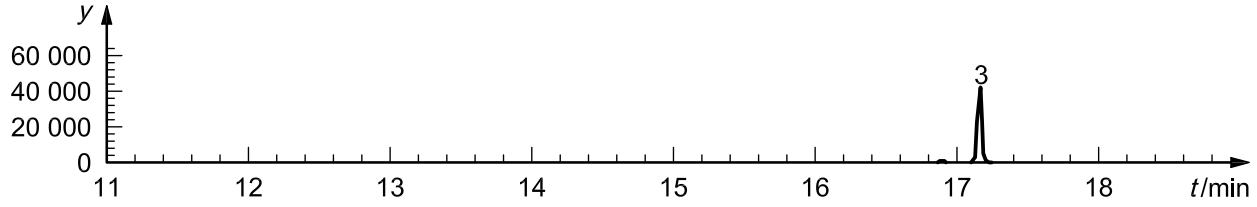
1) Product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.



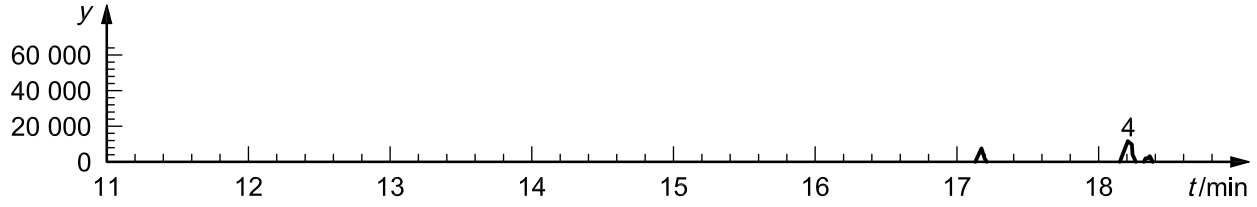
a) Ion 171



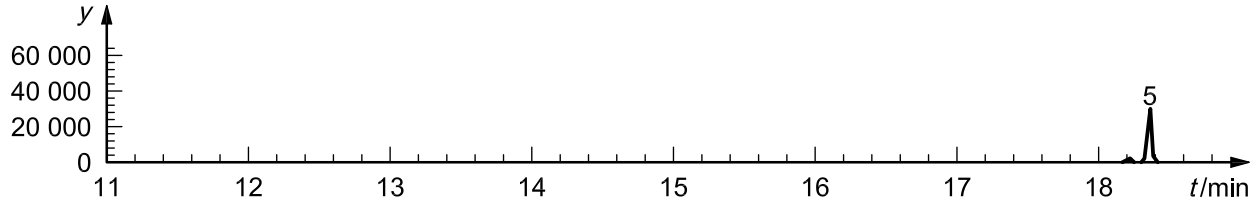
b) Ion 172



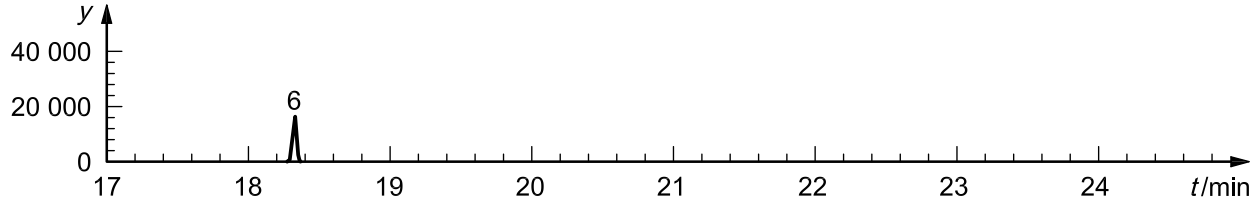
c) Ion 186



d) Ion 201



e) Ion 200



f) Ion 181

Figure A.2 (*continued*)

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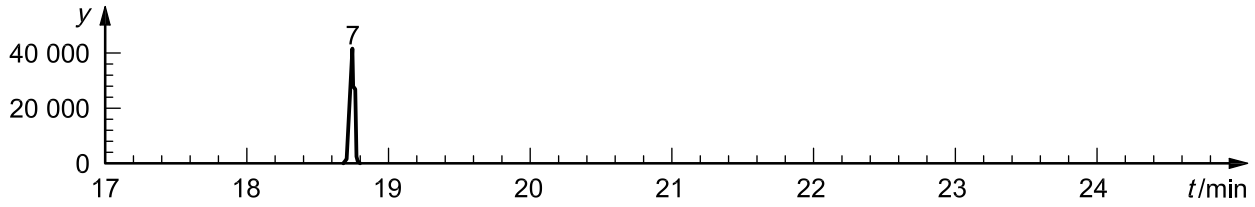
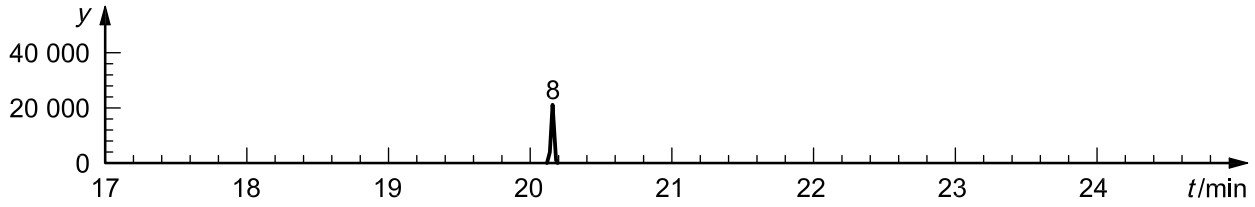
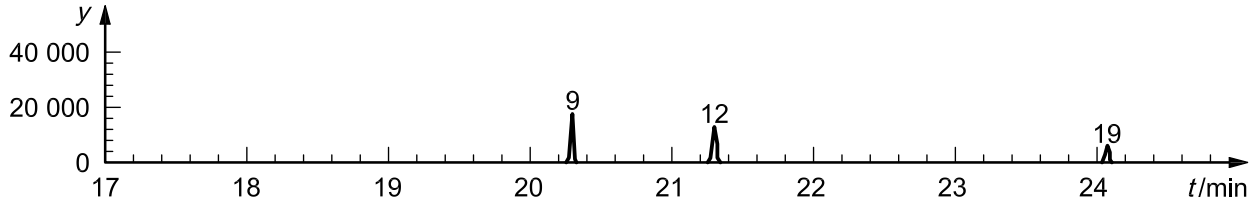


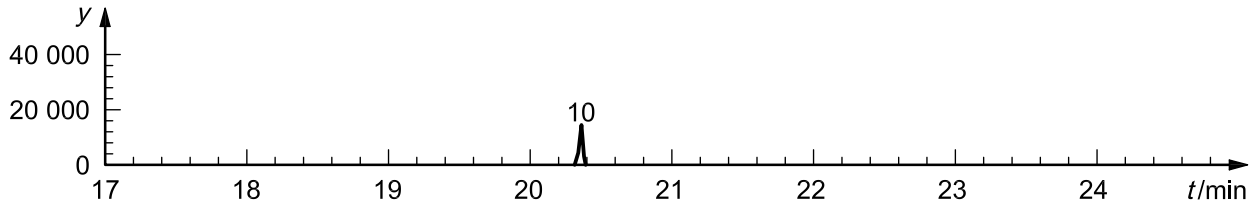
g) Ion 214



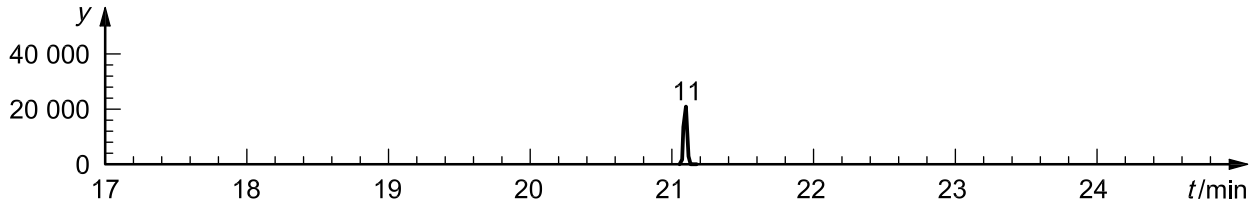
h) Ion 198



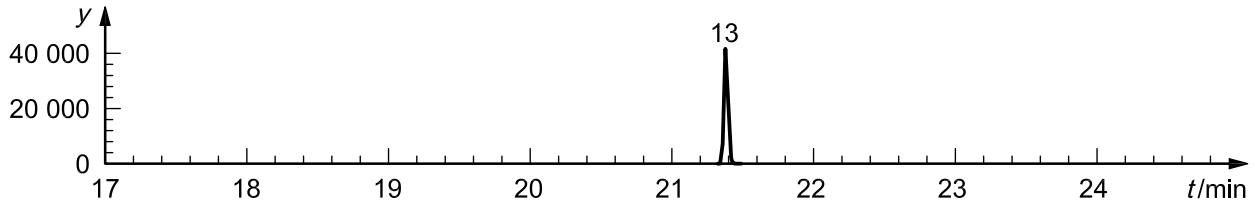
i) Ion 263



j) Ion 272



k) Ion 226



l) Ion 162

Figure A.2 (*continued*)

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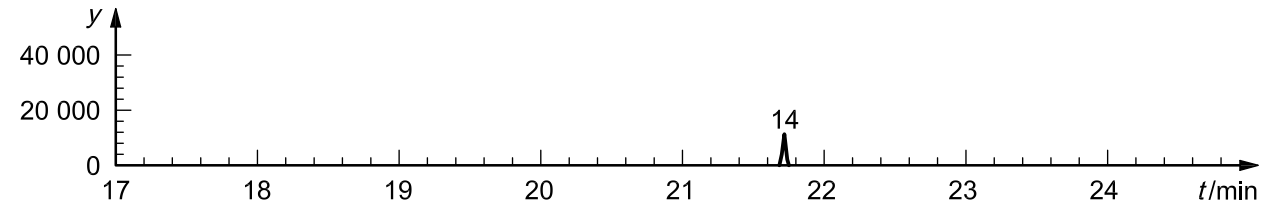
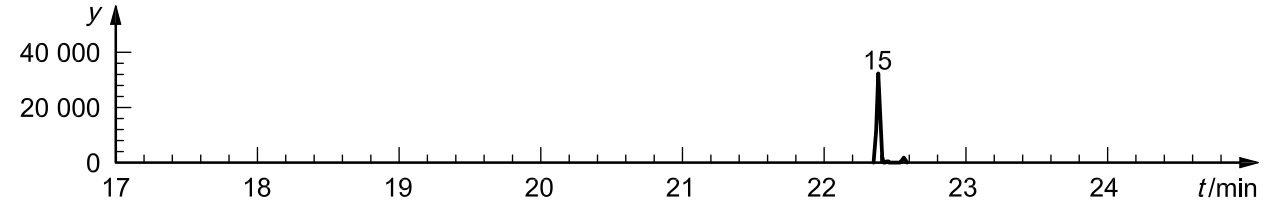
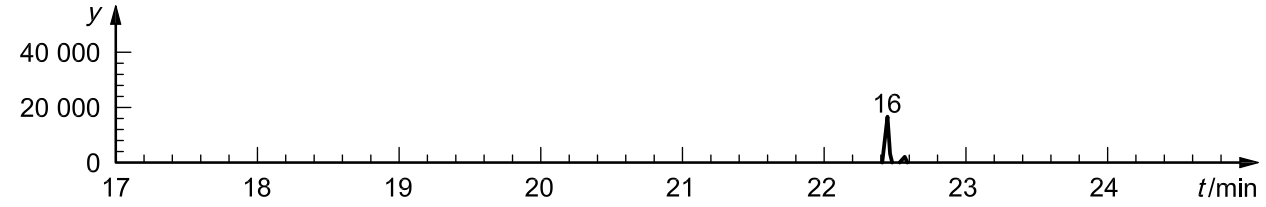


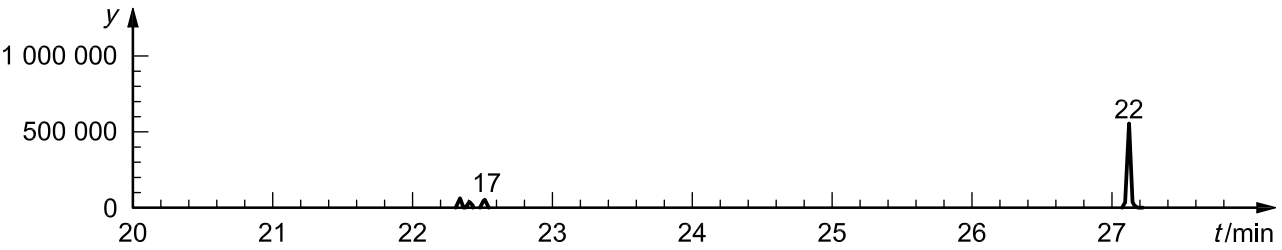
m) Ion 291



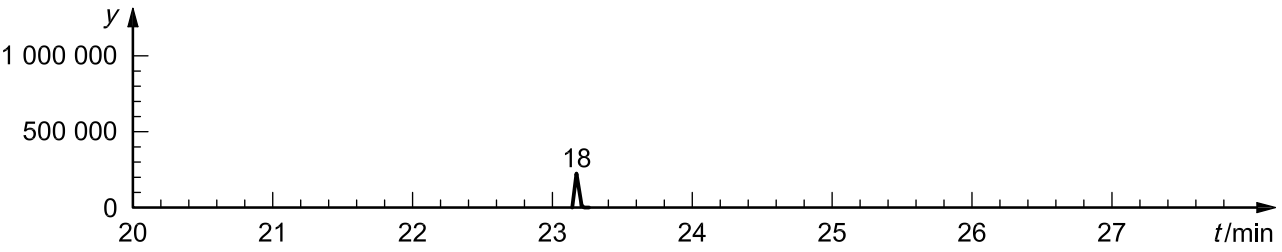
n) Ion 252



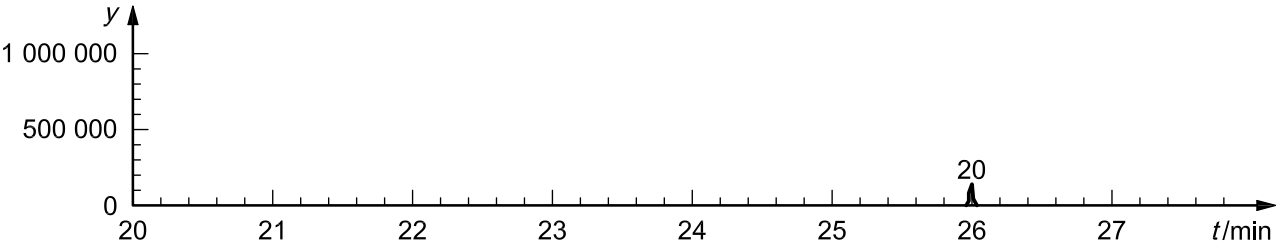
o) Ion 353



p) Ion 252

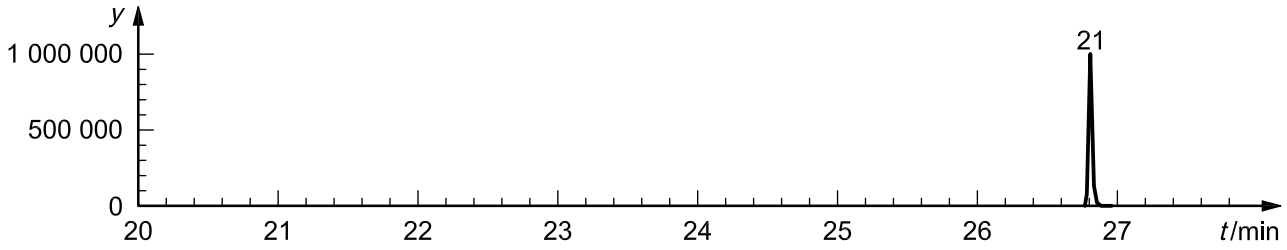


q) Ion 288



r) Ion 340

Figure A.2 (*continued*)



s) Ion 266

GC equipment:	Agilent 6890[2)], Gerstel MPS 2[2)] (fibre rotation), CTC Cycle-Composer software [2)]
SPME conditions:	according to Clause 8 with sample volume: 8 ml, amount NaCl: 2,4 g, fibre: polyacrylate 85 µm [Supelco PA 85[2)], 1 cm, 23 gauge], agitation speed: 250 min^{-1}, extraction time: 60 min
Injection	Gerstel KAS[2)], 60 °C; 10 °C/s to 280 °C; 10 min; splitless (6 min)
Capillary column:	ZB 5 (Phenomenex) [2)], 30 m × 0,25 mm × 0,10 µm
Range of concentration:	0,25 µg/l to 0,35 µg/l
Carrier gas:	helium (5,0); 0,9 ml/min
GC oven temperature:	60 °C, 2 min; 7 °C/min to 200 °C; 20 °C/min to 300 °C; 2 min
MS detector:	Agilent 5973 MSD [2)], quadrupole MS, EI 70 eV, SIM, transfer line 280 °C

Key

1 dichlobenil
2 desethylatrazine
3 desethylterbutylazine
4 simazine
5 atrazine
6 lindane
7 terbutylazine
8 metribuzine
9 parathion-methyl
10 heptachlor
11 terbutryn
12 aldrin
13 metolachlor
14 parathion-ethyl
15 pendimethalin
16 *exo*-Heptachlorepoxide
17 *endo*-Heptachlorepoxide
18 triclosan
19 dieldrin
20 carfentrazone-ethyl
21 diflufenican
22 mefenpyr-diethyl
t time
y abundance

Figure A.2 — Gas chromatogram 2

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

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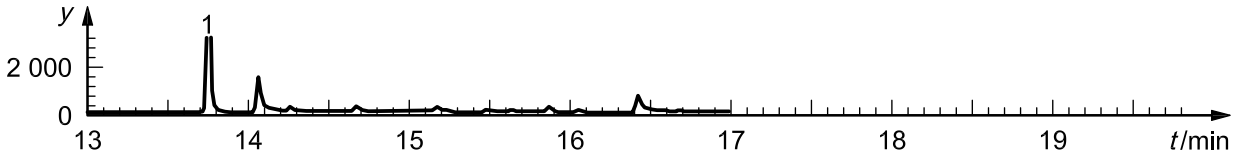
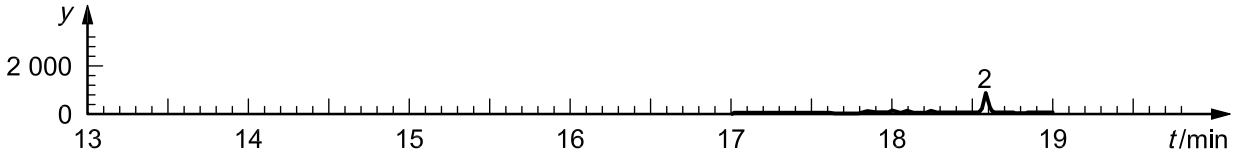
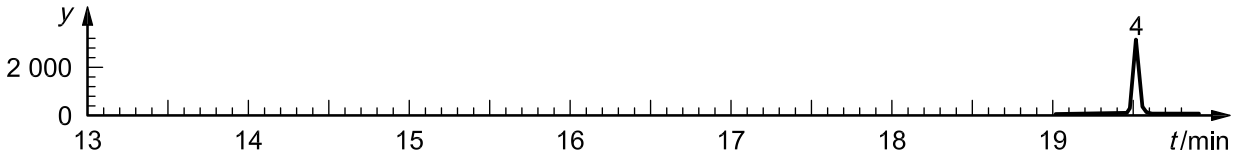


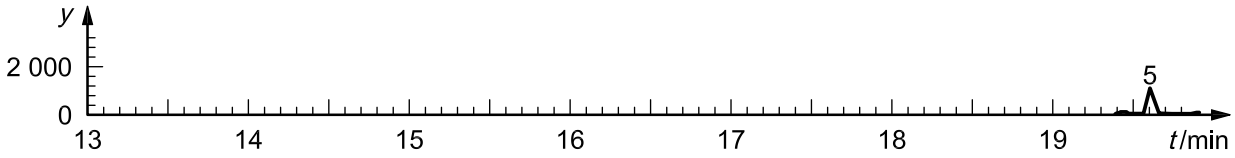
a) Ion 171



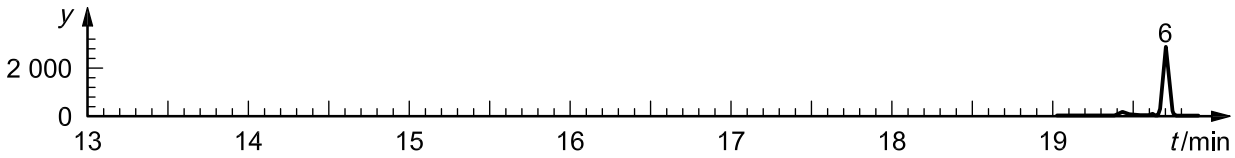
b) Ion 186



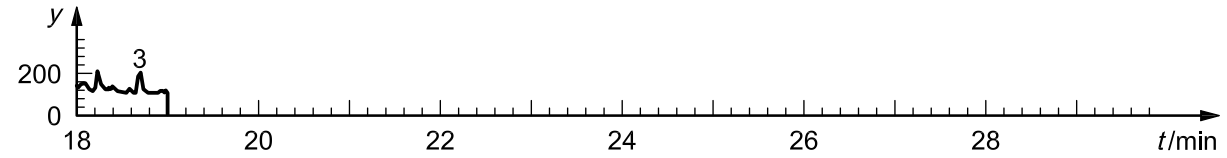
c) Ion 205



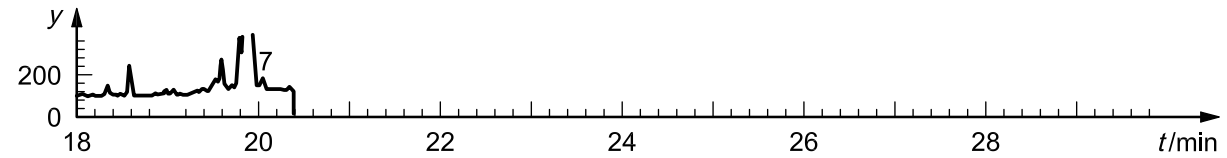
d) Ion 200



e) Ion 214

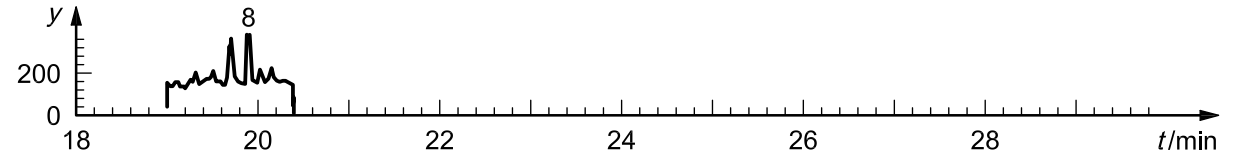


f) Ion 172

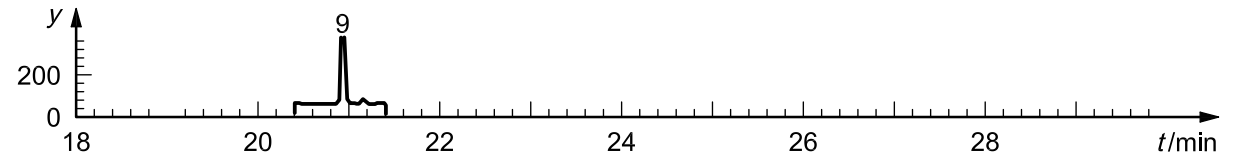


g) Ion 201

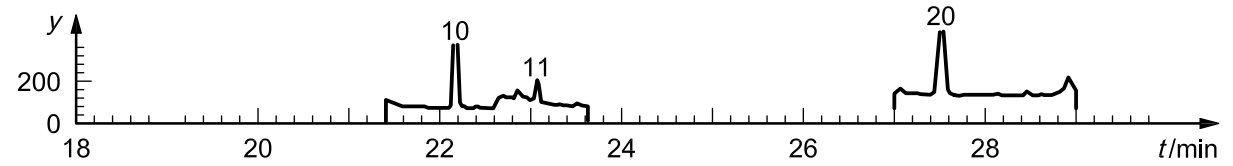
Figure A.3 (*continued*)



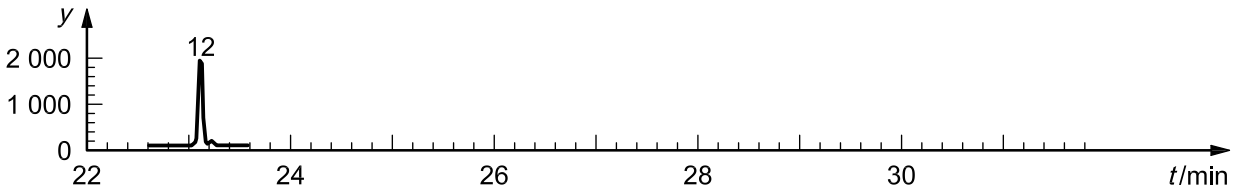
h) Ion 181



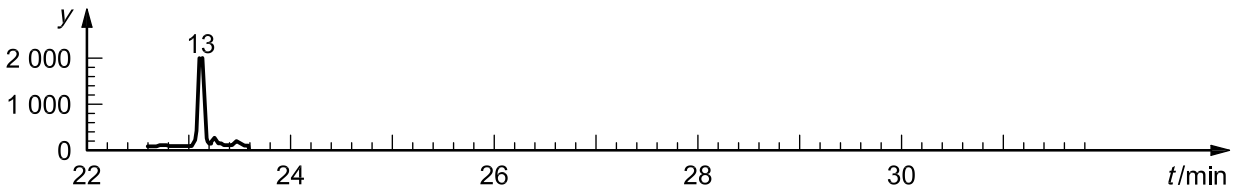
i) Ion 272



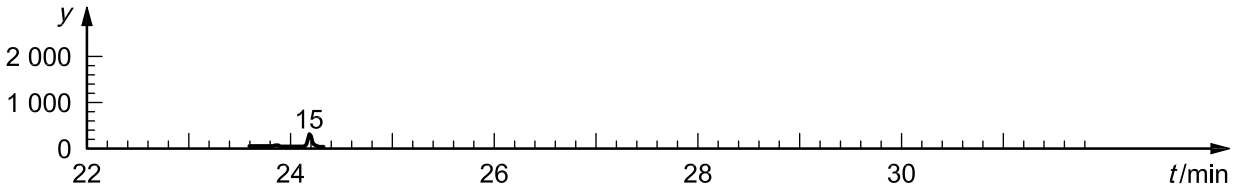
j) Ion 263



k) Ion 226



l) Ion 162



m) Ion 291

Figure A.3 (*continued*)

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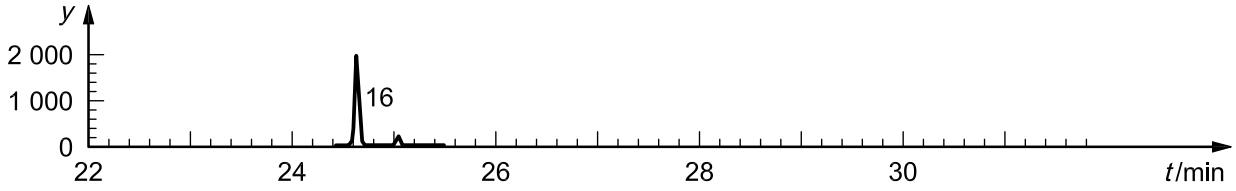
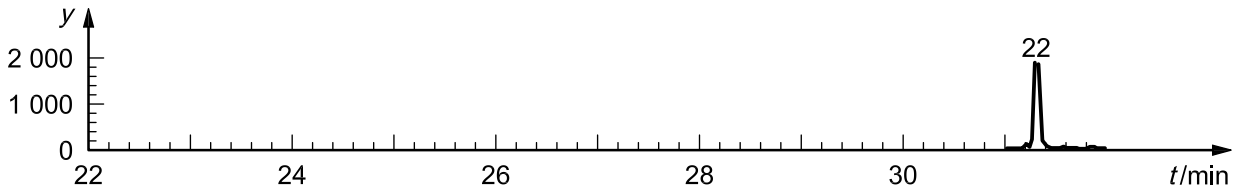
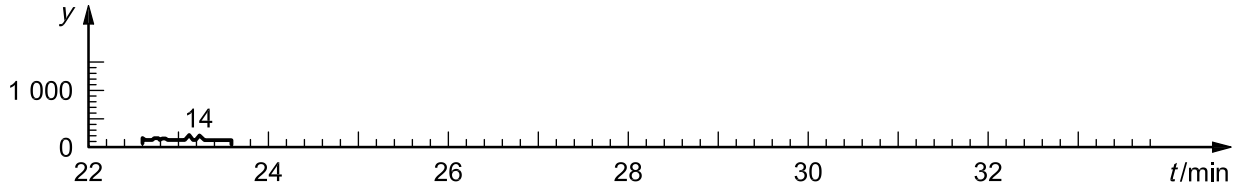


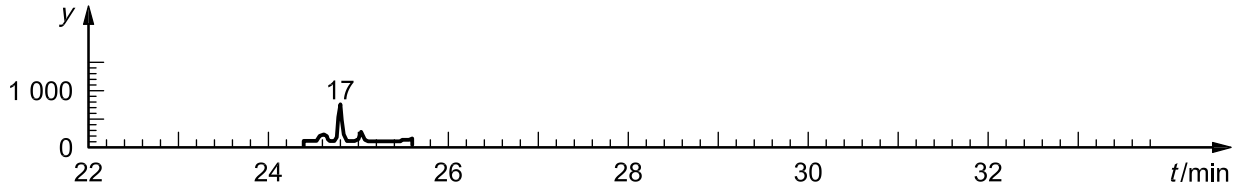
n) Ion 353



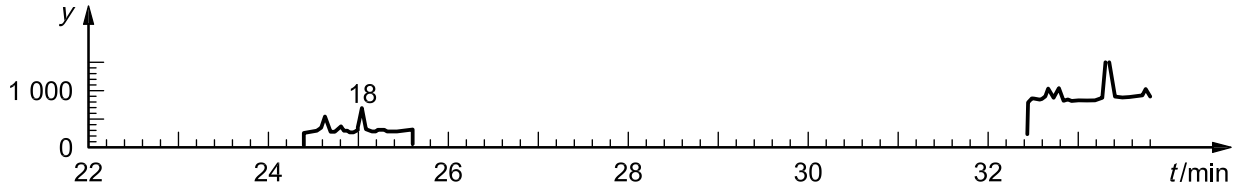
o) Ion 266



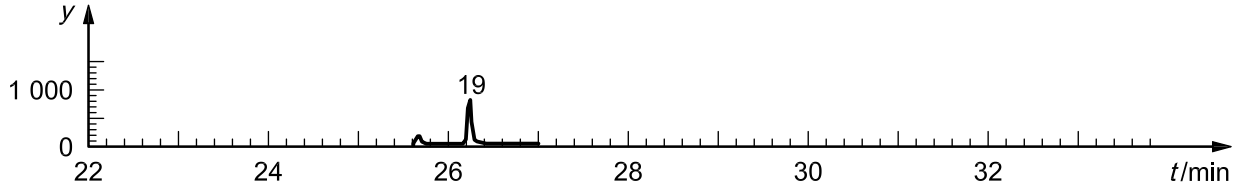
p) Ion 198



q) Ion 252



r) Ion 253



s) Ion 288

Figure A.3 (*continued*)

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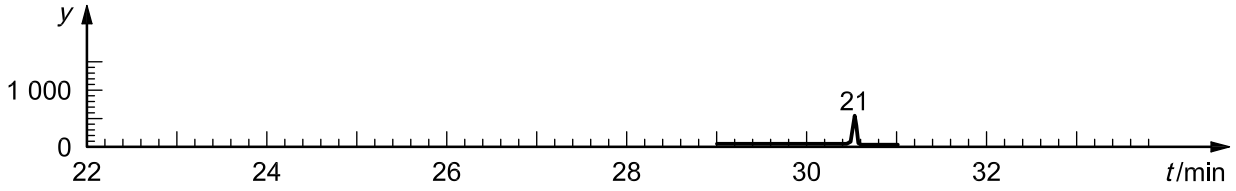

t) Ion 340

GC equipment:	Agilent 6890[3)], Gerstel MPS 2[3)] (fibre rotation), CTC Cycle-Composer software[3)]
SPME conditions:	according to Clause 8 with sample volume: 8 ml, amount NaCl: 2,4 g, fibre: polyacrylate 85 µm [Supelco PA 85[3)], 1 cm, 23 gauge], agitation speed: 250 min^{-1}, extraction time: 60 min
Injection	Gerstel KAS[3)], 60 °C; 10 °C/s to 280 °C; 10 min; splitless (6 min)
Capillary column:	ZB 50 [Phenomenex[3)]], 30 m × 0,25 mm × 0,25 µm
Range of concentration:	0,08 µg/l to 0,01 µg/l
Carrier gas:	helium (5,0); 1,0 ml/min
GC oven temperature:	60 °C, 2 min; 10 °C/min to 180 °C; 4 °C/min to 260 °C, 20 °C/min to 300 °C; 2 min
MS detector:	Agilent 5973 MSD[3)], quadrupole MS, EI 70 eV, SIM, transfer line 280 °C

Key

1 dichlobenil
2 desethylterbutylazine
3 desethylatrazine
4 atrazine-d_5
5 atrazine
6 terbutylazine
7 simazine
8 lindane
9 heptachlor
10 aldrin
11 parathion-methyl
12 terbutryn
13 metolachlor
14 metribuzine
15 parathion-ethyl
16 *exo*-Heptachlorepoxide
17 pendimethalin
18 *endo*-Heptachlorepoxide
19 triclosan
20 dieldrin
21 carfentrazone-ethyl
22 diflufenican
23 mefenpyr-diethyl
t time
y abundance

Figure A.3 — Gas chromatogram 3

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

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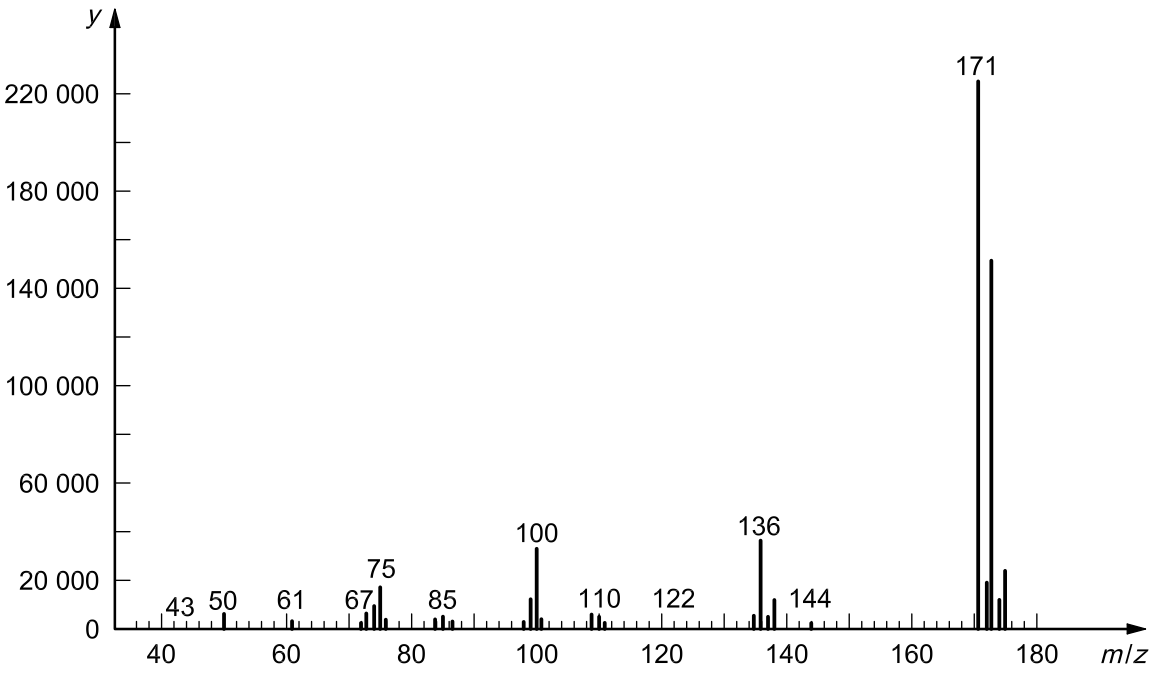
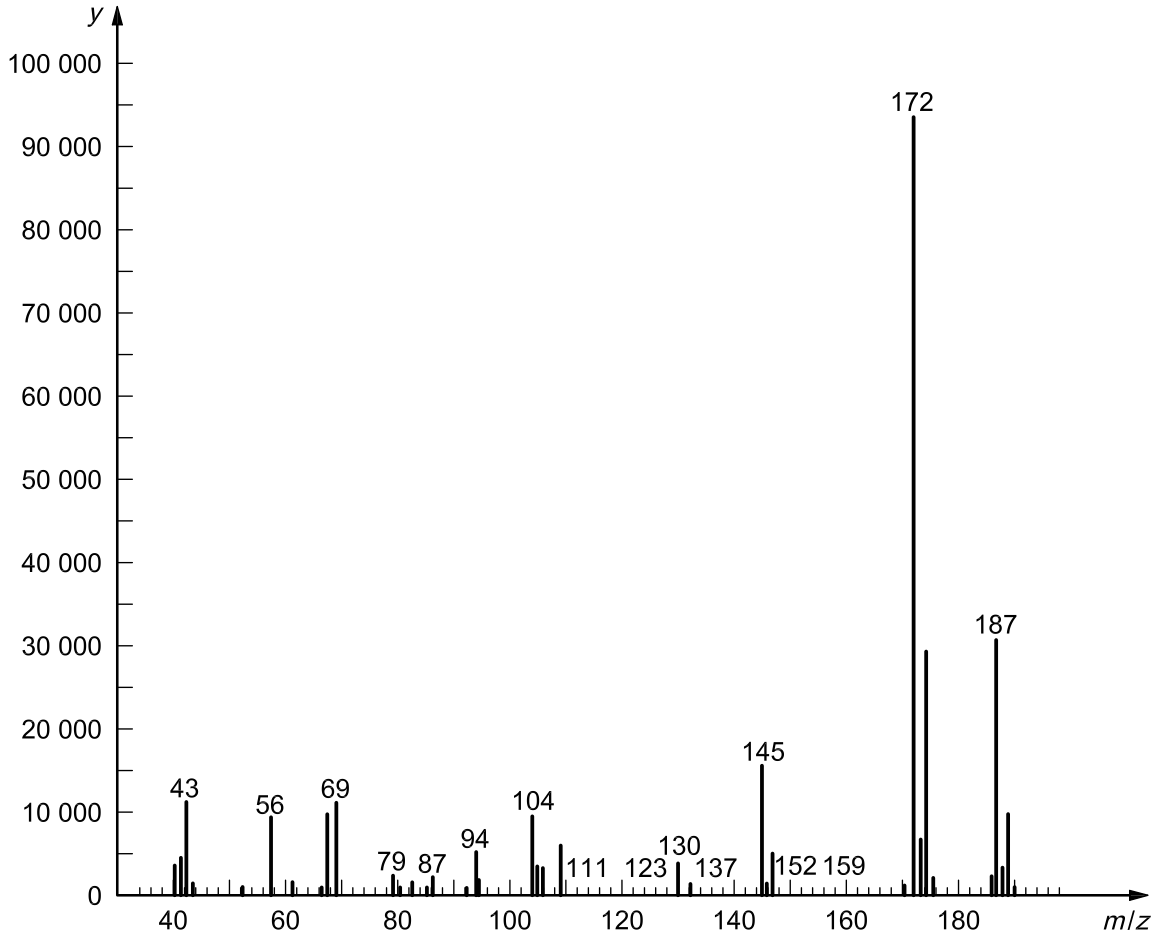
Annex B
(informative)

Mass spectra of compounds of Table 1 (full-scan, EI, 70 eV)



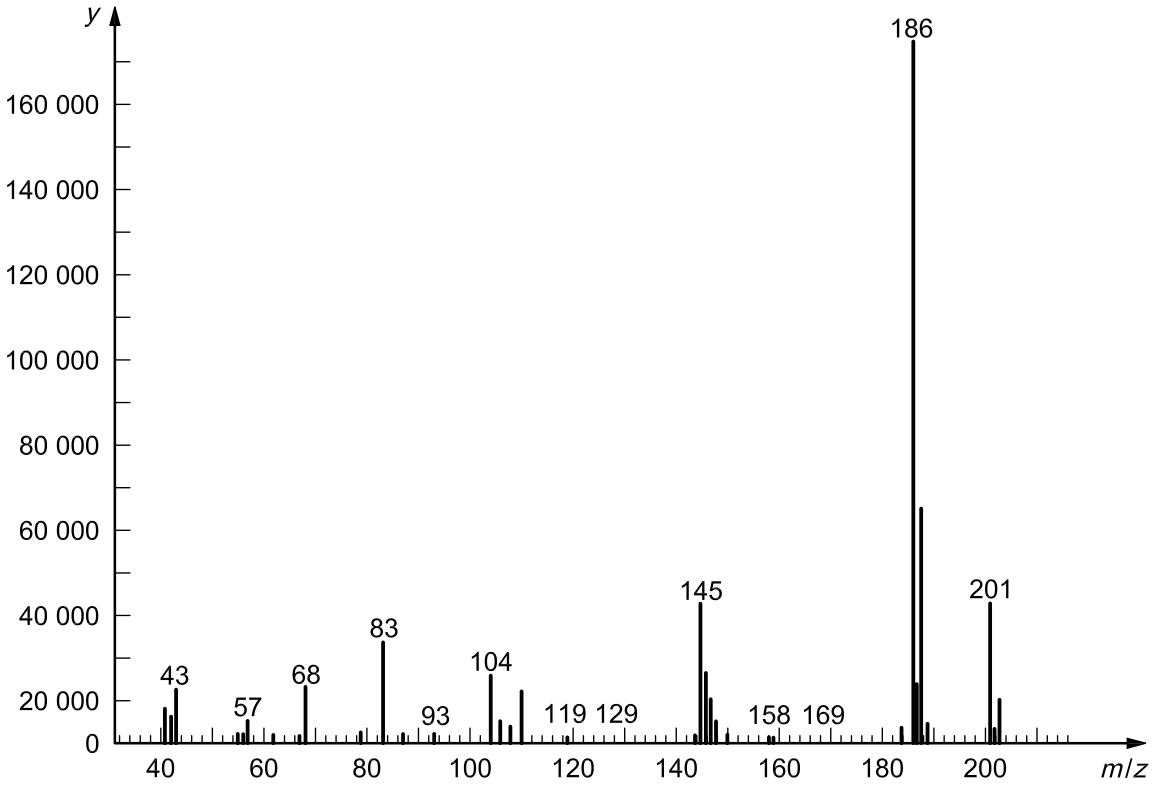
a) Dichlobenil

Figure B.1 (*continued*)

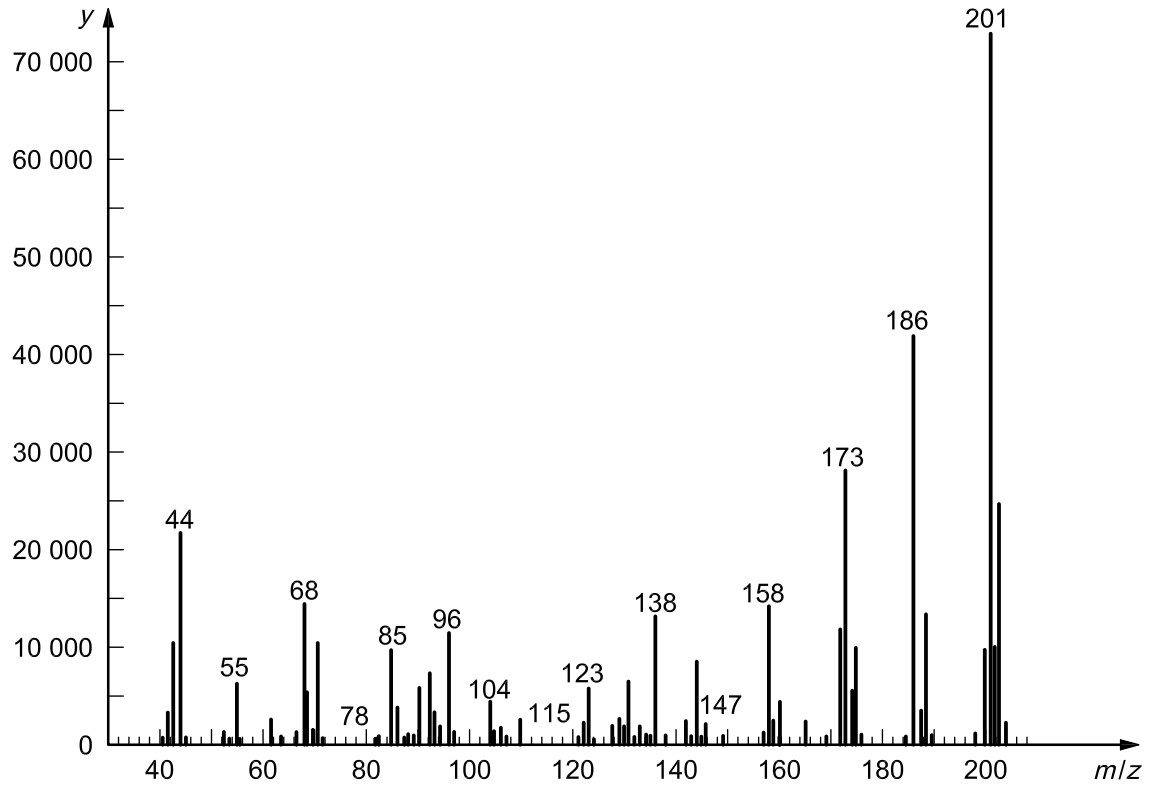


b) Desethylatrazine

Figure B.1 (*continued*)

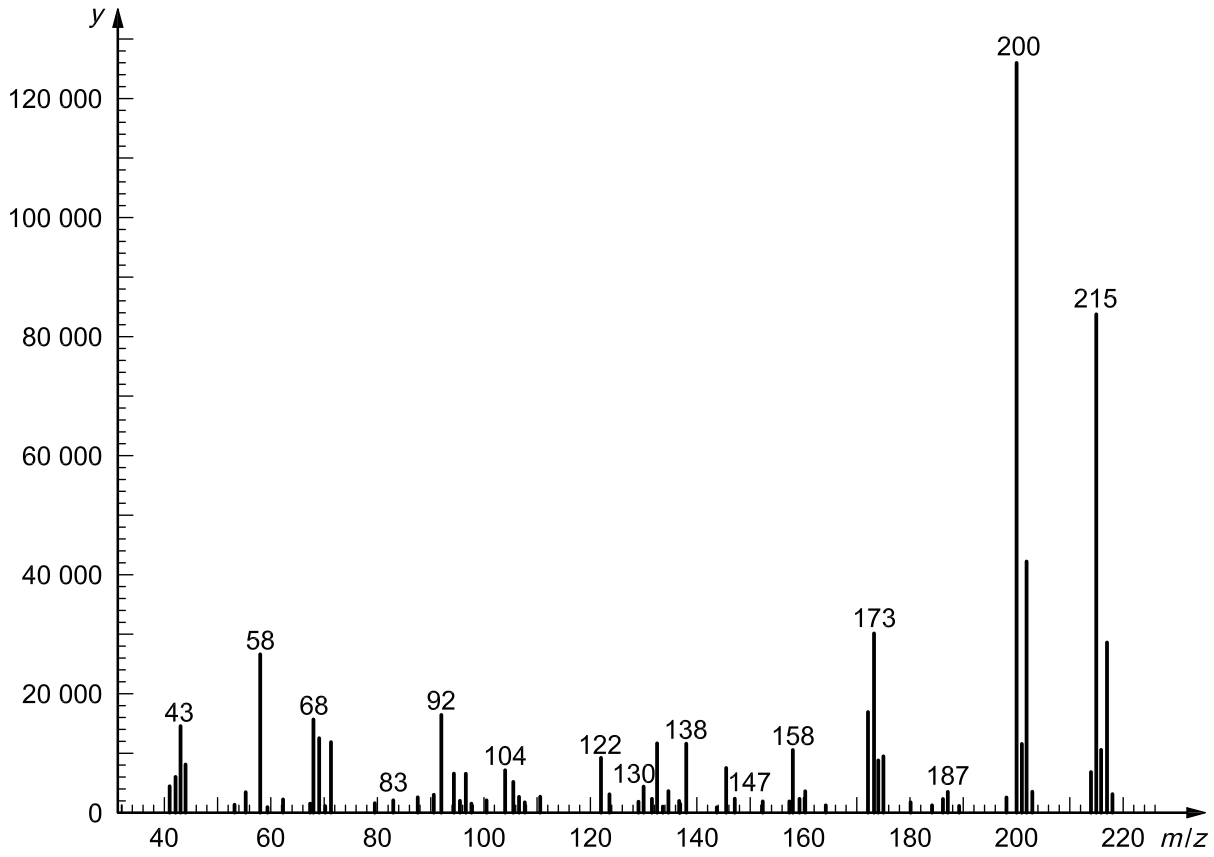


c) Desethylterbutylazine

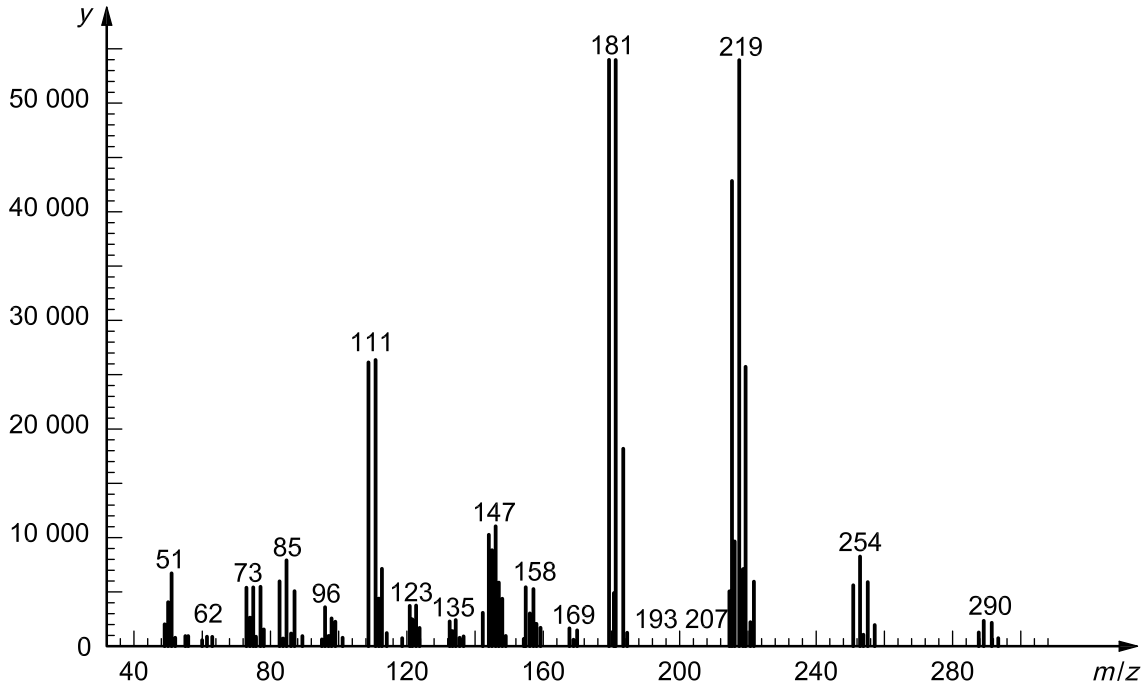


d) Simazine

Figure B.1 (*continued*)

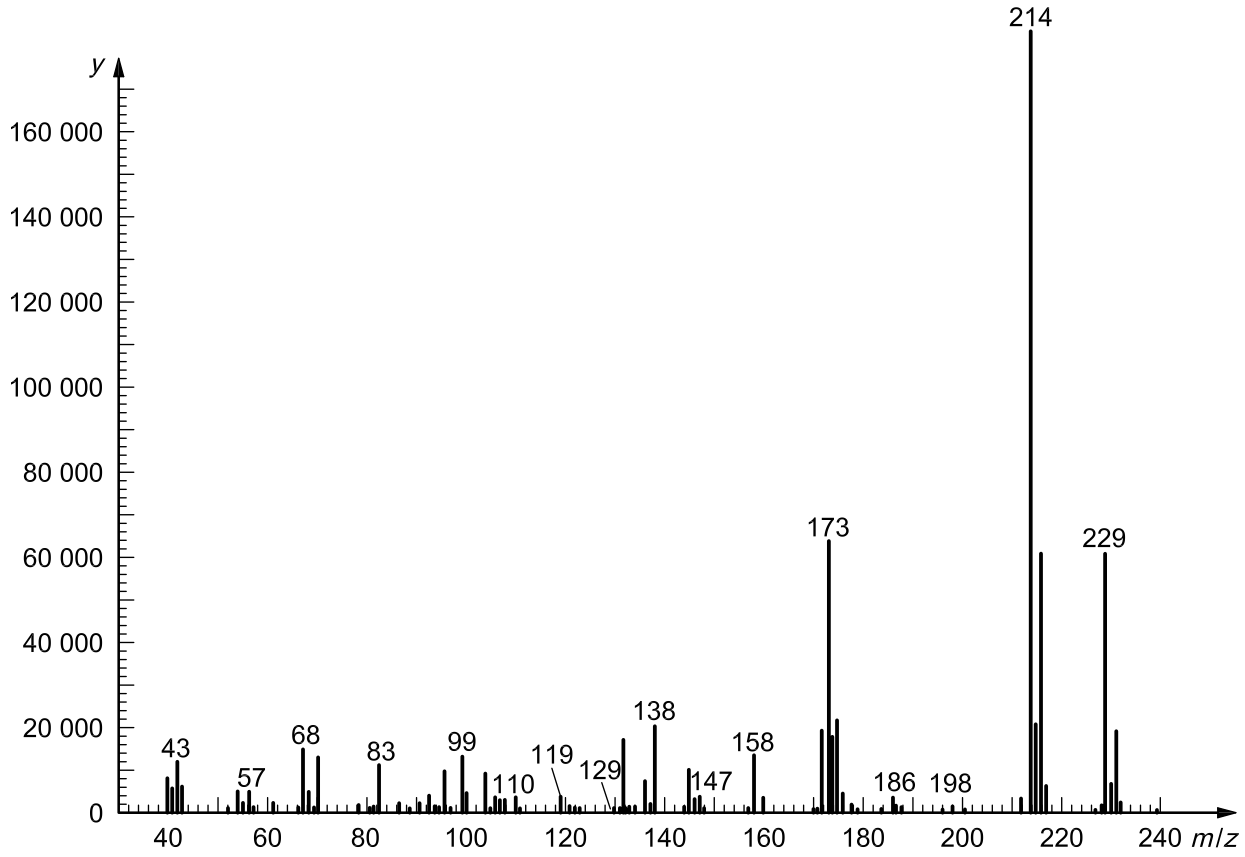


e) **Atrazine**

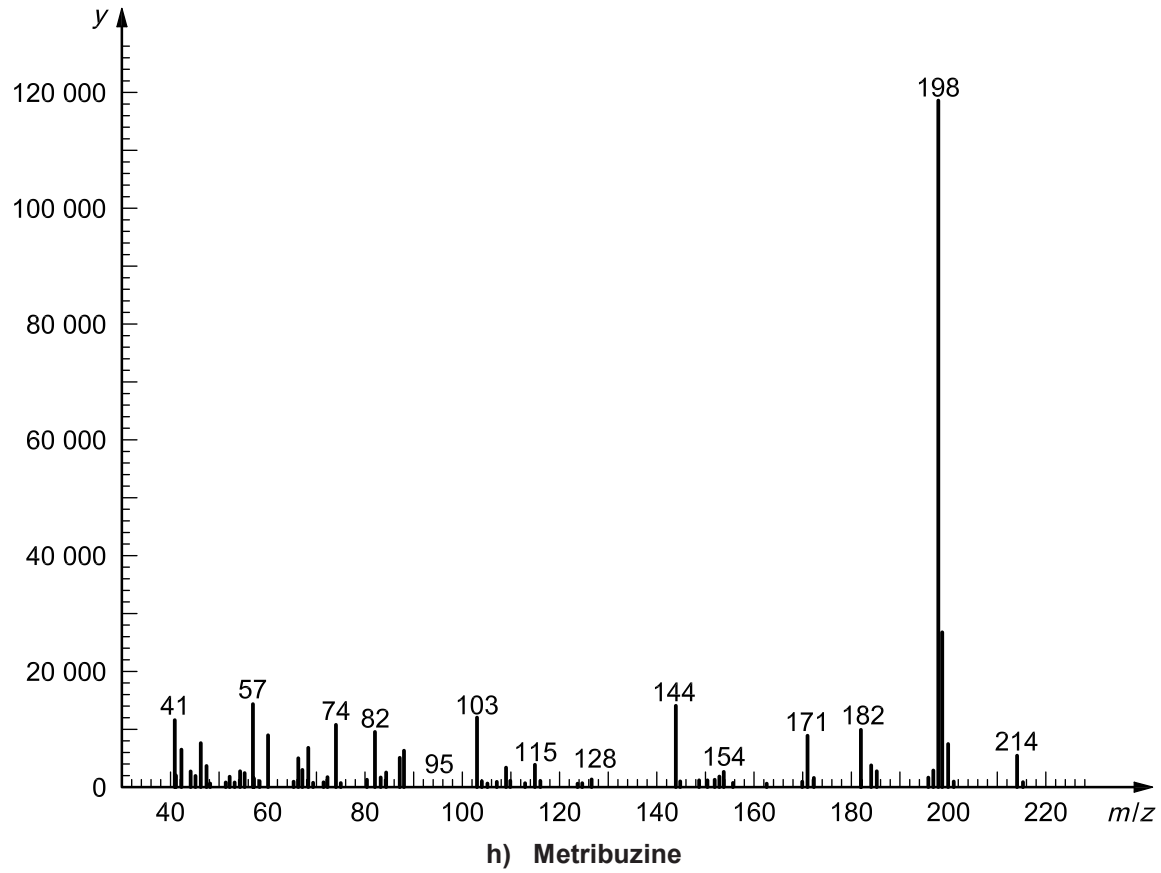


f) **Lindane**

Figure B.1 (*continued*)

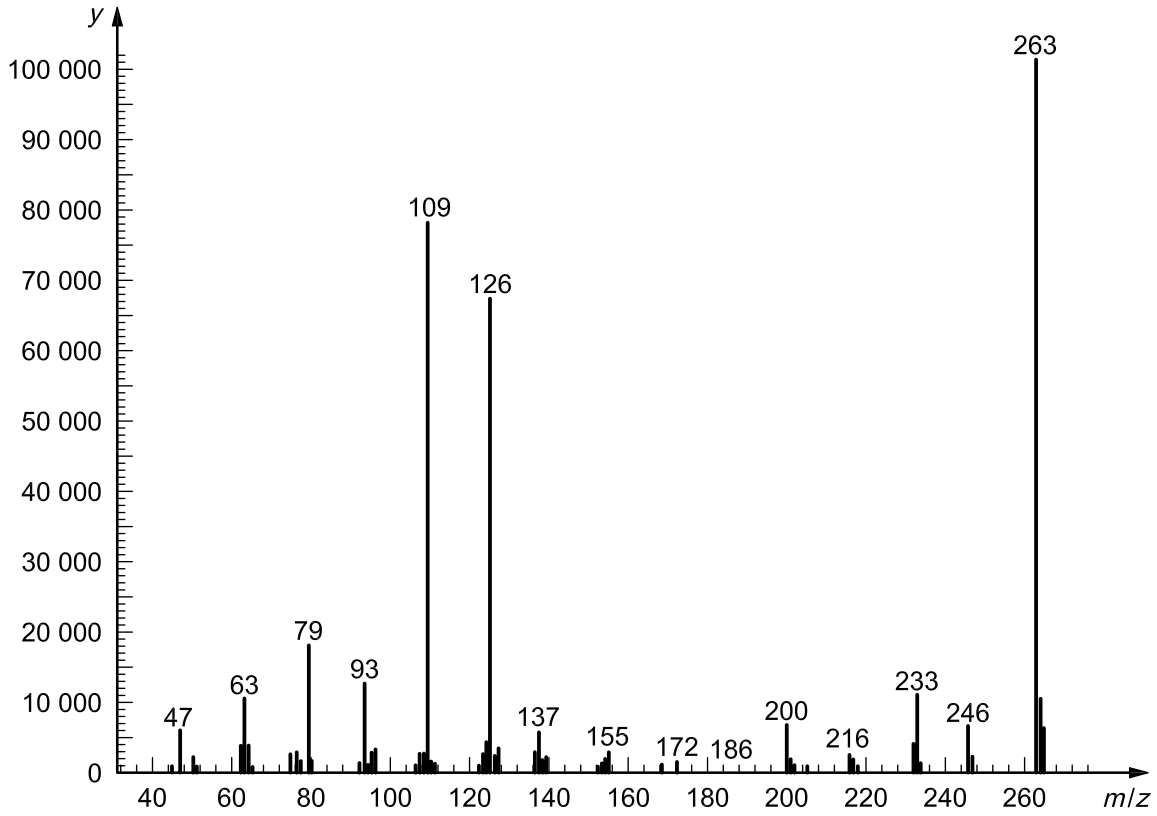


g) Terbutylazine

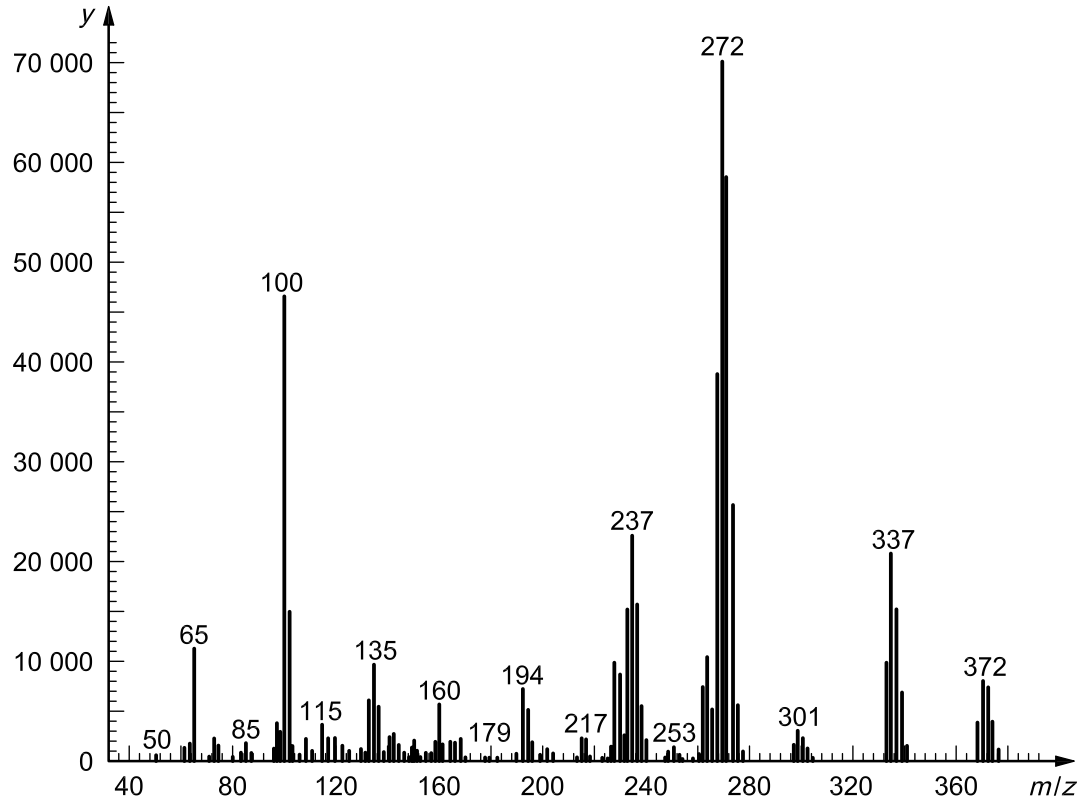


h) Metribuzine

Figure B.1 (*continued*)

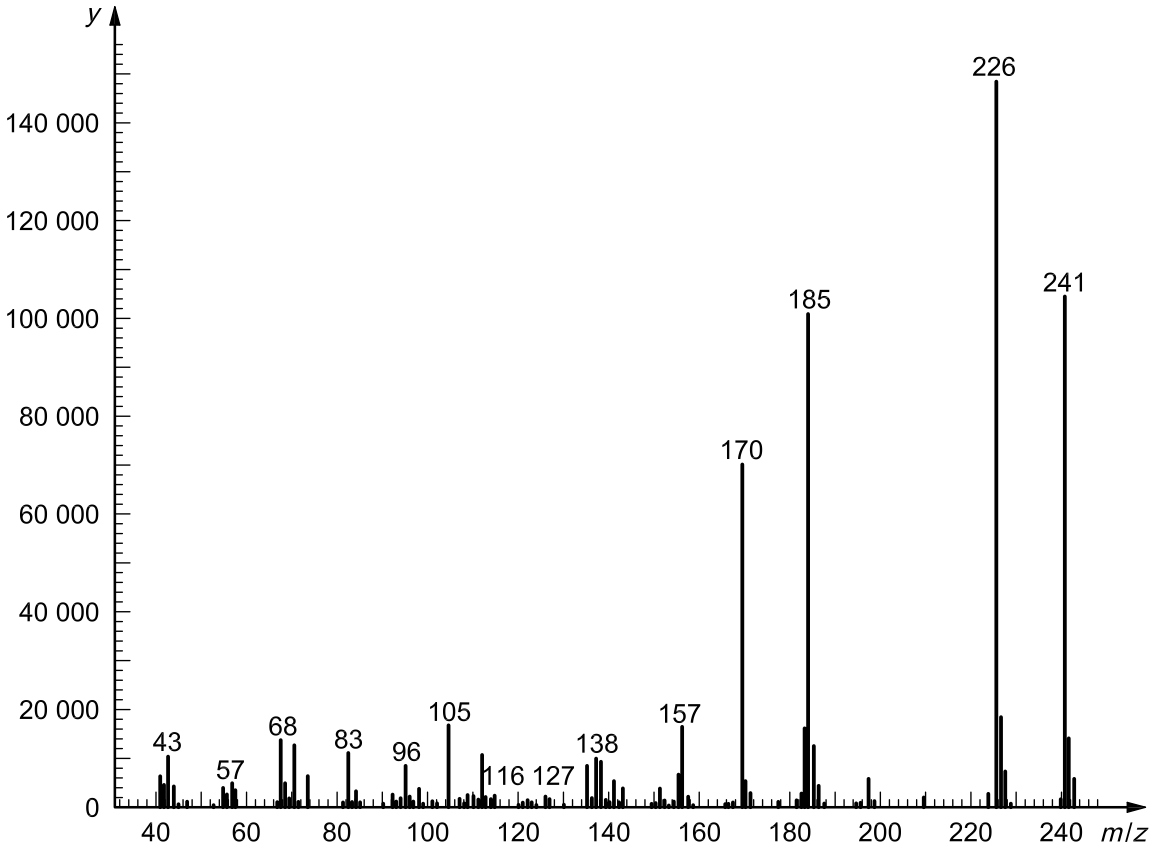


i) **Parathion-methyl**



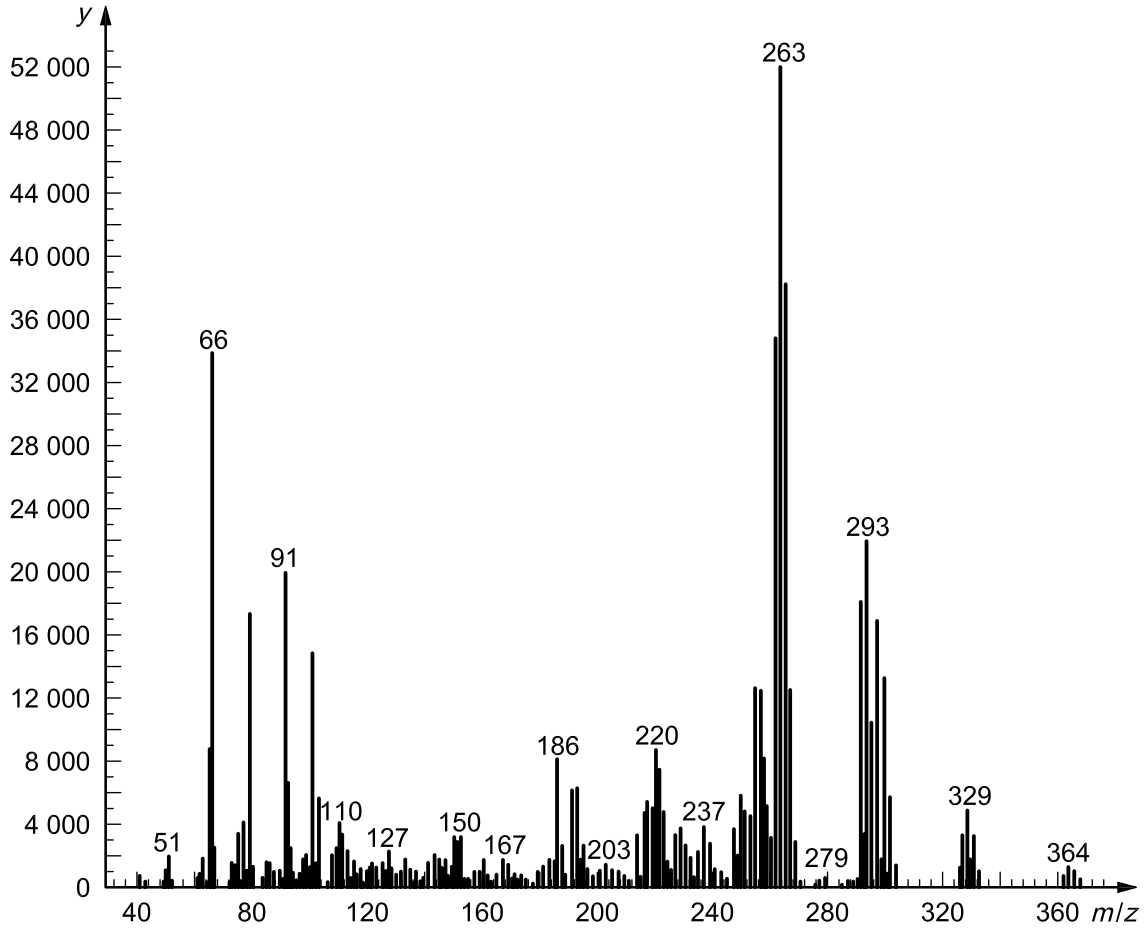
j) **Heptachlor**

Figure B.1 (*continued*)



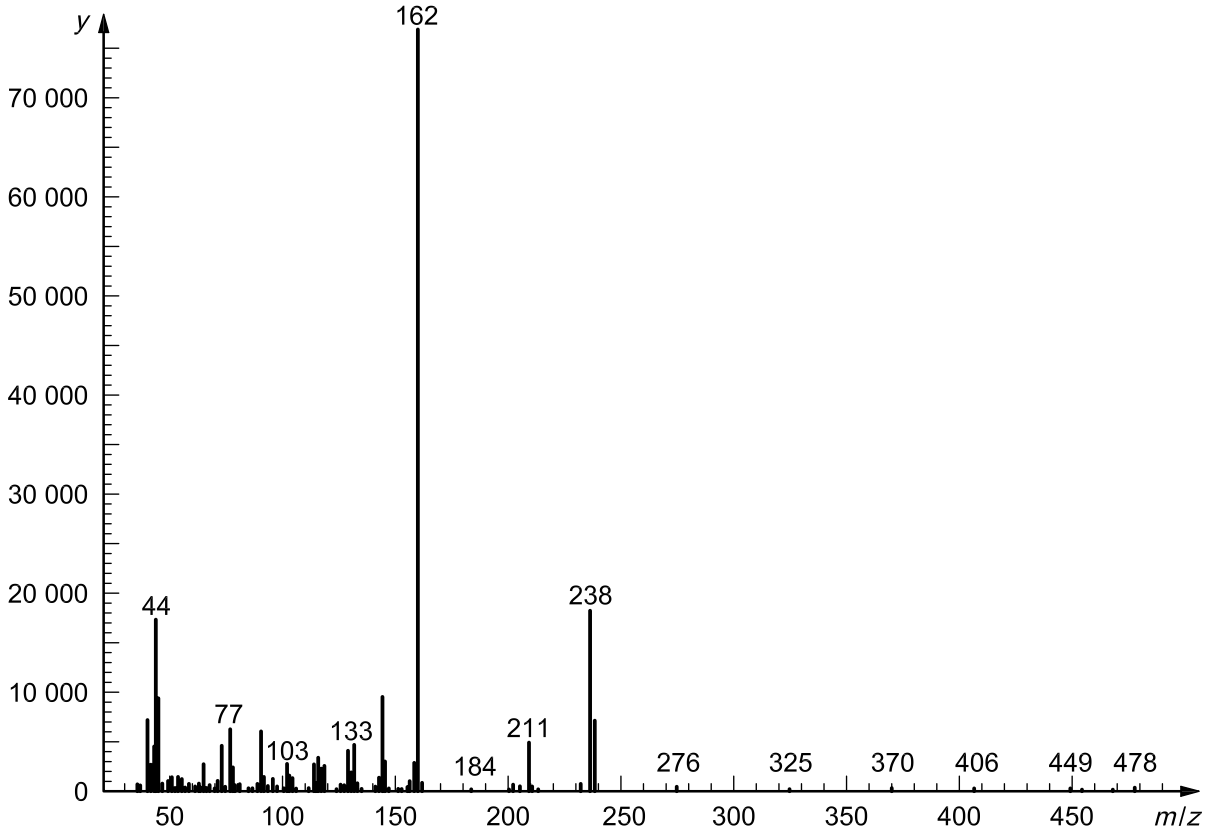
k) Terbutryn

Figure B.1 (*continued*)

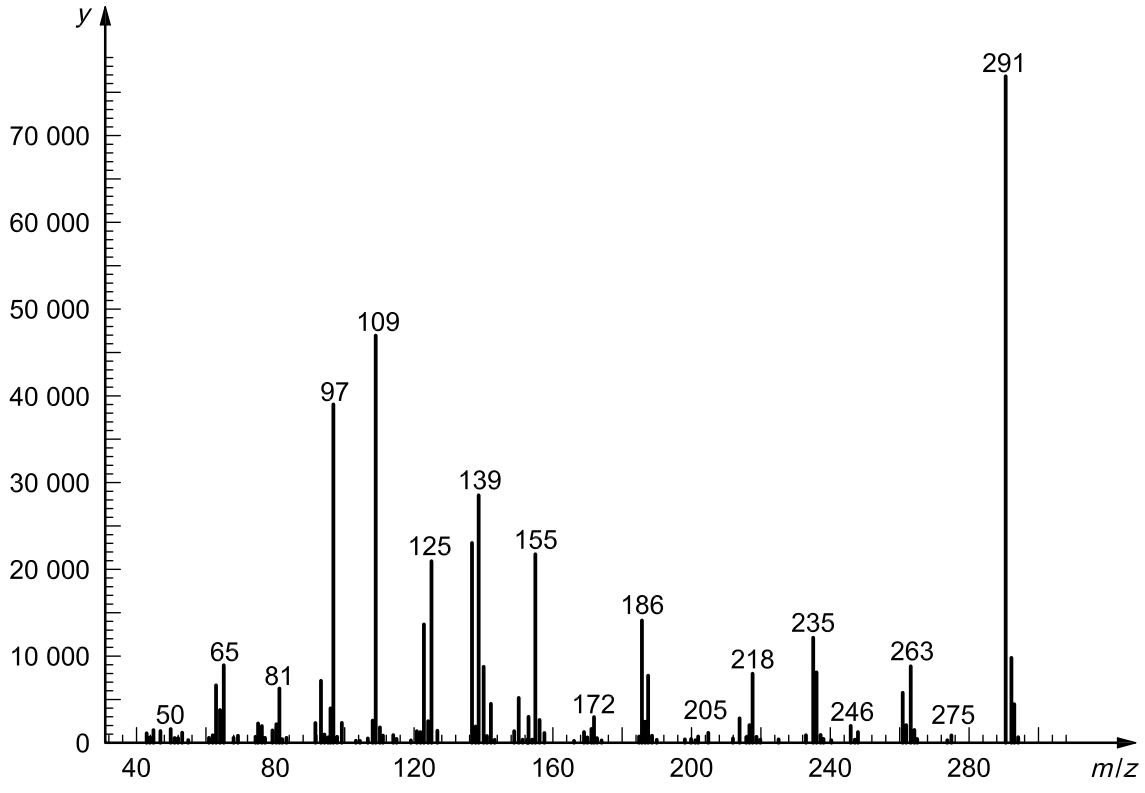


l) Aldrin

Figure B.1 (*continued*)

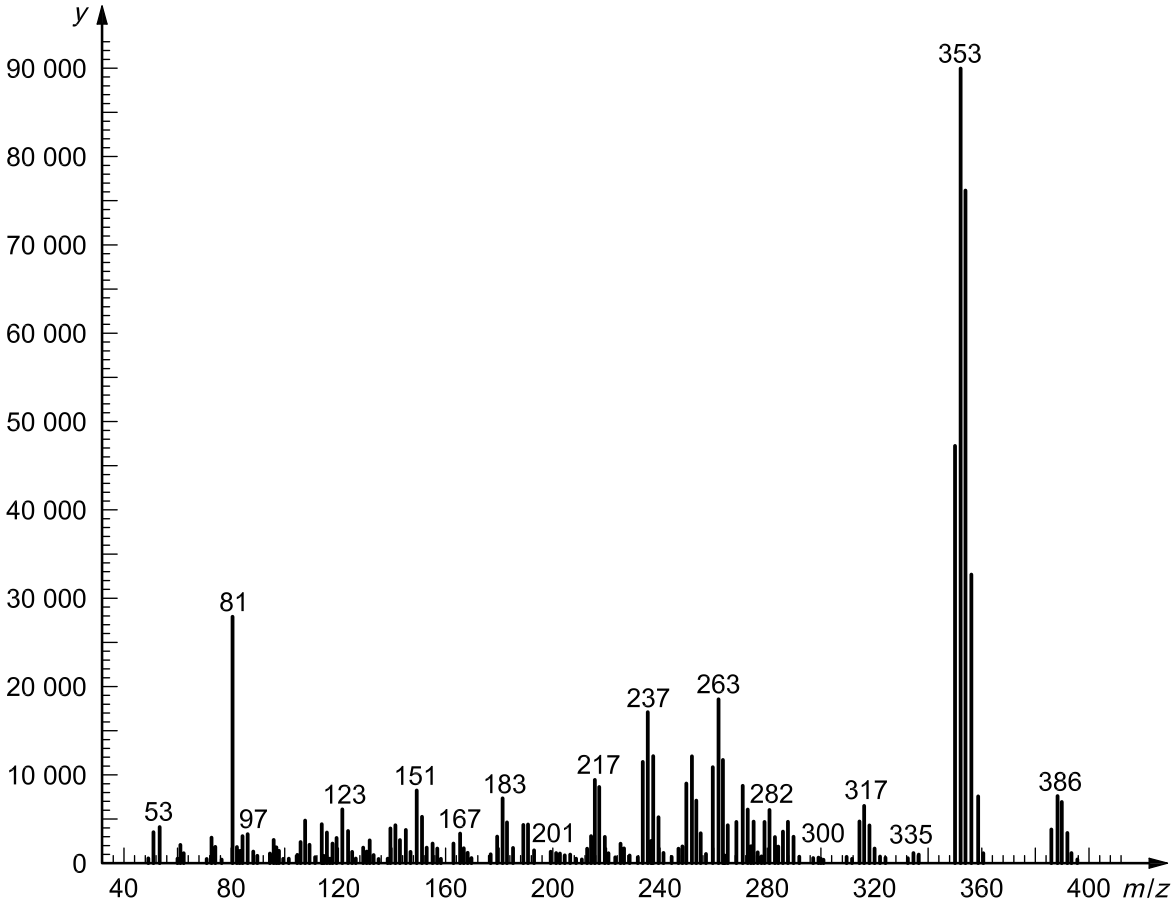


m) Metolachlor



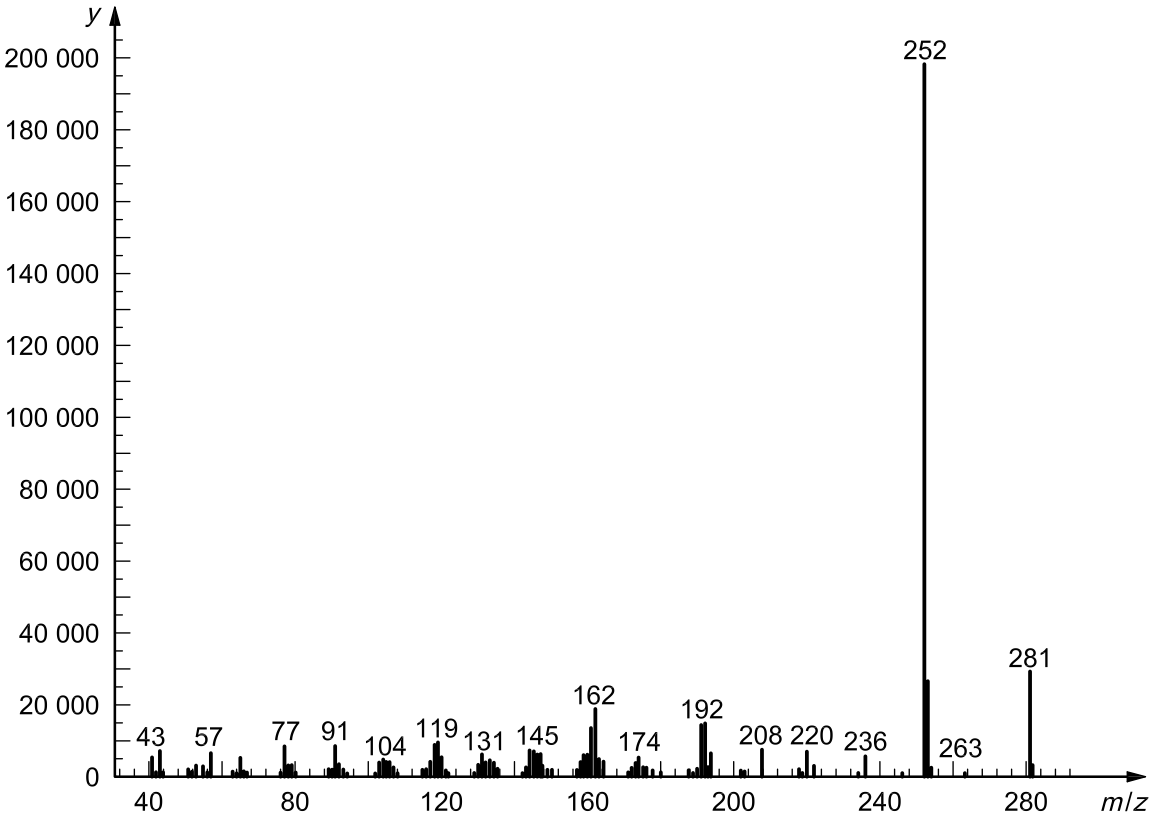
n) Parathion-ethyl

Figure B.1 (*continued*)

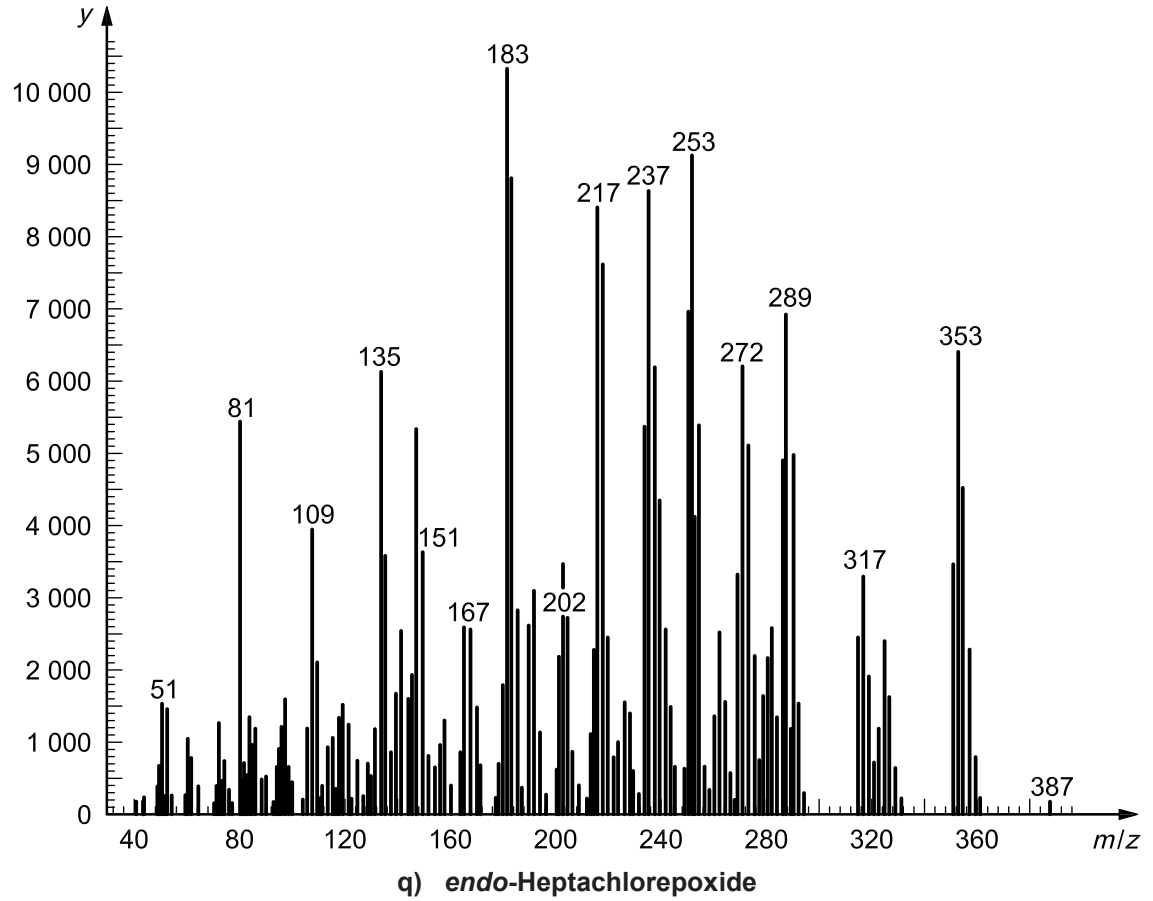


o) *exo*-Heptachlorepoxide

Figure B.1 (*continued*)

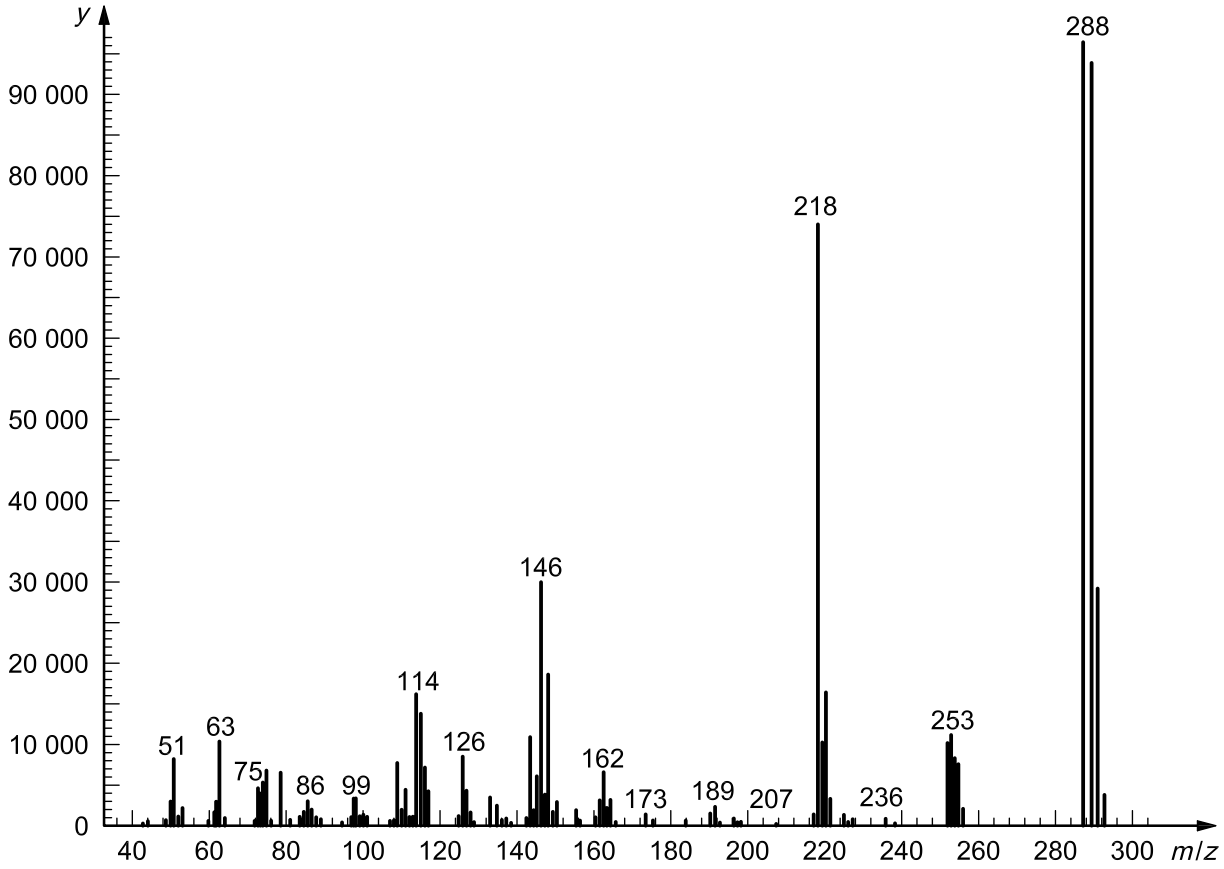


p) Pendimethalin

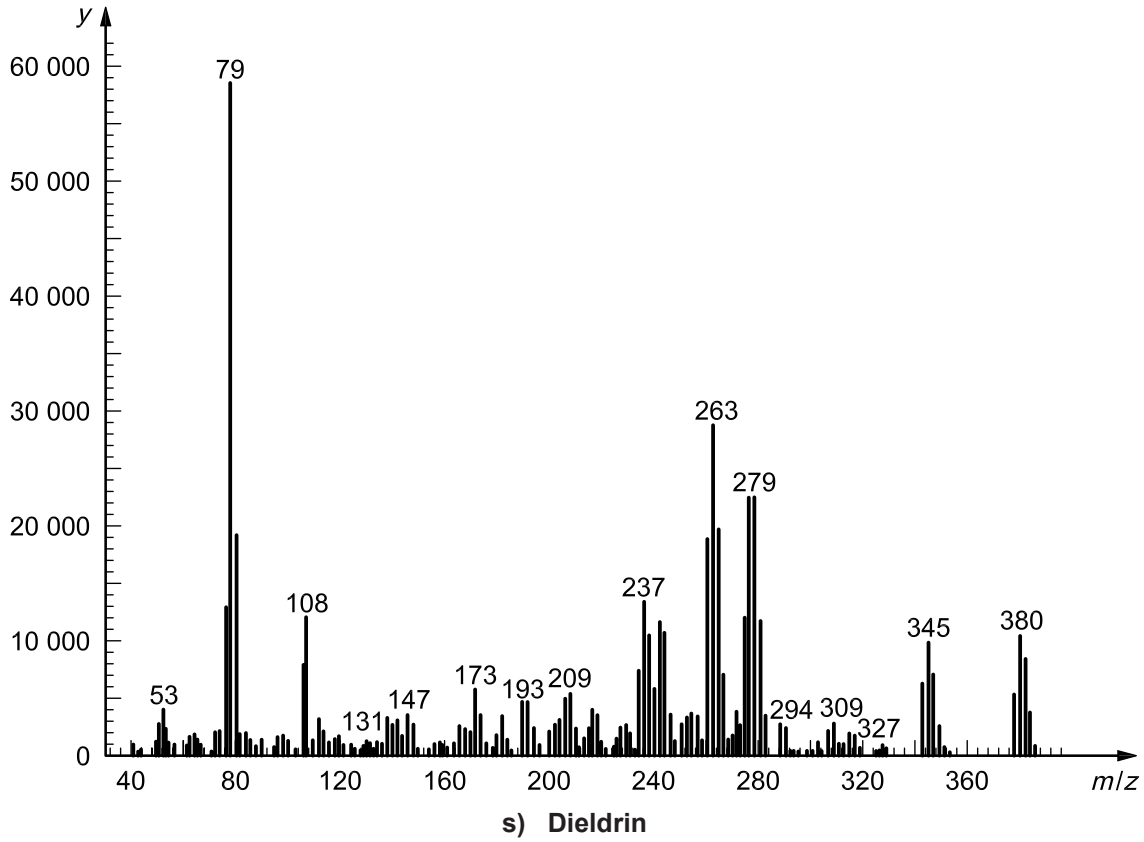


q) *endo*-Heptachlorepoxide

Figure B.1 (*continued*)

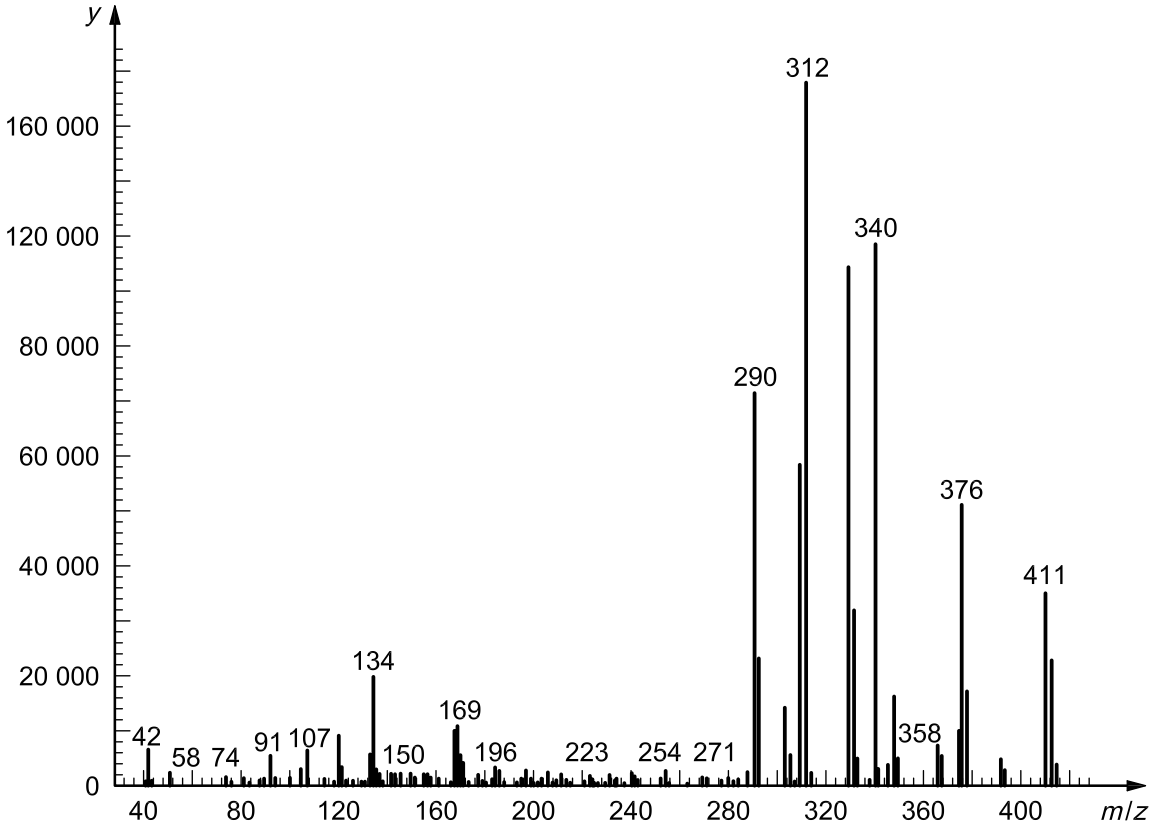


r) Triclosan

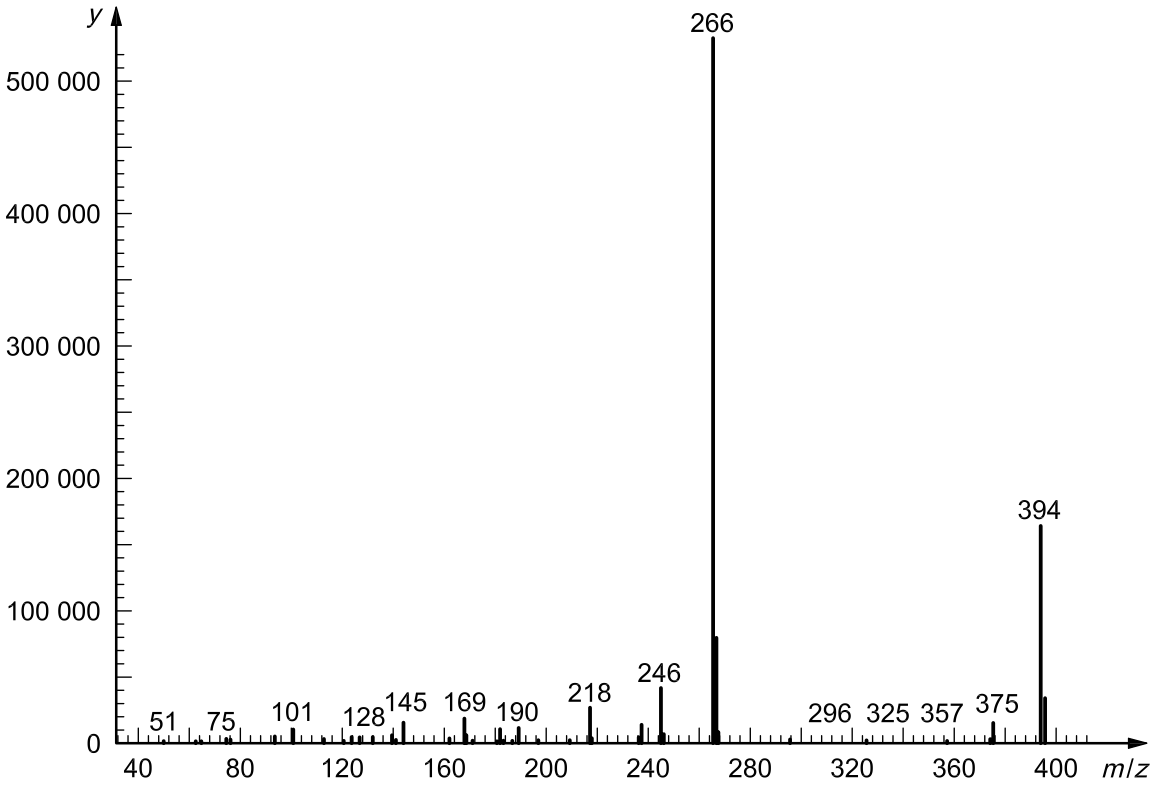


s) Dieldrin

Figure B.1 (*continued*)

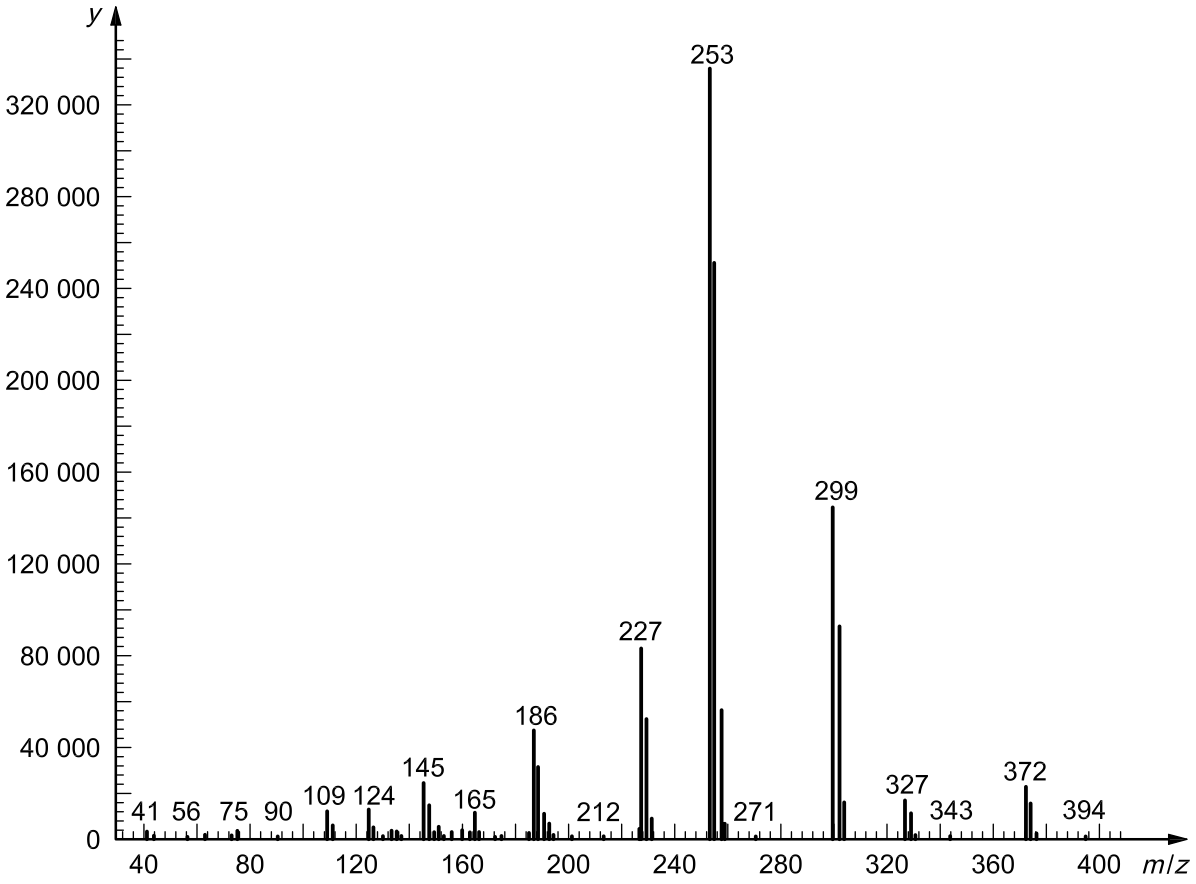


t) Carfentrazone-ethyl



u) Diflufenican

Figure B.1 (*continued*)



v) Mefenpyr-diethyl

Key

m/z mass/charge ratio

Y intensity

Figure B.1 — Mass spectra of selected plant treatment agents and biocide products

Annex C
(informative)

Precision data

The precision data given in Table C.1 were determined in an interlaboratory trial carried out in Germany in June 2005. The data do not cover all of the substances in the scope for all matrices tested. However, the technical working group did experiments on all substances listed in Table 1 during the validation work using spiked surface water, ground water, and drinking water samples. The results indicate that the method is applicable to all matrices.

Table C.1 — Precision data for drinking water, ground water, and surface water for the compounds listed in Table 1

Sample	Compound	p	n	n_{OP}	$\bar{\bar{\rho}}$	ρ_{ref}	η	s_R	$C_{V,R}$	s_r	$C_{V,r}$
				%	µg/l	µg/l	%	µg/l	%	µg/l	%
Drinking water, spiked	Dichlobenil	13	52	13,3	0,051	0,052	97,6	0,0074	14,5	0,0023	4,5
	Desethylterbutylazine	13	52	13,3	0,076	0,080	95,4	0,0107	14,0	0,0040	5,2
	Lindane	14	56	6,7	0,145	0,135	107,3	0,0175	12,1	0,0090	6,2
	Heptachlor	12	48	0,0	0,077	0,122	62,7	0,0273	35,6	0,0100	13,1
	Aldrin	15	59	0,0	0,074	0,084	88,4	0,0259	34,9	0,0115	15,5
	Pendimethalin	13	52	5,5	0,084	0,108	77,6	0,0181	21,6	0,0084	10,0
	Dieldrin	12	48	14,3	0,096	0,111	86,4	0,0160	16,7	0,0089	9,2
	Mefenpyr-diethyl	13	52	7,1	0,073	0,071	102,6	0,0166	22,8	0,0052	7,2
Ground water, spiked	Desethylatrazine	12	46	9,8	0,211	0,244	86,6	0,0412	19,5	0,0180	8,5
	Simazine	15	59	1,7	0,136	0,140	97,2	0,0299	22,0	0,0120	8,8
	Terbutylazine	14	54	8,5	0,100	0,106	94,7	0,0116	11,5	0,0051	5,1
	Parathion-methyl	13	52	7,1	0,175	0,206	84,8	0,0265	15,2	0,0175	10,0
	Metolachlor	12	48	18,6	0,121	0,131	92,5	0,0214	17,6	0,0044	3,6
	Parathion-ethyl	15	60	0,0	0,187	0,199	93,9	0,0291	15,6	0,0147	7,9
	endo-Heptachlorepoxide	14	54	0,0	0,196	0,218	90,1	0,0529	27,0	0,0177	9,0
	Carfentrazone-ethyl	9	35	12,5	0,127	0,141	90,3	0,0196	15,4	0,0094	7,4
Surface water, spiked	Dichlobenil	15	59	0,0	0,148	0,157	94,1	0,0274	18,5	0,0068	4,6
	Desethylterbutylazine	13	52	11,9	0,180	0,183	98,5	0,0349	19,4	0,0077	4,3
	Atrazine	14	54	8,5	0,152	0,158	96,4	0,0241	15,8	0,0080	5,3
	Metribuzine	12	48	0,0	0,307	0,293	104,7	0,0985	32,1	0,0271	8,8
	Terbutryn	15	57	1,7	0,219	0,214	102,2	0,0382	17,5	0,0132	6,0
	exo-Heptachlorepoxide	14	55	0,0	0,156	0,226	68,8	0,0431	27,7	0,0147	9,5
	Triclosan	13	51	7,3	0,220	0,295	74,6	0,0712	32,4	0,0200	9,1
	Diflufenican	14	54	1,8	0,143	0,199	71,8	0,0415	29,0	0,0099	7,0

p	number of laboratories after outlier rejection	η	recovery
n	number of analytical results after outlier rejection	s_R	reproducibility standard deviation
n_{OP}	percentage of outliers	$C_{V,R}$	coefficient of variation of reproducibility
$\bar{\bar{\rho}}$	total mean of all results (outliers excluded)	s_r	repeatability standard deviation
ρ_{ref}	reference value	$C_{V,r}$	coefficient of variation of repeatability

Annex D
(informative)

General information about SPME

When using the SPME technique, the analytes are not completely extracted from the water sample. A distribution equilibrium forms between the analytes in the aqueous solution and those absorbed by the stationary polymer phase. It may take several hours for an equilibrium condition to be reached. However, it is not necessary in practice to wait for equilibrium, as long as the extraction times (e.g. 60 min) and other variables which may influence the results (e.g. extraction temperature, fibre coating, amount of salt added, stirring speed) are kept constant (see Clause 8). The transport into the polymer phase is diffusion controlled and is not primarily dependent on the sample volume. The number of analyte molecules passing into the polymer phase is directly proportional to the distribution coefficient, the volume of the stationary phase, and the concentration of the individual analytes in the water sample (see References [7][8][9]).

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Bibliography

[1] ISO 648, *Laboratory glassware — Single-volume pipettes*

[2] ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

[3] ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

[4] 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*

[5] ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

[6] ISO 22892, *Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry*

[7] BELARDI, R. P., PAWLISZYN, J.B. The application of chemically modified fused silica fibers in the extraction of organics from water matrix samples and their rapid transfer to capillary columns. *Water Pollution Res. J. Can*. 1989, **24**, pp. 179-191

[8] ARTHUR, C. L., POTTER, D. W., BUCHHOLZ, K. D., MOTLAGH, S., PAWLISZYN, J. Solid-phase microextraction for the direct analysis of water: Theory and practice. *LC-GC* 1992, **10**, pp. 656-661

[9] LOUCH, D., MOTLAGH, S., PAWLISZYN, J. Dynamics of organic compound extraction from water using liquid-coated fused silica fibers. *Anal. Chem*. 1992, **64**, pp. 1187-1199

[10] EISERT, R., LEVSEN, K. Determination of organophosphorus, triazine and 2,6-dinitroaniline pesticides in aqueous samples via solid-phase microextraction (SPME) and gas chromatography with nitrogen-phosphorus detection. *Fresenius J. Anal. Chem*. 1995, **351**, pp. 555-562

[11] SCHEYER, A., MORVILLE, S., MIRABEL, P., MILLET, M. Analysis of trace levels of pesticides in rainwater using SPME and GC–tandem mass spectrometry. *Anal. Bioanal. Chem*. 2006, **384**, pp. 475-487

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