

ICS 65.120

CCS B46



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

GB 21034-2017

Replacing GB/T 21034-2007

Feed additive-Calcium salt of hydroxyl analogue of methionine

饲料添加剂 蛋氨酸羟基类似物钙盐

Issued on: October 14, 2017

Implemented on: May 1, 2018

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

Table of Contents

1 Scope

1.0 Heavy metals (Pb)/(mg/kg) $\leq$

2 Normative references

3 Requirements

4.2 Identification test

4.3.1 Reagents and materials

4.3.2 Equipment

4.3.3 Analysis method

4.6.1 Reagents and solutions

4.6.1.6 Ammonia solution (10%) Prepared according to GB/T 603

4.6.2 Analysis steps

15.0 Loss on drying /% $\leq$

20 Total arsenic (As)/(mg/kg) ≤ 2

Foreword

The first chapter of this standard, Chapter 3 and Chapter 5 is mandatory, the rest are recommended. This standard was drafted in accordance with the rules given in GB/T 1.1-2009. This standard replaces GB/T 21034-2007 "feed additive hydroxy methionine calcium." This standard compared with GB/T 21034-2007, the main technical differences are as follows:

- The standard name changed to "feed additive methionine hydroxy analog calcium salt";
- The appearance and traits changed to "This product is white, light gray or taupe powder or granules, a unique stench";
- Increased product methionine hydroxy analog identification method (high performance liquid chromatography);
- Remove the technical indicators in the requirements of granularity;
- Test rules to add a "type test." This standard is proposed and managed by the National Feed Industry Standardization Technical Committee (SAC/TC76). This standard drafting unit. China Agricultural University, China Feed Industry Association, Novartis (China) Investment Co., Ltd.. The main drafters of this standard. Shi Shi Yan, Sun Xiangli, had Xiangfang, Xie Chunyuan, Hou Chengli, Wu Yubo. This standard replaces the standards

previously issued as.

--- GB/T 21034-2007. Feed additive methionine hydroxy analog calcium salt

1 Scope

GB 21034-2017 is the Chinese national standard on feed additive-calcium salt of hydroxyl analogue of methionine, 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 14 October 2017 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 May 2018. Classification: ICS 65.120, CCS B46. 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 feed additive methionine hydroxy analog calcium salt requirements, test methods, inspection rules and labeling, packaging, transportation Loss, storage and shelf life. This standard applies to methionine hydroxy analogues and calcium hydroxide as the main raw material feed additive methionine hydroxy analogues Calcium salt. Chemical name .2-hydroxy-4-methylthiobutyric acid calcium salt; 2-hydroxy-4-methylthiobutyric acid calcium; hydroxy methionine calcium. Molecular formula $(\text{CH}_3\text{SCH}_2\text{CH}_2\text{CHOHCOO})_2\text{Ca}$. Relative molecular mass .338.44 (By International Relative Atomic Weight in.2007).

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version applies to this article Pieces. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 601 chemical reagent standard titration solution preparation

GB/T 603 chemical reagent test methods used in the preparation of preparations and products

GB/T 6435 Determination of moisture in feed

GB/T 6436 Determination of calcium in feed

GB/T 6682 analytical laboratory water specifications and test methods

GB/T 9725 chemical reagent potentiometric titration General GB 10648 feed label

GB/T 13079 Determination of total arsenic in feed

GB/T 14699.1 feed sampling

GB/T 19371.2 feed methionine hydroxy analogues Determination of high performance liquid chromatography

3 Requirements

3.1 Appearance and traits This product is white, light gray or taupe powder or granules, a unique stench.

3.2 Specifications Technical indicators should be consistent with the provisions of Table 1. Table 1 technical indicators Project Indicators Methionine hydroxy analog calcium salt content (dry basis) /% $\geq$

95.0 Methionine hydroxy analog content (dry basis) /% $\geq$

84.0 Table 1 (continued) Project Indicators Calcium /% 11.0 ~

4.2 Identification test

4.2.1 Reagents and solutions Anhydrous copper sulfate saturated sulfuric acid solution. Take anhydrous copper sulfate by adding concentrated sulfuric acid until precipitation appears.

4.2.2 Amino acid identification method Weigh the sample 25mg in a dry test tube, add anhydrous copper sulfate saturated sulfuric acid solution (4.2.1) 1mL, the solution was immediately yellow, and then turn into yellow-green.

4.2.3 methionine hydroxy analog qualitative identification

4.2.3.1 Reagents and solutions 4.2.3.1.1 Standard. Methionine hydroxy analog calcium salt standard (purity 99%). 4.2.3.1.2 methionine hydroxy analog calcium salt standard solution (100mg/L). Weigh methionine hydroxy analog calcium salt standard (4.2.3.1.1) 0.1g (accurate to 0.0001g), add water after ultrasonic solubilization volume to 100mL, take the solution 10mL, water volume to 100mL.

4.2.3.2 Identification method Weigh the sample 0.1g (accurate to 0.0001g), add water ultrasonic assisted solution volume to 100mL, take the solution 10mL, water to volume 100mL. According to GB/T 19371.2 determination, the sample solution of methionine hydroxy analog calcium salt solution peak retention time and methionine hydroxyl Analogues calcium salt standard solution (4.2.3.1.2) the same, and the hydroxy analog content $\geq$ 80%.

4.3 methionine hydroxy analog calcium salt and methionine hydroxy analog content determination

4.3.1 Reagents and materials

4.3.1.1 Acid solution. Glacial acetic acid. water. concentrated hydrochloric acid = 50 10 3.

4.3.1.2 potassium bromate - potassium bromide standard titration solution, $c(6\text{KBrO}_3)$ is about

0.6 mol/L. Weigh 17.5g potassium bromate (accurate to 0.0001g) and 112.5g potassium bromide (accurate to 0.0001g), dissolved in water after the volume to 1L. According to GB/T 601 provisions of the method of calibration, this solution stored in a brown bottle, valid for one month.

4.3.2 Equipment

4.3.2.1 automatic potentiometer or pH meter.

4.3.2.2 platinum saturated calomel electrode composite electrode.

4.3.3 Analysis method

4.3.3.1 Principle In acidic media, methionine hydroxy analogues undergo the following redox reactions. $3\text{RSR}'\text{BrO}_3 \rightarrow 3\text{RSOR}'\text{Br}$ In the test solution into a platinum saturated calomel electrode composed of composite electrode (or Pt saturated calomel composite electrode), with the titration The addition of liquid, the concentration of ions to be tested constantly changing, the electrode potential also occurred in the corresponding changes occur at the equivalence point potential over, in order to determine the titration end.

4.3.3.2 Procedure Weigh 0.5g (accurate to 0.0001g) crushed, dried ($105\text{ }^\circ\text{C} \pm 2\text{ }^\circ\text{C}$) to a constant weight of the sample in a 150mL beaker, add 50mL acid solution (4.3.1.1), ultrasonic dissolved beaker set magnetic stirrer, the electrode into the solution, adjust the stirring speed to the solution Fully vortex, potassium bromate - potassium bromide standard titration solution (4.3.1.2) titration, according to GB/T 9725, confirm the titration volume.

4.3.4 Calculation Results Methionine hydroxy analog calcium salt

$[(\text{CH}_3\text{SCH}_2\text{CH}_2\text{CHOHCOO})_2\text{Ca}]$ content X_1 mass fraction, the value is expressed in% According to equation (1) calculation. $X_1 = c_1 \times V_1 \times 0.0846 \text{ m}_1 \times 100$ (1) In the formula. c_1

--- potassium bromate - potassium bromide standard titration solution concentration in units of moles per liter (mol/L); V_1

--- titration sample consumed potassium bromate - potassium bromide standard titration solution volume, in milliliters (mL); 0.0846

--- and 1.0mL potassium bromate - potassium bromide standard titration solution [$c(6\text{KBrO}_3) = 1.0\text{mol/L}$] equivalent, in grams The methionine hydroxy analog calcium salt mass; m_1