SIST

SLOVENSKI STANDARD
SIST EN ISO 6468:1998

01-januar-1998

Kakovost vode - Določevanje nekaterih organoklornih insekticidov, polikloriranih bifenilov in klorobenzenov - Plinska kromatografska metoda po tekočinski ekstrakciji (ISO 6468:1996)

Water quality - Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes - Gas chromatographic method after liquid-liquid extraction (ISO 6468:1996)

Wasserbeschaffenheit - Bestimmung bestimmter Organochlorinsektizide, Polychlorbiphenyle und Chlorbenzole - Gaschromatographisches Verfahren nach Flüssig -Flüssig-Extraktion (ISO 6468:1996)

Qualité de l'eau - Dosage de certains insecticides organochlorés des polychlorobiphényles et des chlorobenzenes - Méthode par chromatographie en phase gazeuse apres extraction liquide-liquide (ISO 6468:1996)

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Water quality - Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes - Gas chromatographic method after liquid-liquid extraction (ISO 6468:1996)

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard ISO 6468:1996 has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with 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of an identical text or by endorsement, at the latest by June 1997, and conflicting national standards shall be withdrawn at the latest by June 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 6468:1996 was approved by CEN as a European Standard without any modification.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

Annex ZA (normative)
Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

Publication	Year	Title	EN	Year
ISO 5667-1	1980	Water quality - Sampling - Part 1: Guidance on the design of sampling programmes	EN 25667-1	1993
ISO 5667-2	1991	Water quality - Sampling - Part 1: Guidance on sampling techniques	EN 25667-2	1993

INTERNATIONAL STANDARD

ISO 6468

First edition
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Water quality — Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes — Gas chromatographic method after liquid-liquid extraction

Qualité de l'eau — Dosage de certains insecticides organochlorés, des polychlorobiphényles et des chlorobenzènes — Méthode par chromatographie en phase gazeuse après extraction liquide-liquide

Reference number
ISO 6468:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 6468 was prepared by Technical Committee ISO/TC 147, *Water quality,* Subcommittee SC 2, *Physical, chemical, biochemical methods.*

Annex A forms an integral part of this International Standard. Annexes B to H are for information only.

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Water quality — Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes — Gas chromatographic method after liquid-liquid extraction

WARNING AND SAFETY PRECAUTIONS — This method makes use of flammable and toxic organic solvents. Observe the safety regulations in effect.

The electron-capture detector (ECD) contains radionuclides. Adequate safety precautions and legal requirements must be observed.

The halogenated hydrocarbons and chloropesticides, used for the preparation of the calibration standards are toxic. Therefore, the safety regulations pertaining must be strictly observed.

1 Scope

This International Standard describes a method for determining certain organochlorine insecticides, polychlorinated biphenyls (PCBs) and chlorobenzenes (except the mono- and dichlorobenzenes) in drinking waters, ground waters, surface waters and waste waters.

The method is applicable to samples containing up to 0,05 g/l of suspended solids. In the presence of organic matter, suspended matter and colloids, interferences are more numerous and consequently the detection limits are higher.

The method described in this International Standard only gives information on specific PCB compounds but no information on the level of total PCBs.

According to the types of compounds to be detected and the source of the water, the detection limits given in table 1 are applicable for the method described in this International Standard, with waters of low organic contents.

Given the very low concentrations normally present in the waters, the problem of contamination is extremely important. The lower the level measured, the more precautions have to be observed; below concentrations of 10 ng/l, special care is necessary.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design on sampling programmes.*

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

3 Principle

Liquid-liquid extraction of organochlorine insecticides, chlorobenzenes and PCBs by an extraction solvent. After the concentration of the components with low volatility and after any clean-up steps which may be necessary, the sample extracts are analysed by gas chromatography, using an electron-capture detector.

Table 1 — Detection limits

Acronyms	Chemical names (IUPAC)	Detection limits
Organochlorine insecticides:		
HCH	1, 2, 3, 4, 5, 6-hexachlorocyclohexane, five stereoisomers: alpha-HCH, beta-HCH, gamma-HCH, delta-HCH, epsilon-HCH	1 ng/l to 10 ng/l depending on the compound
Lindane		
o,p′-DDE	1,1-dichloro-2-(2-chlorophenyl 1)-2-(4-chlorophenyl)ethylene	
p,p′-DDE	1,1-dichloro-2,2-bis(4-chlorophenyl)ethylene	
o,p′-TDE	1,1-dichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethane (= *o,p′*-DDD)	
p,p′-TDE	1,1-dichloro-2, 2-bis(4-chlorophenyl)ethane (= *p,p′*-DDD)	
o,p′-DDT	1,1,1-trichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethane	
p,p′-DDT	1,1,1-trichloro-2,2-bis(4-chlorophenyl)ethane	
Methoxychlor	1,1,1-trichloro-2,2-bis(4-methoxyphenyl)ethane	
Aldrin	(1*R*, 4*S*, 4a*S*, 5*S*, 8*R*, 8a*R*)-1, 2, 3, 4, 10, 10-hexachloro-1, 4, 4a, 5, 8, 8a-hexahydro-1, 4 : 5,8-dimethanonaphthalene	
Dieldrin	(1*R*, 4*S*, 4a*S*, 5*R*, 6*R*, 7*S*, 8*S*, 8a*R*)-1, 2, 3, 4, 10, 10-hexachloro-1, 4, 4a, 5, 6, 7, 8, 8a-octahydro-6, 7-epoxy-1, 4 : 5,8-dimethanonaphthalene	
Endrin	(1*R*, 4*S*, 4a*S*, 5*S*, 6*S*, 7*R*, 8*R*, 8a*R*)-1, 2, 3, 4, 10, 10-hexachloro-1, 4, 4a, 5, 6, 7, 8, 8a-octahydro-6, 7-epoxy-1, 4 : 5,8-dimethanonaphthalene	
Heptachlor[1]	1, 4, 5, 6, 7, 8, 8-heptachloro-3a, 4, 7, 7a-tetrahydro-4, 7-methanoindene[1]	
Heptachlor-epoxide	1, 4, 5, 6, 7, 8, 8-heptachloro-2,3-epoxy-3a,4,7,7a-tetrahydro-4,7-methanoindane	
Endosulfan[1) 2)]	1, 4, 5, 6, 7, 7, 7-hexachloro-8, 9, 10-trinorborn-5-en-2, 3-ylene-dimethyl-enesulfite: alpha-Endosulfan, beta-Endosulfan	
Chlorobenzenes:		
TrCB	trichlorobenzene	1 ng/l to 10 ng/l depending on the compound
TeCB	tetrachlorobenzene	
PeCB	pentachlorobenzene	
HCB	hexachlorobenzene	
PCNB (Quintozene)	pentachloronitrobenzene	
Polychlorinated biphenyls:		
PCB 28	2,4,4′-trichlorobiphenyl	1 ng/l to 50 ng/l depending on the compound
PCB 52	2,2′,5,5′-tetrachlorobiphenyl	
PCB 101	2,2′,4,5,5′-pentachlorobiphenyl	
PCB 138	2,2′,3,4,4′,5′-hexachlorobiphenyl	
PCB 153	2,2′,4,4′,5,5′-hexachlorobiphenyl	
PCB 180	2,2′,3,4,4′,5,5′-heptachlorobiphenyl	
PCB 194	2,2′,3,3′,4,4′,5,5′-octachlorobiphenyl	

1) The analysis of α and β - endosulfan as well as heptachlor requires special care due to its low stability.

2) The name "endosulfan" is not acceptable for use in Italy, as it is in conflict with a trade mark registered there.

Any substance capable of producing a response on the electron-capture detector, at a retention time indistinguishable from any compound of interest, will interfere. In practice, many potentially interfering substances will be removed during the extraction and clean-up procedures.

NOTE 1 In general, the use of two capillary columns of different polarity is sufficient for the organochlorine compounds analysed according to this International Standard. The results so calculated should be considered as the maximum concentrations, possibly still influenced by coeluting substances. It is possible that there will be cases where a more definite identification is required.

4 Reagents and materials

All reagents shall be sufficiently pure to not give rise to significant interfering peaks in the gas chromatograms of the blanks. The purity of reagents used in the procedure shall be checked by blank determinations (7.6).

NOTE 2 Commercial "pesticide grade" solvents are available. The use of these products is recommended only after verifying their quality. The quality of a solvent is checked by evaporation of about 200 ml down to 1 ml and analysis of the concentrate to determine the compounds subsequently analysed. The solvent should be considered acceptable if it does not give any detectable interfering peaks in the chromatogram for the substance of interest.

4.1 Water purified, for example, using ion-exchange or carbon-column adsorption.

4.2 Extraction solvent.

Hexane, petroleum ether or heptane are suitable.

NOTE 3 Any other solvents meeting the requirements of 8.3 (recovery rate ⩾ 60 %) may be used.

4.3 Sodium sulfate (Na_2SO_4), anhydrous.

Heat a portion of about 250 ml to 300 ml of sodium sulfate powder at 500 °C ± 20 °C for 4 h ± 30 min, cool to about 200 °C in a muffle furnace and then to ambient temperature in a desiccator containing magnesium perchlorate or an equivalent alternative.

4.4 Decane ($C_{10}H_{22}$) or **dodecane** ($C_{12}H_{26}$), or any keeper which is not detected by the electron-capture detector.

4.5 Dry alumina.

Heat a batch of inert alumina, containing particles of size 50 µm to 200 µm and of maximum mass 500 g, at 500 °C ± 20 °C for 4 h ± 30 min on a silica dish in a muffle furnace. Cool to about 200 °C in the furnace and then to ambient temperature in a desiccator. Store in a sealed glass container.

4.6 Deactivated alumina.

Weigh a portion of dry alumina (4.5) into a sealable all-glass container and add 7 % ± 0,2 % (*m*/*m*) of water (4.1). Seal and agitate for at least 2 h to ensure uniformity. Store in a sealed glass container.

Once the seal has been broken, storage time is normally about one week. After the maximum storage time, reprocess batches as described in 4.5 and this subclause.

4.7 Alumina/silver nitrate.

Dissolve 0,75 g ± 0,01 g of silver nitrate in 0,75 ml ± 0,01 ml of water (4.1) using a microburette. Add 4,0 ml ± 0,2 ml of acetone followed by 10 g ± 0,2 g of deactivated alumina (4.6). Mix thoroughly by shaking in an open-topped conical flask, protected from light. Allow the acetone to evaporate at room temperature and prevent condensation, for example by warming with the hand.

Store in the dark and use within 4 h after preparation.

4.8 Silica gel, of particle size 63 µm to 200 µm, heated at 500 °C ± 30 °C in batches not larger than 500 g, for about 14 h. Cool to about 200 °C in the furnace and then to ambient temperature in a sealed flask which is placed in a desiccator without desiccant. Use this material within one week. Deactivate the silica gel by weighing a suitable quantity of silica and adding 3 % (*m*/*m*) of water (4.1). Agitate for at least 2 h to ensure uniformity and store in a sealed glass container.

The deactivated silica gel shall be used within 24 h.

4.9 Toluene.

4.10 Diethylether, free from peroxides.

4.11 Anti-bumping granules, washed with acetone.

4.12 Standard stock solutions.

Pure or certified standards of organochlorine insecticides, chlorobenzenes, and PCBs shall be used for the preparation of standard stock solutions.

NOTE 4 Suitable solvents for the preparation of standard stock solutions are acetone, pentane, hexane, dimethylbenzene or isooctane.

The containers containing the solutions shall be marked or weighed so that any evaporation losses of the solvent may be recognized. The solutions shall be stored in volumetric flasks with ground-glass stoppers at a temperature of 4 °C in the dark. Prior to use, they shall be brought to ambient temperature and the level of solvent shall be adjusted, if necessary.

NOTE 5 A convenient concentration of standard stock solution is obtained by weighing 50 mg of each determinand and dissolving it in 100 ml of the solvent.

The solution is stable for about 1 year.

4.13 Intermediate standard solutions.

Prepare intermediate standard solutions by a suitable dilution of the stock solution (4.12) with the extraction solvent (4.2).

A typical value is 10 µg/ml.

Store the intermediate standard solutions at about 4 °C in the dark. These solutions are stable for six months.

4.14 Working standard solutions.

Prepare at least five different concentrations by suitable dilutions of the intermediate standard solutions (4.13) with the extraction solvent (4.2).

Suitable concentrations are in the nanograms per millilitre range.

Store the solutions at about 4 °C in the dark. These solutions are stable for at least one month.

4.15 Cotton wool or glass wool, washed with extraction solvent.

4.16 Water-miscible solvent.

NOTE 6 Acetone, methanol or dimethylformamide may be used.

5 Apparatus

5.1 Gas chromatograph, with an electron-capture detector (ECD) and suitable for use with capillary columns. This shall be operated in accordance with the manufacturer's instructions. On-column or glass-lined injection systems can be used. The oven shall be suitable for isothermal and temperature-programmable operation.

5.2 Capillary columns, glass or fused-silica capillaries, with an inside diameter of less than 0,4 mm and a length of 25 m to 60 m, coated with stationary phases capable of separating the compounds of interest.

Annex B provides examples of gas chromatographic conditions (tables B.1, B.2 and B.3) and the corresponding gas chromatograms (figures B.1 and B.2).

5.3 Separating funnels, of nominal capacities 1 litre to 5 litres, with a glass tap washed by hexane or a polytetrafluoroethylene (PTFE) tap.

5.4 High-speed stirrer and magnetic stirring bar, washed with hexane and coated with polytetrafluoroethylene (PTFE).

5.5 Microseparator, see example in figure C.1.

5.6 Kuderna-Danish evaporator, see example in figure D.1.

5.7 Snyder microcolumn.

5.8 Rotary evaporator or any suitable system of evaporation.

5.9 Column for drying the extract, filled with 5 g to 7 g of sodium sulfate (4.3) giving a height of about 7 cm to 10 cm. For example, the dimensions are 10 mm internal diameter and 250 mm length (see figure E.1).

5.10 Column for the alumina-alumina/silver nitrate clean-up, for example, the dimensions are 10 mm internal diameter and 250 mm length (see figure E.1).

5.11 Macrocolumn for the silica gel clean-up, for example, the dimensions are 19 mm internal diameter and 400 mm length (see figure E.1).

5.12 Microcolumn for the silica gel clean-up, for the dimensions see figure F.1.

5.13 Microlitre syringes.

5.14 Miscellaneous glassware.

Laboratory glassware shall be cleaned using a cleaning agent (laboratory detergent) followed, for example, by either a treatment with chromium(VI)/sulfuric acid mixture, or peroxodisulfate/sulfuric acid mixture and subsequently washed by hexane or heated for at least 12 h at 200 °C, except for the calibrated glassware.

The efficiency of the treatment shall be experimentally checked at random by blank determinations to ensure that no interfering contamination has occurred.