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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13080-2018

Replacing GB/T 13080-2004

Determination of lead in feeds - Atomic absorption spectrometry

饲料中铅的测定 原子吸收光谱法

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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 13080-2004 "Determination of lead in feeds - Method using atomic absorption spectrometry". Compared with GB/T 13080-2004, in addition to the editorial modifications, the main technical changes of this Standard are as follows: - Add the quantification limit for flame atomic absorption spectrometry (see Chapter 1 of this Edition); - Modify the expression of dry ashing method (see

7.1.1.1 of this Edition,

7.1.1 of Edition 2004); - Add the requirements to buckle the background (see

7.1.2 of this Edition); - Modify the test data processing formula (see

7.1.4 of this Edition,

8.1 of Edition 2004); - Modify the requirements for precision (see

7.1.5 of this Edition,

8.3 of Edition 2004); - Add graphite furnace atomic absorption spectrometry (see

7.2 of this Edition). This Standard was proposed by and shall be under the jurisdiction of National Technical Committee on Feed Industry of Standardization Administration of China (SAC/TC 76). The drafting organizations of this Standard. Sichuan Provincial Feed Work Station [Feed Quality Supervision, Inspection and Testing Center of the Ministry of Agriculture (Chengdu)]. Main drafters of this document. Li Yun, Zeng Xiaofang, Meng Fancui, Hao Qunhua, Zhao Lijun, Lin Shunquan, Zhang Jing, Cheng Chuanmin. Versions of standard substituted by this document are. - GB/T 13080-1991, GB/T 13080-2004. Determination of lead in feeds - Atomic absorption spectrometry

1 Scope

GB/T 13080-2018 is the Chinese national standard on determination of lead in feeds - atomic absorption spectrometry, 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 flame atomic absorption spectrometry and graphite furnace atomic absorption spectrometry for the determination of lead in feeds. This Standard is applicable to the determination of lead in compound feeds, concentrated feeds, additive premix feeds, concentrate supplement and feed raw materials. When the sampling volume is 5 g and the constant volume is 50 mL, the quantification limit for flame atomic absorption spectrometry is 2 mg/kg. When the sampling volume is 1 g and the constant volume is 10 mL, the quantification limit for graphite furnace atomic absorption spectrometry is 100 μg/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 602, Chemical reagent - Preparations of standard solutions for impurity

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

GB 6819-2004, Dissolved acetylene

GB/T 14699.1, Feeding stuffs - Sampling

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

3 Principle

After the specimen is dry ashed, acid dissolved or wet digested, the lead is dissolved. Use an atomic absorption spectrometer to measure the absorbance value at 283.3 nm.

4 Reagents or materials

WARNING -- All kinds of strong acids shall be handled with care. Dilution and use shall be carried out in a fume hood.

4.1 Water. Grade one water in GB/T 6682.

4.2 Nitric acid. Guaranteed reagent.

4.3 Perchloric acid. Guaranteed reagent.

4.10 Magnesium nitrate solution (

0.6 mg/mL). Weigh

60.0 mg of magnesium nitrate. Use water to dissolve and dilute to 100 mL. Mix well.

4.11 Lead standard stock solution (

1.0 mg/mL). Accurately weigh

1.598 g of lead nitrate [Pb(NO3)2]. Add 10 mL of nitric acid solution (4.8) to completely dissolve. Transfer to a 1000 mL volumetric flask.

4.12 Lead standard intermediate solution (10.0 μg/mL). Accurately pipette

1.00 mL of lead standard stock solution (4.11) into a 100 mL volumetric flask. Add water to dilute to the scale. Mix well. Prepare when it is required.

4.13 Lead standard working solution (100 ng/mL). Accurately pipette

1.00 mL of lead standard intermediate solution (4.12) into a 100.0 mL volumetric flask. Use nitric acid solution (4.7) to dilute and set the volume constant to the scale.

4.14 Acetylene. Meet the requirements of

3.1 in GB 6819-2004.

5 Instruments and equipment

5.1 Atomic absorption spectrophotometer. with flame or/and graphite furnace atomizer, lead hollow cathode lamp. acid solution (4.5) to cook for 2 h before use. Use water to wash clean.

5.6 Adjustable hot plate or adjustable electric furnace.

5.7 Flat bottom cylindrical PTFE crucible. 60 cm

3.Soak in hydrochloric acid solution (4.5) overnight before use. Use water to wash clean.

5.8 Glassware. Soak in hydrochloric acid solution (4.5) overnight before use. Use water to wash clean.

6 Sample

Conduct feed sampling according to GB/T 14699.1.Prepare specimens according to GB/T 20195.Crush. Pass a 1 mm nylon sieve. Mix well and load in an airtight container for future use.

7.1.1 Specimen processing

7.1.1.1 Dry ashing method It is suitable for feed raw materials, compound feeds, concentrated feeds and concentrate supplements that contain more organic matter.

7.1.1.2 Perchloric acid digestion WARNING -- Be careful not to burn dry when using perchloric acid. Be careful of explosion.

7.1.1.3 Hydrochloric acid dissolution method It is suitable for additive premix feeds without organic matter. Do two tests in parallel. According to the expected content, weigh 1 g ~ 5 g of specimen

7.1.2 Standard curve drawing Set the instrument to buckle background mode. Respectively pipette 0 mL,

8.0 mL of lead standard intermediate solutions (4.12) into

50.0 mL volumetric flasks. Add 1 mL of hydrochloric acid solution (4.6).

7.1.3 Determination Under the same test conditions, measure the absorbance value of reagent blank and specimen solution. Compare with the standard curve and quantify.

7.2.1 Specimen dissolution Do two tests in parallel. Weigh

1.0 g of the specimen (accurate to 0.0001g) in a porcelain crucible. Heat slowly on an adjustable electric heating plate or adjustable electric furnace at 100°C~300°C to carbonize