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

SN/T 5219-2019

**Determination of Aminopyralid and Amilopic Acid Residues in
Exported Food Liquid Chromatography-Mass Spectrometry /
Mass Spectrometry**

出口食品中氨基吡啶酸、氯氨基吡啶酸残留量的测定 液相色谱-质谱质谱法
(English Translation)

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Foreword

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

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organization of this standard. Shenzhen Customs of the People's Republic of China.

1 Scope

This standard specifies the sample preparation and liquid chromatography-mass spectrometry/mass spectrometry methods for amlpicolinic acid and clopyralid in exported food.

This standard applies to Chinese cabbage, spinach, tomatoes, Chinese kale, apples, bananas, dragon fruit, pears, rice, wheat, pork, the determination and confirmation of the

residues of amphipalic acid and clopyralic acid in foods such as beef, chicken, shrimp, and crucian.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

After extracting the aminopyridine acid and the aminopyridine acid in the sample with acidified acetonitrile, the modified QuEChERS (150 mg C18 900 mg Na2SO4) reagent, after purifying the extract, the liquid chromatography tandem mass spectrometer was used for qualitative and quantitative analysis.

Standard method for quantification.

4 Reagents and materials

Unless otherwise specified, the reagents used are all analytically pure, and the water is first-grade water that meets the requirements of GB/T 6682.

4.1

Acetonitrile. chromatographically pure.

4.2

Methanol. chromatographically pure.

4.3

Formic acid.

4.4

n-hexane.

4.5

Anhydrous magnesium sulfate.

4.6

C18 powder.

4.7

Sodium chloride.

4.8

Ammonium acetate.

4.9

Anhydrous sodium sulfate.

4.10

Apiclopyridine (Picloram, $C_6H_3Cl_3N_2O_2$, CAS No. 1918-02-1). the purity is greater than or equal to 99.0%.

4.11

Aminopyralid (Aminopyralid, $C_6H_4Cl_2N_2O_2$, CAS No. 150114-71-9). The purity is greater than or equal to 99.0%.

4.12

Standard stock solution. accurately weigh out 10 mg of shampyridine acid (4.10) and shampyridine acid (4.11) respectively, and use methanol (4.2) Dissolve and dilute to 10 mL. Store in the dark at $-18^{\circ}C$. The shelf life is 6 months.

4.13

Mixed standard solution. Pipette 100 μL of each substance standard stock solution (4.12), add the blank matrix extract to make the volume up to 10 mL, Store in the dark at $-18^{\circ}C$, and the shelf life is 1 month.

4.14

Microporous membrane. 0.22 μm , organic.

5 Apparatus and equipment

5.1

Liquid chromatography tandem mass spectrometer. equipped with an electrospray ion source (ESI).

5.2

Analytical balance. Sensitivity is 0.1 mg and 0.01 g respectively.

5.3

Homogenizer.

5.4

High-speed centrifuge. the speed is not less than 9 500 r/min.

5.5

Nitrogen blowing instrument.

5.6

Vortex mixer.

5.7

Organize the masher.

6.1 Sample preparation

Take 500 g of a representative sample, use a tissue masher to process the sample into a slurry, mix well, put it into a clean sample container, and seal it.

And mark it.

6.2 Sample storage

The samples should be frozen and stored below -18°C.

During the operation of sample preparation and storage, the samples should be prevented from being contaminated or from changing the content of residues.

6.3.1 Fruit and vegetable samples

Weigh 10.0 g of the sample, accurate to 0.01 g, and place it in a 50 mL centrifuge tube, add 10 mL of 1% formic acid acetonitrile, 9 500 r/min After 5 min, add 4g of anhydrous methyl sulfate (4.5) and 1g of sodium chloride (4.7), vortex for 10 min, and centrifuge at 9 500 r/min for 5 min.

6.3.2 Grain samples

Weigh 5.0 g of the sample, accurate to 0.01 g, place it in a 50 mL centrifuge tube, add 3 mL of water, vortex to soak the sample, add 10 mL of 1% formic acid acetonitrile, 9 500 r/min, homogenize for 5 minutes, then add 4 g of anhydrous sulfuric acid (4.5), 1 g of sodium chloride (4.7), and vortex.

Shake for 10 min, centrifuge at 9 500 r/min for 5 min.

6.3.3 Meat samples

Weigh 2.0 g of the sample, accurate to 0.01 g, place it in a 50 mL centrifuge tube, add 2 mL of 5 mmol/L ammonium acetate buffer, and vortex After spin-soaking the sample, add 10