

ICS 67.050

CCS B 47



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 23412-2009

**Determination of residues of 19 quinolones in honey --
LC-MS/MS method**

蜂蜜中19种喹诺酮类药物残留量的测定方法 液相色谱-质谱/质谱法

(English Translation)

Issued on: March 28, 2009

Implemented on: July 1, 2009

Issued by: Standardization Administration of China

Table of Contents

- Foreword
- 1 Scope
- 2 Normative references
- 3 Method summary
- 4 Reagents and materials

Foreword

The Standard Appendix A, Appendix B, Appendix C and Appendix D is an informative annex.

This standard is proposed and managed by the State Administration of Quality Supervision Inspection and Quarantine of People's Republic of China.

This standard was drafted. People's Republic of China Jiangsu Entry-Exit Inspection and Quarantine Bureau.

Drafters of this standard. Shen Chong-yu, Chen Huilan, Ding Tao, Zhang Xiaoyan, Liu Yan, Zhang Jing, Huang Juan, Jiang Yuan, Wang Jin Tao, Shen Yuk.

Honey 19 quinolone residues Determination of liquid chromatography - mass spectrometry/mass spectrometry

1 Scope

This standard specifies the honey enrofloxacin (referred to ENR, the same below), ciprofloxacin (referred to as CIP, the same below), norfloxacin (referred to NOR, the same below), ofloxacin (referred OFL, the same below), double difloxacin (referred to as the DIF, the same below), quinoline acid (referred to as OXO, the same below), fluorometholone Quinoline (referred FLU, the same below), sarafloxacin (referred to as the SAR, the same below), sparfloxacin (referred to as the SPA, the same below), Dan difloxacin (referred to DAN, the same below), fleroxacin (referred FLE, the same below), marbofloxacin (referred to MAR, the same below), enoxacin (referred ENO, the same below), Ao Bisha Star (referred ORB, the same below), PPA (referred to as PIP, the same below), pefloxacin (referred to as the PEF, the same below), lomefloxacin (referred to LOM, under Same), cinoxacin (referred to CIN, the same below) and nalidixic acid (referred to as the NAL, the same below) quinolone residues in 19 kinds of liquid chromatography - mass spectrometry/mass spectrometry Measurement method. Above 19 quinolone English name, structural formula, CAS Number and molecular weight Appendix A.

This standard applies to the determination of quinolones residues in honey.

2 Normative references

The following documents contain provisions which, through reference in this standard and become the standard terms. For dated references, subsequent Amendments (not including errata content) or revisions do not apply to this standard, however, encourage the parties to the agreement are based on research Whether the latest versions of these documents. For undated reference documents, the latest versions apply to this standard.

GB/T 6682 analytical laboratory use specifications and test methods (GB/T 6682-2008, ISO 3696. 1987, MOD)

3 Method summary

After the sample is added the internal standard solution of sodium hydroxide dissolved honey samples, ionized quinolones residue after anion exchange solid phase extraction Enrichment and purification by liquid chromatography - mass spectrometry/mass spectrometry, gradient, internal standard.

4 Reagents and materials

Unless otherwise noted, all reagents were of analytical grade water as a water GB/T 6682 regulations.

4.1

Methanol. HPLC grade.

4.2

sodium hydroxide.

4.3

formic acid (85%).

4.4

anion exchange column (PAX). 60mg, 3mL, or equivalent person.

4.5

isotope internal standard. NOR-D5. purity $\geq 98\%$.

4.6

ENR, CIP, NOR, OFL, DIF · HCl, SAR · HCl, SPA, DAN, OXO, FLU, FLE, MAR, ENO, ORB, PIP, PEF, LOM, CIN and NAL standard. Purity $\geq 99\%$.

4.7

quinolones standard stock solution. 1mg/mL. Accurately weighed amount of quinolone standard (4.6), (4.1) paired with methanol 1.0mg/mL standard stock solution. Stock solution stored at 4 °C refrigerator for up to 6 months.

4.8

deuterated norfloxacin (NOR-D5) internal standard stock solution. 100µg/mL. Weigh the right amount of NOR-D5 internal standard (4.5), methanol (4.1) dubbed 100µg/mL stock solution, the stock solution stored at 4 °C refrigerator.

4.9

deuterated Norfloxacin (NOR-D5) of internal standard working solution. 1µg/mL. Take appropriate internal standard stock solution (4.8) was diluted with methanol (4.1) to 1µg/mL working solution of internal standard working solution was stored at 4 °C.

4.10

quinolones standard solution. methanol - water (8 + 2, volume ratio) were formulated at concentrations ranging from 1ng/mL ~ 60ng/mL standard