

ICS 67.060
CCS X 11

SN

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

SN 0596-1996

Replacing SN 0596-1996

**Determination of sethoxydim residue in cereals and oilbean for
exports. GC/MS
(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 0596-1996 "cereals for export sethoxydim residues in." this standard compared with SN 0596-1996, in addition to editorial changes, the major technical changes are as follows.

- Modify the standard test method using gas chromatography - mass spectrometry method instead of the gas chromatography method;
- Modify the scope of the standard, the standard scope expanded from corn to corn, rice, soybeans, peanuts, and buckwheat;
- Modify the extraction and purification process, was extracted with acetonitrile, eighth and activated carbon, amino solid phase extraction (SPE);
- Measured by the lower limit of 0.04mg/kg was changed to 0.01mg/kg;
- Omitted sampling procedure.

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

This standard was drafted. People's Republic of China Liaoning Province Exit Inspection and Quarantine.

1 Scope

This standard specifies the cereals for export sethoxydim residual gas chromatography - mass spectrometry and confirmation methods.

This standard is applicable to the export of corn, rice, soybeans, peanuts and buckwheat

SETHOXYDIM measured residues.

2 Method summary

Sample sethoxydim residue was extracted with acetonitrile extract through C18 and Envi-Carb/NH₂ SPE column (SPE) purification, gas Chromatography - mass spectrometry (GC-MS) detection, external standard.

3 Reagents and materials

Unless otherwise specified, the reagents were HPLC grade water to comply with GB/T 6682 secondary water.

3.1

n-hexane.

3.2

acetonitrile.

3.3 Toluene

3.4 acetonitrile - toluene solution (31 volume). acetonitrile and taken 3.1 mixing ratio by volume of toluene.

3.5 anhydrous sodium. AR; 650 °C burning 4h, stored in a sealed container in the back.

3.6 sethoxydim standards (Sethoxydim, C₁₇H₂₉NO₃S, CAS number. 74051-80-2). purity of greater than or equal to 98%.

3.7 sethoxydim standard solution. Accurately weigh an adequate amount of sethoxydim standard, dubbed the concentration of toluene as a standard stock solution 100µg/mL of.

The required standard stock solution was diluted to the appropriate concentration of standard working solution with n-hexane. Stock standard solution and standard working solution in the dark Stored at 0 °C ~ 4 °C condition.

4 Instruments and equipment

4.1

Gas chromatography - mass spectrometry. source with electron impact (EI).

4.2

rotary evaporator.

4.3

Centrifuge. 5000r/min.

4.4

high-speed homogenizer.

4.5

C18 solid phase extraction column. 12mL/2000mg, or equivalent person; prior to use pre-rinsed with 10mL acetonitrile.

4.6

Carb/NH₂ SPE 6mL/500mg/500mg, or equivalent person; prior to use about 2cm high anhydrous sodium sulfate, used 10mL of acetonitrile pre-rinse.

4.7

tube. 100mL, glass.

5 Sample Preparation and Storage

Take a representative sample of about 500g, and crushed it all through a pore size of 2.0mm sieve samples. Mix evenly into clean