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中华人民共和国出入境检验检疫行业标准

SN/T 2222—2008

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

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

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前　言

本标准的附录A为规范性附录，附录B和附录C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。

本标准由中华人民共和国辽宁出入境检验检疫局、中华人民共和国浙江出入境检验检疫局、中国检验检疫科学研究院、中华人民共和国黑龙江出入境检验检疫局负责起草。

本标准主要起草人：林维宣、谢文、康庆贺、孙兴权、肖珊珊、田苗、奚君阳、于灵。

本标准系首次发布的出入境检验检疫行业标准。

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

1 范围

本标准规定了进出口动物源性食品中曲安西龙、泼尼松龙、氢化可的松、泼尼松、地塞米松、氟米松、曲安奈德残留量测定液相色谱-质谱/质谱检测方法。

本标准适用于猪肉、猪肾中糖皮质激素类兽药残留量的检测和确证。

2 方法提要

样品先加入醋酸铵缓冲溶液和β-盐酸葡萄糖醛甙酶-芳基硫酸酯酶水解，再用乙酸乙酯提取，提取液经 HLB 固相萃取小柱净化，液相色谱-质谱/质谱测定和确证，外标法定量。

3 试剂和材料

除另有规定外，所有试剂均为分析纯，水为二次蒸馏水。

3.1 乙腈：高效液相色谱级。

3.2 正己烷：高效液相色谱级。

3.3 乙酸乙酯：高效液相色谱级。

3.4 甲醇：高效液相色谱级。

3.5 乙酸铵。

3.6 冰乙酸。

3.7 无水硫酸钠：650 ℃灼烧 4 h，在干燥器内冷却至室温，贮于密封瓶中备用。

3.8 β-盐酸葡萄糖醛甙酶-芳基硫酸酯酶：含 β-盐酸葡萄糖醛甙酶 134 600 U/mL，芳基硫酸酯酶 5 200 U/mL。

3.9 甲醇-水（3＋7，体积比）：30 mL 甲醇与 70 mL 水混合。甲醇-水（1＋1，体积比）。

3.10 甲醇-水（5＋5，体积比）：50 mL 甲醇与 50 mL 水混合。

3.11 0.02 mol/L 醋酸铵缓冲溶液：溶解 1.54 g 醋酸铵于 950 mL 水中，用冰乙酸调节溶液 pH 值到 5.2，最后用水稀释至 1 L。

3.12 标准品纯度大于等于 97％。标准品信息见附录 A 表 A.1。

3.13 糖皮质激素标准储备溶液：称取适量标准品（3.12），用甲醇溶解配制成浓度为 100 μg/mL 的标准储备溶液，－18℃冷冻保存，有效期 3 个月。

3.14 标准工作溶液：根据需要用空白样品溶液将标准储备液稀释成地塞米松溶液浓度分别为 1、2、4、6 和 8 ng/mL，泼尼松龙的溶液浓度（猪肉）分别为 6、8、16、24 和 40 ng/mL，曲安西龙、氢化可的松、泼尼松、氟米松、曲安奈德混合溶液浓度分别为 10、20、40、80 和 100 ng/mL。临用前现配。

3.15 无水硫酸钠柱：80 mm×40 mm（内径）筒形漏斗，底部垫 5 mm 脱脂棉，再装 40 mm 无水硫酸钠。

3.16 固相萃取柱：Oasis（HLB）500 mg，或相当者。

3.17 有机相微孔滤膜：0.45 μm。

4 仪器和设备

4.1 高效液相色谱-质谱/质谱仪：配有电喷雾离子源。

4.2　旋转蒸发器。

4.3　粉碎机。

4.4　均质器。

4.5　旋涡混合器。

4.6　离心机：7 000 r/min。

4.7　氮吹仪。

4.8　天平：感量为 0.01 g 和 0.000 1 g。

4.9　固相萃取装置。

4.10　恒温箱。

5　试样制备与保存

从所取全部样品中取出有代表性样品约 500 g，用粉碎机粉碎，混合均匀，均分成两份，分别装入洁净容器作为试样，密封，并标明标记。将试样于－18 ℃冷冻保存。

在抽样和制样的操作过程中，应防止样品污染或发生残留物含量的变化。

6　测定步骤

6.1　提取

称取 5 g 试样（精确到 0.01 g）置于 50 mL 具塞塑料离心管中，加 1.5 mL 甲醇，再加入 23.5 mL 醋酸铵缓冲溶液（3.11）和 40 μL β-盐酸葡萄糖醛甙酶-芳基硫酸酯酶（3.8），以 2 000 r/min 混匀 1 min，于恒温箱中 37 ℃培养 16 h，以 6 000 r/min 离心 5 min，量取 10.0 mL 上清液，加入 20 mL 乙酸乙酯，以 2 000 r/min 混匀 1 min，以 4 000 r/min 离心 5 min，将上层乙酸乙酯提取液过无水硫酸钠柱，滤液收集于浓缩瓶中，样品残渣再加入 20 mL 乙酸乙酯，重复上述操作，合并乙酸乙酯提取液，在 45 ℃以下水浴减压浓缩至近干。

6.2　净化

OasisHLB 柱（3.16）使用前依次用 5 mL 甲醇和 5 mL 水预洗。用 5 mL 醋酸铵缓冲溶液（3.11）溶解残渣，将溶液转移至 OasisHLB 柱，弃去流出液，用 5 mL 水和 5 mL 甲醇水溶液（3.9）依次洗涤，弃去流出液，负压抽干，8 mL 甲醇洗脱，收集全部洗脱液，在 50 ℃以下水浴减压浓缩至近干，用 1.0 mL 甲醇-水（3.10）定容，混匀，将溶液通过 0.45 μm 滤膜，供液相色谱-质谱/质谱仪测定。

6.3　测定

6.3.1　液相色谱-质谱/质谱条件

a）　色谱柱：C_8 柱，150 mm×4.6 mm（内径），5 μm 或相当者；

b）　流动相：乙腈-水，梯度见表 1；

表 1　梯度洗脱程序

时间/min	乙腈/%	水/%
0	20	80
8	50	50
15	50	50
18.5	60	40
20	60	40
21	90	10
25	90	10
25.5	20	80
28.5	20	80

c) 流速：0.4 mL/min；

d) 进样量：50 μL；

e) 离子源：电喷雾离子源；

f) 扫描方式：正离子扫描；

g) 检测方式：多反应监测；

h) 雾化气、气帘气、辅助气、碰撞气均为高纯氮气；使用前应调节各气体流量以使质谱灵敏度达到检测要求，参考条件参见附录 B；

i) 监测离子对(m/z)：参见附录 B 表 B.1。

6.3.2 高效液相色谱-质谱/质谱测定

根据试样中被测样液的含量情况，选取待测物的响应值在仪器线性响应范围内的浓度进行测定，如超出仪器线性响应范围应进行稀释。在上述色谱条件下曲安西龙、泼尼松龙、氢化可的松、泼尼松、地塞米松、氟米松、曲安奈德的参考保留时间约分别为 9.0、11.9、12.1、12.2、13.7、14.0、14.8 min，标准溶液的选择性离子流图参见附录 C 中图 C.1。

6.3.3 液相色谱-质谱/质谱确证

按照液相色谱-质谱/质谱条件测定样品和标准工作溶液，样品中待测物质的保留时间与标准溶液中待测物质的保留时间偏差在±2.5%之内。定量测定时采用标准曲线法。定性时应当与浓度相当标准工作溶液的相对丰度一致，相对丰度允许偏差不超过表 2 规定的范围，则可判断样品中存在对应的被测物。

表 2 定性确证时相对离子丰度的最大允许偏差

相对离子丰度/%	>50	>20～50	>10～20	≤10%
允许的相对偏差/%	±20	±25	±30	±50%

6.3.4 空白试验

除不加试样外，均按上述操作步骤进行。

7 结果计算和表述

用色谱数据处理机或按式(1)计算试样中糖皮质激素残留含量，计算结果需扣除空白值：

$$X_i = \frac{c_i \times V}{m} \qquad \cdots\cdots(1)$$

式中：

X_i——试样中糖皮质激素类残留量，单位为微克每千克(μg/kg)；

c_i——从标准曲线上得到的糖皮质激素溶液浓度，单位为纳克每毫升(ng/mL)；

V——样液最终定容体积，单位为毫升(mL)；

m——最终样液代表的试样质量，单位为克(g)。

8 测定低限(LOQ)和回收率

8.1 测定低限(LOQ)

猪肉：地塞米松方法测定低限为 0.75 μg/kg，泼尼松龙方法测定低限为 4 μg/kg，曲安西龙、氢化可的松、泼尼松、氟米松、曲安奈德方法测定低限为 10 μg/kg。

猪肾：地塞米松方法测定低限为 0.75 μg/kg，曲安西龙、泼尼松龙、氢化可的松、泼尼松、氟米松、曲安奈德方法测定低限为 10 μg/kg。

8.2 回收率

在不同添加浓度的回收率数据见表 3。

表 3　回收率数据

基质	化合物	添加浓度/(μg/kg)	回收率/%
猪肉	曲安西龙	10	71.2～94.6
		20	70.0～96.5
		40	70.7～97.7
	泼尼松龙	4	61.5～98.5
		8	63.7～105.0
		16	70.6～101.9
	氢化可的松	10	74.9～100.4
		20	71.0～93.0
		40	71.5～101.2
	泼尼松	10	71.0～91.2
		20	73.0～97.0
		40	71.5～93.0
	地塞米松	0.75	61.3～94.7
		1.5	71.3～102.0
		3	67.7～101.3
	氟米松	10	70.1～96.3
		20	71.5～91.0
		40	70.0～97.7
	曲安奈德	10	71.0～92.0
		20	70.0～97.0
		40	70.7～85.7
猪肾	曲安西龙	10	71.4～91.2
		20	72.5～105.0
		40	70.7～92.7
	泼尼松龙	10	70.0～96.7
		20	70.0～96.5
		40	70.0～95.7
	氢化可的松	10	72.1～98.4
		20	71.0～96.5
		40	71.0～99.0
	泼尼松	10	71.7～94.5
		20	71.5～98.0
		40	70.0～93.0
	地塞米松	0.75	62.6～97.3
		1.5	64.0～96.7
		3	71.7～97.0
	氟米松	10	70.6～94.1
		20	71.5～96.5
		40	71.0～93.5
	曲安奈德	10	71.2～96.3
		20	71.5～98.0
		40	70.7～94.0

附　录　A

（规范性附录）

糖皮质激素标准品信息

表 A.1　糖皮质激素类药物标准品信息

名　　称	英文名称	CAS 号	分子式	相对分子质量
曲安西龙	Ttriamcinolone	124-94-7	$C_{21}H_{27}FO_6$	394.43
泼尼松龙	Prednisolone	50-24-8	$C_{21}H_{28}O_5$	360.4
氢化可的松	Hydrocortisone	50-23-7	$C_{21}H_{30}O_5$	362.5
泼尼松	Prednisone	53-03-2	$C_{21}H_{26}O_5$	358.4
地塞米松	Dexamethasone	50-02-2	$C_{22}H_{29}FO_5$	392.5
氟米松	Flumethasone	2135-17-3	$C_{22}H_{28}F_2O_5$	410.45
曲安奈德	Triamcinolone acetonide	76-25-5	$C_{24}H_{31}FO_6$	434.5

附　录　B
（资料性附录）
API 4000 LC-MS/MS 系统电喷雾离子源参考条件[1]

监测离子对及电压参数：

a） 电喷雾电压(IS)：4 500 V；

b） 雾化气压力(GS1)：262.01 kPa(38 psi)；

c） 气帘气压力(CUR)：186.165 kPa(27 psi)；

d） 辅助气流速(GS2)：310.275 kPa(45 psi)

e） 离子源温度(TEM)：525 ℃；

f） 碰撞气(CAD)34.475 kPa：(5 psi)；

g） 离子对、去簇电压(DP)、碰撞能量(CE)碰撞室出口电压(CXP)见表 B.1。

表 B.1　离子对、去簇电压(DP)碰撞气能量(CE)碰撞室出口电压(CXP)

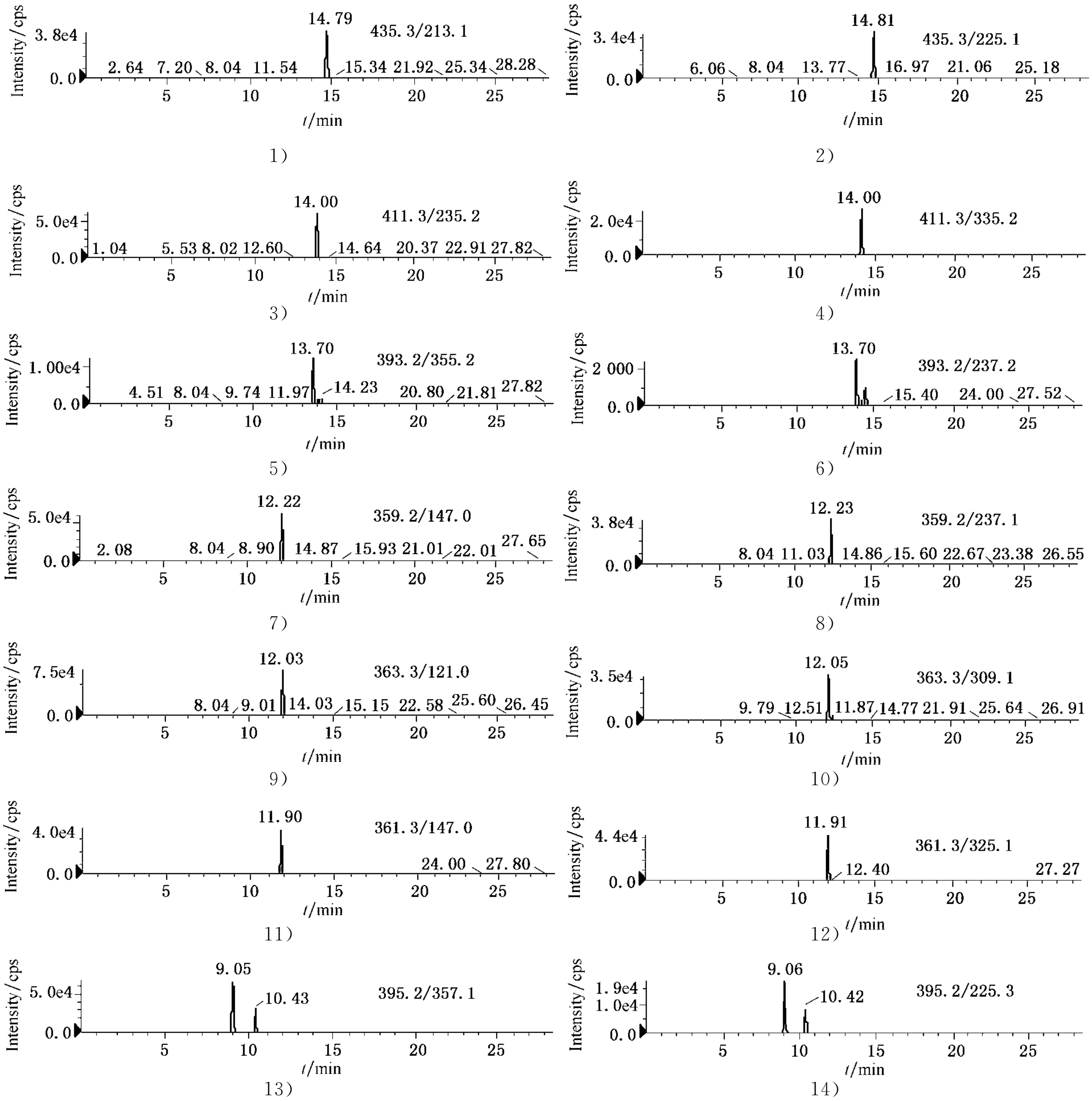
名　　称	离子对 m/z	去簇电压(DP)/V	碰撞气能量(CE)/V	碰撞室出口电压(CXP)/V
曲安西龙	395.2/357.1[a] 395.2/225.3	65	19 29	10 5
泼尼松龙	361.3/147.0[a] 361.3/325.1	61	35 16	7 9
氢化可的松	363.3/121.0[a] 363.3/309.1	90	40 25	5 7
泼尼松	359.2/147.0[a] 359.2/237.1	77	30 38	10
地塞米松	393.2/355.2[a] 393.3/237.2	63	18 28	10 12
氟米松	411.3/253.2[a] 411.3/335.2	77	26 18	6 9
曲安奈德	435.3/213.1[a] 435.3/225.1	62	39 36	10 11
[a] 为定量离子对。				

1） 非商业性声明：附录 B 所列参数是在 API 4000 质谱仪完成的，此处列出试验用仪器型号仅是为了提供参考，并不涉及商业目的，鼓励标准使用者尝试不同厂家和型号的仪器。

附　录　C

（资料性附录）

标准质量色谱图



1)　2)　3)　4)　5)　6)　7)　8)　9)　10)　11)　12)　13)　14)

图 C.1　曲安西龙、泼尼松龙、氢化可的松、泼尼松、地塞米松、氟米松、曲安奈德标准品的选择性离子流图

Foreword

Annex A of this standard is normative annex, Annex B and C of this standard are informative annex.

This standard was proposed by and is under the charged of certification and accreditation administation of the People's Republic of China.

This standard was drafted by Liaoning Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Zhejiang Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Chinese Academy of Inspection and Quarantine, Heilongjiang Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

The standard was mainly drafted by Lin Weixuan, Xie Wen, Kang Qinghe, Sun Xingquan, Xiao Shanshan, Tian Miao, Xi Junyang, Yu Ling.

This standard is a professional standard for entry-exit inspection and quarantine promulgated for the first time.

Note: This English Version, a translation from the Chinese text, is solely for guidance.

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

1 Scope

The standard specifies the determination by LC-MS/MS of triamcinolone, prednisolone, hydrocortisone, prednisone, dexamethasone, flumethasone, triamcinolone acetonide residues in foodstuffs of animal origin.

This standard is applicable to the determination of glucocorticosteroids residues in pork meat and pork kidney.

2 Principle

Sample is added with ammonium acetate buffer and β-glucuronidase. Then it is extracted from the sample by ethyl acetate. It is cleaned up with HLB column. Finally it is determined and confirmed by LC-MS/MS. External standard method is used.

3 Reagents and materials

Unless otherwise specified, all the reagent used should be analytical grade, "water" is double distilled water.

3.1 Acetonitrile: HPLC grade.

3.2 *n*-hexane: HPLC grade.

3.3 Ethyl acetate: HPLC grade.

3.4 Methanol: HPLC grade.

3.5 Ammonium acetate: HPLC grade.

3.6 Acetic acid.

3.7 Anhydrous sodium sulphate: Ignite for 4 h at 650 ℃, cool to room temperature in desiccator and keep in a tightly closed container.

3.8 β-glucuronidase: 100 U/mL.

3.9 Methanol-water(3+7, *V*/*V*):30 mL methanol mix with 70 mL water.

3.10 Methanol-water (5+5, *V*/*V*):50 mL methanol mix with 50 mL water.

3.11 0.02 mol/L ammonium acetate buffer: Dissolve 1.54 g ammonium acetate in 950 mL water, adjust to pH 5.2 with acetic acid and dilute to 1 L.

3.12 Standards: Purity≥97%. Standard information see table A.1 in annex A.

3.13 Stock standard solution: Accurately weigh appropriate standard(3.12), dissolve with methanol, the concentration of solution is 100 μg/mL. It should be stored in brown volumtric flask at −18 ℃ in refrigerator. Stock standard solution is stable for 3 months.

3.14 Calibration curve standard working solutions: Working solutions were prepared in blank sample solution. The concentration of dexamethasone is 1, 2, 4, 6 and 8 ng/mL, prednisone (pork meat) is 6, 8, 16, 24 and 40 ng/mL, triamcinolone, prednisolone, hydrocortisone, prednisone, dexamethasone, flumethasone, triamcinolone acetonide is 10, 20, 40, 80 and 100 ng/mL. It is prepared before using.

3.15 Column of anhydrous sodium sulfate: 80 mm × 40 mm (i.d.) cylinder funnel, pack with ca 5 mm absorbent cotton at the bottom of the column and fill in 40 mm anhydrous sodium sulfate.

3.16 Column: Oasis(HLB)C_{18} 500 mg, or equivalent.

3.17 Organic filter: 0.45 μm.

4 Apparatus and equipment

4.1 Liquid chromatograhy with electrospray ionization mass spectrometry.

4.2 Rotary vacuum evaporator.

4.3 Blend.

4.4 Homogenizer.

4.5 Vortex mixer.

4.6 Centrifuge: 7 000 r/min.

4.7 Nitrogen evaporator.

4.8 Balances (0.01 g, 0.000 1 g).

4.9 SPE-12G Column Processor.

4.10 Incubator.

5 Preparation of test sample

Take the representative portions from the whole sample. It is about 500 g and ground in a blender. Keep the prepared sample into two sample bottles, seal and label. The rest sample is stored in −18 ℃ refrigerator.

In the course of sample preparation, precautions must be taken to avoid contamination or any factors, which may cause the change of residue content.

6 Analytical Procedure

6.1 Extraction

Weigh ca 5 g of the test sample (accurate to 0.01 g) into a 50 mL centrifuge tube. Add 1.5 mL methanol. Add 23.5 mL ammonium acetate buffer and 40 μL β-glucuronidase (3.8). Blend for 1 min at 2 000 r/min. The mixture incubate 37 ℃ for 16 h. Centrifuge for 5 min under 6 000 r/min. Transfer 10.0 mL the supernatant layer, add 20 mL ethyl acetate. Blend for 1 min at 2 000 r/min. Centrifuge for 5 min under 4 000 r/min. Ethyl acetate layer was passed through anhydrous sodium sulfate column into flask. Repeat the extraction in the same way with 20 mL ethyl acetate, combined the solution. The solution is evaporated to nearly dryness in a water bath below 45 ℃.

6.2 Clean up

OasisHLB column(3.16)is conditioned with 5 mL methanol followed by 5 mL water. Add 5 mL ammonium acetate buffer (3.11) to dissolve the residues. Transfer the above solution into the OasisHLB column discard the eluate. Rinse the column with 5 mL water and 5 mL methanol-water (3.9), discard the eluate. The cartridge is evacuated continuously to “dryness”. Elute the column with 8 mL methanol. The solution is evaporated to nearly dryness in a water bath below 50 ℃. Add exactly 1.0 mL methanol-water (3.10) to dissolve the residues. The solution is passed through 0.45 μm filter. It is ready for LC-MS/MS determination.

6.3 Determination

6.3.1 LC-MS/MS operating conditions

a) Column: C_8, 150 mm × 4.6 mm (i.d.), 5 μm or the equivalent;

b) Mobile phase: acetonitrile-water see table 1;

Table 1—Gradient of mobile phase

Time/min	acetonitrile/%	water/%
0	20	80
8	50	50
15	50	50
18.5	60	40
20	60	40
21	90	10
25	90	10
25.5	20	80
28.5	20	80

c) Flow rate: 0.4 mL/min;

d) Injection volume: 50 μL;

e) Source: ESI;

f) Polarity: Positive;

g) Mode: Multiple reaction monitoring;

h) Carrier gas: Nitrogen (purity > 99.999%). Instrumental settings may be optimized. Conditionsis in annex B;

i) Transitions (m/z): See table B.1 in annex B.

6.3.2 LC-MS/MS determination

According to the concentrations of analyte in sample solution, content should be within the linear range of the calibration curve. If it is over the range, the solution should be diluted. Under the above LC-MS/MS operating condition, the retention times of triamcinolone, prednisolone, hydrocortisone, prednisone, dexamethasone, flumethasone and triamcinolone acetonide are 9.0, 11.9, 12.1, 12.2, 13.7, 14.0 and 14.8 min. Selected ion chromatograms of the standards see Figure C.1 in annex C.

6.3.3 LC-MS/MS confirmation

Under LC-MS/MS conditions, the working solution and sample solution are injected. The retention time of the analyte in sample solution shall correspond to that of the analyte in standard solution. Tolerance is within ±2.5%. Calibration curve method is used for quantitative measurement. The relative intensities of sample transitions shall correspond to those of standard solution transitions for confirmation. The concentration of standard solution should be same with those of sample solution. The permitted tolerances listed in table 2, and then the corresponding analyte must be present in sample.

Table 2—**Maximum permitted tolerances for relative ion intensities while confirmation**

Relative intensity/%	>50	>20～50	>10～20	≤10
Permitted/%	±20	±25	±30	±50

6.3.4 Blank test

The operation of the blank test is the same as the described in the method of determination, but with the omission of sample addition.

7 Calculation and expression of result

Calculation the content of glucocorticosteroids residues in the test sample by LC-MS/MS data processor or according to the formula(1), the blank value should be subtracted from the above result of calculation.

$$X_i = \frac{c_i \times V}{m} \qquad \cdots\cdots\cdots\cdots (1)$$

Where:

X_i —the residue content of glucocorticosteroids residues in the test sample, μg/kg;

c_i —the concentration of glucocorticosteroids residues is from calibration curve, ng/mL;

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

m —mass of test sample of final sample solution, g.

8 Limit of quantification(LOQ)and recovery

8.1 Limit of quantification

Pork meat: The limit of quantification for dexamethasone is 0.75 μg/kg, for prednisolone is 4 μg/kg, for triamcinolone, hydrocortisone, prednisone, flumethasone, triamcinolone acetonide is 10 μg/kg.

Pork kidney: The limit of quantification for dexamethasone is 0.75 μg/kg, for is 4 μg/kg, for triamcinolone, prednisolone, hydrocortisone, prednisone, flumethasone, triamcinolone acetonide is 10 μg/kg.

8.2 Recovery

According to the experimental data, the corresponding recoveries of fortifying concentrations arein Table 3.

Table 3—The recoveries of glucocorticosteroids in different matrix

Matrix	Compound	Spike level /（μg/kg）	Recovery/%	Matrix	Compound	Spike level /（μg/kg）	Recovery/%
Pork meat	Triamcinolone	10	71.2～94.6	Pork kidney	Triamcinolone	10	71.4～91.2
		20	70.0～96.5			20	72.5～105.0
		40	70.7～97.7			40	70.7～92.7
	Prednisolone	4	61.5～98.5		Prednisolone	10	70.0～96.7
		8	63.7～105.0			20	70.0～96.5
		16	70.6～101.9			40	70.0～95.7
	Hydrocortisone	10	74.9～100.4		Hydrocortisone	10	72.1～98.4
		20	71.0～93.0			20	71.0～96.5
		40	71.5～101.2			40	71.0～99.0
	Prednisone	10	71.0～91.2		Prednisone	10	71.7～94.5
		20	73.0～97.0			20	71.5～98.0
		40	71.5～93.0			40	70.0～93.0
	Dexamethasone	0.75	61.3～94.7		Dexamethasone	0.75	62.6～97.3
		1.5	71.3～102.0			1.5	64.0～96.7
		3	67.7～101.3			3	71.7～97.0
	Flumethasone	10	70.1～96.3		Flumethasone	10	70.6～94.1
		20	71.5～91.0			20	71.5～96.5
		40	70.0～97.7			40	71.0～93.5
	Triamcinolone acetonide	10	71.0～92.0		Triamcinolone acetonide	10	71.2～96.3
		20	70.0～97.0			20	71.5～98.0
		40	70.7～85.7			40	70.7～94.0

Annex A

(normative annex)

The information of glucocorticosteroids standards

Table A. 1—The information of glucocorticosteroids standards

Compound	CAS	Formula	Molecular weight
Ttriamcinolone	124-94-7	$C_{21}H_{27}FO_6$	394. 43
Prednisolone	50-24-8	$C_{21}H_{28}O_5$	360. 4
Hydrocortisone	50-23-7	$C_{21}H_{30}O_5$	362. 5
Prednisone	53-03-2	$C_{21}H_{26}O_5$	358. 4
Dexamethasone	50-02-2	$C_{22}H_{29}FO_5$	392. 5
Flumethasone	2135-17-3	$C_{22}H_{28}F_2O_5$	410. 45
Triamcinolone acetonide	76-25-5	$C_{24}H_{31}FO_6$	434. 5

Annex B

（informative annex）

API 4000 LC-MS/MS conditions[1]

Instrumental settings：

a） IS：4 500 V；

b） GS1：262.01 kPa（38 psi）；

c） CUR：186.165 kPa（27 psi）；

d） GS2：310.275 kPa（45 psi）；

e） TEM：525 ℃；

f） CAD：34.475 kPa：（5 psi）；

g） Transitions see table B.1.

Table B.1 Transitions，DP，CE and CXP and CE

Compound	Transitions m/z	DP/V	CE/V	CXP/V
Ttriamcinolone	395.2/357.1[a] 395.2/225.3	65	19 29	10 5
Prednisolone	361.3/147.0[a] 361.3/325.1	61	35 16	7 9
Hydrocortisone	363.3/121.0[a] 363.3/309.1	90	40 25	5 7
Prednisone	359.2/147.0[a] 359.2/237.1	77	30 38	10
Dexamethasone	393.2/355.2[a] 393.3/237.2	63	18 28	10 12
Flumethasone	411.3/253.2[a] 411.3/335.2	77	26 18	6 9
Triamcinolone acetonide	435.3/213.1[a] 435.3/225.1	62	39 36	10 11

[a] quantification.

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[1] Non-commercial statement：the equipments and their models involved in the standard method are not related to commercial motive. The analysts are encouraged to use different equipments and models.

Annex C

(informative annex)

Selected ion chromatograms of glucocorticosteroids standards

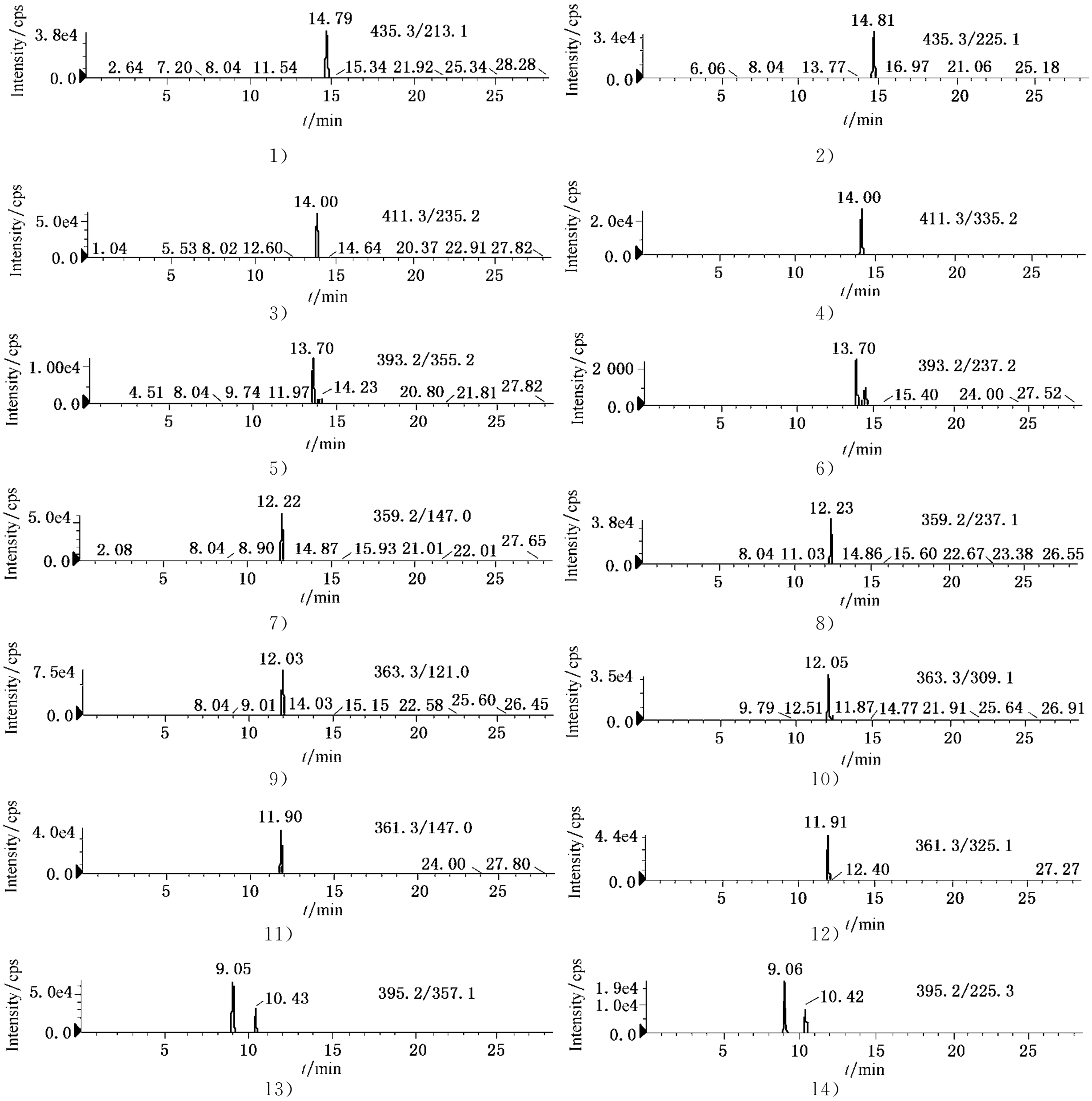

Figure C. 1—Selected ion chromatograms of glucocorticosteroids standards

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