

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

SN/T 2222-2008

**Determination of glucocorticosteroids residues in foodstuffs of
animal origin for import and export. LC-MS/MS method**

进出口动物源性食品中糖皮质激素类兽药残留量检测方法 液相色谱-质谱/质谱法
(English Translation)

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Foreword

Appendix A of this standard is a normative appendix, Appendix B and Appendix C are informative appendices.

This standard is proposed and managed by the National Certification and Accreditation Administration Committee.

This standard by the People's Republic of China Liaoning Entry-Exit Inspection and Quarantine People's Republic of China, Zhejiang, China seized Inspection and Quarantine Science Research Institute, Heilongjiang People's Republic of China Exit Inspection and Quarantine is responsible for drafting.

1 Scope

This standard specifies the import and export of animal-derived foods triamcinolone, prednisolone, hydrocortisone, prednisone, dexamethasone, betamethasone fluorine, Triamcinolone acetonide Determination of Residual liquid chromatography - mass spectrometry/mass spectrometry methods.

This standard applies to pork, pig kidney glucocorticoid Residues in the detection and confirmation.

2 Method summary

First sample was added ammonium acetate buffer solution of hydrochloric acid and beta-glucuronidase enzymes glycosides - aryl sulfatase hydrolysis, and then extracted with ethyl acetate extract it was dried HLB SPE column purification, liquid chromatography - mass spectrometry/mass spectrometry and validation, external standard.

3 Reagents and materials

Unless otherwise specified, all reagents were of analytical grade, double distilled water.

3.1

acetonitrile. HPLC fractions.

3.2

hexane. HPLC fractions.

Ethyl acetate 3.3. HPLC grade.

3.4

Methanol. HPLC fractions.

3.5

ammonium acetate.

3.6

glacial acetic acid.

3.7

anhydrous sodium. 650 °C burning 4h, cooled in a desiccator to room temperature and stored in a sealed bottle aside.

3.8

beta- hydrochloric acid glucuronide glycoside enzymes - arylsulfatase. hydrochloric acid containing beta- glucuronidase enzyme glycosides 134600U/mL, arylsulfatase 5200U/mL.

3.9

Methanol - water (7 + 3, volume ratio) .30mL 70mL of methanol and water. Methanol - water (1 + 1, volume ratio).

3.10

methanol - water (5 + 5, volume ratio) .50mL 50mL of methanol and water.

3.11 0.02mol/L ammonium acetate buffer solution. 1.54g of ammonium acetate was dissolved in 950mL of water, the solution was adjusted to pH with glacial acetic acid 5.2, and finally diluted with water to 1L.

3.12

standard purity not less than 97%. Standard information, see Appendix A, Table A. 1.

3.13

glucocorticoid standard stock solution. Weigh the right amount of standard (3.12), dissolved in methanol formulated in a concentration of 100µg/mL standard Quasi stock solution, -18 °C cryopreservation, valid for three months.

3.14

standard solution. According to the need to use a blank sample solution was diluted to a stock standard solution of dexamethasone concentration of 1,2,4, respectively, 6 and 8ng/mL, the concentration of prednisolone solution (pork) were 6,8,16,24 and 40ng/mL, triamcinolone, hydrocortisone, splash Nison, Flumetasone, triamcinolone acetonide concentration of the mixed solution were 10,20,40,80 and 100ng/mL. Prior to use the existing service.

3.15

anhydrous sodium sulfate column. 80mm x 40mm (inner diameter) cylindrical funnel bottom pad 5mm cotton, refill 40mm anhydrous sodium sulfate.

3.16

SPE. Oasis (HLB) 500mg, or equivalent person.

The organic phase was 3.17 microporous membrane. 0.45µm.

4 Instruments and equipment

4.1

High performance liquid chromatography - mass spectrometry/mass spectrometer. with electrospray ionization source.