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**Method for the determination of oxytetracycline,
tetracycline, chlortetracycline and doxycycline
residues in honey - LC-MS-MS method**

蜂蜜中土霉素、四环素、金霉素、强力霉素残留量的测定方法

液相色谱-串联质谱法

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Foreword

This Part of GB/T 18932 modifies and adopts Canadian Standard ACC-042-V1.0 *Determination of Tetracycline Residue in Honey - LC-MS-MS Method*. The main modifications are as follows:

- Eluent is changed from methanol to ethyl acetate;
- Carboxylic acid anion exchange column is added for purification;
- The single quadrupole mass spectrometry detector is changed to tandem quadrupole mass spectrometry detector.

Annex A and B of this Part are informative.

This Part was proposed by State Administration for Quality Supervision and Inspection and Quarantine (AQSIQ).

This Part shall be under the jurisdiction of All China Federation of Supply and Marketing Cooperatives.

Drafting organization of this Part: Qinhuangdao Entry-Exit Inspection and Quarantine Bureau of PRC.

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This Part is issued as a national standard for the first time.

Method for the determination of oxytetracycline, tetracycline, chlortetracycline and doxycycline residues in honey - LC-MS-MS method

1. Scope

This Part of GB/T 18932 specifies the method for the determination of oxytetracycline, tetracycline, chlortetracycline and doxycycline residues in honey.

This Part is applicable to the determination of oxytetracycline, tetracycline, chlortetracycline and doxycycline residues in honey.

Detection limits for the method in this Part: for oxytetracycline and tetracycline, it is 0.001 mg/kg ; for chlortetracycline and doxycycline, it is 0.002 mg/kg.

2. Normative references

The articles contained in the following documents have become the articles of this Part when they are quoted in this Part of GB/T 18932. For the dated documents so quoted, all the subsequent modifications (excluding all corrections) or revisions made thereafter do not apply to this Part. However, the parties who reach an agreement according to this Part are encouraged to study whether the latest versions of these documents may be used. For the undated documents so quoted, the latest versions (including all modification sheets) apply to this document.

GB/T 6379 Precision of test methods - Determination of repeatability and reproducibility for a standard test method by interlaboratory tests (GB/T 6379-1986, neq ISO 5725:1981)

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-1992, neq ISO 3696:1987)

3. Principle

For tetracycline antibiotic residue in the specimen, use 0.1 mol/L Na₂EDTA-McIlvaine (pH=4.0±0.05) buffer solution to extract the extraction solution; after centrifugation, purify the supernatant with Oasis HLB or equivalent solid phase extraction column and anion exchange column; determine the tetracycline antibiotics in the specimen; Use liquid chromatography - tandem mass spectrometer to determine. And adopt the external

oxytetracycline, tetracycline, chlortetracycline and doxycycline (4.15); respectively use methanol to prepare 0.1 mg/mL standard stock solutions. Store the stock solutions in -18°C.

4.17 Matrix mixed standard working solutions of oxytetracycline, tetracycline, chlortetracycline and doxycycline: according to needs, pipette appropriate standard stock solutions of oxytetracycline, tetracycline, chlortetracycline and doxycycline (4.16); use blank sample extracting solution to dilute to be the matrix mixed standard working solutions of appropriate concentrations; store the matrix standard solutions at 4°C; it may be used in three days.

5. Apparatuses

5.1 LC-tandem quadrupole mass spectrometer, equipped with electrospray ionization source.

5.2 Analytical balances: 2 balances of which the sensitivity is 0.1 mg and 0.01 g respectively.

5.3 Liquid mixer.

5.4 Solid phase extraction vacuum device.

5.5 Liquid reservoir: 50mL.

5.6 Micro-syringe: 25uL and 100uL.

5.7 Graduated sample tube: 5mL, precision is 0.1mL.

5.8 Vacuum pump: vacuum degree shall reach 80kPa.

5.9 Centrifuge tube: 50mL.

5.10 Flat-bottomed flask: 100mL.

5.11 pH meter: measurement precision is ± 0.02 .

6. Preparation and storage of sample

6.1 Sample preparation

For non-crystallized laboratory sample, stir it evenly; For crystallized sample, in a enclosed circumstance, place it in the water bath at not more than 60°C; warm it; oscillate it; after it is totally melted, stir it; rapidly cool it to room temperature. Take 0.5 kg as the sample. Place the prepared sample in the sample flask, seal and mark it.

oxytetracycline, tetracycline, chlortetracycline and doxycycline are shown in Table 3.

If the difference exceeds the repeatability limit, it shall discard the test results and carry out two independent tests again.

9.2 Reproducibility

Under reproductive conditions, the absolute difference between two independent test results shall not exceed reproducibility limit (R). The content range and reproducibility equation of oxytetracycline, tetracycline, chlortetracycline and doxycycline are shown in Table 3.

Table 3 Content range AND repeatability and reproducibility equations

Name	Content Range (mg/kg)	Repeatability Limit r	Reproducibility Limit R
Oxytetracycline	0.002~0.100	$\lg r = 0.9668 \lg m - 0.8686$	$\lg R = 1.0096 \lg m - 0.6597$
Tetracycline	0.002~0.100	$\lg r = 0.9484 \lg m - 0.8370$	$\lg R = 1.1611 \lg m - 0.2317$
Chlortetracycline	0.002~0.100	$\lg r = 0.8395 \lg m - 1.0872$	$\lg R = 0.9060 \lg m - 0.8136$
Doxycycline	0.002~0.100	$\lg r = 0.9209 \lg m - 0.8462$	$\lg R = 0.9899 \lg m - 0.6504$

Note: m is the arithmetic mean value of two test results.

Annex B**(Informative)****Recovery rate**

The test data of adding concentrations and average recovery rates of oxytetracycline, tetracycline, chlortetracycline and doxycycline are shown in Table B.1.

Table B.1 Test data of adding concentrations and average recovery rates of oxytetracycline, tetracycline, chlortetracycline and doxycycline

Drug Name	Adding Concentration / (mg/kg)	Average Recovery Rate / (%)
Oxytetracycline	0.002	88.0
	0.010	95.3
	0.050	93.6
	0.100	95.8
Tetracycline	0.002	81.9
	0.010	82.6
	0.050	84.5
	0.100	89.3
Chlortetracycline	0.002	87.2
	0.010	86.0
	0.050	86.6
	0.100	90.8
Doxycycline	0.002	85.2
	0.010	85.3
	0.050	86.8
	0.100	87.9

END

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