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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13079-2022

Replacing GB/T 13079-2006

Determination of total arsenic in feeds

饲料中总砷的测定

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1 Scope

GB/T 13079-2022 is the Chinese national standard on determination of total arsenic 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 silver salt method, hydride generation-atomic fluorescence spectrometry and inductively coupled plasma mass spectrometry for the determination of total arsenic in feeds. This document is applicable to the determination of total arsenic in compound feeds, concentrated feeds, concentrate supplements, additive premixed feeds,

feedstuffs and feed additives. When the sampling size is 5 g and the constant volume is 50 mL, the detection limit of the silver salt method is

0.25 mg/kg and the quantitation limit is

0.50 mg/kg; when the sampling size is 5 g, the constant volume is 50 mL and the dilution factor is 20, the detection limit of the hydride generation-atomic fluorescence spectrometry and inductively coupled plasma mass spectrometry is

0.02 mg/kg, and the quantitation limit is

0.05 mg/kg.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 20195 Animal Feed - Preparation of Test Samples

3 Terms and Definitions

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

4 Silver Salt Method (arbitration method)

4.1 Principle After a specimen is treated by acid digestion, direct acid dissolution or dry ashing method, use potassium iodide and stannous chloride to reduce high-valent arsenic to trivalent arsenic, then, generate arsine with the new ecological hydrogen produced by zinc particles and acid, which is absorbed by the silver diethyldithiocarbamate (Ag-DDTC)-triethylamine-chloroform solution to form a red jelly, whose color shade is proportional to the arsenic content.

4.2 Reagents or Materials WARNING

---handle all kinds of strong acids with caution. Dilute and use them in a fume hood. When using perchloric acid, be careful not to burn it to dryness, and be careful of explosion.

4.3 Instruments and Equipment

4.3.1 Spectrophotometer. with a wavelength accuracy of 2 nm.

4.3.2 Analytical balance. with an accuracy of 0.0001 g.

4.3.3 Electric drying oven. with a temperature control accuracy of 5 C.

4.4 Sample In accordance with GB/T 20195, prepare the sample of at least 200 g. Make it all pass through the analysis sieve with a pore size of

0.425 mm, thoroughly mix it, put it into a closed container and set it aside.

4.5.2.1 Preparation of standard series of solutions Accurately transfer-take

0.00 mL,

10.00 mL of the arsenic standard intermediate solution (4.2.20), respectively place them in arsenic reaction bottle, and use water to dilute to 30 mL. Add 10 mL of hydrochloric acid (4.2.3), mix it well, and follow the steps specified in 4.5.2.2, starting from the addition of 2 mL of potassium iodide solution (4.2.12).

4.6 Test Data Processing The total arsenic content w1 in the specimen, which is expressed in mass fraction and (mg/kg), is calculated in accordance with Formula (1).

4.7 Precision The ratio of the absolute difference between the two independent determination results obtained under repeatability conditions and their arithmetic mean, and the arithmetic mean shall comply with the requirements of Table 1.

5 Hydride Generation-atomic Fluorescence Spectrometry

5.1 Principle After a specimen is treated by acid digestion, direct acid dissolution or dry ashing method, add thiourea to pre-reduce pentavalent arsenic to trivalent arsenic, then, in an acidic environment, reduce it with potassium borohydride to generate gaseous arsine, which is loaded into a quartz atomizer by argon gas and decomposed into atomic arsenic. Under the excitation of the emitted light of an arsenic hollow cathode lamp, generate atomic fluorescence, whose fluorescence intensity is proportional to the concentration of arsenic in the determined solution. Adopt the external standard method for quantitative determination.

5.2 Reagents or Materials WARNING

---handle all kinds of strong acids with caution. Dilute and use them in a fume hood. When using perchloric acid, be careful not to burn it to dryness, and be careful of explosion. Unless otherwise specified, use only analytically pure reagents.

5.2.1 Water. GB/T 6682, Grade-1.

5.2.2 Nitric acid. guaranteed reagent.

5.2.3 Hydrochloric acid. guaranteed reagent.

5.2.4 Sulfuric acid. guaranteed reagent.

5.2.5 Hydrogen peroxide.

5.2.6 Magnesium oxide. Total Arsenic Content (w1)/(mg/kg) Relative Deviation/%

5.2.7 Magnesium nitrate.

5.2.8 Potassium hydroxide. guaranteed reagent.

5.2.9 Potassium borohydride. guaranteed reagent.

5.3 Instruments and Equipment

5.3.1 Atomic fluorescence spectrophotometer.

5.3.2 Analytical balance. with an accuracy of 0.0001 g.

5.4 Sample Same as 4.4.

5.5 Test Procedures

5.5.4 Instrument reference conditions The instrument reference conditions are as follows:

5.5.5 Determination Adjust the atomic fluorescence spectrophotometer to the optimum working condition, successively determine the fluorescence intensity value of the standard series of solutions, blank solution and specimen solution. Take the concentration of the standard series of solutions as the x-coordinate and the fluorescence intensity value as the y-coordinate to draw a standard curve, with a correlation coefficient of r 0.995.When the fluorescence value of the specimen solution exceeds the linear range of the curve, reduce the dispensing volume of the specimen solution, in accordance with

5.5.3 and 5.5.4, re-perform the determination. In order to ensure good repeatability of the determination results, it is recommended that the number of sequences for each determination should not exceed 30 (including the number of curve sequences).

5.6 Test Data Processing The total arsenic content w2 in the specimen, which is expressed in mass fraction and (mg/kg), is calculated in accordance with Formula (2).

6 Inductively Coupled Plasma Mass Spectrometry

6.1 Principle After a specimen is treated by acid digestion, direct acid dissolution or dry ashing method, dilute it into a specimen test solution, which is atomized and sent into the ICP torch by the carrier gas. Through evaporation, dissociation, atomization and ionization, convert it into charged ions, which enter the mass spectrometer through the ion collection system.

6.2 Reagents or Materials WARNING

---handle all kinds of strong acids with caution. Dilute and use them in a fume hood. When