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

GB/T 13081-2022

Replacing GB/T 13081-2006

Determination of mercury in feeds

饲料中汞的测定

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Foreword

This document was drafted in accordance with the provisions of GB/T 1.1-2020 Directives for standardization - Part

1.Rules for the structure and drafting of standardizing documents. This document replaces GB/T 13081-2006 Determination of mercury in feeds. Compared with GB/T 13081-2006, in addition to structural adjustments and editorial changes, the main technical changes are as follows:

- a) The scope of application has been increased to concentrate supplements and feed ingredients; the quantitation limits of atomic fluorescence spectrometry and cold atomic absorption spectrometry are added (see Chapter 1; see Chapter 1 of the 2006 edition);
- b) The treatment methods of high-pressure tank digestion and microwave digestion are changed (see 4.5.1.1 and 4.5.1.2; see 4.4.1.2 of the 2006 edition);
- c) The reference conditions of microwave digestion are changed (see 4.5.1.2; see

4.4.1.2 of the 2006 edition);

d) The reference conditions of the atomic fluorescence spectrometry instrument are changed (see 4.5.2; see

4.4.3 of the 2006 edition);

e) The test data processing is changed (see

4.6 and 5.6; see

5.5 of the 2006 version);

f) The expression of precision is changed (see

1 Scope

GB/T 13081-2022 is the Chinese national standard on determination of mercury in feeds, 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 30 December 2022 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 July 2023. 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 document describes the atomic fluorescence spectrometry, cold atomic absorption spectrometry, and direct injection methods for the determination of total mercury in feeds. This document is applicable to the determination of total mercury in compound feeds, concentrated feeds, concentrate supplements, additive premixed feeds, feed raw materials, and feed additives. Atomic fluorescence spectrometry. When the sample weight is

0.5 g and the constant volume is 50 mL, the method detection limit is 0.6 µg/kg and the quantification limit is 3 µg/kg. Cold atomic absorption spectrometry. When the sample weight is 2 g and the constant volume is 100 mL, the method detection limit is 3 µg/kg and the quantitation limit is 10 µg/kg. Direct injection method. When the sample weighing amount is

0.1 g, the detection limit of the method is 0.3 µg/kg, and the quantification limit is 1 µg/kg.

2 Normative references

The contents of the following documents constitute essential provisions of this document through normative references in the text. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated

referenced documents, only the latest version (including all the amendments) is applicable to this document.

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

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

3 Terms and definitions

There are no terms or definitions that need to be defined in this document.

4 Atomic fluorescence spectrometry (arbitration method)

4.1 Principle After the sample is digested by acid heating, mercury is reduced to atomic mercury by potassium borohydride (KBH₄) in the acidic medium, which is brought into the atomizer by argon gas. Under the irradiation of a special mercury hollow cathode lamp, the ground-state mercury atoms are excited to a high energy state and then deactivated back to the ground state, which emit fluorescence with a characteristic wavelength. Its fluorescence intensity is proportional to the mercury content and can be quantified by the external standard method.

4.2 Reagents or materials WARNING -- Mercury is highly toxic. Pay attention to ventilation when handling and avoid contact with skin and clothing. All kinds of strong acids shall be handled carefully and shall be handled in a fume hood.

4.3 Instruments and equipment

4.3.1 Atomic fluorescence spectrometer. It is equipped with a mercury hollow cathode lamp.

4.3.2 Analytical balance. The sensitivity is 0.0001 g.

4.3.3 High-pressure digestion tank. It is equipped with a 100 mL polytetrafluoroethylene digestion tube.

4.3.4 Microwave digestion instrument. It is equipped with a 50 mL polytetrafluoroethylene digestion tube.

4.3.5 Constant temperature drying oven. The temperature control accuracy is ± 2 °C.

4.3.6 Ultrasonic cleaner. NOTE. Glassware and polytetrafluoroethylene digestion tubes need to be soaked in nitric acid solution I (4.2.4) for 24 hours, and then rinsed repeatedly with water.

4.4 Samples The sample is prepared according to GB/T 20195. The solid sample shall be at least 200 g. It is crushed so that it all passes through an analysis sieve with a pore size of 0.425 mm; it is mixed thoroughly and stored in an airtight container for later use.

4.5.1 Sample solution preparation

4.5.3 Determination Adjust the atomic fluorescence spectrometer to the best working condition and start measurement after stabilizing for 10 to 20 minutes. Use nitric acid solution III (4.2.6) as the carrier fluid and potassium borohydride solution (4.2.8) as the reducing agent, and use nitric acid solution II (4.2.5) to continuously inject samples; after the reading is stable, measure the standard series of solutions.

4.6 Test data processing The mercury content w in the sample is expressed as a mass fraction, in milligrams per kilogram (mg/kg), and is calculated according to formula (1).

4.7 Precision Under repeatability conditions, the ratio of the absolute difference between the two independent measurement results and their arithmetic mean to the arithmetic mean, that is, the relative deviation r , shall meet the requirements of Table 3.

5 Cold atomic absorption spectrometry

5.1 Principle The sample is digested with nitric acid-sulfuric acid, and the mercury is converted into an ionic state. Under strong acid conditions, mercury ions are reduced to elemental mercury by stannous chloride, which is blown out by dry clean air. The absorbance is measured at a wavelength of 253.7 nm and quantified by the external standard method.

5.2 Reagents or materials **WARNING** -- Mercury is highly toxic. Pay attention to ventilation when handling and avoid contact with skin and clothing. All kinds of strong acids shall be handled carefully and shall be handled in a fume hood. Unless otherwise specified, use only guaranteed reagents.

5.2.1 Water. The second-grade water specified in GB/T 6682.

5.2.2 Nitric acid.

5.2.3 Sulfuric acid.

5.2.9 Mercury standard working solution (0.1 $\mu\text{g/mL}$). Accurately pipette 1 mL of mercury standard intermediate solution (5.2.8), place it in a 100 mL volumetric flask, add the mixed acid solution (5.2.6) to dilute to the mark, and mix well. Prepare it fresh just before use.

5.3 Instruments and equipment

5.3.1 Cold atom mercury analyzer. It is equipped with a 50 mL reducing bottle.

5.3.2 Analytical balance. The sensitivity is 0.0001 g.

5.5 Test procedures

5.5.1 Preparation of sample solution Do two experiments in parallel. Weigh 1 g~5 g of the