



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

GB 23200.64-2016

Replacing SN/T 2623-2010

**Food safety national standard -- Determination of Residual
Potassium Acetate in Food by Liquid Chromatography -- Mass
Spectrometry / Mass Spectrometry**

食品中吡丙醚残留量的测定 液相色谱-质谱 质谱法

(English Translation)

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Foreword

This standard replaces SN/T 2623-2010 "Liquid Chromatography-Mass Spectrometry/Mass Spectrometry" for the determination of pyruvate residues in food for import and export.

Compared with SN/T 2623-2010, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "import and export food" to "food";

- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 2623-2010.

National standards for food safety Determination of Residual Potassium Acetate in Food by Liquid Chromatography - Mass Spectrometry/Mass Spectrometry

1 Scope

This standard specifies the preparation and determination of the content of pyruvate residues in food.

This standard applies to rice, soybeans, spinach, lemon, tea, mushrooms, beef, liver and milk in the detection of dibutyl ether residues, Other food can refer to the implementation.

2 Normative reference documents

The following documents are indispensable for the application of this document. For dated references, only the dated edition applies to this article Pieces. For undated references, the latest edition (including all modifications) applies to this document.

GB 2763 National Standard for Food Safety - Maximum Residue Limit of Pesticides in Foodstuffs.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle

The residual pyridyl ether in the sample was extracted with acidic acetonitrile under the action of sodium acetate buffer and purified by PSA packing. The liquid chromatography-mass spectrometry Determination of the external standard method.

4 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade and water is in accordance with the primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

anhydrous magnesium sulfate (MgSO_4).

4.1.2

anhydrous sodium acetate ($\text{C}_2\text{H}_3\text{O}_2\text{Na}$).

4.1.3

acetic acid (C₂H₄O₂). chromatography grade.

4.1.4

Acetonitrile (C₂H₃N). Chromatographic grade.

4.1.5

N-propyl ethylenediamine (PSA) filler. 50 m, chromatographic grade.

4.1.6

Formic acid (CH₂O₂). Chromatographic grade.

4.1.7

Ammonium acetate (C₂H₇NO₂).

4.2 Solution preparation

4.2.1 0.025% formic acid aqueous solution (containing 5 mmol/L ammonium acetate). accurately absorb 0.25 mL formic acid and weigh 0.386 g ammonium acetate in 1 L volumetric flask, dissolved in water and diluted to 1 L.

4.2.2 1% acetic acid in acetonitrile solution.

4.3 Standards

4.3.1

Pyridine ether standard (Pyriproxyfen, C₂₀H₁₉NO₃, CAS. 95737-68-1). purity > 99%.

4.4 Standard solution preparation

4.4.1

Pyridyl ether standard stock solution. accurately weighed the appropriate amount of pyridyl ether standard, dissolved in acetonitrile and diluted with 100 µg/mL standard Quasi-stock solution, stored below 4 °C.

4.4.2

Pyridyl ether standard working solution. If necessary, with 10% acetonitrile aqueous solution diluted to the appropriate concentration of standard working fluid, 4 °C below Save

4.5 Materials

4.5.1

Microporous membrane. 0.2 m, organic phase type.

5 Instruments and equipment

5.1

Liquid Chromatography-Mass Spectrometry/Mass Spectrometer. Electrospray (ESI) ion source.

5.2

Centrifuge. 4 000 r/min.

5.3

Analysis of balance. 0.0001 g and 0.01 g.

5.4

plugs with polypropylene centrifugal tubes. 2 mL and 50 mL.

5.5

Crusher.

6.1 Rice, soybeans, spinach, lemon, tea, mushrooms, beef, liver

Take a representative sample of about 500 g, with the tissue crusher fully mashed and mix, are divided into two, respectively, into the clean container as a test kind. Sealed and marked with mark. The sample was stored at -18 ° C and protected from light.

6.2 Milk

Take a representative sample of about 500 g, fully mixed, are divided into two, respectively, into a clean container as a sample, sealed, and marked mark. Store the sample below 4 ° C and store it in dark.

During the operation of the sample preparation, the sample should be protected from contamination or changes in the content of the residue.

Note: The above sample sampling site according to GB 2763 Appendix A implementation.

7.1.1 Tea, soybeans, mushrooms, rice samples

Weigh the sample 5 g (accurate to 0.01 g) in a 50 mL polypropylene plastic centrifuge tube, add 10 mL of water, mix and let stand for 30 min.

The centrifuge tube was placed in an ice bath and then 6 g of anhydrous magnesium sulfate and 1.5 g of anhydrous sodium acetate were added. After returning to room temperature, 15.0 mL 1% acetic acid in acetonitrile solution, shake extraction 4 min, 4000 r/min centrifugation 5 min.