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NATIONAL STANDARD
OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13081-2006

Replacing GB/T 13081-1991

Determination of mercury in feeds

饲料中汞的测定

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Foreword

This standard is a revised version of GB/T 13081-1991 "Determination of mercury in feeds".

The main differences between this standard and GB/T 13081-1991 are as follows:

- ADD the atomic fluorescence spectrometry and USE it as an arbitration method;
- REVISE the preparation of the stannous chloride solution in the cold atomic absorption spectrometry: PREPARE it in accordance with JJG 548-2004.

The standard shall, from the date of implementation, replace GB/T 13081-1991.

This standard was proposed by AND shall be under the jurisdiction of the National Feed Industry Standardization Technical Committee.

The drafting organizations of this standard: State Feed Quality Supervision and Inspection Center (Wuhan), Jiangsu Yixing Tianshi Feed Co., Ltd.

The main drafters of this standard: He Yifan, Zou Sanyuan, Liu Xiaomin, Yang Lin, Yang Xiankui.

This standard was first published in 1991 as the national standard GB 13081-1991. It is ranked as a non-mandatory standard in 1997, AND its number is changed into GB/T 13081-1991. This revision is the first revision of the standard.

Determination of mercury in feeds

1 Scope

This standard specifies the method of determination of mercury in mixed feed, concentrated feed, premixed feed, and feed additives.

This standard is applicable to the determination of mercury in compound feed, concentrated feed, premix feed and feed additive.

Atomic fluorescence spectrometry: the detection limit is 0.15 µg/kg, the best linear range of standard curve is 0 µg/L ~ 60 µg/L; the cold atomic absorption detection limit: pressure digestion method is 0.4 µg/kg, other digestion method is 10 µg/kg.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 602-2002 Chemical reagent – Preparations of standard solutions for impurity

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

GB/T 14699.1 Feeding stuffs - Sampling

GB/T 20195 Animal feeding stuffs – Preparation of test samples

JJG 548-2004 Verification regulation of mercury analyzers

3 Sampling and sample preparation

Sampling shall be carried out in accordance with the provisions of GB/T 14699.1. The preparation of the samples shall be carried out in accordance with the provisions of GB/T 20195.

4 Method 1: Atomic fluorescence spectrometry (arbitration method)

4.1 Principles

4.5.3 Repeatability

The same analyst performs two determinations against the same sample simultaneously or continuously, AND the difference between the results obtained shall be:

- Not more than 100% of the average when the mercury content is less than or equal to 0.020 mg/kg;
- Not more than 50% of the average when the mercury content is more than 0.020 mg/kg and less than 0.100 mg/kg;
- Not more than 20% of the average when the mercury content is more than 0.100 mg/kg.

5 Method 2: Cold atomic absorption spectrometry

5.1 Principles

In the atomic absorption spectrum, the mercury atoms have a strong absorption effect on the resonant lines having a wavelength of 253.7 nm. Samples are subjected to nitric acid - sulfuric acid digestion to convert the mercury into ionic state. In the strong acid, stannous chloride reduces the mercury ion into elemental mercury, which is blown out by the carrier of dry and clean air, subjected to cold atom absorption, AND it is quantified by comparison with the standard series.

5.2 Reagents and solutions

Unless otherwise specified, the reagents used in this standard are analytical pure, AND water is deionized water or water of equivalent purity, which shall comply with the level II requirements of GB/T 6682.

5.2.1 Nitric acid (excellent grade pure).

5.2.2 Hydrochloric acid (excellent grade pure).

5.2.3 Sulfuric acid (excellent grade pure).

5.2.4 10% stannous chloride solution: PREPARE it in accordance with JJG 548-2004; WEIGH 10 g of stannous chloride (excellent grade pure); ADD 20 mL of concentrated hydrochloric acid (5.2.2); slightly HEAT it to make it dissolve and transparent; ADD water to dilute it to 100 mL; PREPARE it before use.

5.2.5 Mixed acid solution: MEASURE 10 mL of sulfuric acid (5.2.3); ADD 10 mL of nitric acid (5.2.1); slowly POUR it into 50 mL water; COOL it down; ADD water to dilute it to 100 mL.

5.2.6 Mercury standard stock solution: same as 4.2.8.

5.4.2 Drawing of standard curve

PIPETTE 0 mL, 0.10 mL, 0.20 mL, 0.30 mL, 0.40 mL, and 0.50 mL of mercury standard solution (equivalent to 0 µg, 0.01 µg, 0.02 µg, 0.03 µg, 0.04 µg, 0.05 µg of mercury); PLACE them into the reduction bottle (5.3.7); respectively ADD 10 mL of mixed acid solution (5.2.4); ADD 2 mL of stannous chloride solution (5.2.4); immediately COVER the reduction bottle tightly (5.3.7) for 2 min; RECORD the maximum absorbance of the mercury analyzer reading indicator. USE the absorbance as the ordinate and the mercury concentration as the abscissa, to draw the standard curve.

5.4.3 Determination

ADD 10 mL of the sample digestion solution in the reduction bottle (5.3.7); ADD 2 mL of stannous chloride solution (5.2.4); immediately COVER the reduction bottle tightly (5.3.7) for 2 min; RECORD the maximum absorbance of the mercury analyzer reading indicator.

5.5 Determination results

5.5.1 Calculation

The mercury content in the sample is calculated in accordance with the formula (2):

$$\omega = \frac{(m_1 - m_0) \times 1\,000}{m \times \frac{V_2}{V_1} \times 1\,000} = \frac{V_1(m_1 - m_0)}{mV_2} \quad \dots\dots\dots(2)$$

Where:

w - The content of mercury in the sample, in milligrams per kilogram (mg/kg);

m₁ - The mass of mercury in the sample digestion solution for the determination purposes, in micrograms (µg);

m₀ - The mass of mercury in the reagent blank solution, in micrograms (µg);

m - The sample mass, in grams (g);

V₁ - The total volume of the sample digestion solution, in milliliters (mL);

V₂ - The volume of the digestion solution for the determination purposes, in milliliters (mL).

5.5.2 Expression of analysis results

Each sample is subjected to 2 times of parallel determination, AND the arithmetic mean is used as the result.

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Contact: Wayne Zheng, Sales@ChineseStandard.net

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