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中华人民共和国出入境检验检疫行业标准

SN/T 1626—2005

进出口肉及肉制品中甲硝唑、替硝唑、奥硝唑、罗硝唑、二甲硝咪唑、塞克硝唑残留量测定方法　高效液相色谱法

Determination of metronidazole, tinidazole, ornidazole, ronidazole, dimetridazole and secnidazole residues in meat and meat products for import and export—HPLC

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ing to formula (1)

$$X = \frac{(A - A_0) \cdot c \cdot V}{A_s \cdot m} \quad \cdots\cdots(1)$$

where:

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

A——the peak area of analyte of the sample solution, mm^2;

A_0——the peak area of blank test, mm^2;

A_s——the peak area of analytes of the standard solution, mm^2;

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

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

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

4 Limit of determination and recovery

4.1 Limit of determination

The limit of determination of this method is 1 μg/kg.

4.2 Recovery

The recovery range of DMOH is 72.70% to 83.86%; the recovery range of RMZ is 65.7% to 86.35%; the recovery range of AMZ is 69.85% to 87.14%; the recovery range of SNZ is 72.70% to 83.86%; the recovery range of DMZ is 75.75% to 91.46%; the recovery range of TNZ is 81.00% to 90.10%; the recovery range of ONZ is 92.73% to 117.40%, when fortified at the concentration of 0.1 mg/kg, 0.5 mg/kg and 1.0 mg/kg.

The recovery range of DMOH is 81.10% to 93.50%; the recovery range of RMZ is 62.20% to 82.40%; the recovery range of AMZ is 76.68% to 89.60%; the recovery range of SNZ is 76.80% to 88.60%; the recovery range of DMZ is 73.40% to 97.20%; the recovery range of TNZ is 93.50% to 99.50%; the recovery range of ONZ is 79.503% to 94.00%, when fortified at the concentration of 0.1 mg/kg, 0.5 mg/kg and 1.0 mg/kg.

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：中华人民共和国河北出入境检验检疫局。

本标准主要起草人：王凤池、马振栋、郭春海、吕红英、艾连峰。

本标准系首次发布的出入境检验检疫行业标准。

3.2.17 SCX solid-phase extraction cartridge: 5 mL acetic acid-ethyl acetate (5+95) pass the cartridge to condition. If there is interference in blank test, the cartridge should be washed as follows: 10 mL ammonium hydroxide-acetonitrile (5+95), 0.1 mol/L hydrochloric acid, 20 mL deionized water, 3 mL methenol and 5 mL acetic acid-ethyl acetate (5+95). In this process, the SCX cartridge should keep wet and leave about 1 cm high solution in cartridge. Then connect a reservoir the bottom of which is stuffed with degrease cotton to the top of cartridge.

3.3 Apparatus and equipment

3.3.1 High-performance chromatography: equipment with UV detector.

3.3.2 Ultrasonic cleanser.

3.3.3 Vortex shaker.

3.3.4 Rotary vacuum evaparator.

3.3.5 Nitrogen evaparator.

3.3.6 Homogenizer.

3.3.7 Centrifuge: the max rotate speed is 5 000 r/min.

3.3.8 Vacuum manifold processing station.

3.4 Procedure

3.4.1 Extraction

Weigh ca 5 g of test sample (accurate to 0.01 g) into a 50 mL centrifuge tube, add 20 mL ethyl acetate, homogenize 1 min, extract for 5 min in ultrasonic bath and centrifuge for 5 min at 3 000 r/min. Transfer the extraction into the heart-shape flask and extract once more with another 20 mL ethyl acetate and combine the extraction into the same flask.

3.4.2 Clean-up

Evaporate the exaction to almost dryness (not complete dryness) using rotary vacuum evaparator at 40 ℃. Dissovle the residues with 5 mL acetic acid-ethyl acetate(5+95), transfer the solution into the reservoir above the conditioned SCX cartridge. Dissolve the residues again with 5 mL acetic acid-ethyl acetate(5+95) and pour into the same reservoir. Then let the solution pass the cartridge at the speed of 2 mL/min. After drop over, wash the cartridge with 2.5 mL acetone, 5 mL methanole, 5 mL acetontrile in turn, finally elute the cartridge with 5 mL ammonium hydroxide-acetonitrile (5+95), discard the prime 2 mL elution and collect the left elution with 10 mL glass flask. Evaporate elution to dryness with nitrogen evaporator at 40 ℃. Residues are dissolved with 1.0 mL deionized water in ultrasonic bath and mix well by vortex shaker. Then the solution is passed through 0.45 μm

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1　范围

本标准规定了肉及肉制品中罗硝唑、二甲硝咪唑及其共同的代谢物、替硝唑、奥硝唑、塞克硝唑、甲硝唑7种硝基咪唑残留量的高效液相色谱测定方法。

本标准适用于鸡肉、猪肉中罗硝唑、二甲硝咪唑及其共同的代谢物、替硝唑、奥硝唑、塞克硝唑、甲硝唑残留量的测定。

2　样品处理

将实验室样品用绞肉机全部绞碎。样品量太大时从每块样品上取代表性的部分，然后全部绞碎，混合均匀后，分为两份，一份进行测定，另一份装入密闭容器内，−18℃冷冻避光保存。

3　测定方法

3.1　方法提要

试样中的硝基咪唑残留用乙酸乙酯提取，通过SCX柱净化后，用配有紫外检测器的高效液相色谱仪测定，外标法定量。

3.2　试剂和材料

除另有规定外，所用试剂均为HPLC级，水为高纯水。

3.2.1　乙腈。

3.2.2　甲醇。

3.2.3　丙酮。

3.2.4　乙酸乙酯。

3.2.5　浓盐酸：优级纯。

3.2.6　冰乙酸：优级纯。

3.2.7　氨水：分析纯。

3.2.8　盐酸：0.1 mol/L，吸取浓盐酸0.83 mL，用水定容于100 mL容量瓶中。

3.2.9　乙酸-乙酸乙酯(5+95)：5 mL冰乙酸用乙酸乙酯定容至100 mL。

3.2.10　氨水-乙腈(5+95)：5 mL氨水用乙腈定容至100 mL。

3.2.11　0.45 μm滤膜。

3.2.12　7种硝基咪唑标准品：纯度≥98.0%，其中罗硝唑和二甲硝咪唑的代谢物为1-甲基-2-羟甲基-5-硝基咪唑(简写为DMZOH)。

3.2.13　标准储备液：分别称取每种硝基咪唑标准品各10 mg(准确至0.1 mg)，用甲醇溶解，并分别定容到10 mL棕色容量瓶中，混匀，该溶液的浓度为1 mg/mL。避光−18℃冷冻保存，保存期为1年。

3.2.14　混合标准中间液Ⅰ：取每种标准储备液各1 mL，移入100 mL棕色容量瓶中，用甲醇定容。该溶液浓度为10 μg/mL。避光−18℃冷冻保存，保存期为1年。

3.2.15　混合标准中间液Ⅱ：取1 mL混合标准中间液Ⅰ于100 mL棕色容量瓶中，用水稀释至刻度。该溶液浓度为100 ng/mL。置于冰箱中1℃～4℃下避光保存，保存期为3个月。