# INTERNATIONAL STANDARD

# ISO 6468

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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 | |
| **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 | 1 ng/l |
| *o,p'*-TDE | 1,1-dichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethane (= *o,p'*-DDD) | to |
| *p,p'*-TDE | 1,1-dichloro-2, 2-bis(4-chlorophenyl)ethane (= *p,p'*-DDD) | 10 ng/l |
| *o,p'*-DDT | 1,1,1-trichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)ethane | depending |
| *p,p'*-DDT | 1,1,1-trichloro-2,2-bis(4-chlorophenyl)ethane | on the |
| **Methoxychlor** | 1,1,1-trichloro-2,2-bis(4-methoxyphenyl)ethane | compound |
| **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-dimethylenesulfite: alpha-Endosulfan, beta-Endosulfan | |
| **Chlorobenzenes:** | | |
| **TrCB** | trichlorobenzene | 1 ng/l |
| **TeCB** | tetrachlorobenzene | to |
| **PeCB** | pentachlorobenzene | 10 ng/l |
| **HCB** | hexachlorobenzene | depending on |
| **PCNB (Quintozene)** | pentachloronitrobenzene | the compound |
| **Polychlorinated biphenyls:** | | |
| **PCB 28** | 2,4,4′-trichlorobiphenyl | |
| **PCB 52** | 2,2′,5,5′-tetrachlorobiphenyl | 1 ng/l |
| **PCB 101** | 2,2′,4,5,5′-pentachlorobiphenyl | to |
| **PCB 138** | 2,2′,3,4,4′,5′-hexachlorobiphenyl | 50 ng/l |
| **PCB 153** | 2,2′,4,4′,5,5′-hexachlorobiphenyl | depending on |
| **PCB 180** | 2,2′,3,4,4′,5,5′-heptachlorobiphenyl | the compound |
| **PCB 194** | 2,2′,3,3′,4,4′,5,5′-octachlorobiphenyl | |

1) The analysis of $\alpha$ and $\beta$ - 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 $\geqslant$ 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.