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GB/T 19371-2025

Replacing GB/T 19371.2-2007

Determination of methionine hydroxy analogue in feeds - High-performance liquid chromatography

饲料中蛋氨酸羟基类似物的测定 高效液相色谱法

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents". Drafting.

This document supersedes GB/T 19371.2-2007 "Determination of Methionine Hydroxyl Analogs in Feed - High Performance Liquid Chromatography".

Compared with GB/T 19371.2-2007, apart from structural adjustments and editorial changes, the main technical changes are as follows.

a) The scope of application has been expanded to include concentrate supplements and mixed feed additives (see Chapter 1);

b) The mobile phase was changed to formic acid acetonitrile solution (see 5.4, 4.5 in the 2007 edition);

c) The requirements and related regulations for standard products have been revised (see 5.8, 4.6 of the 2007 edition);

d) Requirements for standard series solutions have been added (see 5.9);

e) Sampling was removed, and sample preparation was changed (see Chapter 7, Chapter 6 in the 2007 edition);

f) The formula for calculating calcium hydroxymethionine has been removed (see Chapter 8 in the 2007 edition);

g) The precision requirements have been changed (see Chapter 10, Chapter 9 of the 2007 edition).

Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents.

This document was proposed and is under the jurisdiction of the National Technical Committee on Standardization of Feed Industry (SAC/TC76).

This document was drafted by: Institute of Agricultural Quality Standards and Testing Technology, Chinese Academy of Agricultural Sciences; Tongwei Agricultural Development Co., Ltd.; and Shaanxi Province.

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The release history of this document and the document it replaces is as follows.

- First published in 2004 as GB/T 19371.2-2003, and revised for the first time in 2007;

- This is the second revision.

Determination of methionine hydroxy analogues in feed High performance liquid chromatography

1 Scope

This document describes a high-performance liquid chromatography method for the determination of methionine hydroxy analogues in feed.

This document applies to methionine in compound feed, concentrated feed, concentrate supplements, additive premixes, and mixed feed additives.

Determination of hydroxy analogs and/or calcium salts of methionine hydroxy analogs

(calculated as methionine hydroxy analogs).

The quantitative limits for compound feed, concentrated feed, and concentrate supplements in this document are 0.03%. The quantitative limits for additive premixed feed and mixed feed additives are also specified.

The limit of quantification for the additive is 0.1%.

2 Normative references

GB/T 6682

GB/T 20195

3 Terms and Definitions

This document does not contain any terms or definitions that need to be defined.

4 Principles

Methionine hydroxy analogues in the sample were extracted with acetonitrile solution, hydrolyzed with potassium hydroxide solution, determined by high performance liquid chromatography, and quantified by external standard method.

5 Reagents or materials

Unless otherwise specified, use only analytical grade reagents.

5.1 Water. GB/T 6682, Grade I water.

5.2 Acetonitrile. chromatographic grade.

5.3 Formic acid. chromatographic grade.

5.4 Formic acid and acetonitrile solution. Take 0.5 mL of formic acid and 50 mL of acetonitrile, add 950 mL of water, mix well, and degas by sonication.

5.5 10% acetonitrile solution. Measure 100 mL of acetonitrile and 900 mL of water, and mix well.

5.6 50% phosphoric acid solution. Measure 100 mL of phosphoric acid and 100 mL of water, and mix well.

5.7 50% potassium hydroxide solution. Weigh 50g of potassium hydroxide, add 100mL of water to dissolve, and mix well.

5.8 1 mg/mL Standard Stock Solution (calculated as methionine hydroxy analog). Weigh an appropriate amount (accurate to 0 0001 g) of methionine hydroxy analog.

Calcium sulfate (CAS No.. 4857-44-7, purity greater than 98%) standard was dissolved in a 100 mL volumetric flask with 10% acetonitrile solution (5.5).

Mix thoroughly; or weigh an appropriate amount (accurate to 0 0001 g) of methionine hydroxy analogue (CAS No.. 583-91-5, purity greater than 88%) and mix well.

Dissolve and dilute to volume with 10% acetonitrile solution (5.5) in a 100 mL volumetric flask, and mix well. Store below -18°C; shelf life is 6 months. Alternatively, use a standard... Quasi-materials.

Note. If necessary, use the correction standard stock solution for determining the content of methionine hydroxy analogues according to GB 7300.103.

5.9 Standard Series Solutions. Accurately transfer appropriate amounts of the standard stock solution (5.8), dilute and bring to volume with 10% acetonitrile solution (5.5), mix well, and prepare...

A series of standard solutions with mass concentrations of 5 μg/mL, 10 μg/mL, 20 μg/mL, 40 μg/mL, and 80 μg/mL were prepared. (Prepared for immediate use) Prepared on the spot.

5.10 Microporous filter membrane. 0.45μm, organic system.

6 Instruments and Equipment

6.1 High-performance liquid chromatograph. equipped with an ultraviolet detector or a diode array detector.

6.2 Analytical balance. with an accuracy of 0.01g and 0.0001g.

6.3 Centrifuge. speed not less than 8000 r/min. 6.4 Vortex Oscillator. 6.5 Ultrasonic cleaner. 6.6 Reciprocating oscillator.

7 Samples

Prepare samples according to GB/T 20195, at least.200g, crush them so that they all pass through a test sieve with a 0.425mm aperture, mix thoroughly, and load into... In a sealed container.

8.1.1 Compound feed, concentrated feed, and concentrate supplement

Perform two parallel tests. Weigh 5g of the sample (accurate to 0.0001g) and place it in a 150mL Erlenmeyer flask with a stopper. Accurately add 50mL of [unspecified liquid].

Mix 10% acetonitrile solution (5.5), vortex for 30 seconds, shake vigorously on a shaker for 30 minutes, and centrifuge at 8000 rpm for 5 minutes. Collect the supernatant. spare.