



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

GB 23200.44-2016

Replacing SN 0353-1995

**Food safety national standard -- Determination of carbon
disulfide, carbon tetrachloride and dibromoethane residues in
grain**

粮谷中二硫化碳、四氯化碳、二溴乙烷残留量的检测方法

(English Translation)

Issued on: December 18, 2016

Implemented on: June 18, 2017

**Issued by: National Health and Family Planning Commission of the
People 's Republic of China**

Table of Contents

Foreword
1 Scope
2 Normative reference documents
3 Principle
4 Reagents and materials
4.1 Reagents
4.2 Standard solution preparation
5 Instruments and equipment
6 Preparation and storage of samples
6.1 Preparation of the sample
6.2 Sample storage
7 Analysis steps
7.1 Extraction
7.2 Determination
7.3 Blank experiment
8 Results are calculated and expressed
9 Precision
10.1 Quantitation limits
10.2 Recovery rate

Foreword

This standard replaces SN 0355-95 "Method for the determination of carbon disulfide, carbon tetrachloride and dibromoethane residues in export cereals".

This standard compared with SN 0355-95, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "export grain" to "grain";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN 0353-95.

National standards for food safety Determination of carbon disulfide, carbon tetrachloride

and dibromoethane residues in grain

1 Scope

This standard specifies the sample preparation of carbon disulfide, carbon tetrachloride and dibromoethane residues in grain and gas chromatographic method.

This standard applies to the determination of carbon disulfide, carbon tetrachloride and dibromoethane residues in maize, and other foodstuffs can be used for reference.

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 heated and azeotroped with the isooctane and sulfuric acid solution in a distillation extractor. Carbon disulfide, carbon tetrachloride, dibromoethane and isooctane, the water vapor is distilled off and cooled, and the isooctane extract is separated from water in a collection tube of a distillation extractor. The extract is fixed after dehydration.

It was determined by gas chromatograph with electron capture detector and quantified by external standard method.

4.1 Reagents

4.1.1

distilled water. boiled before use for 20min, cooling standby. Take 300 ml of distilled water in a flask of a distillation extractor (see figure below) Add 15 ml of isooctane, according to the extraction step (7.1) operation. Under the specified chromatographic conditions, 1 l of the isooctane extract was chromatographed, should be the same as the octane other than the chromatographic peaks.

4.1.2

Isooctane (C₈H₁₈). analytical grade, in the same chromatographic conditions with the determination of injection 1ul for chromatography, should be no octane Other than the peaks.

Note: Isooctane purification. 1000ml flask by adding 500ml isooctane, and then add metal sodium tablets 5-10g, connected to the grinding of the condenser, reflux 6-8h, and then use the whole glass

distiller was distilled to collect fractions between 97.5 and 99.5 ° C.

4.1.3

anhydrous sodium sulfate (Na_2SO_4). analysis of pure, 650 °C burning 4h, after cooling stored in a closed container.

4.1.4

concentrated sulfuric acid (H_2SO_4). analytical grade.

4.1.5

sulfuric acid aqueous solution. 10% (V/V).

4.1.6 Carbon disulfide (CS_2). Analytical pure (d

1.263)

4.1.7 Carbon tetrachloride (CCl_4). Analytical purity (d

1.594)

4.1.8 Dibromoethane ($\text{C}_2\text{H}_4\text{Br}_2$). Analytical pure (d

2.177)

4.2 Standard solution preparation

4.2.1

Standard stock solution. accurately measure the appropriate volume of carbon disulfide, carbon tetrachloride, dibromoethane, and weigh the weight Calculate, with isooctane, respectively, with the concentration of 1.00mg/ml of the standard stock solution.

4.2.2 Standard working solution

If necessary, standard stock solutions are diluted with isooctane to form a mixed standard working solution at the following concentrations (Table 1).

5 Instruments and equipment

5.1

Gas Chromatograph. Equipped with an electron capture detector.

5.2

Distillation extractor (see figure).

5.3

Microinjector. 10ul or 5ul.

5.4

capacity bottle. 25ml.

5.5

anhydrous sodium sulfate column. cylindrical funnel, 22mm (diameter) built-in 5cm high anhydrous sodium sulfate (4.3).

5.6

electric sets. thermostat type, 500ml,.200w.

5.7

All glass system distillation unit.

5.8

Analytical balance. 0.01 g and 0.0001 g.

6.1 Preparation of the sample

The samples taken, the sampling site according to GB 2763 Appendix A implementation, with a sub-sampling device or quadruple to 1kg, all grinding and through 20 Mesh sieve, mix, are divided into two, immediately into the clean container, sealed, marked mark.

6.2 Sample storage

Store the sample below 0 ° C.

Note: (1) shrink the sample, the operation should be as fast as possible to prevent the loss of fumigant.

During the operation of the sample preparation, it is necessary to prevent the sample from being contaminated or the change in the content of the residue.

7.1 Extraction

A sulfuric acid aqueous solution (10%, V/V) was added to the return port from the receiving tube of the distillation extractor and the gas inlet. even Connect the condenser and gas absorption wells, add 5 mL of isooctane from the mouth of the condenser to the gas absorption trap. Access to cooling water. Quickly take the mix 50.0 g (accurate to 0.1 g) of the sample was placed in a flask to connect the flask and extractor. Add 12 mL of isooctane from the flotation port of the flask 300 m1 sulfuric acid aqueous solution (10%, V/V). Close the dosing port and seal the dosing with a small amount of sulfuric acid solution (10%, V/V). Will be used to heat sets the flask was heated and the isooctane extract was slowly distilled