

INTERNATIONAL STANDARD

**ISO
15303**

First edition
2001-09-15

Animal and vegetable fats and oils — Detection and identification of a volatile organic contaminant by GC/MS

*Corps gras d'origines animale et végétale — Détection et identification d'un
contaminant organique volatil par CPG/SM*



Reference number
ISO 15303:2001(E)

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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.

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.

International Standard ISO 15303 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Annex A of this International Standard is for information only.

Animal and vegetable fats and oils — Detection and identification of a volatile organic contaminant by GC/MS

1 Scope

This International Standard specifies a method for the detection and identification of a volatile organic contaminant in edible oils.

It is applicable to the identification of volatile industrial chemicals in both crude and refined edible oils that are suspected of being contaminated. It also enables determination of the concentration of the contaminant.

This International Standard is not applicable to the determination of the concentration of chemicals that may react with the edible oil or with one of its natural components. In these cases, the presence of the contaminant may sometimes be established on a qualitative basis. Also, this International Standard is not applicable to non-volatile chemicals.

This method has been shown to be applicable for the identification of the following compound classes:

- saturated halogenated hydrocarbons;
- unsaturated halogenated hydrocarbons;
- esters; aldehydes; alcohols; amines; ketones; ethers;
- cyclic and aromatic compounds;
- nitrogen compounds;
- acrylates; etc.

The method has been evaluated for concentrations in the range of 1 mg/kg to 10 mg/kg.

2 Normative reference

The following normative document contains 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 edition of the normative document 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 661:1989, *Animal and vegetable fats and oils — Preparation of test sample*

3 Principle

A deuterated reference compound with a GC retention time close to that of the suspected contaminant is added to the oil at a concentration close to that of the suspected contaminant. A sample of the oil is then introduced into a

thermal cold-trapping inlet of a GC/MS apparatus. Volatile components evaporate from the oil and are held in the cold trap. The trap is then flash-heated to 160 °C and the chemicals released from the trap are swept into the GC/MS for analysis.

4 Reagents

Use only reagents of recognized analytical grade, and distilled or demineralized water or water of equivalent purity.

4.1 Standard reference compound, corresponding to suspected contaminating compound (99 % pure).

4.2 Methanol, Analar grade.

4.3 Fully deuterated internal standards of benzene, ethyl benzene or naphthalene (99 % pure).

4.4 Helium, chemically pure grade.

4.5 Refined, bleached, deodorized (RBD) groundnut oil, or a similar stable vegetable liquid oil, known to be free of industrial chemicals related to the analyte under consideration.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Capillary gas chromatograph.

5.2 Mass spectrometer.

5.3 Thermal desorption cold-trapping device¹⁾.

5.4 Capillary column, of length 50 m, methyl polysiloxane with OV101 (or equivalent) stationary phase, 0,5 µm film thickness and 0,32 mm internal diameter.

5.5 Gas chromatograph/mass spectrometer, operating under the following conditions.

a) Thermal desorption cold-trapping temperature programme (at injection):

- thermal cold trap oven 160 °C;
- thermal cold trap below –40 °C.

The trap is held at a temperature below –40 °C for 5 min and is then flash-heated to 160 °C. The GC temperature programme is started simultaneously with the flash-heating of the trap. The trap is held at 160 °C for 3 min.

b) GC temperature programme:

- initial temperature 50 °C for 5 min;
- increase at 7,5 °C per min;
- final temperature 250 °C for 5 min.

¹⁾ The Chrompack Purge and Trap System 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 the product.