

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

SN 0683-1997

Replacing SN 0683-1997

**Determination of tricyclazole in cereals for export. HPLC-MS/MS
method**

(English Translation)

Issued on: January 1, 1970

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Foreword

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

This standard replaces SN 0683-1997 "cereals for export tricyclazole residues in."

Compared with SN 0683-1997, this standard technological change as follows.

--- Pretreatment method from the original artificial filler column purification to the solid phase extraction (SPE) method;

--- Instrument method from the original GC - NPD and gas chromatography - mass spectrometry method validation, to high performance liquid chromatography - mass Spectrum/mass spectrometry;

--- Detection matrix range from the original expansion of rice corn, barley, brown rice, rice, wheat.

Please note that some of the content of this document may involve patents. Release mechanism of the present document does not assume responsibility for the identification of these patents.

This standard is proposed and managed by the National Certification and Accreditation Administration Committee.

This standard was drafted. People's Republic of China Shanghai Entry-Exit Inspection and Quarantine Bureau, People's Republic of China Entry-Exit Inspection and Tibet Quarantine Bureau.

1 Scope

This standard specifies the method for determination of cereals for export tricyclazole residues.

This standard applies to corn, barley, brown rice, rice, wheat tricyclazole residue in the determination and confirmation.

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version suitable for use herein Member. For undated references, the latest edition (including any amendments) applies to this document.

Laboratory use specifications and test methods GB/T 6682 Analysis SN/T 0800.1 and export grain, feed inspection sampling and sample preparation methods Principle 3 Sample remaining tricyclazole shaking extraction with acetone, over neutral alumina solid phase extraction (SPE) for liquid chromatography - mass spectrometry/mass spectrometry Determination, external standard.

4 Reagents and materials

Unless otherwise specified, reagents were of analytical grade water as a water GB/T 6682 regulations.

4.1

acetone. chromatography.

4.2

dichloromethane. chromatography.

4.3

Methanol. chromatographically pure.

4.4

acetonitrile. chromatography.

4.5

formic acid. Chromatographic pure.

Ammonium acetate 4.6. HPLC grade.

4.7 5mmol/L ammonium acetate aqueous solution (containing 0.1% formic acid). Weigh 0.385g of ammonium acetate, dissolved in 1L of water was added, was added 1mL Formic acid mix.

4.8

dichloromethane - methanol solution (991, volume ratio).

4.9

acetonitrile - water (11, volume ratio).

4.10

Acetonitrile -5mmol/L ammonium acetate aqueous solution (containing 0.1% formic acid) (11, volume ratio).

4.11

Standard. tricyclazole (Tricyclazole, CASN o.41814-78-2), purity $\geq 99\%$.

4.12

standard stock solution. weigh the amount tricyclazole standards, with acetonitrile formulated into standard stock solution 10 μ g/mL of, 0 °C ~ 4 °C insurance Deposit, valid for 12 months. Acetonitrile -5mmol/L ammonium acetate solution (containing 0.1% formic acid) (11 volume) required in the standard configuration Interstitial fluid and the working standard solution.

4.13

neutral alumina SPE. 1g/3mL.

4.14

and 0.22 μ m syringe needle organic phase filters.