



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

GB 23200.43-2016

Replacing SN/T 1017.5-2002

**Food safety national standard -- Determination of residual
chloroquine phosphate in grain and oilseeds -- Gas
chromatographic method**

粮谷及油籽中二氯喹啉酸残留量的测定 气相色谱法

(English Translation)

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Foreword

This standard replaces SN/T 1017.5-2002 "Test method for the presence of quinclorac residues in grain and oilseeds".

Compared with SN/T 1017.5-2002, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;

- the name "export grain and oilseed" in the standard name is replaced by "grain and oilseed";

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

This standard replaced the previous version of the standard release.

-SN/T 1017.5-2002.

National standards for food safety Determination of quercetin acid residues in grain and oilseeds - Gas chromatographic method

1 Scope

This standard specifies the method for the determination of quinclorac residues in grain and oilseeds by gas chromatography.

This standard applies to brown rice, soybeans, corn, wheat, the determination of residual chlorine quinolines, 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 file. 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 sample was extracted with acetone, evaporated to dryness and dissolved in water. The methylene chloride was extracted with methylene chloride and purified by methyl ester Purification, gas chromatography, external standard method quantitative.

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

Acetone (C₃H₆O). Chromatographic pure.

4.1.2

Sodium bicarbonate (NaHCO_3). Chromatographic purity.

4.1.3

Sodium hydroxide (NaOH).

4.1.4

dichloromethane (CH_2Cl_2). chromatographic purity.

4.1.5

Sulfuric acid (H_2SO_4).

4.1.6

anhydrous sodium sulfate (Na_2SO_4). 650 °C burning 4 h, set the dryer in the spare.

4.1.7

petroleum ether.

4.1.8

ether ($\text{C}_4\text{H}_{10}\text{O}$). re-distillation.

4.1.9

Potassium hydroxide (KOH).

4.2 Solution preparation

4.2.1

sulfuric acid solution. 3 mol/L.

4.2.2

Sodium hydroxide solution. 1 mol/L.

4.2.3

Potassium hydroxide solution. 10 mol/L.

4.2.4

Diazomethane. Set 4 mL of ether and 2 mL of potassium hydroxide solution in the reaction tube, add 5 mL of ether dissolved 2 g N-methyl-N-nitroso-4-toluenesulfonic acid amine solution, blowing nitrogen for 5 min, collecting the ether solution in the reaction tube.

4.3 Standards

4.3.1

Quinclorac acid standard. purity $\geq 99\%$.

4.4 Standard solution preparation

4.4.1

Quinclorac acid standard stock solution. accurately weighed the right amount of quinolinic acid standard, with a small amount of acetone dissolved, and petroleum ether Prepared as standard stock solution of 1 000 µg/mL, stored at 0 °C ~ 4 °C.

4.4.2

Quinclorac Acid Standard working solution. According to the work need to use petroleum ether diluted to the appropriate concentration of standard working solution.

4.5 Materials

4.5.1

Magnesium silicate solid phase extraction column. 6 mL/1 g, or equivalent.

5 Instruments and equipment

5.1

Gas chromatograph, with electron capture detector.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Oscillator.

5.4

Rotary Evaporator.

5.5

separatory funnel.

5.6

Solid phase extraction unit with vacuum pump.

6.1 Preparation of the sample

Take a representative sample 500 g, the sampling site according to GB 2763 Appendix A implementation, crushed with a pulverizer. Mix, are divided into two as a test Type, into a clean sample container, sealed and marked.