INTERNATIONAL STANDARD

ISO 10382

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Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection

Qualité du sol — Dosage des pesticides organochlorés et des biphényles polychlorés — Méthode par chromatographie en phase gazeuse avec détection par capture d'électrons

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10382 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Annexes A, B, C and D of this International Standard are for information only.

Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection

1 Scope

This International Standard specifies a method for quantitative determination of seven polychlorinated biphenyls and seventeen organochlorine pesticides in soil.

This International Standard is applicable to all types of soil.

Under the conditions specified in this International Standard, limits of detection of 0,1 µg/kg to 4 µg/kg (expressed as dry matter) can be achieved.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 10381-1, *Soil quality — Sampling — Part 1: Guidance on the design of sampling programmes*

ISO 10381-2, *Soil quality — Sampling — Part 2: Guidance on sampling techniques*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 14507, *Soil quality — Pretreatment of samples for the determination of organic contaminants*

3 Principle

After pretreatment, the soil test sample is extracted with a hydrocarbon solvent.

The extract is concentrated; polar compounds are removed by passing the concentrated extract through a column filled with aluminium oxide. The eluate is concentrated.

Elemental sulfur is removed from the concentrated extract, if necessary, by treatment with tetrabutylammonium sulfite reagent.

The extract is analysed by gas chromatography. The various compounds are separated using a capillary column with an immobile phase of low polarity. Detection occurs with an electron-capture detector (ECD).

Polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs) are assigned and quantified by comparison of relative retention times and relative peak heights (or peak areas) with respect to injection standards

added, with the corresponding variables of an external standard solution. The efficiency of the procedure depends on the composition of the soil that is investigated. The described procedure does not take account of incomplete extraction due to the structure and composition of the soil sample.

The limit of detection is dependent on the determinands, the equipment used, the quality of chemicals used for extraction of the soil sample, and the clean-up of the extract.

NOTE 1 For confirmation of the identity of detected compounds and the concentrations found, further investigation is necessary. Confirmation can be carried out by repeating the gas chromatographic analysis using a column of different polarity and/or using gas chromatography/mass spectrometry (GC/MS).

NOTE 2 Other non-volatile organochlorine compounds, e.g. some chlorobenzenes, can also be identified and quantified by this method.

4 Reagents

All reagents shall be of recognized analytical grade. The purity of the reagents used shall be checked by running a blank determination as described in 8.1.

4.1 Petroleum ether, boiling range 40 °C to 60 °C.

4.2 Acetone.

4.3 n-Hexane.

4.4 Diethyl ether.

Diethyl ether can contain peroxides which may oxidize some of the determinands. Check for the absence of peroxides, e.g. by shaking with a freshly prepared 10 % (mass fraction) KI solution.

4.5 Anhydrous sodium sulfate, heated for at least 6 h to 550 °C ± 20 °C, cooled to about 200 °C in a furnace and then to ambient temperature in a desiccator containing magnesium perchlorate or a suitable alternative.

The anhydrous sodium sulfate shall be kept carefully sealed.

4.6 Aluminium oxide, basic or neutral, areic mass 200 m^2/g, activity Super I according to Brockmann.

4.7 Aluminium oxide, deactivated with 10 % water.

To 90 g of aluminium oxide (4.6) add 10 g of water. Shake until all lumps have disappeared. Allow the aluminium oxide to condition before use for approximately 16 h, sealed from the air.

4.8 Silica gel, particle size 60 µm to 200 µm, deactivated with 5 % water.

Heat 95 g of silica gel for at least 24 h in an oven at 150 °C. Then allow to cool in a desiccator and add 5 g of water. Shake until all lumps have disappeared. Allow the silica gel to condition before use for approx. 16 h, sealed from the air.

For each new batch of aluminium oxide or silica gel, the elution pattern should be checked against a standard solution of PCB and OCP. If necessary, the deactivation of the adsorbent should be adjusted (see 8.4).

4.9 Standards.

4.9.1 Polychlorinated biphenyls.

PCB- 28: 2,4,4'-trichlorobiphenyl	CAS number[1]: 7012-37-5
PCB- 52: 2,2',5,5'-tetrachlorobiphenyl	CAS number: 35693-99-3
PCB-101: 2,2',4,5,5'-pentachlorobiphenyl	CAS number: 37680-73-2
PCB-118: 2,3',4,4',5-pentachlorobiphenyl	CAS number: 31508-00-6
PCB-138: 2,2',3,4,4',5'-hexachlorobiphenyl	CAS number: 35065-28-2
PCB-153: 2,2',4,4',5,5'-hexachlorobiphenyl	CAS number: 35065-27-1
PCB-180: 2,2',3,4,4',5,5'-heptachlorobiphenyl	CAS number: 35065-29-3

NOTE The numbers 28, 52, etc. correspond with the sequential numbers of chlorobiphenyls according to the IUPAC rules for the nomenclature of organic compounds.

4.9.2 Organochlorine pesticides.

Hexachlorobenzene (HCB)	CAS number: 118-74-1
α-Hexachlorocyclohexane (α-HCH)	CAS number: 319-84-6
β-Hexachlorocyclohexane (β-HCH)	CAS number: 319-85-7
γ-Hexachlorocyclohexane (γ-HCH)	CAS number: 58-89-9
Aldrin	CAS number: 309-00-2
Dieldrin	CAS number: 60-57-1
Endrin	CAS number: 72-20-8
Heptachlor	CAS number: 76-44-8
Heptachloro epoxide (exo-, *cis*- or a-isomer)	CAS number: 28044-83-9
Heptachloro epoxide (endo-, *trans*- or b-isomer)	CAS number: 1024-57-3
α-Endosulfan	CAS number: 959-98-7
p,p'-DDE	CAS number: 72-55-9
o,p'-DDD	CAS number: 53-19-0
o,p'-DDT	CAS number: 784-02-6
p,p'-DDD	CAS number: 72-54-8
o,p'-DDE	CAS number: 3424-82-6
p,p'-DDT	CAS number: 50-29-3

1) Registration used by the Chemical Abstracts Service.

4.9.3 Injection standards.

PCB-155: 2,2',4,4',6,6'-hexachlorobiphenyl CAS number: 33979-03-2

Select a second injection standard, not interfering with the analytes, from the following substances:

PCB-143: 2,2',3,4,5,6'-hexachlorobiphenyl CAS number: 68194-15-0

PCB-207: 2,2',3,3',4,4',5,6,6'-nonachlorobiphenyl CAS number: 52663-79-3

Mirex CAS number: 2385-85-5

4.10 Tetrabutylammonium reagent (TBA sulfite reagent).

Saturate a solution of tetrabutylammonium hydrogen sulfate in a mixture of equal volumes of water and 2-propanol, $c[(C_4H_9)_4NHSO_4] = 0{,}1$ mol/l, with sodium sulfite.

NOTE 25 g of sodium sulfite is normally sufficient for 100 ml of solution.

4.11 n-Heptane.

5 Apparatus

5.1 Customary laboratory glassware.

All glassware to be used shall be thoroughly cleaned, preferably in a dishwasher using a customary cleaning procedure, followed by rinsing with acetone and a subsequent rinsing with petroleum ether or hexane.

5.2 Glass sample bottles, of nominal capacity 1 l, with screw top and polytetrafluoroethene seal (PTFE).

5.3 Shaking device, with horizontal movement (200 to 300 strokes per minute).

5.4 Water bath, capable of being heated to 100 °C.

5.5 Shaking funnels, with a capacity of 2 l.

5.6 Conical flasks, with a capacity of 500 ml.

5.7 Evaporator, Kuderna Danish (see Figure 1).

Other evaporators, e.g. a rotary evaporator, may be used if found to be equally suitable.

5.8 Quartz wool or silanized glass wool, rinsed with petroleum ether or hexane.

WARNING Working with quartz wool imposes a risk to health through the release of fine quartz particles. Prevent inhalation of these by using a fume cupboard and wearing a dust mask.

5.9 Boiling chips, of glass or porcelain beads, rinsed with petroleum ether or hexane.

5.10 Calibrated test tubes, with a capacity of 15 ml and ground glass stoppers.

5.11 Chromatography tubes (see Figure 2).

5.12 Gas chromatograph, equipped with a non-discriminating injection system, capillary column and electron-capture detector (ECD) based on ^{63}Ni.

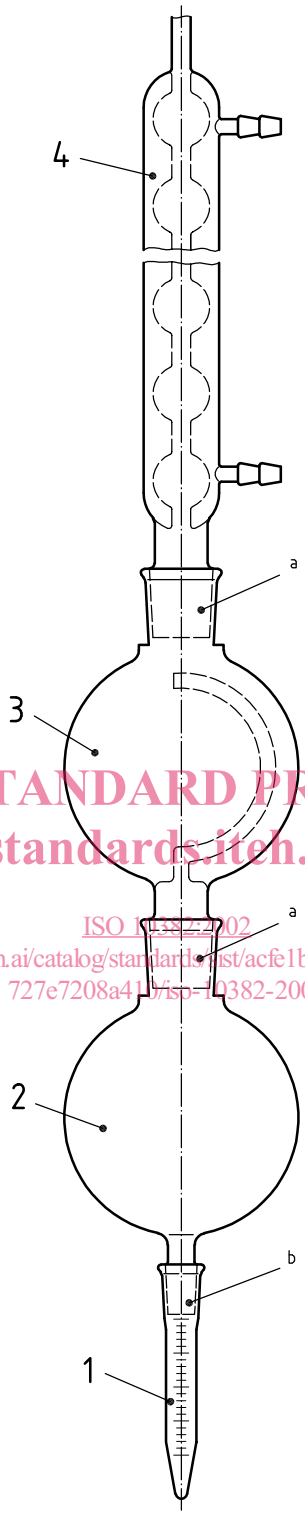
NOTE 1 Working with an encapsulated radioactive source such as that present in an ECD requires a licence in accordance with the appropriate national regulations.

NOTE 2 Gas chromatographs equipped with two detectors and with facilities for connecting two capillary columns to the same injection system are very well suited for this analysis; with such apparatus the confirmatory analysis can be performed simultaneously.

5.13 Capillary column, of fused silica, with a length of 50 m and an internal diameter of about 0,25 mm coated with a film of cross-linked polysiloxane.

Other columns can also be used, although in some cases unsatisfactory separation is obtained. A column coated with a moderate polar phase, e.g. CP-Sil 19, OV 1701 etc., shall be used to confirm the result obtained.

NOTE The retention times for PCB and OCP on capillary columns coated with CP-Sil 8 and CP-Sil 19 are given in annex A.



Key

1 Graduated test tube, capacity 15 ml
2 Distillation flasks
3 Receiver flask
4 Reflux condenser

[a] ISO 383 29/32
[b] ISO 383 14/23

All joints shall be in accordance with ISO 383.

Figure 1 — Example of evaporator (Kuderna Danish)

Dimensions in millimetres

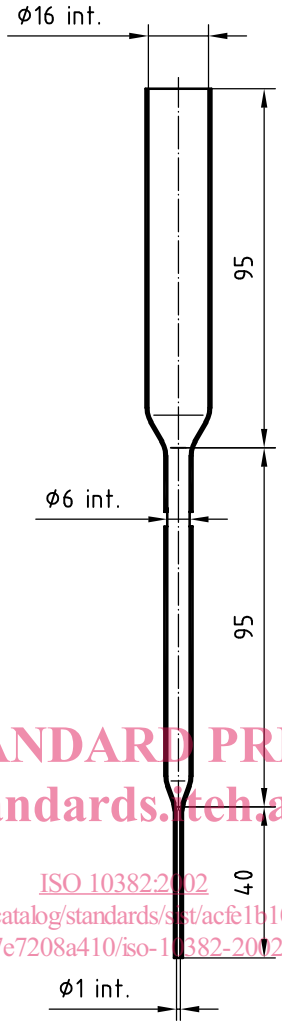


Figure 2 — Example of chromatography tube

6 Preparation of standard solutions of PCB and OCP

Prepare individual concentrated primary standard solutions of mass concentration about 0,4 mg/ml in n-heptane by weighing approx. 10 mg of each of the standards (4.9) to the nearest 0,1 mg and dissolving them in 25 ml of n-heptane.

Check the purity of the primary standard solutions by means of a gas chromatogram of the solutions concerned. Preferably a relatively non-specific detector, such as a flame ionization detector (FID) or a heat conductivity detector (TCD), shall be used.

Combine small quantities (2 ml to 10 ml) of the individual primary standard solutions into a mixed standard solution of PCB and OCP including the injection standards (see annex B). Using this solution, prepare the working standard solutions in accordance with annex B by dilution.

Components present in mixed standard solutions should be completely separated by the gas chromatographic columns used.

Store the primary and diluted standard solutions in a dark place at a temperature of less than 4 °C.

NOTE The solutions are stable for at least one year, provided that evaporation of solvent is negligible.