

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

SN 0591-1996

Replacing SN 0591-1996

**Determination of dioxathion residues in cereals and oil seeds for
export--GC and GC-MS method
(English Translation)**

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Foreword

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

This standard replaces SN 0591-1996 "Method for the determination of dioxathion residues in export grain and oilseeds".

Compared with SN 0591-1996, the main technical changes are as follows.

--- standard name from the "export grain and oilseed in the amount of dioxathion residues test method" revised to "export grain and oil seeds in the dioxane Determination of phosphorus residues by gas chromatography and gas chromatography - mass spectrometry";

- to expand the scope of application of standard testing;

--- cut the sampling part;

Improved sample pretreatment technology;

--- increased gas chromatography - mass spectrometry confirmation method;

- reduce the detection limit, and for different detection objects, given the different matrix to add different concentrations of standard material recovery range.

1 Scope

This standard specifies the preparation of samples for the detection of dioxathion residues in grain and oilseeds, gas chromatography and gas chromatography-mass spectrometry and confirmatory methods.

This standard applies to rice, wheat, barley, corn, sorghum, mung bean, red beans, peanuts, soybeans, rapeseed residues in the determination of dioxathion Test and confirm.

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. For undated references, the latest edition (including all modifications) applies to this document.

GB/T 6682 Analytical laboratory water specifications and experimental methods

3 Principle

The samples were immersed in water and extracted with acetonitrile. The samples were purified by solid phase extraction (SPE) and confirmed by gas chromatography or gas chromatography-mass spectrometry.

External standard method.

4 Reagents and materials

Unless otherwise specified, all reagents are analytical grade and water is a secondary water specified in GB/T 6682.

4.1

acetonitrile. pure chromatography.

4.2

n-hexane. pure chromatography.

4.3

Acetone. Chromatographic pure.

4.4

toluene. pure chromatography.

4.5

Sodium chloride.

4.6

anhydrous sodium sulfate. 650 °C burning 4h, stored in a dryer, cooling and standby.

4.7

acetonitrile - toluene solution (3:1, volume ratio). measure 300mL of acetonitrile and 100mL toluene, mix.

4.8

Acetone-n-Hexane (1 1, volume ratio). Measure 100 mL of acetone and 100 mL of n-hexane.

4.9

Dioxathion standard. Purity. 97.0%. Molecular formula. $C_{12}H_{26}O_6P_2S_4$, CAS. 78-34-2.

4.10

Dioxazosulfuride standard stock solution. accurately weighed dioxin sulfide standard 0.01031g, dissolved with acetone to set capacity to 50mL, dubbed 200mg/L of the standard stock solution, 0 °C ~ 4 °C refrigerator stored in the dark, valid for 6 months.

4.11

Dioxazosine standard intermediate. according to the need to take the appropriate volume of dioxathion standard stock solution, with acetone - n-hexane (4.8) preparation 2.0mg/L standard intermediate liquid, 0 °C ~ 4 °C refrigerator stored in the dark, valid for 1 month.

4.12

Dioxazophos standard working solution. according to the needs of the standard solution of dioxathion standard with acetone - n-hexane solution prepared into a standard working solution, the working fluid is now available.

4.13

C18 solid phase extraction column..2000mg/12mL or equivalent.