
**Eggs and egg products —
Determination of fipronil and
metabolites residues — Liquid
chromatography tandem mass
spectrometry method**

*Œufs et ovoproduits — Dosage des résidus de fipronil et de ses
métabolites — Méthode de chromatographie en phase liquide couplée
à la spectrométrie de masse en tandem*





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Published in Switzerland

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 6, *Meat, poultry, fish, eggs and their products*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Eggs and egg products — Determination of fipronil and metabolites residues — Liquid chromatography tandem mass spectrometry method

1 Scope

This document specifies a liquid chromatography tandem mass spectrometry method (LC-MS/MS) for the determination of fipronil and metabolites (including fipronil-desulfinyl, fipronil-sulfide and fipronil-sulfone) residues in eggs and egg products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

fipronil and metabolites content

ω

mass fraction of fipronil and metabolites residues in egg and egg products

Note 1 to entry: The fipronil and metabolites content is determined according to the procedure specified in this document.

Note 2 to entry: The fipronil and metabolites content is expressed in micrograms per kilogram.

4 Principle

The test portion is extracted with acetonitrile. The extract is cleaned up with dispersive solid phase extraction. The fipronil and metabolites are determined and confirmed by LC-MS/MS, and are quantified with an internal standard method.

5 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in CAC/GL 50-2004.

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Start from a representative sample of at least 1 kg.

Store the sample in such a way that deterioration and change in composition are prevented.

6 Preparing and storing the test sample

6.1 Preparing

6.1.1 Fresh eggs and egg products with shell

Wash approximately 15 to 20 fresh eggs (with a mass of about 1 kg) and remove the shells. Smash and homogenize the egg samples with the appropriate equipment ([7.6.6](#)). Place the sample in a polyethylene bottle.

Weigh about 1 kg of egg products (with shells) and then remove the shells. Smash and homogenize the egg samples with the appropriate equipment ([7.6.6](#)). Place the sample in a polyethylene bottle.

6.1.2 Other egg products

Homogenize the egg product samples. Place each sample in a polyethylene bottle.

6.2 Storing

Store the test sample and a standby sample at a temperature of $-20\text{ }^{\circ}\text{C}$ to $-16\text{ }^{\circ}\text{C}$, respectively.

7 Liquid chromatography tandem mass spectrometry method

7.1 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

7.1.1 Water, conforming to grade 1 of ISO 3696.

7.1.2 Acetonitrile, HPLC grade.

7.1.3 Methanol, HPLC grade.

7.1.4 Formic acid, HPLC grade.

7.1.5 Ammonium acetate, HPLC grade.

7.1.6 Anhydrous magnesium sulfate.

7.1.7 Sodium chloride.

7.1.8 Anhydrous sodium sulfate.

7.2 Preparation of solutions

7.2.1 Formic acid solution, 1 ml/l.

Pipette 1 ml formic acid ([7.1.4](#)) to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.