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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13883-2023

Replacing GB/T 13883-2008

Determination of selenium in feeds

饲料中硒的测定

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1 Scope

GB/T 13883-2023 is the Chinese national standard on determination of selenium 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 6 August 2023 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 March 2024. Classification: ICS 65.120, CCS B 46. 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 determination of selenium in feeds by hydride generation-atomic fluorescence spectrometry, fluorescence spectrophotometry and inductively coupled plasma-mass spectrometry. In this document, hydride generation-atomic fluorescence spectrometry and fluorescence spectrophotometry are applicable to the determination of selenium in compound feed, concentrated feed, concentrate supplement, additive premix feed and feed raw materials. Inductively coupled plasma mass spectrometry is applicable to

the determination of selenium in compound feed, concentrated feed, concentrate supplement and feed raw materials (except mineral feed raw materials). When the sample size is 1 g and the fixed volume is 50 mL, the detection limit of hydride generation-atomic fluorescence spectrometry and fluorescence spectrophotometry is

0.01 mg/kg and the quantification limit is

0.02 mg/kg. When the sample size is

0.5 g and the fixed volume is 50 mL, the detection limit of inductively coupled plasma mass spectrometry is

0.01 mg/kg and the quantification limit is

0.02 mg/kg.

2 Normative references

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

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

There are no terms or definitions that require definition in this document.

4 Hydride generation-atomic fluorescence spectrometry (arbitration method)

4.1 Principle After the specimen is digested with acid, the selenium in the specimen digestion solution is reduced to tetravalent selenium in the hydrochloric acid medium. Potassium borohydride is used as a reducing agent to reduce tetravalent selenium to hydrogen selenide in the hydrochloric acid medium.

4.2 Reagents or materials Warning -- All strong acids shall be handled with caution. Dilution and use shall be carried out in a fume hood. When using perchloric acid, be careful not to burn it dry. Be careful of explosion.

4.2.17 Argon. the purity is not less than 99.995%.

4.3 Instruments and equipment

4.4 Sample Prepare the specimen according to GB/T 20195, at least 200 g. Crush the compound feed, concentrated feed, concentrate supplement and feed raw materials so that

they all pass through a

0.425 mm analysis sieve. Crush the additive premix feed so that they all pass through a

0.25 mm analysis sieve. Mix well. Put into a sealed container and set aside.

4.5.1 Preparation of specimen solution

4.5.4 Determination Adjust the instrument to the best working state. Start the measurement after stabilizing for 15 min~20 min. Use hydrochloric acid solution II (4.2.10) as the carrier and potassium borohydride solution (4.2.12) as the reducing agent. Use the carrier solution for continuous injection.

4.6 Test data processing The selenium content w1 in the specimen is expressed as mass fraction. The value is expressed in milligrams per kilogram (mg/kg), calculated according to formula (1).

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

5 Fluorescence spectrophotometry

5.1 Principle The specimen is digested with mixed acid, and selenium (Se4+) and 2,3-diaminonaphthalene (DAN) generate 4,5-phenylbenzoselenadiazole in a slightly acidic solution. The resulting complex is extracted with cyclohexane.

5.2 Reagents or materials Warning -- All strong acids shall be handled with caution. Dilution and use shall be carried out in a fume hood. When using perchloric acid, be careful not to burn it dry. Be careful of explosion.

5.2.1 Water. GB/T 6682, Grade II.

5.2.2 Perchloric acid. guaranteed reagent. Selenium content/(mg/kg) Relative deviation/%

5.2.3 Nitric acid. guaranteed reagent.

5.3 Instruments and equipment

5.4 Sample Same as 4.4.

5.5.1.1 Wet digestion

5.5.2 Extraction Add 2 drops of cresol red indicator (5.2.12) to the sample digestion solution. Adjust with ammonia solution (5.2.7) until the solution turns yellow. Adjust with hydrochloric acid solution III (5.2.8) until the solution turns orange (pH 1.5~2), then add 3 mL of hydroxylamine hydrochloride-ethylenediaminetetraacetic acid disodium solution (5.2.10). Shake well. Add 2 mL of 2,3-diaminonaphthalene solution (5.2.11).

5.6 Test data processing The selenium content w2 in the specimen is expressed as mass fraction. The value is expressed in milligrams per kilogram (mg/kg), calculated according to formula (2).

6 Inductively coupled plasma mass spectrometry

6.1 Principle The specimen is digested with acid to dissolve selenium. Under certain acidic conditions, it is introduced into an inductively coupled plasma mass spectrometer for determination.

6.2 Reagents or materials Selenium content/(mg/kg) Relative deviation/% Warning -- All strong acids shall be handled with caution. Dilution and use shall be done in a fume hood. When using perchloric acid, be careful not to burn it dry. Be careful of explosion.

6.2.5 Hydrogen peroxide.

6.2.6 Nitric acid solution I. Take 5 mL of nitric acid (6.2.3) and add it to 95 mL of water. Mix well.

6.2.7 Nitric acid solution II. Measure 30 mL of nitric acid (6.2.3) and add it to 70 mL of water. Mix well.

6.4 Sample Same as 4.4.

6.5.1 Preparation of specimen solution Perform two tests in parallel. Weigh

0.5 g of the specimen (accurate to 0.0001

g) into a digestion tank. Add 8 mL of nitric acid (6.2.3). Soak for 10 min. Add 2 mL of hydrogen peroxide (6.2.5).

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

6.5.3 Determination Adjust the instrument to the best working state. Calibrate with rhodium (103Rh) as internal standard. Add rhodium internal standard element working solution (6.2.13) online.