Annex A
(informative)
mass chromatogram

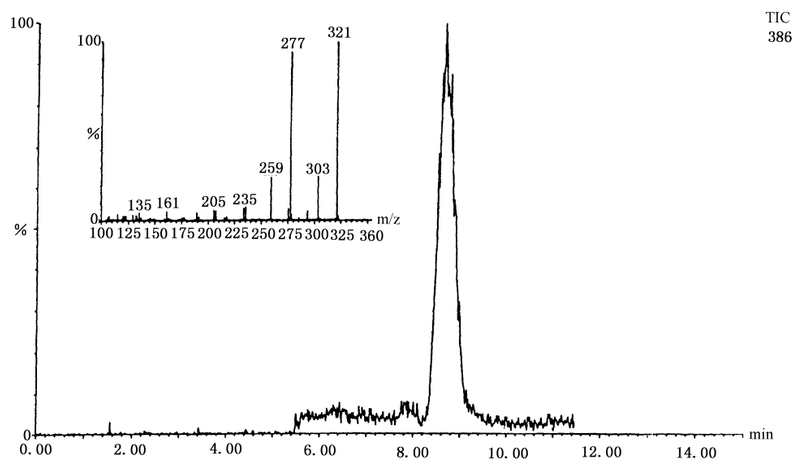

Figure A. 1 Chromatogram and mass spectrum of zeranol standard(conc. 0. 02 μg/mL)

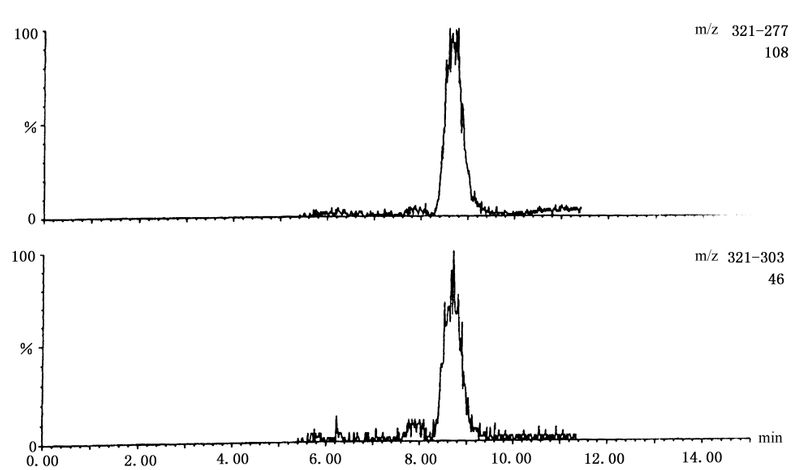

Figure A. 2 Mass chromatograms of zeranol standard using MRM mode(conc. 0. 02 μg/mL)

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中华人民共和国出入境检验检疫行业标准

SN/T 1544—2005

进出口动物源性食品中玉米赤霉醇残留量的检验方法 高效液相色谱-质谱/质谱法

Inspection of zeranol residues in animal original products for import and export—HPLC-MS/MS method

2005-02-17 发布 2005-07-01 实施

中华人民共和国
国家质量监督检验检疫总局 发布

3.4.3 Blank test

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

3.4.4 Calculations

The concentration of zeranol in sample is calculated with chromatographic data processor or with the following equation (1):

$$X = c \times \frac{V}{m} \qquad \cdots\cdots(1)$$

Where:

X——the concentration of analyte of interest in sample, μg/kg;

c——the concentration of analyte from calibration curve, ng/mL;

V——final volume of sample extract, mL;

m——weight of sample, g.

Note: The blank value should be subtracted from the above result of calculation.

4 Limit of Quantitation and Recovery

4.1 Limit of quantitation

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

4.2 Recovery

According to the experimental data, the fortifying concentration of zeranol and its corresponding recoveries are:

1 μg/kg, recovery 72%.

5 μg/kg, recovery 74%.

50 μg/kg, recovery 80%.

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um acetate buffer (3.2.13) and 25 μL of β-glucuronidase, mix well, and then incubate at 37℃ for 8 h～14 h.

After hydrolysis, add 10 mL of diethyl ether, cap and vortex for 3 min, and then centrifuge at 3 500 r/min for 10 min. Pipette the upper ether phase into a 15 mL conical centrifuge tube. Evaporate the ether phases nearly to dryness under nitrogen stream at 40℃. Repeat the above extraction one time. Dissolve the residue in 1 mL of chloroform. Extract zeranol from chloroform using 2×2.5 mL of sodium hydroxide solution (3.2.12). Vortex and centrifuge at 3 500 r/min for 5 min. Transfer the upper sodium hydroxide phase into a 10 mL centrifuge tube. Acidify the combined alkaline solution with 0.5 mL of phosphoric acid solution (3.2.11), and load onto a C_{18} solid-phase extraction cartridge (the cartridge was conditioned with 3 mL of methanol and 3 mL of sodium acetate buffer). After the alkaline solution percolates through, rinse the cartridge with 2 mL of sodium acetate buffer (3.2.13) and 2 mL of methanol-water solution (3.2.10). Apply 2.0 mL of methanol to the cartridge and collect the eluate into a 5 mL centrifuge tube (draw for 2 min to make the cartridge gradually dry). Evaporate the eluate to dryness under nitrogen stream at 55℃. Resuspend the residue with 200 μL of acetonitrile for analysis.

3.4.2 Determination

3.4.2.1 Liquid Chromatographic Conditions

a) Column: Zorbax Extend-C_{18} column (150 mm×2.1 mm, 5 μm i.d.), or equivalent.
b) Mobile phase: acetonitrile-water (40+60) containing 0.025% acetic acid.
c) Flow rate: 0.20 mL/min.
d) Column temperature: 22℃.
e) Injection volume: 10 μL.

3.4.2.2 Mass Spectrometric Conditions

a) Ionization mode: negative electrospray.
b) Cone voltage: 50 V.
c) Capillary voltage: 3 kV.
d) Collision voltage:20 V.
e) Analyzer vacuum: <3 mPa(3×10^{-5} mbar).
f) Collision gas: argon.
g) Cone gas flow: 80 L/h.
h) Nebuliser gas flow: 480 L/h.
i) Multiple reaction monitoring (MRM) mode: m/z 321→303, m/z 321→277. m/z 321→277 is used for quantitation.

3.4.2.3 LC-MS/MS detection

Analyze matrix-matched standards (3.2.16) in order of increasing concentration, obtaining the matrix-matched calibration curve of peak area against concentration. The detector response of sample should be in the range of calibration curve. Under such LC-MS/MS conditions, the retention time of zeranol is ca. 8.7 min. The mass chromatograms list in appendix A (appendix A.1 and A.2).

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:中华人民共和国上海出入境检验检疫局。
本标准主要起草人:方晓明、陈家华、倪昕路、唐毅锋。
本标准系首次发布的出入境检验检疫行业标准。