SN

中华人民共和国出入境检验检疫行业标准

SN/T 2221—2008

进出口动物源性食品中氮哌酮及其代谢产物残留量的检测方法 气相色谱-质谱法

Determination of azaperone and its metabolite residues in foodstuffs of animal origin for import and export—GC-MS method

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前言

本标准的附录A和附录B均为资料性附录。

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、中华人民共和国辽宁出入境检验检疫局。

本标准主要起草人:李波、郭德华、王传现、卫锋、杨惠琴、林维宣、邓晓军、朱坚、韩丽。

本标准系首次发布的出入境检验检疫行业标准。

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进出口动物源性食品中氮哌酮及其代谢产物残留量的检测方法 气相色谱-质谱法

1 范围

本标准规定了动物源性食品中氮哌酮及其代谢产物氮哌醇残留量检测的气相色谱-质谱测定方法。

本标准适用于猪肉和猪肾中氮哌酮及其代谢产物氮哌醇残留量的测定和确证。

2 方法提要

试样用乙腈提取，经 C_{18}SPE 柱净化，酸性乙腈洗脱，正己烷液液分配，叔丁基甲醚萃取后，氮哌醇再经硅烷化衍生，用气相色谱-质谱法测定，内标法定量。

3 试剂和材料

除另有规定外，所用试剂均为分析纯，水为蒸馏水。

3.1 甲醇：液相色谱纯。

3.2 正己烷：液相色谱纯。

3.3 乙腈：液相色谱纯。

3.4 乙酸乙酯：液相色谱纯。

3.5 叔丁基甲醚：液相色谱纯。

3.6 硫酸：98%。

3.7 氢氧化钠。

3.8 氯化钠。

3.9 10%氯化钠溶液：称取 100 g 氯化钠溶于适量水中，并用水定容至 1 L。

3.10 硫酸溶液：将 28 mL 硫酸(3.6)缓缓加入装有 500 mL 水的烧杯中，混匀冷却至室温备用。

3.11 0.01 mol/L 硫酸溶液：移取 20 mL 硫酸溶液(3.10)至装有 250 mL 水的 1 L 量杯中，加水至 1 L。

3.12 0.05 mol/L 硫酸溶液：移取 100 mL 硫酸溶液(3.10)至装有 250 mL 水的 1 L 量杯中，加水至 1 L。

3.13 酸性乙腈溶液：在 100 mL 乙腈中加入 1 mL 0.05 mol/L 硫酸溶液(3.12)。

3.14 0.5 mol/L 氢氧化钠溶液：称取 20 g 氢氧化钠溶于适量水，并用水定容至 1 L。

3.15 乙腈饱和正己烷溶液：取 100 mL 正己烷，30 mL 乙腈于分液漏斗中，振荡混匀，待用。

3.16 衍生化试剂：双三甲基硅基三氟乙酰胺(BSTFA)-三甲基氯硅烷(TMCS)(99+1，体积比)。

3.17 2,3,4-2′,4′,5′-六氯联苯(PCB138)：纯度大于等于 98%。

3.18 内标溶液：用乙酸乙酯配制 2,3,4-2′,4′,5′-六氯联苯(PCB138)浓度为 0.10 μg/mL。

3.19 氮哌酮标准品(Azaperone，CAS 号：1649-18-9，分子式：$C_{19}H_{22}FN_3O$)：纯度大于等于 98%。

3.20 氮哌醇(Azaperol，CAS 号：2804-05-9，分子式：$C_{19}H_{24}FN_3O$)标准溶液：10 μs/mL 甲醇溶液，在 −18 ℃下避光保存。

3.21 氮哌酮标准储备溶液：准确称取适量氮哌酮标准品，用甲醇配制成浓度为 10 μg/mL 的标准储备液，在 −18 ℃下避光保存，有效期半年。

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3.22 混合标准工作溶液：各准确移取 0.50 mL 氮哌醇标准溶液(3.20)和氮哌酮标准储备溶液(3.21)于 10 mL 棕色容量瓶并用甲醇定容，溶液浓度约为 0.5 μg/mL。在－18 ℃下避光保存，有效期 3 个月。

3.23 C_{18} SPE 柱：Sep Pak Vac 1 g，6 mL 或相当者。

4 仪器和设备

4.1 气相色谱-质谱仪：四极杆质谱仪，配有 EI 源并具有选择离子功能。

4.2 分析天平：感量 0.1 mg 和 0.01 g。

4.3 旋涡混匀器。

4.4 组织捣碎机。

4.5 旋转蒸发器。

4.6 固相萃取装置。

4.7 离心机：转速不低于 4 000 r/min，配有 10 mL 和 50 mL 塑料离心管。

4.8 氮气吹干仪。

4.9 超声波水浴。

5 试样制备与保存

5.1 试样制备

从所取全部样品中取出有代表性样品约 500 g，充分捣碎均质，均分成两份，分别装入洁净容器内。密封作为试样，标明标记。

5.2 试样保存

将试样于－18 ℃以下保存。

在制样的操作过程中，应防止样品受到污染或发生残留物含量的变化。

6 测定步骤

6.1 提取

称取约 5 g 均匀试样(精确到 0.01 g)，置于 50 mL 塑料离心瓶中，加 20 mL 乙腈，置于旋涡混匀器上涡旋混匀 45 s，再超声提取 10 min，4 000 r/min 离心 10 min。吸取 10 mL 上层乙腈溶液至一已存放 50 mL 10％氯化钠溶液(3.9)的玻璃瓶中，混匀，置 0 ℃～4 ℃存放约 0.5 h，待净化。

6.2 净化

将 C_{18} SPE 柱(3.23)安装到固相萃取装置上，用 10 mL 甲醇和 10 mL 水活化，加入上述提取液(6.1)，控制流速 2 滴/s，用 1 mL 0.01 mol/L 硫酸溶液(3.11)淋洗，并排干柱中液体，再用 3 mL 酸性乙腈溶液(3.13)洗脱并收集于 10 mL 塑料离心管中，于 40 ℃下用氮气吹至约 300 μL。立即加 1 mL 乙腈饱和的正己烷(3.15)，并在旋涡混匀器上高速混匀 30 s 后，4 000 r/min 离心 3 min，弃去正己烷层，再加 1 mL 乙腈饱和的正己烷重复操作一次。残留溶液加入 150 μL 0.5 mol/L 氢氧化钠溶液(3.14)，调节 pH 至 10，慢慢加入 1 mL 叔丁基甲醚，旋涡混匀 30 s 后，4 000 r/min 离心 3 min，移取上层有机相至一玻璃小试管中，再用 2×1 mL 叔丁基甲醚重复提取 2 次，合并提取液于 30 ℃下用氮气吹干。

6.3 衍生化

用 200 μL 甲醇溶解残渣并转移至衍生瓶中，再重复操作一次，合并溶液。同时吸取 0 μL、50 μL、100 μL、200 μL、400 μL、800 μL 混合标准使用溶液(3.22)于另一系列衍生瓶中。同步把样品提取液和混合标准溶液在 30 ℃下用氮气吹干，加入 100 μL 衍生化试剂(3.16)，混匀，于 70 ℃烘箱内衍生 20 min，冷却后用氮气吹干，加 100 μL 内标溶液(3.18)溶解残渣，供气相色谱-质谱测定。

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6.4 测定

6.4.1 气相色谱-质谱条件

a) 色谱柱：HP-5MS，30 m×0.25 mm(内径)×0.25 μm(膜厚)，或相当者；

b) 升温程序：80 ℃保持 1 min，以 20 ℃/min 升至 190 ℃(保持 2 min)，再以 20 ℃/min 升至 250 ℃(保持 15 min)；

c) 载气：氦气，纯度≥99.999%，流速 1.0 mL/min；

d) 进样口温度：250 ℃；

e) 进样方式：无分流进样，1.0 min 后开阀；

f) 进样量：2 μL～3 μL；

g) 电离方式：EI，70 eV；

h) 接口温度：280 ℃；

i) 离子源温度：230 ℃；

j) 溶剂延迟：13.5 min；

k) 测定方式：选择离子监测方式(SIM)；

l) 监测离子：见表 1。

表 1 氮哌酮和氮哌醇衍生物的监测离子及其丰度比

名 称	监测离子(m/z)	监测离子丰度比/%
氮哌醇衍生物	107(定量离子)、294、204、401	100∶21∶16∶7
氮哌酮	107(定量离子)、123、233、327	100∶35∶26∶7
PCB138 内标	360(定量离子)	—

6.4.2 气相色谱-质谱测定

根据样液中被测组分含量，选定浓度相近的标准工作溶液。其响应值均应在仪器检测的线性范围内。对标准工作溶液与样液等体积参插进样测定，以色谱峰面积按内标法定量。在上述色谱条件下，内标 PCB138、氮哌醇衍生物和氮哌酮的参考保留时间分别为 14.2 min、20.2 min、20.6 min，标准溶液的选择离子色谱图参见附录 A 中图 A.1。

6.4.3 气相色谱-质谱确证

在相同实验条件下，试样中待测物质的保留时间与标准工作溶液中对应的保留时间偏差在±2.5%之内；并且被测样液与标准品的质谱图相似，所选择的全部监测离子均出现；而且之间的丰度比也相一致，其允许偏差不超过表 2 规定的范围时，则可确定为样品中存在这种药物残留。标准品的质谱图参见附录 B 中图 B.1 和图 B.2。

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/%	±10	±15	±20	±50

6.5 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)计算样品中氮哌酮和氮哌醇残留量。计算结果需扣除空白值。

$$X_i = \frac{c_s \times c' \times A_i \times A'_s \times V}{c'_s \times A' \times A_s \times m} \qquad \cdots\cdots(1)$$

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式中：

X_i——试样中氮哌酮或氮哌醇残留量，单位为毫克每千克(mg/kg)；

c_s——标准工作溶液中氮哌酮或氮哌醇的浓度，单位为微克每毫升(μg/mL)；

c'——样液中内标物的浓度，单位为微克每毫升(μg/mL)；

A_i——样液中氮哌酮或氮哌醇的峰面积；

A'_s——标准工作溶液中内标物的峰面积；

V——样液最终定容体积，单位为毫升(mL)；

c'_s——标准工作溶液中内标物的浓度，单位为微克每毫升(μg/mL)；

A'——样液中内标物的峰面积；

A_s——标准工作溶液中氮哌酮或氮哌醇的峰面积；

m——最终样液代表的试样质量，单位为克(g)。

本方法氮哌酮的残留量测定结果系指氮哌酮和氮哌醇残留量之和。

8 测定低限和回收率

8.1 测定低限

本方法测定低限：0.01 mg/kg。

8.2 回收率

8.2.1 猪肉

添加水平为0.010 mg/kg时，氮哌酮回收率为72.0%～107%；氮哌醇回收率为68.0%～103%。

添加水平为0.050 mg/kg时，氮哌酮回收率为76.8%～106%；氮哌醇回收率为70.4%～96.8%。

添加水平为0.100 mg/kg时，氮哌酮回收率为76.0%～108%；氮哌醇回收率为72.4%～103%。

8.2.2 猪肾

添加水平为0.010 mg/kg时，氮哌酮回收率为72.0%～108%；氮哌醇回收率为68.0%～106%。

添加水平为0.050 mg/kg时，氮哌酮回收率为78.4%～106%；氮哌醇回收率为68.8%～98.4%。

添加水平为0.100 mg/kg时，氮哌酮回收率为71.2%～105%；氮哌醇回收率为70.0%～101%。

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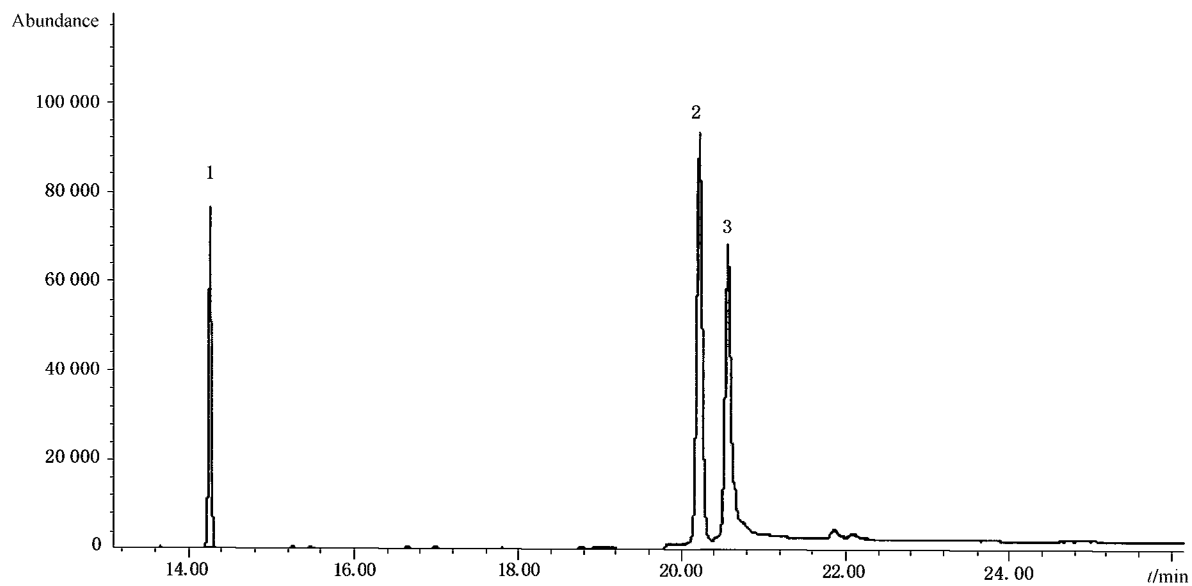
附　录　A
（资料性附录）
标准品选择离子色谱图



1——PCB138；
2——氮哌醇衍生物；
3——氮哌酮。

图 A.1　氮哌醇标准品衍生物、氮哌酮标准品的选择离子色谱图

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附 录 B
（资料性附录）
标准品质谱图

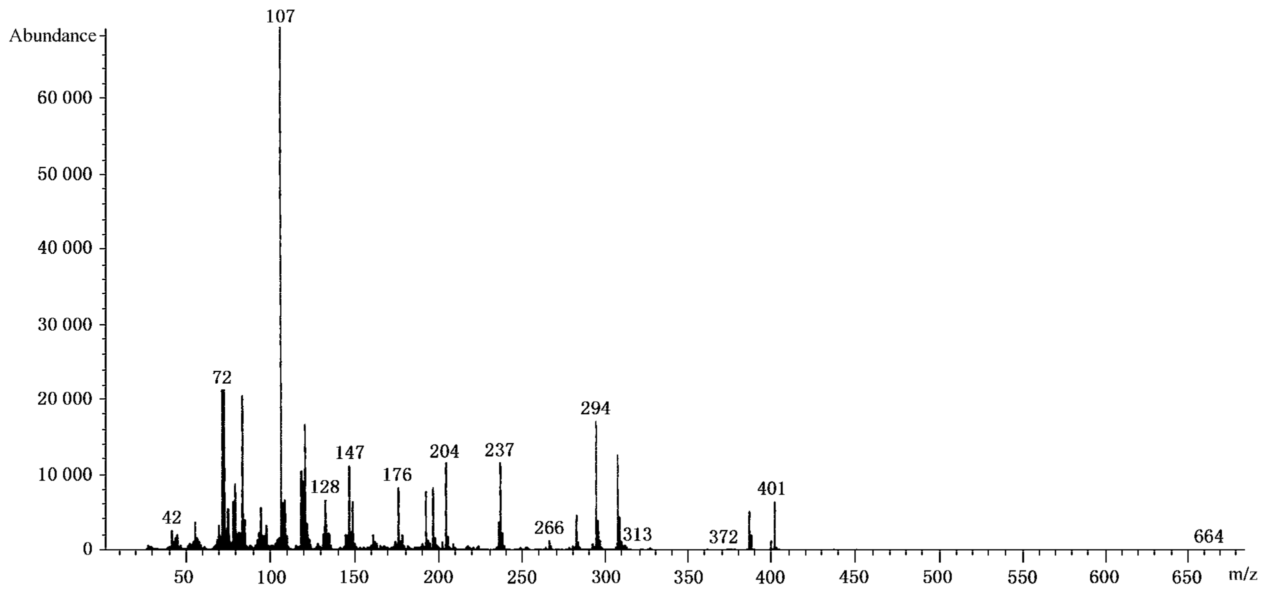

图 B.1 氮哌醇标准品衍生物的全扫描质谱图

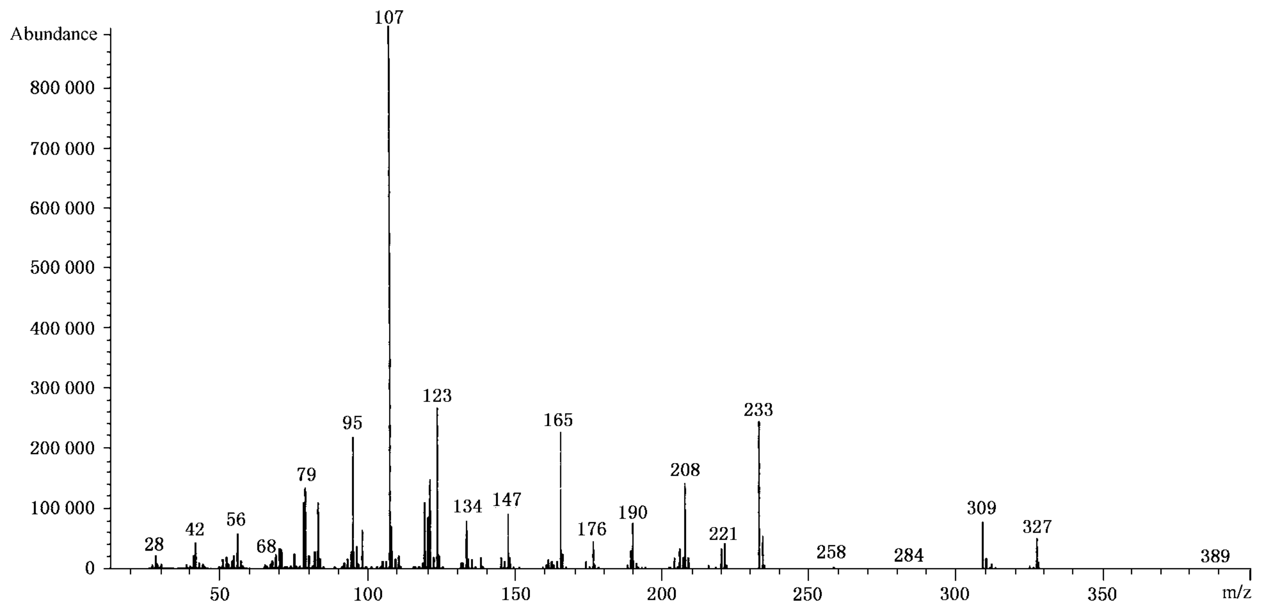

图 B.2 氮哌酮标准品的全扫描质谱图

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Foreword

Annex A and annex B of this standard is an 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 Shanghai Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Liaoning Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

The standard was mainly drafted by Li Bo, Guo Dehua, Wang Chuanxian, Wei Feng, Yang Huiqin, Lin Weixuan, Deng Xiaojun, Zhu Jian, Han Li.

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.

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Determination of azaperone and its metabolite residues in foodstuffs of animal origin for import and export—GC-MS method

1 Scope

The standard specifies the methods of sample preparation and determination of azaperone and its major metabolite,azaperol residues by GC-MS in foodstuffs of animal origin.

This standard is applicable to the determination and confirmation of azaperone and its major metabolite,azaperol residues in pork and kidney.

2 Principle

Azaperone and Azaperol residues were extracted from the sample with acetonitrile,extract through C_{18} SPE cartridge. Re-extracted with tert-butylmethyl ether after defatting with hexane. The residues derivatized with BSTFA-TMCS,determined by GC-MS,and quantified by internal standard method.

3 Reagents and materials

Unless otherwise specified,all the reagent used should be analytical grade,"water" was Milli-Q quality.

3.1 Methanol:HPLC grade.

3.2 Hexane:HPLC grade.

3.3 Acetonitrile:HPLC grade.

3.4 Ethyl acetate:HPLC grade.

3.5 Tert-butylmethyl ether(TBME):HPLC grade.

3.6 Sulfuric acid.

3.7 Sodium hydroxide.

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3.8 Sodium chloride.

3.9 10% sodium chloride solution:100 g sodium chloride was dissolved in 1 L water.

3.10 Sulfuric acid solution:28 mL sulfuric acid (3.6) was poured into 500 mL water slowly and carefully.

3.11 0.01 mol/L,sulfuric acid solution:20 mL sulfuric acid solution (3.10) was added to 250 mL water carefully,and water was added to 1 L.

3.12 0.05 mol/L sulfuric acid solution:100 mL sulfuric acid solution (3.10) was added to 250 mL water carefully,and water was added to 1 L.

3.13 Acidified acetonitrile:1 mL 0.05 mol/L sulfuric acid solution (3.12) was added to 100 mL acetonitrile.

3.14 0.5 mol/L sodium hydroxide solution:20 g sodium hydroxide was dissolved in 1 L water.

3.15 Hexane solution saturated with acetonitrile:30 mL acetonitrile was added to 100 mL hexane, vibrated and separated.

3.16 Silylating agent:BSTFA-TMCS(99+1,*V*/*V*).

3.17 PCB138:Purity≥98%.

3.18 Internal standard solution:prepared about 0.10 μg/mL of PCB 138 in ethyl acetate.

3.19 Azaperone:Purity≥98%.

3.20 Azaperol standard solution:10 μg/mL methanol,store at −18 ℃ in black.

3.21 Azaperone stock standard:Prepared the azaperone stock standard solution at the concentration of 10 μg/mL with methanol,store at −18 ℃ in black.

3.22 Mixture working standard solutions:Dilute azaperol standard solution (3.20) and azaperone stock standard (3.21) to 0.5 μg/mL in methanol,store at −18 ℃ in black.

3.23 C_{18} SPE cartridge:Sep Pak Vac 1 g,6 mL

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4 Apparatus and equipment

4.1 Gas chromatography combined with a quardrupole mass spectrometer.

4.2 Balances:0.1 mg and 0.01 g.

4.3 Vortex mixer.

4.4 Sample processor.

4.5 Rotary vacuum evaporator.

4.6 Apparatus of SPE.

4.7 Centrifuge:4 000 r/min.

4.8 Nitrogen evaporator.

4.9 Ultrasonator.

5 Preparation and storage of test sample

5.1 Preparation of sample

About 500 g representative samples should be taken from all samples, then grinded and blended to produce homogenous samples, divided into two equal portions and put in suitable clean containers, sealed and labeled.

5.2 Storage of sample

The prepared samples should be stored in −18 ℃ refrigerator.

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

6 Procedure

6.1 Extraction

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Weigh ca 5 g of the test sample (accurate to 0.01 g) into a 50 mL centrifuge plastic tubes with a plug, add 20 mL acetonitrile, mix on a vortex mixer for 45 s and in ultrasonator for 10 min . After that, the tubes were centrifuged at 4 000 r/min for 10 min. Transfer 10 mL of the supernatant acetonitrile to a clean bottle which contained 50 mL 10% sodium chloride solution (3.9), then mix for clean-up.

6.2 Clean up

Transfer the above solution (6.1) into the C_{18} SPE cartridge (3.23) which was conditioned with 10 mL methanol and 10 mL water, control the rate of 2 drop/s. Add 1 mL 0.01 mol/L sulfuric acid solution (3.11) to the reservoir and elute; discard the eluate. Elute the analytes with 3 mL acidified acetonitrile (3.13) in a 10 mL centrifuge plastic tube, the collected eluate was concentrated to about 300 μL under a stream of nitrogen gas at 40 ℃. Add 1 mL hexane (3.15), mix on a vortex mixer for 30 s, centrifuged at 4 000 r/min for 3 min, and discard the hexane phase. Repeat defatted procedure with another 1 mL hexane (3.15). Add 150 μL 0.5 mol/L sodium hydroxide solution (3.14) and adjust pH to 10, and then add 1 mL tert-butylmethyl ether, mix on a vortex mixer for 30 s, centrifuged at 4 000 r/min for 3 min, move the upper level to the clean tube, repeat extracting with another two times, then combine the ether phase, which was evaporated to dryness under a stream of nitrogen gas at 30 ℃.

6.3 Derivatization

The residue was transferred to a vial of 2 mL volume with two 200 μL methanol, and in the meanwhile withdraw 0 μL, 50 μL, 100 μL, 200 μL, 400 μL, 800 μL mixture working standard solutions (3.22) to a series vials. The solvent was evaporated to dryness and the residue derivatized with 100 μL of Silylating agent (3.16) for 20 min at 70 ℃. The extracts were evaporated to dryness and the residues dissolved in 100 μL of internal standard solution (3.18), the solutions are ready for GC-MS determination.

6.4 Determination

6.4.1 GC-MS operating conditions

a) Column: HP-5MS, 30 m × 0.25 mm (i.d.) × 0.25 μm, or the equivalent;

b) Column temperature: 80 ℃ for 1 min, rise to 190 ℃ at 20℃/min, hold for 2 min, rise to 250 ℃ at 20 ℃/min, hold for 15 min;

c) Carrier gas: Helium, purity≥99.999%, 1.0 mL/min;

d) Injection port temperature: 250 ℃;

e) Injection mode: Splitless, purge after 1.0 min;

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f) Injection volume:2 μL～3 μL;

g) Ionisation mode:EI,70 eV;

h) Transfer line temperature:280 ℃;

i) Source temperature:230 ℃;

j) Solvent delay:13.5 min;

k) Monitor mode:SIM;

l) Monitor ions:see table 1.

Table 1—The monitor ions and relative abundance for Azaperone and Azaperol-TMS

Compound	The monitor ions(m/z)	The relative abundance/%
Azaperol-TMS	107(the quantitative ion),294,204,401	100∶21∶16∶7
Azaperone	107(the quantitative ion),123,233,327	100∶35∶26∶7
PCB138	360(the quantitative ion)	—

6.4.2 GC-MS determination

According to the approximate concentration of component determination in the test sample solution, select the standard working solution with similar concentration. The responses of the analyte in the standard working solution and the sample solution should be within the linear range of the instrument detection. The standard working solution should be injected randomly in between the injections of sample solution of equal volume. Quantified by internal standard. Under the above GC-MS operating condition, the retention time of PCB138, AZL-TMS and AZN is about 14.2 min, 20.2 min and 20.6 min, the selected ion chromatograms of the standards see Figure A.1 in annex A.

6.4.3 GC-MS confirmation

Determinated under the same GC-MS conditions, the retention times of sample chromatogram peaks are consistent with that of working solution, and the relative abundance ratio tolerance is listed in table 2, it is safe to conclude that this compound do exist in the sample. Mass spectrogram of the standards see FigureB.1 and B.2 in annex B.

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

Relative intensity/%	>50	>20～50	>10～20	≤10
Permitted tolerances/%	±10	±15	±20	±50

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6.5 Blank test

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

7 Calculation and expression of result

Calculation the content of Azaperone or Azaperol residue in the test sample by GC-MS data processor or according to the formula (1). The blank value should be subtracted from the above result of calculation.

$$X_i = \frac{c_s \times c' \times A_i \times A'_s \times V}{c'_s \times A' \times A_s \times m} \qquad \cdots\cdots (1)$$

Where:

X_i—the residue content of Azaperone or Azaperol in the test sample, mg/kg;
c_s—the concentration of Azaperone or Azaperol in standard working solution, μg/mL;
c'—the concentration of PCB138 in sample solution, μg/mL;
A_i—the peak area of Azaperone or Azaperol in sample solution;
A'_s—the peak area of PCB138 in standard working solution;
V—the final volume of the sample solution, mL;
c'_s—the concentration of PCB138 in standard working solution, μg/mL;
A'—the peak area of PCB138 in sample solution;
A_s—the peak area of Azaperone or Azaperol in standard working solution;
m—mass of test sample of final sample solution, g.

The test result means the sum of Azaperone or Azaperol.

8 Limit of quantitation (LOQ) and recovery

8.1 Limit of quantitation

The limit of quantitation (LOQ) of the method is 0.01 mg/kg.

8.2 Recovery

8.2.1 Pork

When spiking level was 0.010 mg/kg, the recovery of azaperone range from 72.0%～107%; the recovery of azaperol range from 68.0%～103%.

When spiking level was 0.050 mg/kg, the recovery of azaperone range from 76.8%～106%; the re-

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covery of azaperol range from 70.4%～96.8%.

When spiking level was 0.100 mg/kg, the recovery of azaperone range from 76.0%～108%; the recovery of azaperol range from 72.4%～103%.

8.2.2 kidney

When spiking level was 0.010 mg/kg, the recovery of azaperone range from 72.0%～108%; the recovery of azaperol range from 68.0%～106%.

When spiking level was 0.050 mg/kg, the recovery of azaperone range from 78.4%～106%; the recovery of azaperol range from 68.8%～98.4%.

When spiking level was 0.100 mg/kg, the recovery of azaperone range from 71.2%～105%; the recovery of azaperol range from 70.0%～101%.

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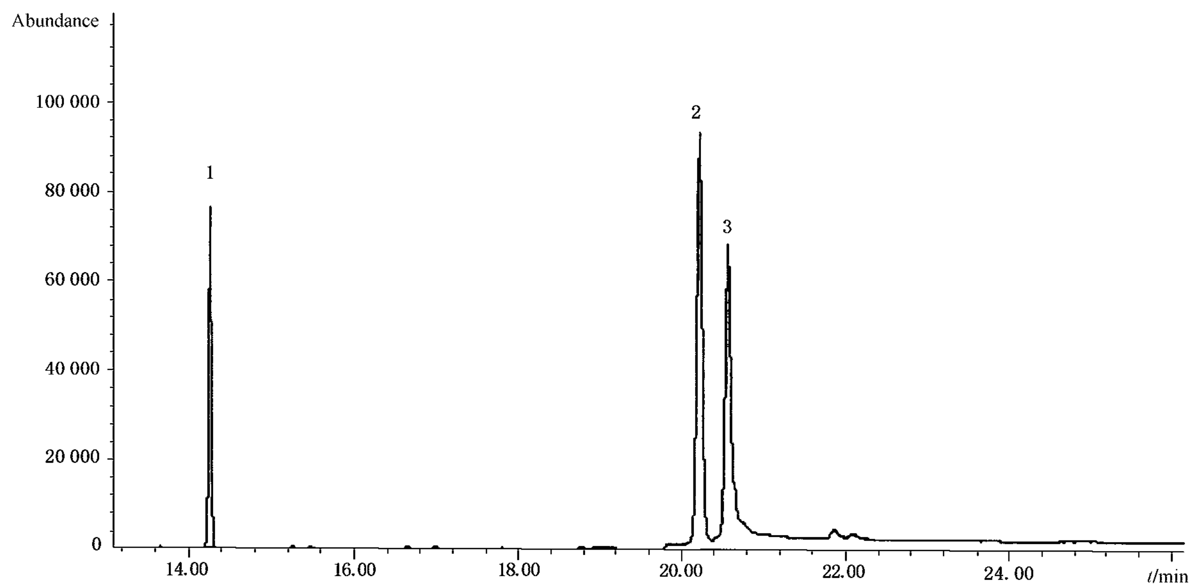
Annex A
(informative annex)
Selected ion chromatograms of the standards



1—PCB138;

2—Azaperol-TMS;

3—Azaperone.

Figure A. 1—The selected ion chromatiograms of the standards

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Annex B

(informative annex)

Mass spectrogram of the standards

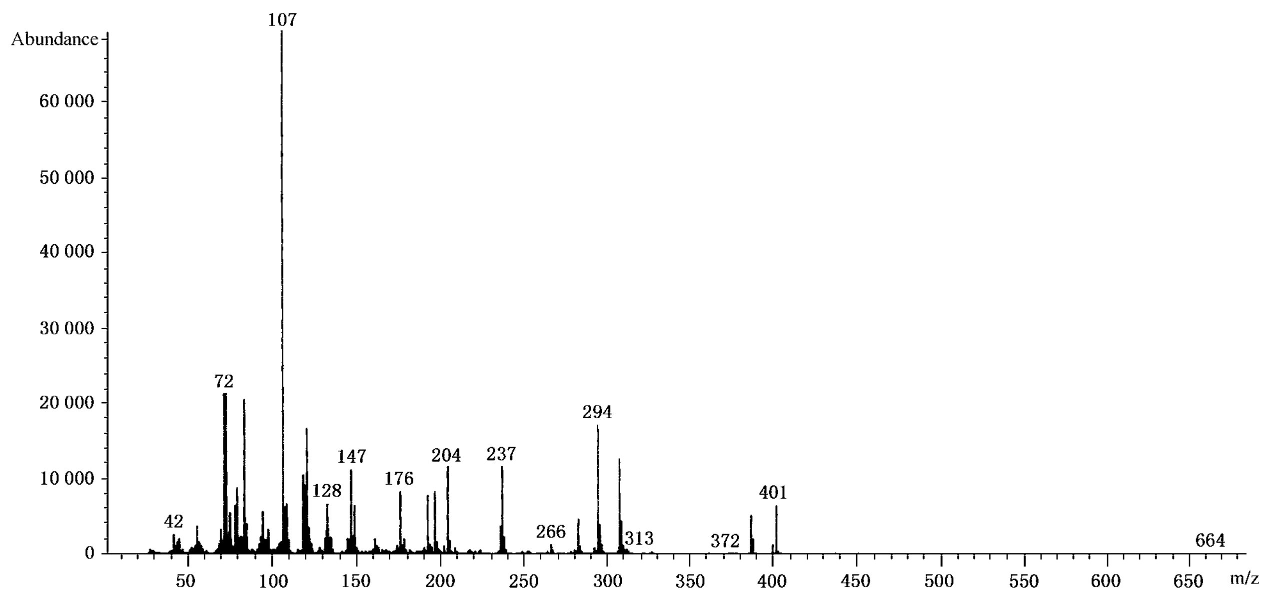


Figure B. 1—GC-MS(EI)spectrum of Azaperol-TMS standard

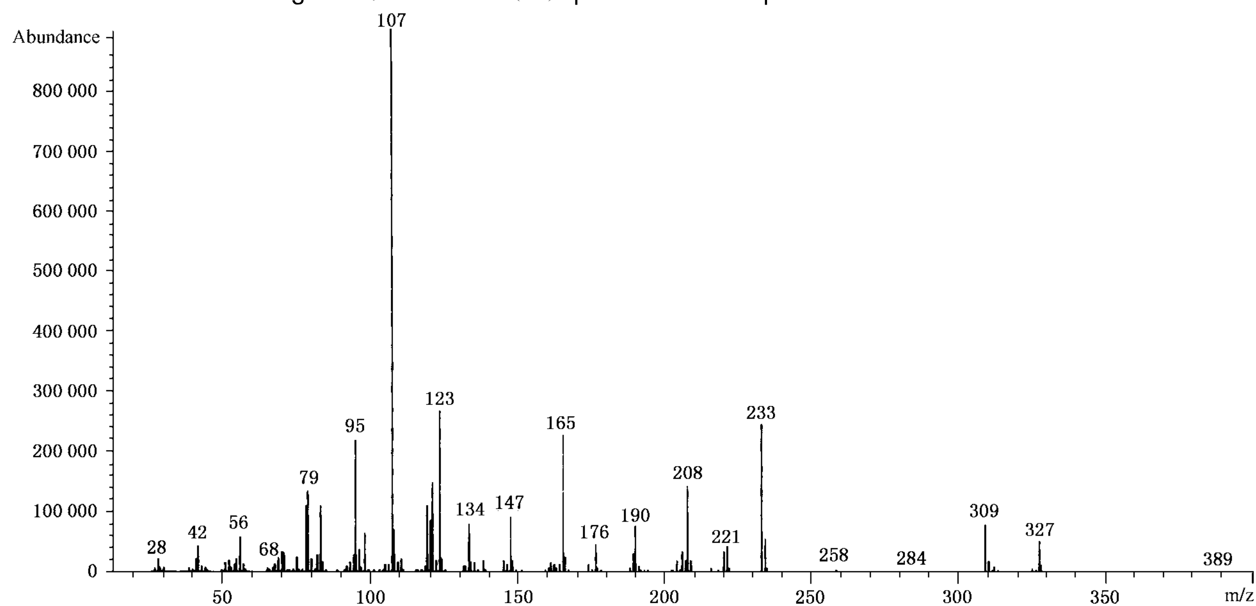
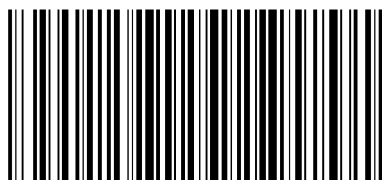


Figure B. 2—GC-MS(EI)spectrum of Azaperone standard



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