ICS 65.100.10
CCS G25

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 29384-2012

Acephate technical material

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Issued on: December 31, 2012 **Implemented on: July 1, 2013**

**Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;
Standardization Administration of China**

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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. Sanonda Co., Ltd. Hubei, Zhejiang Jiahua Group Co., Ltd., Jiangsu blue abundance of chemicals Co., Ltd., Hangzhou Agrochemical Co., Ltd. Qingfeng. The main drafters of this standard. High Scenic, Li Dong, Liao Yan, Li Da Yuan, Zhang Shurong, India Xie Gang, He Hongdong. Acephate original drug

1 Scope

GB 29384-2012 is the Chinese national standard on acephate 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 65.100.10, 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 requirements of the original drug acephate, Test Method for marking, labeling, packaging, storage, and acceptance period. This standard applies to impurities derived from the production of the composition and acephate acephate original drug.

Note. Acephate acetamide and impurities, other names methamidophos, O, O, S- trimethyl phosphorothioate, structural formulas and physicochemical parameters of the basic see Appendix Record 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. Preparation

GB/T 601 standard titration solution Chemicals

GB/T 1600 Pesticide Method Moisture

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

3.1 Appearance White crystalline powder, no visible foreign matter.

3.2 Technical Specifications Acephate original drug should also meet the requirements of Table 1. Table 1 acephate original drug control project indicators Item Index Acephate mass fraction /% >=

97.0 Acetamide mass fraction /% <=

0.3 Methamidophos mass fraction /% <=

0.3 O, O, S- trimethyl phosphorothioate mass fraction a /% <=

0.1 Moisture content /% <=

4.2 Sampling Carried out in

GB/T 1605-2001 of "original drug product sampling" approach. Determined by sampling a random number table method packages, the amount of the final sample Not less than 100g.

4.3 Identification Test The present liquid chromatography

--- identification test can be carried out simultaneously with the determination of the mass fraction of acephate. In the same chromatographic operating conditions , The peak of a retention time in the sample solution acephate chromatographic peak retention time of standard solution, which should be in the relative difference 1.5% or less. Infrared Spectroscopy Infrared

--- sample and standard sample in the range of 4000cm-1 ~ 400cm-1 absorption spectrum should be no difference, see figure 1. 1 acephate standard infrared spectrum of FIG.

4.4 Determination of acephate, acetamide and mass fraction methamidophos

4.4.1 Method summary Sample dissolved in mobile phase, acetonitrile-water (pH = 3) as the mobile phase, used as a filler C18 stainless steel column and UV detector (210nm), the sample acephate, methamidophos acetamide and reverse phase high performance liquid chromatography, external standard.

4.4.2 Reagents and solutions Acetonitrile. HPLC grade; Water. The new secondary steam distilled water; Phosphoric acid; Acephate standards. a known mass fraction, w >=99.0%; Acetamide standard. a known mass fraction, w >=98.0%; Methamidophos standards. a known mass fraction, w >=98.0%; Sample preparation and methamidophos acetamide standard solution. Weigh acetamide standard 0.06g,

0.1 g of Sample methamidophos (accurate to 0.0002g) was placed in a 50mL volumetric flask, add the mobile phase to dissolve and dilute to the mark.

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

0.45 m; Microsyringe. 250μL; Ultrasonic cleaning.

4.4.4 HPLC operating conditions Mobile phase. Psi. after (acetonitrile water) = 10.90, with phosphoric acid water adjusted to pH 3, mixed well, filtered through a filter membrane, and Degassing; Flow rate. 1.0mL/min; Column temperature. room temperature (temperature change should be less than 2 deg.] C); Detection wavelength. 210nm; Injection volume. 20μL; Retention time. acetamide 3.6min, methamidophos 5.0min, acephate 5.7min. 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 HPLC acephate TC shown in Figure 2. 1

--- acetamide; 2

--- methamidophos; 3

--- acephate. Figure 2 HPLC original drug acephate

4.4.5.1 Preparation of standard solution Sample Weigh

0.06 g of acephate (accurate to 0.0002g) was placed in a 50mL volumetric flask, with a pipette pipetted 2.0mL acetyl Methamidophos and amine standards in the standard solution in the same flask, dissolved in mobile phase and dilute to the mark.

4.4.5.2 Preparation of sample solution Weigh

0.06 g of acephate-containing (accurate to 0.0002g) sample, placed in a 50mL volumetric flask, and dilute with mobile phase to dissolve 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 acephate relative peak area After the change is less than 1.2%, measured according to the standard solution, sample solution, the sample solution, the order of standard solution.

4.4.5.4 computing The two doses of the standard sample solution before and after the two-pin and a sample solution is measured acephate (acetamide, methamidophos) peak area, respectively, Averaged. Acephate sample (acetamide, methamidophos) mass fraction, according to equation (1). w1 = A2 · m1 · w A1 · m2 x f (1) Where. W1

--- sample acephate (acetamide, methamidophos) the mass fraction in%; A2

--- sample solution, an average value acephate (acetamide, methamidophos) peak area; M1

--- Sample mass in grams (G); W

--- Sample acephate (acetamide, methamidophos) the mass fraction in%; f

--- dilution factor, acephate f = 1, and methamidophos acetamide f = 0.04; A1

--- the standard solution, the average value of acephate (acetamide, methamidophos) peak area;

--- M2 sample mass, in grams (g).

4.4.5.5 allow poor Acephate differential mass fraction of the two parallel measurement result should be less than 1.2%, and methamidophos acetamide relative difference should