

ICS 65.100.30
CCS G 25

NY

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

NY/T 3598-2020

**Fosetyl-aluminium wettable powder
(English Translation)**

Issued on: March 20, 2020

Implemented on: July 1, 2020

**Issued by: Ministry of Agriculture and Rural Affairs of the People's
Republic of China**

Table of Contents

Foreword...	3
1 Scope...	4
2 Normative references...	4
3 Requirements...	5
4 Test methods...	5
5 Acceptance and quality assurance periods...	12
6 Marks, labels, packaging, storage and transportation...	13

1 Scope

This Standard specifies the requirements, test methods, acceptance and quality assurance periods, as well as marks, labels, packaging, storage and transportation for fosetyl-aluminium wettable powder.

This Standard applies to fosetyl-aluminium wettable powder processed from fosetyl-aluminium original drug, additives and fillers.

NOTE: Please refer to Annex A for other names, structural formulas and basic physical and chemical parameters of fosetyl-aluminium.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 601, Chemical reagent -- Preparations of reference titration solutions

GB/T 603, Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 1600, Testing method of water for pesticides

GB/T 1601, Determination method of pH value for pesticides

GB/T 1604, Commodity pesticide regulations for acceptance

GB/T 1605-2001, Sampling method for commodity pesticides

GB 3796, General rule for packing of pesticides

GB/T 5451, Testing method for the wettability of dispersible powders of pesticides

GB/T 6682, Water for analytical laboratory use -- Specification and test methods

GB/T 8170-2008, Rules of rounding off for numerical values and expression and judgement of limiting values

GB/T 14825-2006, Determination method of suspensibility for pesticides method to determine the packages to be sampled. The final sample size shall be no less than 300 g.

4.3.1 Ion chromatography

This identification test can be carried out simultaneously with the determination of the mass fraction of fosetyl-aluminium. Under the same chromatographic operating conditions, the relative difference between the retention time of the main chromatographic peak in the specimen solution and the retention time of the ethyphosphonate chromatographic peak in the standard solution shall be within 1.5%.

4.3.2 Aluminum ion identification

Please refer to Annex B for qualitative identification of aluminum ions.

4.4.1 Method summary

The specimen is dissolved in mobile phase. Sodium carbonate and sodium bicarbonate solutions are used as mobile phases. Use anion chromatography column and conductivity detector to perform ion chromatography separation of ethyphosphonate in the specimen. Use the external standard method for quantification (chemical titration method can also be used to determine the mass fraction of aluminum triacetate. See

Annex C for details).

4.4.2 Reagents and solutions

Water: ultrapure.

Sodium carbonate: guaranteed pure.

Sodium bicarbonate: guaranteed pure.

Standard sample of fosetyl-aluminium: known mass fraction of fosetyl-aluminium, $\omega \geq 97.0\%$.

4.4.3 Instruments

Ion chromatograph: equipped with conductivity detector and suppressor.

Integrator or electronic data acquisition system.

Chromatographic column: 250 mm x 4 mm (inner diameter) anion separation column (filled

with polyvinylbenzene/ethylvinylbenzene/polyvinyl alcohol matrix, with alkyl quaternary ammonium or alkanol quaternary ammonium functional groups) and anion protection column; particle size is 6 μm (or an equivalent chromatographic column).

Weigh 0.05 g (accurate to 0.0001 g) of fosetyl-aluminium standard sample into a 100 mL volumetric flask. Add 20 mL of water. Place in an ultrasonic cleaner and sonicate for 30 min. Cool to room temperature. Use mobile phase to dilute to volume. Shake well. Use a pipette to transfer 1 mL of the above solution into another 100 mL volumetric flask. Dilute to the graduation with mobile phase. Shake well. Filter.

4.4.5.2 Preparation of specimen solution

Weigh a specimen containing 0.05 g (accurate to 0.0001 g) of fosetyl-aluminium into a 100 mL volumetric flask. Add 20 mL of water. Place in an ultrasonic cleaner and sonicate for 30 min. Cool to room temperature. Dilute to the graduation with mobile phase. Shake well. Use a pipette to transfer 1 mL of the above solution into another 100 mL volumetric flask. Dilute to the graduation with mobile phase. Shake well. Filter.

4.4.5.3 Determination

Under the above operating conditions, after the instrument is stable, continuously inject several needles of the standard solution until the relative change in the peak area of two adjacent ethyphosphonate peaks is less than 1.5%. Measure in the order of standard sample solution, specimen solution, specimen solution, and standard sample solution.

4.4.5.4 Calculation

Average the ethosphonate peak areas in the measured two injections of sample solution and the two injections of standard solution before and after the specimen. The mass fraction of fosetyl-aluminium in the specimen is calculated according to formula (1).

Where,

ω_1 - the mass fraction of fosetyl-aluminium in the specimen, in percent (%);

A_2 - the average value of the ethyphosphonate peak area in the specimen solution;

m_1 - the mass of the standard sample, in grams (g);

ω - the mass fraction of fosetyl-aluminium in the fosetyl-aluminium standard sample, in percent (%);

A_1 - the average value of the ethyphosphonate peak area in the standard sample solution;

m_2 - the mass of the specimen, in grams (g).

4.4.6 Allowable difference

The difference between the two parallel determination results of the mass fraction of fosetyl-aluminium shall be no more than 0.6% for 40% and no more than 1.0% for 80%.

Take the arithmetic mean as the measurement result.

4.5.1 Method summary

The specimen is dissolved in mobile phase. Sodium carbonate and sodium bicarbonate solutions are used as mobile phases. Using an anion chromatography column and a conductivity detector, the phosphite in the specimen is separated by ion chromatography.

Conduct quantification by external standard method.

4.5.2 Reagents and solutions

Water: ultrapure.

Sodium carbonate: guaranteed pure.

Sodium bicarbonate: guaranteed pure.

Sodium phosphite pentahydrate standard: known mass fraction $\omega \geq 98.0\%$.

4.5.3 Instruments

Ion chromatograph: equipped with conductivity detector and suppressor.

Integrator or electronic data acquisition system.

Chromatographic column: 250 mm x 4 mm (inner diameter) anion separation column (filled with polyvinylbenzene/ethylvinylbenzene/polyvinyl alcohol matrix with alkyl quaternary ammonium or alkanol quaternary ammonium functional groups) and anion protection column; particle size is 6 μm (or an equivalent chromatographic column).

Filter: the pore size of the filter membrane is approximately 0.45 μm .

Microsampler: 50 μL .

Quantitative injection tube: 5 μL .

Ultrasonic cleaner.

4.5.4 Ion chromatography operating conditions

Mobile phase: carbonate eluent [$c(\text{Na}_2\text{CO}_3) = 4.5 \text{ mmol/L} + c(\text{NaHCO}_3) = 0.8 \text{ mmol/L}$].

Weigh 477 mg of sodium carbonate and 67.2 mg of sodium bicarbonate and dissolve them in 1000 mL of water. Mix well.