
**Tobacco and tobacco products —
Determination of organochlorine pesticide
residues — Gas chromatographic method**

*Tabac et produits du tabac — Dosage des résidus de pesticides
organochlorés — Méthode par chromatographie en phase gazeuse*



Reference number
ISO 4389:2000(E)

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Foreword

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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.

International Standard ISO 4389 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 4389:1997). As a result of extensive examination, the scope of this edition has been extended to tobacco products. Technical Corrigendum 1:1998 has been included.

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Tobacco and tobacco products — Determination of organochlorine pesticide residues — Gas chromatographic method

1 Scope

This International Standard specifies a method for the gas chromatographic determination of pesticide residues in leaf tobacco, manufactured tobacco and tobacco products.

The method is applicable to the determination of the organochlorine pesticides listed in Table 1. The method is particularly recommended for determination of the substances within the detection limits listed in Table 1.

NOTE 1 The method has been demonstrated to be free from errors that may arise from interfering peaks on the chromatogram that originate from non-organochlorine pesticide substances, when applied to various styles of leaf tobacco. However, the preceding statement cannot be assumed to apply to all forms and variants of tobacco and tobacco products and care should be taken in the interpretation of any unexpected, apparently positive results. Some analysts advocate the use of mass spectrometric confirmation of the chemical structure of compounds detected by chromatography in such circumstances.

NOTE 2 ISO 1750 contains the systematic chemical names and structures corresponding to the common names given in Table 1.

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 648, *Laboratory glassware — One-mark pipettes*.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*.

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

ISO 4874, *Tobacco — Sampling of batches of raw material — General principles*.

ISO 8243, *Cigarettes — Sampling*.

3 Principle

The pesticide residues from a dried and milled sample, mixed with Florisil®¹⁾, are extracted by *n*-hexane in a special Soxhlet extractor. They are determined without any further clean-up with a gas chromatographic apparatus equipped with an electron-capture detector.

4 Reagents

All reagents shall be suitable for pesticide residue analysis. All solvents shall be checked for purity before use by carrying out a blank determination using exactly the same procedure (extraction and gas chromatography) as used for the test sample. The chromatogram obtained from the solvents shall have a baseline without noticeable peaks that could interfere with those from the pesticide residues being determined.

4.1 Water, degassed, in accordance with at least grade 2 of ISO 3696.

4.2 *n*-Hexane.

4.3 Toluene.

4.4 Reference substances: certified reference materials of minimum purity 95 % (mass fraction) of the substances listed in Table 1.

NOTE *trans*-Chlordane is used as an indicator for chlordane (technical mixture). If α -endosulfan is detected by this method, it is advisable to determine residues of the sum of α -endosulfan, β -endosulfan and endosulfan sulfate by a method suitable for such determinations.

4.5 Internal standard stock solution

Use Mirex,²⁾ an obsolete pesticide which has been superseded (see reference [2]).

NOTE Mirex is a generic name for dodecachloropentacyclo[5.2.1.0^{2,6}.0^{3,9}.0^{5,8}]decane.

Weigh, to the nearest 0,000 1 g, 0,02 g of Mirex into a 100 ml volumetric flask. Dilute to the mark with *n*-hexane (4.2).

4.6 Internal standard solution

Pipette 5 ml of the internal standard stock solution (4.5) into a 200 ml volumetric flask and dilute to the mark with *n*-hexane (4.2) to give a solution containing approximately 5 µg/ml of Mirex. Store the internal standard solution at between 0 °C and +4 °C and exclude light. Under these conditions the internal standard solution is stable for at least 6 months.

1) Florisil® is an example of a suitable 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.

2) Mirex is an example of a suitable 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.