International Standard

ISO 23883

First edition
2026-05

Meat, fish and their products — Determination of fluoroquinolone residue content — High performance liquid chromatography-tandem mass spectrometry method

Viandes, poissons et leurs produits dérivés — Détermination de la teneur en résidus de fluoroquinolone — Méthode par chromatographie liquide à haute performance couplée à la spectrométrie de masse en tandem

Reference number
ISO 23883:2026(en)

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Published in Switzerland

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Foreword

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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.

Introduction

Fluoroquinolones are synthetic broad-spectrum antimicrobial agents extensively used as a veterinary drug for the prevention and treatment of bacterial infections in food-producing animals. Their widespread application, particularly in husbandry and aquaculture, has raised concerns about the presence of residual amounts in edible animal-derived products. Residues of fluoroquinolones in food can cause adverse health effects for human beings, including allergies and disturbances to the intestinal flora. More critically, prolonged exposure to sub-therapeutic levels of these compounds through the food chain is considered to be a contributing factor to the emergence and dissemination of antimicrobial resistance.

To safeguard public health and ensure the safety of animal-derived food, many regulatory authorities have established maximum residue limits (MRLs) for various fluoroquinolone compounds in meat, fish and related products. The effective enforcement of these limits depends on the availability of sensitive, accurate and validated analytical methods capable of detecting trace levels of residues in complex biological matrices.

Several analytical approaches have been developed for the determination of fluoroquinolone residues, including microbiological inhibition tests, immunoassays and instrumental techniques. While microbiological and immunological methods are relatively simple and cost-effective, they often suffer from limited specificity and sensitivity, as well as susceptibility to matrix interferences. In contrast, instrumental methods such as high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) offer significant advantages in terms of selectivity, precision and detection capability. The ability of HPLC-MS/MS to simultaneously quantify multiple fluoroquinolone compounds with high sensitivity makes it particularly suitable for regulatory monitoring and confirmatory analysis across a variety of food matrices.

Meat, fish and their products — Determination of fluoroquinolone residue content — High performance liquid chromatography-tandem mass spectrometry method

1 Scope

This document specifies the determination of fluoroquinolone residue content in meat, fish and their products by high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) method.

This document is applicable to the determination of enrofloxacin, ciprofloxacin, norfloxacin, ofloxacin and pefloxacin residues in meat, fish and their products, including livestock and poultry.

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

No terms and definitions are listed in this document.

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/

4 Principle

The fluoroquinolone residues in the test sample are extracted with acidified acetonitrile, purified by n-hexane extraction, determined by high performance liquid chromatography-tandem mass spectrometry, and quantified by an internal standard method.

5 Reagents and materials

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

5.1 Water, conforming to ISO 3696, grade 1 or equivalent.

5.2 Methanol, LC grade.

5.3 Acetonitrile, LC grade.

5.4 Formic acid, LC grade.

5.5 Ammonium acetate, LC grade.