

ICS 67.180.10

CCS X31



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

GB/T 18932.26-2005

**Method for the determination of metronidazole, ronidazole and
dimetridazole residues in honey-Liquid chromatographic
method**

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Issued on: February 4, 2005

Implemented on: August 1, 2005

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;
Standardization Administration of China**

Table of Contents

Foreword...	3
1 Scope...	4
2 Normative references...	4
3 Principle...	4
4 Reagents and materials...	5
5 Instruments...	5
6 Sample preparation and preservation...	6
7 Determination steps...	6
8 Result calculation...	8
10 Annex B...	11

Foreword

Annex A and Annex B in this Part of GB/T 18932 are informative. This Part was proposed by Qinhuangdao Entry-exit Inspection and Quarantine Bureau of the People's Republic of China. The Part shall be under the jurisdiction of China Supply and Marketing Cooperatives. 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, Liu Yongming, Cao Yanzhong, Jia Guangqun, Fan Chunlin, Zhang Jinjie, Li Xuemin and Shi Yuqiu. The Part is first-time released national standard. Method for the determination of metronidazole, ronidazole and dimetridazole residues in honey - Liquid chromatographic method

1 Scope

GB/T 18932.26-2005 is the Chinese national standard on method for the determination of metronidazole, ronidazole and dimetridazole residues in honey-liquid chromatographic method, in the field of food technology. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 4 February 2005 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 August 2005. Classification: ICS 67.180.10, CCS X31. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in

the document itself, which is delivered complete in English translation.

This Part of GB/T 18932 specifies the liquid chromatographic determination method for the residues of metronidazole, ronidazole and dimetridazole in honey. This Part applies to the determination of the residues of metronidazole, ronidazole and dimetridazole in honey. The detection limit of the method in this Part. 0.0010mg/kg for metronidazole, ronidazole and dimetridazole.

2 Normative references

The provisions in following documents become the provisions of this Part through reference in this Part of

GB/T 18932. For dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Part, however, parties who reach an agreement based on this Part are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

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

The nitroimidazole residues in the sample are extracted by ethyl acetate; after the extracted solution is evaporated to dryness, dissolve with water, purified with Oasis HLB solid phase extraction column and BAKERBOND Carboxylic Acid solid phase extraction column; determined by liquid chromatography ultraviolet detector; quantified by external standard method.

5.2 Analytical balances. 2 balances with sensitivity of 0.1mg and 0.01g respectively.

5.3 Liquid mixer.

5.4 Solid phase extraction vacuum device.

5.6 Glass centrifuge tube with stoppers. 50mL.

5.7 Vacuum pump. the vacuum shall reach 80kPa.

5.9 Rotary evaporator.

5.10 Sample tube. 5mL.

5.11 pH meter. measurement accuracy is ± 0.02 .

5.12 Pear-shaped flask. 200mL.

6 Sample preparation and preservation

6.1 Sample preparation For non-crystallized laboratory samples, stir them evenly. For samples with crystallization, under hermetic conditions, PLACE them in the water bath not more than 60°C to warm; SHAKE; STIR evenly until it is fully melt; rapidly cool to room temperature; TAKE 0.5kg as the sample. The prepared sample is placed in flask, sealed and marked.

6.2 Sample preservation The test sample shall be preserved under room temperature.

7 Determination steps

7.1 Extraction WEIGH 6g of sample (accurate to 0.01g); PLACE in a 50mL glass centrifuge tube; ADD 6mL of water; MIX in liquid mixer; ADD 20mL of ethyl acetate; OSCILLATE in the oscillator for 20min; CENTRIFUGE; TAKE the supernatant to a pear-shaped flask. Again, EXTRACT with 20mL of ethyl acetate; MERGE the supernatant; USE a rotary evaporator, on water bath of 45°C, to reduce the pressure and evaporate to dryness; DISSOLVE with 5mL of water; WAIT to be purified.

9.1 Repeatability Under repeatability conditions, the absolute difference between two independent test results obtained shall not exceed the repeatability limit (r). The content range and repeatability equation of metronidazole, ronidazole and dimetridazole in honey are shown in Table 2. Table

2 Content range and repeatability and reproducibility equations Names Content range/ (mg/kg) Repeatability limit Reproducibility Metronidazole 0.010~

0.100 $r = 0.0303m + 2.1673$ $R = 0.0807m - 1.0408$ Ronidazole 0.010~

0.100 $r = 0.0608m + 1.8727$ $R = 0.0620m + 1.7190$ Dimetridazole 0.010~

0.100 $r = 0.0627m + 1.4411$ $lgR = 0.7099lgm - 0.4377$

Note. m is the arithmetic mean of two determination results. If the difference exceeds the repeatability limit, it shall abandon the test results and re- complete the determination of two single tests.

9.2 Reproducibility Under reproducibility conditions, the absolute difference between two independent test results obtained shall not exceed reproducibility limit (R). The content range and reproducibility equation of metronidazole, ronidazole and dimetridazole in honey are shown in Table 2.

Annex B

(Informative) Recovery rate Test data of the added concentration and the average recovery rate of metronidazole, ronidazole and dimetridazole in this method is shown in Table B.1.

Table B.1 Test data of the added concentration and the average recovery rate of metronidazole, ronidazole and dimetridazole

Name of chemicals	Added concentration/($\mu\text{g/kg}$)	Average recovery rate/(%)
Metronidazole	0.010	86.27
	0.020	87.13
	0.050	87.35
Ronidazole	0.010	84.64
	0.020	89.38
	0.050	82.87

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