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

GB/T 18932.19-2003

**Method for the determination of chloramphenicol residues in
honey -- LC-MS-MS method
(English Translation)**

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Foreword

This Part of GB/T18932 is modified on the basis of adopting the Canadian Standard ACC-062-V1.0 "Method for the determination of chloramphenicol residues in honey - LC-MS method". The main modified contents are as follows.

- Purification column is modified from C18 solid phase extraction column to Oasis HLB solid phase extraction column;
- Eluent is modified from acetonitrile + water (3+7) to ethyl acetate;
- Single quadrupole mass-spectrometry detector is modified to tandem quadrupole mass-spectrometry detector;
- Internal standard method is modified to the external standard method.

Appendix A and Appendix B of this Part are informative.

This Part was proposed by Qinhuangdao Entry - Exit Inspection and Quarantine Bureau of the People's Republic of China.

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

The drafting organization of this Part. Qinhuangdao Entry - Exit Inspection and Quarantine Bureau of the People's Republic of China.

Main drafters of this Part. Pang Guofang, Cao Yanzhong, Zhang Jinjie, Jia Guangqun, Fan Chunlin, Li Xuemin, Liu Yongming and Shi Yuqiu.

This Part was issued for the first time.

Method for the determination of chloramphenicol
residues in honey - LC-MS-MS method

1 Scope

This Part of GB/T 18932 specifies the method for the determination of chloramphenicol residues in honey - LC-MS-MS method.

This Part is applicable to the determination of chloramphenicol residues in honey.

The method detection limit of this Part. chloramphenicol is 0.10 µg/kg.

2 Normative References

The following standards contain the provisions which, through reference in this Part of the GB/T18932, constitute the provisions of this Part. For dated references, all subsequent amendments (excluding corrections) or revisions do not apply to this Part. However, the parties who enter into agreement based on this Part are encouraged to investigate whether the latest versions of these standards are applicable. For undated reference documents, the latest versions apply to this Part.

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

3 Principles

Use ethyl acetate to extract the sample. After the extract is concentrated, dissolve it in water.

4.5 Acetonitrile + water (1+7). measure 20 mL of acetonitrile and mix it with 140 mL of water.

4.8 Chloramphenicol standard stock solution. 0.1 mg/mL. Accurately weigh an adequate amount of chloramphenicol reference material (4.7), and use methanol to make 0.1 mg/mL standard stock solution. Store the stock solution in the refrigerator at 4°C; it shall be used for two months.

5.1 Liquid chromatography - 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.

6.1 Sample preparation

Evenly stir the non-crystallized laboratory sample. For crystallized sample, in a enclosed circumstance, place it in the water bath at not more than 60°C; warm it; oscillate it till it is totally melted;

6.2 Sample storage

Store the sample at room temperature.

7.1 Extraction

Weigh 5 g of sample, accurate to 0.01 g. Place it into a 50 mL stoppered centrifuge tube; add 5 mL of water. Quickly mix it in the liquid mixer for 1 min till it is completely dissolved.

7.2 Purification

Pour the extract (7.1) into a liquid reservoir that is connected to Oasis HLB column (4.6) in the lower. The solution flows through the Oasis HLB solid phase extraction column at the flow rate ≤ 3 mL/min.

7.3.2 Mass-spectrometry conditions

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7.3.3 LC-MS-MS determination

Respectively inject the chloramphenicol standard working solutions (4.9) under LC-MS-MS conditions. Take the peak area as ordinates, and take working solution concentration (ng/mL) as abscissa; draw the seven-point standard working curve.

7.4 Parallel test

Follow the previous steps to carry out the parallel test for the same sample.

7.5 Blank test

Follow the previous steps, except weighing the sample.

8 Calculation of results

The result is calculated according to equation (1).

9 Precision

The precision data in this Part is determined in accordance with the provisions of GB/T 6379. The repeatability and reproducibility values are calculated in 95% confidence.

9.1 Repeatability

Under conditions of repeatability, the content of chloramphenicol in honey is between 0.19g/kg~4.0μg/kg. The absolute difference between two independent test results obtained shall not exceed the repeatability limit (r). The repeatability limit of this Part is calculated according to equation (2).

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