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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13085-2018

Replacing GB/T 13085-2005

Determination of nitrite in feeds - Colorimetry method

饲料中亚硝酸盐的测定 比色法

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Foreword

This Standard was drafted in accordance with the rules given in GB/T 1.1-2009. This Standard replaces GB/T 13085-2005, Determination of nitrite in feed - Method using colorimetric analysis. Compared with GB/T 13085-2005, the main technical changes, except editorial changes, are as follows: -- Modify the detection limit of the method, and add the quantitation limit of the method (see Chapter 1; Chapter 1 of the 2005 edition); -- Modify the preparation method of test solution (see 7.1;

7.1 of the 2005 edition); -- Modify the preparation method of standard curve (see 7.2;

7.2 of the 2005 edition); -- Modify the measurement method of absorbance (see 7.3;

7.3 of the 2005 edition); -- Modify the requirements for precision (see Chapter 9;

8.3 of the 2005 edition). This Standard shall be under the jurisdiction of National Technical Committee 76 on Feed Industry of Standardization Administration of China (SAC/TC 76). Drafting organization of this Standard. Huazhong Agricultural University. Main drafters of this Standard. Qi Desheng, Zhang Niya, He Qiongyu, Wang Jun, Tao Can, Zhu Luoyi. The previous versions - replaced by this Standard - of the standard are. -- GB/T 13085-1991, GB/T 13085-2005. Determination of nitrite in feeds - Colorimetry method

1 Scope

GB/T 13085-2018 is the Chinese national standard on determination of nitrite in feeds - colorimetry method, in the field of agriculture. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 17 September 2018 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 April 2019. 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 colorimetry method for the determination of nitrite in feeds. This Standard applies to the determination of nitrite in feed raw materials, compound feeds, concentrated feeds and concentrate supplements. The detection limit of this method is

0.70 mg/kg, and the quantitation limit is

2 Normative references

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

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

GB/T 14699.1, Feeding stuffs - Sampling

3 Principle

The sample removes protein under weakly alkaline conditions, and the nitrite in the sample reacts with p-aminobenzenesulfonic acid under weakly acidic conditions to form diazo compounds, which are then coupled with N-1-naphthylethylenediamine to form purple compounds.

4 Reagents or materials

Unless otherwise specified, only use reagents confirmed to be of analytical grade in the analysis.

4.1 Water. grade-II water as specified in GB/T 6682.

4.2 Dilute hydrochloric acid solution. Take 5 mL of hydrochloric acid; add it to a 100 mL volumetric flask; use water to dilute to a constant volume; mix well.

4.7 Acetic acid solution. Measure 600 mL of glacial acetic acid in a 1 000 mL volumetric flask; use water to dilute to the mark; mix well.

4.8 P-aminobenzenesulfonic acid solution. Weigh

5.0 g of p-aminobenzenesulfonic acid; dissolve it in 700 mL of water and 300 mL of glacial acetic acid; mix well; store in a brown bottle. The validity period is 7 days.

4.9 N-1-Naphthylethylenediamine solution. Weigh

0.1 g of N-1- naphthylethylenediamine; add acetic acid solution (4.7) to dissolve and dilute to a constant volume of 100 mL; mix well; put it in a brown bottle; store it in a refrigerator at 4 °C ~ 10 °C. The validity period is 7 days.

4.10 Color-developing agent. Mix equal volumes of p-aminobenzenesulfonic acid solution (4.8) and N-1-naphthylethylenediamine solution (4.9) before use.

4.12 Sodium nitrite standard working solution (5.0 μg/mL). Accurately pipette

1.00 mL of sodium nitrite standard stock solution (4.11) into a 100 mL volumetric flask; add water to dilute to a constant volume to the mark; mix well. Prepare when necessary.

5 Instruments and equipment

5.1 Spectrophotometry.

5.2 Water bath. The temperature control range is 25 °C ~ 100 °C; the temperature control accuracy is ±1 °C.

6 Sample

Collect samples according to GB/T 14699.1, and prepare samples according to GB/T 20195.

7 Test steps

7.1 Preparation of test solution Perform two tests in parallel. Weigh about 5 g of sample, accurate to

0.001 g; place it in a 200 mL conical flask; add 70 mL of water and

1.2 mL of sodium hydroxide solution (4.6); mix well; use sodium hydroxide solution (4.6) to adjust the pH to 8 ~ 9; add 10 mL of zinc sulfate solution (4.5);

7.2 Preparation of nitrite standard curve Accurately pipette 0 mL,

0.4 mL,

0.8 mL,

2.0 mL of sodium nitrite standard working solution (4.12), respectively, into 25 mL brown volumetric flasks (equivalent to 0 μg, 2 μg, 4 μg, 6 μg, 8 μg, 10 μg of sodium nitrate); after respectively adding

4.5 mL of ammonium chloride buffer solution (4.4) and

2.5 mL of acetic acid solution (4.7), immediately add

5.0 mL of color-developing agent (4.10);

7.3 Determination of test solution Pipette two parts of

10.0 mL test solution (7.1) into 25 mL volumetric flasks a and b, respectively. Add

4.5 mL of ammonium chloride buffer solution (4.4) and

2.5 mL of acetic acid solution (4.7) into the volumetric flask a; then, use water to dilute to the mark; mix well.

8 Test data processing

The content of nitrite in the sample is calculated according to the mass fraction w of sodium nitrite ($NaNO_2$); the value is expressed in milligrams per kilogram (mg/kg), and calculated according to Formula (1).