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

SN/T 3155-2012

**Determination of multi-veterinary drugs residues in pork,
shrimp and honey for export. LC-MS/MS method**

出口猪肉、虾、蜂蜜中多类药物残留量的测定 液相色谱-质谱/质谱法

(English Translation)

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Foreword

This standard was drafted in accordance with GB/T 1.1-2009 given rules.

Please note that some of the content of this document may involve patents. Distribution of

this document Institutions do not assume the responsibility to identify these patents.

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

This standard was drafted. People's Republic of China Zhejiang Exit Inspection and Quarantine.

1 Scope

This standard specifies the pork, shrimp and many types of drug residues in honey Determination of sample preparation and liquid chromatography - mass spectrometry/mass spectrometry.

This standard applies to pork, shrimp and honey sulfadiazine, Sulfathiazole, sulfasalazine, sulfamethyldiazine, sulfamethazine, sulfonamide Amine-5- (p) pyrimidine methicillin, sulfamethoxazole thiadiazole sulfa methoxy pyridazine, sulfa chlorine pyridazine, sulfonamides 6- (inter) methoxy pyrimidine sulfonamides o-methoxy-ethyl Piperidine, sulfamethoxazole, sulfamethazine isoxazole, benzoyl sulfonamides, sulfadimethoxine, sulfa quinoline morpholino, hydroxy metronidazole, 2-methyl-5-Nitroimidazole, hydroxy dimethyl Mi Zun nitrate, metronidazole, Dimetridazole, Ronidazole, chlorine metronidazole imidazole, nitrobenzene and imidazole, hydroxy propyl metronidazole, Isopropyl metronidazole, marbofloxacin, enoxacin, ofloxacin, norfloxacin, ciprofloxacin, danofloxacin, enrofloxacin, lomefloxacin, Ao Bisha Star, double difloxacin, sarafloxacin, sparfloxacin, oxolinic acid, nalidixic acid, flumequine, spiramycin, tilmicosin, oleandomycin, tylosin, Erythromycin, roxithromycin, josamycin, guitar neomycin, tiamulin, lincomycin, clindamycin, pirlimycin and praziquantel residues Testing.

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version suitable for use herein Member. For undated references, the latest edition (including any amendments) applies to this document.

Laboratory use specifications and test methods GB/T 6682 Analysis

3 PRINCIPLE OF THE METHOD

Pork, shrimp samples were extracted with acetonitrile, hexane, and then the C18 SPE column purification; honey diluted with phosphate buffer solution after Directly HLB solid phase extraction column purification, liquid chromatography - mass spectrometry/mass spectrometry, quantitative internal standard method or external standard method.

4 Reagents and materials

Unless otherwise indicated, the reagents used were HPLC grade water as a water line with GB/T 6682 regulations.

4.1

acetonitrile.

4.2

methanol.

4.3

formic acid.

4.4

n-hexane.

4.5

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

4.6

sodium dihydrogen phosphate. AR.

4.7

Sodium hydroxide. AR.

4.8 0.1mol/L sodium hydroxide solution. Weigh 4g of sodium hydroxide, and diluted with water to 1L.

4.9

Phosphate buffer solution. 13.8g of sodium dihydrogen phosphate were dissolved in 950mL water and extracted with 0.1mol/L sodium hydroxide solution to adjust the solution pH to 8.0, and finally diluted with water to 1L.

4.10

veterinary standards. sulfa drugs purity greater than or equal to 98%, nitroimidazoles purity greater than or equal to 98%, quinolones Purity of greater than or equal to 96%, macrolides purity of greater than or equal to 95%, lincosamides purity not less than 86% of drug praziquantel Greater than or equal to 98% purity, detailed criteria for substances listed in Table A.1.

4.11

isotope internal standard Standard. sulfadiazine -D4, Sulfathiazole -D6, sulfapyridine -D6,

sulfamethazine -D4, sulfamethoxazole Oxazole -D6, sulfadimethoxine -D4, sulfa quinoline morpholine -D6, hydroxy dimetridazole -D3, dimetridazole -D3, Ronidazole -D3, hydroxyl Isopropyl metronidazole -D3, isopropyl metronidazole -D3, enoxacin -D8, ofloxacin -D3, norfloxacin -D5, ciprofloxacin -D8, enrofloxacin -D5, Double difloxacin -D4, sarafloxacin -D8 isotope standards. purity greater than or equal to 98%, for more information see Table A.1.

4.12

formulated standard stock solution. learn were called commuted right amount standard (4.10) (accurate to 0.1mg), dissolved in methanol to volume, the concentration of various types of compound solution are 1mg/mL, 1 °C ~ 4 °C refrigerator stored.

Preparation 4.13 isotope internal standard stock solution. Weigh the right amount of isotope labeled internal standard (4.11), dissolved in methanol to volume, each the concentration of compound solution are 1mg/mL, 1 °C ~ 4 °C refrigerator stored.

4.14

intermediate standard solution is prepared by mixing. standard stock solution was diluted with methanol (4.12) to a mixed standard solution containing sulfa final Drug concentration of 500ng/mL, nitroimidazole drugs at a concentration of 500ng/mL, a concentration of quinolones 1000ng/mL, a large ring Esters drug concentration of 1500ng/mL, Lim drug concentration amides of 1000ng/mL, praziquantel drug concentration of 150ng/mL, 1 °C ~ 4 °C refrigerator stored.

4.15

blank sample extract. containing no pork, shrimp or honey sample test drug, in accordance with 7.1 blank sample solutions were prepared.

4.16

matrix mixed standard working solution. a mixed intermediate standard solution (4.14) diluted to fit as needed blank matrix solution (4.15) Suitable concentrations of mixed standard working solution, using now.

4.17

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

4.18

C18 SPE column. 500mg, 3mL, or rather who, prior to use 5mL methanol pre-wash.

4.19

Oasis (HLB) SPE column, 500mg, 6mL or equivalent person. Before using successively with 5mL methanol, 5mL phosphate buffer Red solution (4.9) Pre-wash.