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中华人民共和国出入境检验检疫行业标准

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进出口蜂王浆及蜂王浆冻干粉中链霉素残留量检测方法　液相色谱法

Determination of streptomycin residues in royal jelly and royal jelly powder for import and export—Liquid chromatographic method

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streptomycin in the standard working solution and the sample solution should be in the linear range of the instrumental detection. The standard solution should be randomly injected between the injections of sample solution of equal volume. Under the above operating condition, the retention time of streptomycin is about 13.3 min. For the chromatogram of the standard, reference fig A1 in annex A.

3.5 Blank test

The operation of the blank test is the same as that described in the method of determination, but without addition of sample.

3.6 Calculation and expression of result

The calculation of result is carried out according to the following formula:

$$X = \frac{A \times c \times V}{A_s \times m} \quad \cdots\cdots (1)$$

where

X——the residue content of streptomycin in the test sample, mg/kg;

A——the peak area of streptomycin in sample solution;

c——the concentration of streptomycin in the standard working solution, μg/mL;

V——the final volume of sample solution, mL;

A_s——the peak area of streptomycin in the standard working solution;

m——the corresponding mass of the test sample in the final sample solution, g.

4 Limit of determination and recovery

4.1 Limit of determination

The limit of determination of this method is 0.010 mg/kg for royal jelly and 0.020 mg/kg for royal jelly powder.

4.2 Recovery

4.2.1 According to the experiment data, the fortifying concentrations of streptomycin and its corresponding recoveries in royal jelly are:

——0.010 mg/kg. The recovery of streptomycin is between 83.7%～94.5%;

——0.020 mg/kg. The recovery of streptomycin is between 90.4 %～98.5%;

——0.050 mg/kg. The recovery of streptomycin is between 86.4 %～93.8%.

4.2.2 According to the experiment data, the fortifying concentrations of streptomycin and its corresponding recoveries in royal jelly powder are:

——0.02 mg/kg. The recovery of streptomycin is between 82.0 %～94.0%;

——0.05 mg/kg. The recovery of streptomycin is between 85.4%～96.2%;

——0.10 mg/kg. The recovery of streptomycin is between 85.6% ～96.8%.

前　言

本标准的附录 A 为资料性附录。

本标准由国家认证认可监督管理委员会提出并归口。

本标准起草单位:中华人民共和国浙江出入境检验检疫局。

本标准主要起草人:陈笑梅、池浩超、陈晓霞、王栋、施旭霞、刘海山、石磊。

本标准系首次发布的出入境检验检疫行业标准。

3.2.19 Cation exchange column: aromatic sulphonic solid phase extraction(SPE) cartridg, 500 mg, 3 mL. Condition aromatic sulphonic SPE cartridge With 5 mL methanol and 10 mL water before using.

3.2.20 HLB Solid phase extraction(SPE)Cartridge: 500 mg,3 mL. Condition HLB SPE cartridge with 5 mL methanol and 10 mL water before using.

3.2.21 Streptomycin standard: 95.8%.

3.2.22 Streptomycin standard stock solution: Accurately weigh an appropriate amount of streptomycin standard and dissolve with water to prepare a standard stock solution of 100 μg/mL. This standard stock solution should be stored at 4℃.

3.2.23 Streptomycin standard working solution: According to the requirement, pipette adequate amount of standard stock solution and dilute with 0.01 mol/L 1-Heptanesulphonic acid sodium solution to prepare standard working solution of suitable concentrations.

3.3 Apparatus and equipment

3.3.1 High-performance liquid chromatograph, equipped with fluorescence detector and post-column derivation apparatus.

3.3.2 Centrifuge.

3.3.3 Vortex mixer.

3.3.4 Solid phase extraction with vacuum pump.

3.3.5 pH measurer: Capable of measuring ±0.02 unit.

3.3.6 Plastic centrifuge tube with cap: 100 mL.

3.4 Determination Procedure

3.4.1 Extraction

Weigh 10 g royal jelly (accurate to 0.01g) in 50mL volumetric flask. Add 40 mL phosphoric acid solution and agitate on a vortex mixer until the sample was dissolved completely. 2.0 mL of 50% Trichloroacetic acid solution was added to the sample solution and diluted to volume by phosphoric acid solution. Shake lightly and transfer the solution to centrifuge tube. Centrifuge the sample solution for 5 mins(4 000 r/min). The supernatant was filtered. Piptte 25 mL filtrate into a clean container and adjusted it to pH 2.0 by saturated NaOH solution.

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1　范围

本标准规定了进出口蜂王浆及蜂王浆冻干粉中链霉素残留量检测的制样和液相色谱测定方法。

本标准适用于进出口蜂王浆及蜂王浆冻干粉中链霉素残留量的检测。

2　制样

2.1　试样的制备

将抽取的样品充分混匀,均分成两份,分别装入洁净容器内,一份作为留样保存,另一份作为试样供检测用。在抽样和制样的操作过程中,应防止样品受到污染或发生含量的变化。

2.2　试样的保存

试样宜及时检测,在不能及时检测的情况下,应置于−18℃以下冷冻保存。

3　测定方法

3.1　方法提要

试样中的链霉素用稀磷酸提取,三氯乙酸沉淀样品中的蛋白质,清液过阳离子交换柱和 HLB 固相萃取小柱净化。液相色谱柱后衍生荧光检测器测定样品中的链霉素,外标法定量。

3.2　试剂和材料

除另有规定外,试剂均为分析纯,水为重蒸水或去离子水。

3.2.1　甲醇:液相色谱级试剂。

3.2.2　乙腈:液相色谱级试剂。

3.2.3　磷酸:≥85%。

3.2.4　乙酸。

3.2.5　磷酸氢二钾。

3.2.6　磷酸二氢钾。

3.2.7　氢氧化钠。

3.2.8　三氯乙酸。

3.2.9　庚烷磺酸钠($C_7H_{15}NaO_3S.H_2O$):液相色谱纯。

3.2.10　1,2-萘醌-4-磺酸钠。

3.2.11　30%甲醇水溶液:甲醇+水(30+70,V/V)。

3.2.12　稀磷酸溶液:pH=2。1 000 mL 水中滴加磷酸,在 pH 计上调节溶液的 pH 为 2。

3.2.13　磷酸盐缓冲溶液:0.2 mol/L,pH=8。称取 33.46 g 磷酸氢二钾和 1.05 g 磷酸二氢钾于烧杯中,加水溶解后定容至 1 000 mL。用磷酸调节溶液 pH 为 8。

3.2.14　氢氧化钠溶液:0.2 mol/L,称取 8 g 氢氧化钠溶于适量水,再定容至 1 000 mL。

3.2.15　三氯乙酸溶液:50%水溶液。称取 100 g 三氯乙酸溶于适量水,再定容至 100 mL。

3.2.16　饱和氢氧化钠溶液。

3.2.17　庚烷磺酸钠溶液:0.5 mol/L,称取 11 g 庚烷磺酸钠溶于 100 mL 水中。

3.2.18　庚烷磺酸钠酸溶液:0.01 mol/L,pH 3.3。称取 2.2 g 庚烷磺酸钠溶于 900 mL 水中,滴加乙