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Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry

Qualité du sol — Lignes directrices pour l'identification des composés cibles par chromatographie en phase gazeuse et spectrométrie de masse

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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Introduction

In many analytical standards, use is made of gas chromatography (GC) in combination with mass spectrometric (MS) detection. This detector is a powerful tool provided it is properly used. In this International Standard, guidelines are given for the identification of target compounds. This International Standard can be used in combination with specific analytical standards or in combination with any GC-MS procedure. The result of the procedure described is: identified, indicated or absent.

Soil quality — Guidelines for the identification of target compounds by gas chromatography and mass spectrometry

1 Scope

This International Standard gives criteria for gas chromatography and mass spectrometry (GC-MS) identification of target compounds in soil samples. This International Standard is intended for use with standards developed for the determination of specific compounds. The identification criteria are based on the comparison of retention times followed by interpretation of the electron ionization mass spectra, or if necessary, additional mass spectrometric techniques and other relevant factors.

NOTE This International Standard is also applicable for other environmental samples.

2 Principle

A target compound is identified if the measured values meet the criteria specified in this International Standard or in the standard in which the procedures are described to analyse the target compound. Criteria are based on the relative retention times and the intensity of diagnostics ions selected in the scan mode and measured in the selected ion mode (SIM), and other relevant factors. Additional information regarding diagnostic ions from specific international standards on the analysis of the target compound can be used. The principle of identification points is used.

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1
target compound
selected component, the presence or absence of which is being established

NOTE This definition also applies to derivatives of the original compound which are formed during an intentional derivatization procedure or on-line derivatization.

3.2
standard compound
target compound with the highest possible purity, which can be used as a reference during the analysis

NOTE Any impurities should not have any influence on the mass spectrum of the standard compound.

3.3
retention time standard
compound that is added to the sample (or to the sample extract) and to the calibration standard solution, and used to calculate the relative retention times of the target compounds

NOTE The retention time standard may be identical to the internal standard(s).

3.4
relative retention time
ratio between the retention time of the target compound and the retention time of the retention time standard

3.5
lowest concentration for identification
lowest concentration of the target compound, which, if present in the sample, can be identified using the identification criteria in this International Standard

NOTE 1 It requires that the selected diagnostic ion with the lowest intensity is still present in the mass spectrum with a signal to noise ratio (S/N) higher than 3:1.

NOTE 2 This concentration is very dependent on the sensitivity of the instrument and on the performance characteristics of the analytical method.

3.6
diagnostic ion
selected fragment ion, molecular ion or other characteristic ion from the mass spectrum of the target compound with the highest possible specificity

3.7
identification point
result of mass spectrometric investigation or other investigations/information to identify a component in environmental matrices

3.8
selected ion mode
SIM
measuring the intensity of selected diagnostic ions only

4 Apparatus

As this International Standard is complementary to other standards using GC-MS, it is assumed that the instrumentation used meets the requirements of those standards and a detailed description is not within the scope of this International Standard. Suitable quality assurance requirements are set out in ISO/IEC 17025.

Minimum requirements are:

Ionization mode:	Electron ionization.
Electron energy:	Depending on the application (usually 70 eV).
Mass range:	Peaks (masses) with a S/N < 3 are not taken into consideration and the scan range is limited to 35 (to avoid the measurement of oxygen and nitrogen) to the highest mass of the target compound + 10 unified atomic mass units (u) in full scan measurements.
Scan rate:	The scan rate should be 10 times the peak frequency with a minimum of 7 scans per peak.
Scan mode:	Cyclic, linear. Full scan or selected ion monitoring.
Mass resolution:	To be tuned on nominal resolution, the peak width at half-height of every tune mass should not exceed 0,7 u.

5 Procedure

5.1 Retention times

The relative retention time of the target compound shall be determined using a calibration standard solution containing an appropriate number of internal standards. The use of internal standards is often prescribed in the specific standard describing the determination of the target compound. The relative retention times are calculated using the retention time standard(s). The calculated relative retention time shall have a value below 2.

5.2 Mass spectra, selection of diagnostic ions

If available, three diagnostic ions shall be selected for each target compound. Their intensities I_1, I_2, I_3 shall be determined in the calibration standard solution (at least three injections) as the peak area or peak height of the corresponding extracted ion current chromatograms. The relative intensities are calculated as the ratio of the determined peak heights (or areas) and the peak height (or area) of the most intensive diagnostic ion. Annex A gives a table of suitable diagnostic ions for a range of substances. Diagnostic ions may also be specified in the standard method being used.

It is not always possible to obtain three diagnostic ions (for instance, polycyclic aromatic hydrocarbons). In that case, select the available ions.

Diagnostic ions should have a high "uniqueness value" [3]. It is suggested that:

— high m/z values should be preferred due to their higher significance;

— even mass fragments are preferred over odd ones;

— if possible, the molecular ion should be selected as one of the diagnostic ions;

— the intensity of diagnostic ions is preferably higher than 15 % in relation to the base peak in the spectrum;

— if characteristic isotope clusters are present in the mass spectrum (e.g. chlorine), two diagnostic ions should be selected from one isotope cluster. Isotopes can be very characteristic for complex compounds, i.e. organotin;

— if during the sample preparation, the target compounds have been derivatized with a reagent with low specificity, only one of the ions M^+ and $[M\text{-der}]^+$ should be selected as a diagnostic ion (M^+ is the molecular ion of the derivatized target compound);

— in the selection of the diagnostic ions, possible column artefacts have to be taken into consideration, avoiding corresponding masses (e.g. m/z 73, 207, 281);

The peak shape and retention time of all measured diagnostic ions shall be identical. Co-eluting substances may influence the peak shape. As long as the peak of interest can be separately integrated, it may be used. Criteria for the retention time are the peak maxima of the extracted ion current chromatograms.

Diagnostic ions are supposed to originate from the analyte under investigation only. This implies that theoretically all diagnostic ions belonging to one and the same analyte have the same retention time. If the retention time of one selected diagnostic ion differs from the retention times of the other diagnostic ions from the same analyte, a co-eluting substance or a partly-separated substance giving the same mass may be present. In this case, the particular diagnostic ion cannot be used.

The accuracy of the retention time depends on the number of scans within the chromatographic peak and hence, on the scan rate. Because the scan rate is limited, small differences in the retention times of the diagnostic ions should be allowed.

A suitable criterion for the allowed difference in retention times of all diagnostic ions of an analyte shall not be greater than 20 % of the peak width at half the peak height. Therefore, the differences in retention times of the peak maxima of all the selected diagnostic ions in the extracted ion current chromatograms belonging to the same analyte shall not be greater than 20 % of the peak width at half the peak height. For most analyses, this means an acceptable difference of 1 s. These criteria apply for both the calibration standard solution and the sample.

NOTE 1 MTBE and TAME have m/z 73 as diagnostic ion.

NOTE 2 Due to overloading, the ratios of the diagnostic ions can change.

6 Qualification

6.1 GC-MS procedure

The procedure to qualify a component consists of three steps (see the flow scheme in Figure 1).

— **Step 1**: Gas-chromatographic result

The relative retention time shall fulfil the specified criteria (see 6.3, Step 1). Only if Step 1 is positive, can Step 2 be made.

— **Step 2**: Gathering identification points using analytical procedures

For qualification, the principle of identification points is used [1]. Identification points can be obtained from mass spectrometric data, but also using other analytical information.

— **Step 3**: Gathering additional identification points using knowledge and interpretation of this knowledge about the sample or sampling site.

Then the following classification can be obtained.

a) *Identification* (see 6.3.1)

The target compound is present in the analysed extract. At least three identification points are obtained.

b) *Indication* (see 6.3.2)

The target compound may be present. One or two identification points are obtained.

c) *Absence* (below the detection limit) (see 6.3.3)

No identification points are obtained using mass spectrometry.

First, the mass spectrometric results are evaluated. For every ion peak meeting the criteria given in 6.3, Step 2, an identification point is obtained. Three identification points give a positive identification. If less then three ion peaks are available [due to sensitivity (S/N < 3) or absence of fragments (PAH)], additional identification points shall be gathered using additional evidence. Possibilities are given in Table 1 and also explained in Annex B.

Table 1 — Examples of number of identification points, provided criteria are met

Source	Identification points *n*	Remark
Diagnostic ion	1	Every ion S/N > 3
Absence of any other ions in full scan	1	Diagnostic ions in full scan S/N > 3
Column with other polarity [a]	1	GC-criterion (extra retention time value)
Isotope dilution	1	
Component spike/standard addition	1	
Chromatographic pattern	1	i.e. PCB, PAH, dioxins
Other analytical techniques	1	Every other selective detector (i.e. ECD) or technique (i.e. LC)
GC-MS (EI and CI; positive/negative)	3	1 (EI) + 2 (CI)
GC-MS-MS	4	1 precursor and 2 daughters (product ion)
HR-MS (high resolution MS)	2	Every ion S/N > 3
Expectation, plausibility, earlier investigations	1	See 6.2
NOTE More examples with different techniques are found in Reference [1].		
[a] Not valid for non-separated compounds (isomers) with the same mass (chrysene/triphenylene, *m/p*-xylene).		

6.2 Additional information

Interpretation of environmental data is always a combination of data analyses, knowledge about the origin of the sample, knowledge on the behaviour of contaminants and processes that occur or may occur. This is also true for the interpretation of GC-MS analysis. As stated, a component is identified if 3 identification points are obtained. If only 1 or 2 diagnostic ions are present, additional identification points are necessary. In this International Standard, gathering additional identification points using analytical procedures is part of Step 2. Using information about the sample, and interpretation of this information, takes place in Step 3. An extra identification point is obtained if one or more of the following criteria is fulfilled.

NOTE Strictly taken, an identification point obtained in Step 3 is of another order than the identification points obtained in Step 1 and Step 2. They are obtained by interpretation of additional non-analytical information. In this International Standard, the term "identification point" (3.7) is used for the points obtained in all three steps.

Step 2: Gathering identification points using analytical procedures.

— No other ions are visible in full scan mode and this is in agreement with the mass spectrum of the pure component (for instance, PAH).

— Identification is in agreement with the chromatographic pattern normally present or present on that site (for instance, PCB or PAH).

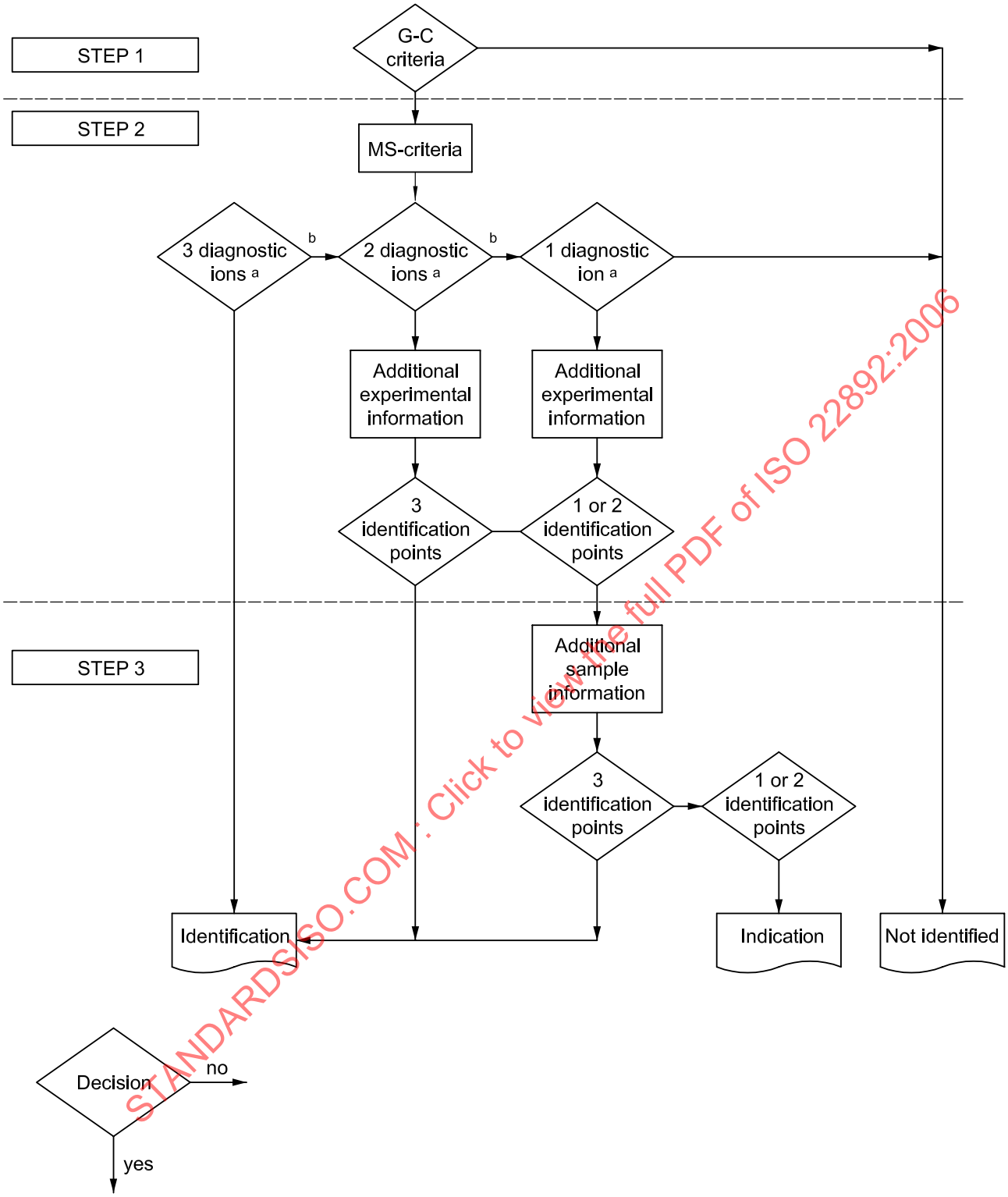
— For volatile compounds, the specificity of the mass fragments in combination with their retention time will generally be sufficient. Their volatility corresponds to a low molecular mass, limiting the number of possible false positive results: there are no low molecular mass compounds with the same retention time on a GC column and also having similar mass spectra.

Step 3: Gathering additional identification points using knowledge and interpretation of this knowledge about the sample or sampling site. If identification points are obtained using Step 3, this shall be reported.

— The component is identified in earlier samples from the same site (for instance, if the sample under investigation has a low concentration and one or two diagnostic ions have S/N < 3 following the biodegradation.

— From historical investigation, it was shown that presence of the component was expected.

— Other samples from the same site give positive identification.



a A diagnostic ion must be present with S/N > 3

b Only allowed if the missing ion has the smallest intensity and S/N < 3, otherwise not identified

Figure 1 — Flow scheme for the identification of a target compound using GC-MS

6.3 Reporting the presence of target compounds

6.3.1 Identification

The analysed target compound is identified if:

Step 1

The relative or absolute retention time measured in the sample shall meet one of the following criteria, when compared to the times measured in the last or following calibration standard solution:

— absolute retention time shall not differ more than 1 s if the absolute retention time is less than 500 s; or,

— relative retention time shall not differ more than ± 0,2 % relative if the absolute retention time is greater than 500 s and less than 5 000 s; or,

— absolute retention time shall not differ more than 6 s if the absolute retention time is greater than 5 000 s; or,

— as otherwise specified in the specific standard in use.

NOTE Absolute retention time criteria (1 s and 6 s) are added because the window of ± 0,2 % is too restrictive at the very beginning of the chromatogram (components will not be identified), and at the end of the chromatogram not specific enough (too many components will be identified).

Step 2

— The relative intensities (relative to the diagnostic peak having the highest response in the calibration standard solution) of all the selected diagnostic ions measured in the sample do not deviate by more than $\pm (0{,}1 \times I_{std} + 10)$ % from the relative intensities determined in the calibration standard solution.

— I_{std} is the relative intensity of the diagnostic ion in the calibration standard solution.

Every diagnostic ion gives 1 identification point. At least 3 points must be obtained in order to achieve identification.

EXAMPLE 1 Identification with GC-MS (three diagnostic ions).

Three selected diagnostic ions satisfy the criteria for identification, as also explained in Annex B.

A total of 3 identification points is obtained.

Step 3

Extra identification points can be gathered as described in Table 1 and Annex B.

EXAMPLE 2 Identification with GC-MS and additional evidence (low concentration).

The most sensitive diagnostic ion is present (S/N > 3)	1 identification point
Column with other polarity (Step 1)	1 identification point
Expected value	1 identification point

EXAMPLE 3 Identification with GC-MS and additional evidence (only 1 diagnostic ion, for instance PAH).

Diagnostic ion with S/N > 3 is present	1 identification point
Pattern of PAH recognized	1 identification point
Result of LC-FLD	1 identification point

6.3.2 Indication

There is an indication for the presence of the analysed target compound in the sample if:

Step 1

The criteria given in 6.3.1 Step 1 are met.

Step 2 and Step 3

Only one or two identification points are obtained. At least one must be based on the GC-MS data.

6.3.3 Absence of the target compound (< detection limit)

The target compound is absent in the sample (not identified and no indication for its presence), if:

— **Step 1**

The criteria given in 6.3.1 Step 1 are not met.

— **Step 2**

The criteria given in 6.3.1 Step 1 are met and no identification points are obtained with MS, providing the proper GC-MS conditions have been used.

7 Test report

The test report in addition to the specifications given in the analytical International Standard, shall contain the following information:

a) a reference to this International Standard, i.e. "ISO 22892:2006";

b) complete identification of the sample;

c) the results of the identification procedure, i.e. *identification*, *indication* or *absence* as performed according to 6.3 of this International Standard;

d) the source of the additional non-analytical identification points (Step 3) which have been used;

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

Annex A
(informative)

Diagnostic ions to be used for identification using GC-MS

Table A.1 gives a list of recommended diagnostic ions. The relative intensities are given in parentheses when ion trap detection is used. These ratios depend on the equipment used and can only be used as an indication. Therefore the ratios are to be determined on each single machine by the user. The precision of the masses used depends on the equipment, and more digits can be necessary.

Table A.1 — List of recommended diagnostic ions

Compound	CAS RN [a]	Diagnostic ion 1 *m/z*	Diagnostic ion 2 *m/z*	Diagnostic ion 3 [b] *m/z*
Volatile compounds				
MTBE	1634-04-4	73 (100)	57 (9)	
TAME	994-05-08	73 (100)	55 (24)	87 (23)
Dichloromethane	75-09-2	84 (33)	86 (7)	49 (100)
Dichlorodifluoromethane	75-01-04	85 (100)	87 (31)	
Vinylchloride	75-71-8	62 (100)	64 (32)	
1,1-Dichloroethane	75-34-3	63 (100)	65 (36)	83 (25)
1,2-Dichloroethane	107-06-2	62 (100)	64 (34)	98 (8)
1,1-Dichloroethene	75-35-4	96 (75)	98 (45)	61(100)
Z-1,2-Dichloroethene	156-59-2	96 (91)	98 (63)	61 (100)
E-1,2-Dichloroethene	156-60-5	96 (94)	98 (63)	61 (100)
1,1-Dichloropropane	78-99-9	77 (100)	79 (33)	78 (10)
1,2-Dichloropropane	78-87-5	62 (85)	63 (76)	76 (100)
1,3-Dichloropropane	142-28-9	76 (100)	78 (33)	63 (7)
2,2-Dichloropropane	594-20-7	77 (100)	79 (33)	97 (27)
1,1-Dichloropropene	563-58-6	75 (100)	110 (45)	77 (31)
Z-1,3-Dichloropropene	10061-01-5	75 (100)	110 (7)	77 (36)
E-1,3-Dichloropropene	10061-02-6	75 (100)	110 (7)	77 (36)
2,3-Dichloropropene-1	78-88-6	75 (100)	110 (23)	77 (33)
Benzene	71-43-2	78 (100)	77 (41)	
Toluene	108-88-3	91 (100)	92 (32)	
Ethylbenzene	100-41-4	91 (100)	106 (21)	
o-Xylene	95-47-6	91 (100)	106 (33)	105 (16)
m-Xylene	108-38-3	91 (100)	106 (33)	105 (20)
p-Xylene	106-42-3	91 (100)	106 (33)	105 (20)

Table A.1 (*continued*)

Compound	CAS RN [a]	Diagnostic ion 1 *m/z*	Diagnostic ion 2 *m/z*	Diagnostic ion 3 [b] *m/z*
Polycyclic aromatic hydrocarbons (PAH)				
Naphthalene	91-20-3	128 (100)	102 (11)	-
Acenaphthene	83-32-9	154 (70)	153 (100)	76 (10)
Acenaphthylene	208-96-8	152 (100)		
Fluorene	86-73-7	166 (81)	165 (100)	
Anthracene	120-12-7	178 (100)	152 (12)	
Phenanthrene	85-01-8	178 (100)	152 (9)	
Fluoranthene	206-44-0	202 (100)	200 (31)	
Pyrene	129-00-0	202 (100)		
Benz[*a*]anthracene	56-55-3	228 (100)		
Chrysene	218-01-9	228 (100)	226 (6)	
Benzo[*b*]fluoranthene	205-99-2	252 (100)	250 (22)	
Benzo[*k*]fluoranthene	207-08-9	252 (100)	250 (22)	
Benzo[*a*]pyrene	50-32-8	252 (100)	250 (18)	[113] (11)
Indeno[1,2,3-*cd*]pyrene	193-39-5	276 (100)	138 (12)	
Dibenz[*a,h*]anthracene	53-70-3	278 (100)	139 (9)	
Benzo[*ghi*]perylene	191-24-2	276 (100)	138 (12)	
Organochlorine pesticides (OCP)				
β-Endosulfan	33213-65-9	195 (100)	241 (85)	159 (56)
Mirex	2385-85-5	272 (100)	237 (88)	119 (37)
Hexachlorobenzene (HCB)	118-74-1	284 (100)	142 (22)	249 (24)
α-Hexachlorocyclohexane (α-HCH)	319-84-6	181 (100)	219 (33)	109 (29)
β-Hexachlorocyclohexane (β-HCH)	319-85-7	181 (97)	219 (54)	109 (49)
γ-Hexachlorocyclohexane (γ-HCH)	58-89-9	181 (97)	219 (34)	109 (33)
Aldrin	309-00-2	66 (100)	263 (78)	293 (41)
Dieldrin	60-57-1	79 (100)	263 (70)	277 (18)
Endrin	72-20-8	81 (100)	263 (70)	245 (55)
Heptachlor	76-44-8	100 (100)	65 (65)	272 (89)
cis-Heptachloro epoxide	28044-83-9	253 (100)	183 (90)	289 (85)
trans-Heptachloro epoxide	1024-57-3	353 (100)	81 (67)	263 (26)
α-Endosulfan	959-98-7	195 (100)	159 (93)	265 (55)
p,p'-DDE	72-55-9	246 (100)	318 (37)	176 (36)
o,p'-DDD	53-19-0	235 (100)	165 (66)	199 (29)
o,p'-DDT	789-02-6	235 (100)	165 (67)	199 (27)
p,p'-DDD	72-54-8	235 (100)	165 (66)	199 (20)
o,p'-DDE	3424-82-6	246 (100)	318 (37)	176 (27)
p,p'-DDT	50-29-3	235 (100)	165 (68)	199 (20)

Table A.1 (*continued*)

Compound	CAS RN [a]	Diagnostic ion 1 *m/z*	Diagnostic ion 2 *m/z*	Diagnostic ion 3 [b] *m/z*
Polychlorinated biphenyls (PCB)				
PCB 28	7012-37-5	186 (100)	258 (74)	256 (82)
PCB 52	35693-99-3	292 (100)	294 (49)	220 (95)
PCB101	37680-73-2	326 (100)	328 (65)	256 (62)
PCB 118	31508-00-6	326 (100)	328 (62)	254 (57)
PCB 138	35065-28-2	290 (100)	358 (42)	360 (94)
PCB 153	35065-27-1	360 (100)	362 (92)	290 (73)
PCB 180	35065-29-3	394 (100)	396 (96)	324 (84)
Herbicides				
Atrazine	1912-24-9	200 (100)	202 (36)	215 (41)
Simazine	122-34-9	138 (62)	186 (66)	201 (100)
Acylated chlorophenols [c]				
Mono		128 (100)	170 (6)	100 (13)
Di		162 (100)	133 (10)	
Tri		196 (100)	238 (6)	160 (25)
Tetra		230 (100)	194 (15)	
Penta		264 (100)		

[a] CAS RN: Chemical Abstracts Service Registration Number.

[b] The choice of ion 3 (*m/z*) depends on the applied mass spectrometric method: quadrupole or ion trap detection, because of the formation of different fragment ions.

[c] Taken from Reference [4]. The relative intensities may vary between mass isomers.

Annex B
(informative)

Examples

B.1 Example 1, β-endosulfan

Compound: β-endosulfan, diagnostic ions 195, 241, 159.

The three selected diagnostic ions have the following relative intensities: 100 %, 80 % and 56 %.

B.1.1 Identification

B.1.1.1 Example based on relative retention time

Table B.1 — Retention times and relative retention times

	Calibration standard 1		Sample 1	
	Retention time s	Relative retention time	Retention time s	Relative retention time
Retention time standard (anthracene-d10)	1 327	—	1 325	—
Ion 1 (*m/z* 195)	1 677	1,264	1 678	1,266
Ion 2 (*m/z* 241)	1 678	1,265	1 677	1,266
Ion 3 (*m/z* 159)	1 677	1,264	1 678	1,266

The relative retention time of ion 1 (195) in sample 1 differs from the relative retention time of calibration standard 1 by – 0,16 %.

$$(1{,}264 - 1{,}266)/1{,}264 \times 100\ \% = -0{,}16\ \%$$

The relative retention time of ion 2 (241) in sample 1 differs from the relative retention time of calibration standard 1 by – 0,08 %.

$$(1{,}265 - 1{,}266)/1{,}265 \times 100\ \% = -0{,}08\ \%$$

The relative retention time of ion 3 (159) in sample 1 differs from the relative retention time of calibration standard 1 by – 0,16 %.

$$(1{,}264 - 1{,}266)/1{,}264 \times 100\ \% = -0{,}16\ \%$$

Each of these differences is less than ± 0,2 % from relative retention time in the last measured calibration standard solution.

B.1.1.2 Example based on maximum allowed deviation of the relative intensities

Table B.2 — Peak area

	Peak area ion 1 ($\times 10^4$)	Peak area ion 2 ($\times 10^4$)	Peak area ion 3 ($\times 10^4$)
Calibration standard 1	22,9	18,3	12,8
Sample 1	5,31	4,25	2,92

Table B.3 — Relative intensity of ion2/ion1 and ion3/ion1 based on peak area

	Relative intensity peak area ion 2/ion 1	Relative intensity peak area ion 3/ion 1
Calibration standard 1	0,80	0,56
Sample 1	0,80	0,55

The relative intensity of ion 2/ion 1, based on peak area of sample 1 is: 4,25/5,31 (see Table B.2) = 0,80.

The relative intensity of ion 3/ion 1, based on peak area of sample 1 is: 2,92/5,31 (see Table B.2) = 0,55.

The deviation of I_2 in sample 1 (based on peak area) is: (0,80 − 0,80)/0,80 × 100 % = 0 %.

The deviation of I_3 in sample 1 (based on peak area) is: (0,56 − 0,55)/0,56 × 100 % = 1,8 %.

The maximum allowed deviation of I_2 is: ± (0,1 × 80 + 10) % = ± 18 %.

The maximum allowed deviation of I_3 is: ± (0,1 × 56 + 10) % = ± 15,6 %.

B.1.1.3 Conclusion

The criteria for both retention time (B.1.1.1) and ion relative intensities (B.1.1.2) have been met. In this case, the target compound is considered as being positively identified.

B.1.2 Indication

B.1.2.1 Example based on relative retention time

Table B.4 — Retention times and relative retention times

	Calibration standard 2		Sample 2	
	Retention time s	Relative retention time	Retention time s	Relative retention time
Retention time standard (anthracene-d10)	1 328	—	1 329	—
Ion 1 (*m/z* 195)	1 680	1,265	1 680	1,264
Ion 2 (*m/z* 241)	1 681	1,265	1 679	1,263
Ion 3 (*m/z* 159)	1 680	1,265	1 680	1,264

The relative retention time of ion 1 (195) in sample 2 differs from the relative retention time in calibration standard 2 by – 0,08 %.

$$(1{,}264 - 1{,}265)/1{,}265 \times 100\ \% = -0{,}08\ \%$$

The relative retention time of ion 2 (241) in sample 2 differs from the relative retention time in calibration standard 2 by – 0,15 %.

$$(1{,}263 - 1{,}265)/1{,}265 \times 100\ \% = -0{,}15\ \%$$

The relative retention time of ion 3 (159) in sample 2 differs from the relative retention time in calibration standard 2 by – 0,08 %.

$$(1{,}264 - 1{,}265)/1{,}265 \times 100\ \% = -0{,}08\ \%$$

Each of these differences is less than the allowable ± 0,2 % from relative retention time in the last measured calibration standard solution.

B.1.2.2 Example above the lowest concentration for identification

All of the selected diagnostic ions are present, but the deviation in the relative intensity does not satisfy the criteria for identification.

Table B.5 — Peak area

	Peak area ion 1 ($\times 10^4$)	Peak area ion 2 ($\times 10^4$)	Peak area ion 3 ($\times 10^4$)
Calibration standard 2	13,3	9,75	12,8
Sample 2	1,42	1,01	1,02

Table B.6 — Relative intensity of ion2/ion1 and ion3/ion1 based on peak area

	Relative intensity peak area ion 2/ion 1	Relative intensity peak area ion 3/ion 1
Calibration standard 2	0,73	0,96
Sample 2	0,71	0,72

The deviation of I_2 in sample 2 (based on peak area) is: (0,71 – 0,73)/0,73 × 100 % = – 2,7 %.

The deviation of I_3 in sample 2 (based on peak area) is: (0,72 – 0,96)/0,96 × 100 % = – 25 %.

The maximum allowed deviation of I_2 is: ± (0,1 × 80 + 10) % = ± 18 %.

The maximum allowed deviation of I_3 is: ± (0,1 × 56 + 10) % = ± 15,6 %.

B.1.2.3 Conclusion

The criteria for positive identification have not been met, but there is an indication for the presence of the analysed compound in sample 2.

B.1.3 Negative result

Table B.7 — Retention times and relative retention times

	Calibration standard		Sample	
	Retention time s	Relative retention time	Retention time s	Relative retention time
Retention time standard (anthracene-d10)	1 327	—		—
Ion 1 (m/z 195)	1 677	1,264	not found	0
Ion 2 (m/z 241)	1 678	1,265	not found	0
Ion 3 (m/z 159)	1 677	1,264	not found	0

B.2 Example 2, Fluoranthene

Fluoranthene, diagnostic ions 202, 200, 100

The three selected diagnostic ions have the following relative intensities: 100 %, 18 % and 7 %.

B.2.1 Identification

B.2.1.1 Based on relative retention time

Table B.8 — Retention times and relative retention times

	Calibration standard 1		Sample 1	
	Retention time s	Relative retention time	Retention time s	Relative retention time
Retention time standard (anthracene-d10)	1 321	—	1 321	—
Ion 1 (*m/z* 202)	1 539	1,165	1 540	1,166
Ion 2 (*m/z* 200)	1 539	1,165	1 540	1,166
Ion 3 (*m/z* 100)	1 539	1,165	1 541	1,167

The relative retention time of ion 1 (202) in sample 1 differs from the relative retention time in calibration standard 1 by 0,09 %.

$$(1{,}166 - 1{,}165)/1{,}165 \times 100\ \% = 0{,}09\ \%$$

The relative retention time of ion 2 (200) in sample 1 differs from the relative retention time in calibration standard 1 by 0,09 %.

$$(1{,}166 - 1{,}165)/1{,}165 \times 100\ \% = 0{,}09\ \%$$

The relative retention time of ion 3 (100) in sample 1 differs from the relative retention time in calibration standard 1 by 0,17 %.

$$(1{,}167 - 1{,}165)/1{,}165 \times 100\ \% = 0{,}17\ \%$$

This is less than ± 0,2 % from relative retention time in the last measured calibration standard solution.

B.2.1.2 Based on maximum allowed deviation of the relative intensities

Table B.9 — Peak area

	Peak area ion 1 ($\times 10^4$)	Peak area ion 2 ($\times 10^4$)	Peak area ion 3 ($\times 10^4$)
Calibration standard 1	29,74	5,48	0,18
Sample 1	18,16	3,09	1,30

Table B.10 — Relative intensity of ion2/ion1 and ion3/ion1 based on peak area

	Relative intensity peak area ion 2/ion 1	Relative intensity peak area ion 3/ion 1
Calibration standard 1	0,18	0,07
Sample 1	0,17	0,07

The relative intensity of ion 2/ion 1, based on peak area of sample 1 is: 3,09/18,16 (see Table B.8) = 0,17.