

ICS 65.100.20

CCS G25



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

GB 29382-2012

Mesotrione technical material

硝磺草酮原药

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Foreword

This standard in Chapter 3, Section 5 is mandatory, the rest are recommended. This standard was drafted in accordance with rules GB/T 1.1-2009 given. Please note that some of the content of this document may involve patents, the issuing authority of this document does not assume responsibility for identifying these patents. The standard proposed by China Petroleum and Chemical Industry Federation. This standard by the National Pesticide Standardization Technical Committee (SAC/TC133). This standard is drafted. Shenyang Chemical Research Institute Co., Ltd. Participated in the drafting of this standard. Shenyang Branch Chemical Co., Ltd. Limin Chemical Co., Ltd., Jiangsu Changqing Agrochemical Co. shares The company, Shijiazhuang City, three rural Chemical Co., Ltd., Jiangsu Ampang Electrochemical Co., Ltd.. The main drafters. Hou Chunqing, Zhang Xuebing, Li Dong, CHENG Chun, Zhang Ruifang, Ji Yuping, Xie Yueqing, Jiang Yutian, Tao Chengbin. Mesotrione TC

1 Scope

GB 29382-2012 is the Chinese national standard on mesotrione technical material, in the field of agriculture. It carries no /T suffix, which in the Chinese system means compliance is mandatory: a product or a practice within its scope has to meet it to be lawfully made, sold or carried out in China. It was issued on 31 December 2012 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 July 2013.

Classification: ICS 65.100.20, CCS G25. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This standard specifies the mesotrione original drug of requirements, test methods and marking, labeling, packaging, storage, and acceptance period. This standard applies to impurities derived from mesotrione composition and production of the original drug mesotrione. NOTE. mesotrione and other names, structural formulas and physicochemical parameters related impurities nitro substantially xanthone see Appendix A.

2 Normative references

The following documents for the application of this document is essential. For dated references, only applies to the version dated paper Pieces. For undated references, the latest edition (including any amendments) applies to this document. Determination 1601 pH value pesticides GB/T

GB/T 1604 pesticide regulations for acceptance of goods

GB/T 1605-2001 sampling commercial pesticides

GB 3796 pesticide packaging General

GB/T 6682-2008 analytical laboratory use specifications and test methods (ISO 3696. 1987, MOD)

GB/T 8170-2008 rounding off means about rules and determining limiting values

GB/T 19138 Determination of acetone insoluble pesticide

3.1 Appearance As a light brown to yellow-brown solid powder, or wet brown solid, without visible foreign matter and added modifier.

3.2 Technical Specifications Mesotrione TC shall comply with the requirements in Table 1.
Table 1 TC mesotrione control program Index Item Index Mesotrione mass fraction (dry basis) /% $\geq$

95.0 Loss on drying /% $\leq$

25 Xanthone, thioxanthone nitro mass fraction a (dry basis)/(mg/kg) $\leq$

4.2 Sampling Carried out in

GB/T 1605-2001 of "original drug product sampling" approach. Determining Sample packages with a random number table method; final sample size Not less than 100g.

4.3 Identification Test Infrared spectroscopy

--- mesotrione sample and standard $\sim 400\text{cm}^{-1}$ in the infrared range of 1 4000 cm^{-1} -absorption spectrum should not expressly Significant difference. Mesotrione standard infrared spectra of Figure 1. FIG 1 mesotrione standard infrared spectrum of FIG. The present liquid chromatography

--- identification test can be conducted simultaneously with the determination of one mass fraction sulcotrione nitrate. Chromatography under the same operating conditions, Main peaks of the sample solution and the retention time of the solution of the mesotrione sample peak retention time scale, the relative difference of 1.5% should be Or less. Determination of mass sulcotrione nitro ketone score 4.4

4.4.1 Method summary After drying in vacuo a sample was dissolved in mobile phase, acetonitrile and water (phosphate adjusted to pH 3) as the mobile phase, used as a filler C 18 Stainless steel column and a UV detector at a wavelength of 270nm, the sample of mesotrione reverse phase high performance liquid chromatography and determination, External standard.

4.4.2 Reagents and solutions Acetonitrile. HPLC grade; Water. The new secondary steam distilled water; Phosphoric acid; Mesotrione Sample. a known mass fraction, $w \geq 99.0\%$.

4.4.3 Instruments HPLC. a variable wavelength UV detector; Chromatographic data processor or chromatography workstation; Column. 250mm x 4.6mm (id) stainless steel column, C18, 5 μm filler contents (or column with equivalent effect); Filter. membrane pore size of about

0.45 μm ; Microsyringe. 50 μL ; Quantitative sampling tube. 5 μL ; Ultrasonic cleaning.

4.4.4 HPLC operating conditions Mobile phase. [Psi] [acetonitrile. water (adjusted to pH 3 phosphate to)] = 40:60, by membrane filtration, and degassing; Flow rate. 1.0mL/min; Column temperature. room temperature (temperature change should be less than 2 deg.] C); Detection wavelength. 270nm; Injection Volume. 5 μL ; Retention time. mesotrione about 10.1min. Are typical operating parameters of the above, according to the characteristics of different instruments, for a given operating parameters appropriately adjusted to obtain the best result. Typical mesotrione HPLC TC shown in Figure 2. 1

--- mesotrione. Figure 2 HPLC mesotrione original drug

4.4.5.1 Preparation of standard solution

0.1 g of weighed (accurate to 0.0002g) mesotrione standard in 50mL volumetric flask, dilute to the mark with acetonitrile, ultrasonic oscillation 5min to dissolve the sample, cooled to room temperature, shaking. Pipette with a pipette 5mL above solution in 50mL volumetric flask, dilute with the mobile phase Release to the mark.

4.4.5.2 Preparation of sample solution Weigh mesotrione containing

0.1 g of (accurate to 0.0002g) sample had been dried (drying method of section 4.5.2) in a 50mL volumetric flask, Washed with acetonitrile to volume, to dissolve the sample ultrasonic vibration 5min, cooled to room temperature, shaking. Pipette with a pipette to the solution 5mL, 50mL volumetric flask, diluted in phase with the flow to the mark.

4.4.5.3 Determination Under the above operating conditions, after the instrument is stable, the number of consecutive doses of the standard solution is injected until the two adjacent needles mesotrione peak area becomes relatively After of less than 1.5%, measured according to the standard solution, sample solution, the sample solution, the order of standard solution.

4.4.5.4 computing The two needle and the sample solution was measured before and after the sample solution two needle mesotrione standard peak area are averaged. Mesotrione sample Oxadiazon mass fraction according to formula (1). $w_1 = A_2 \cdot M_1 \cdot w_{A1} \cdot m_2$ (1)
Where. W_1

- mesotrione sample mass fraction in%; A_2
- sample solution, nitro sulcotrione one average peak area; M_1
- mesotrione standard mass in grams (G); W
- the mass fraction mesotrione standard sample, expressed in%; A_1
- the standard solution, mesotrione average peak area;
- M_2 sample mass, in grams (g).

4.4.6 allows poor Ketone mass difference mesotrione Results replicates grass two fractions should be not more than 1.5%, and the arithmetic mean value as a measurement result.

4.5 Determination of loss on drying

4.5.1 Instruments Vacuum pump; Vacuum oven. $60\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, exemplar vacuum pressure. $-90\text{kPa} \pm 5\text{kPa}$.

4.5.2 Determination Step Weigh 10g weighing bottle with the sample constant weight (accurate to 0.01g), paving, placed in a vacuum oven. Vacuum pump was turned on $60\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, exemplar vacuum dried to constant weight (about 1h) $-90\text{kPa} \pm 5\text{kPa}$ depressed condition in a desiccator, cooled to room temperature, Weighed.

4.5.3 computing Loss on drying the sample by the formula (2). $w_2 = m_0 - m_1 \times 100$ (2)
Where.

- w_2 of the sample mass fraction of drying loss, expressed in%; Mass M_0
- weighing bottle and the sample in grams (G); Mass M_1