GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 65.120

B 46

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

Issued on: September 17, 2018 **Implemented on: April 01, 2019**

Issued by: State Administration for Market Regulation;
Standardization Administration of the People's Republic of China.

Table of Contents

Determination of lead in feeds - Atomic absorption spectrometry

1 Scope

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. Compare with the standard curve and quantify.

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.

Unless otherwise stated, it shall only use the confirmed analytically-pure reagents in the analysis. The reagents and solutions used in the test were prepared according to the provisions of GB/T 602.

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

4.2 Nitric acid: Guaranteed reagent.

4.3 Perchloric acid: Guaranteed reagent.

4.4 Hydrofluoric acid.

4.5 Hydrochloric acid solution (0.6 mol/L): Measure 5 mL of hydrochloric acid. Use water to dilute to 100 mL. Mix well.

4.6 Hydrochloric acid solution (6mol/L): Measure 50 mL of hydrochloric acid. Use water to dilute to 100 mL. Mix well.

4.7 Nitric acid solution (0.5mol/L): Measure 3.6 mL of nitric acid. Use water to dilute to 100 mL. Mix well.

4.8 Nitric acid solution (6mol/L): Measure 43 mL of nitric acid. Use water to dilute to 100 mL. Mix well.

4.9 Ammonium dihydrogen phosphate solution (10.0 mg/mL): Weigh 1.0 g of ammonium dihydrogen phosphate. Use water to dissolve and dilute to 100 mL. Mix well.

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 [$Pb(NO_3)_2$]. Add 10 mL of nitric acid solution (4.8) to completely dissolve. Transfer to a 1000 mL volumetric flask. Add water to dilute and set the volume constant to the scale. Mix well. Store in a PTFE bottle. Store at 4°C. The validity is 6 months. Or purchase certified reference materials to prepare corresponding concentrations.

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

Do two tests in parallel. Weigh 5 g of the specimen (accurate to 0.0001 g) in a porcelain crucible. Heat slowly on an adjustable electric furnace at 100°C~300°C to carbonize the specimen until it is smokeless. Move the crucible into a muffle furnace at 550°C for ashing for 2 h ~ 4 h. After cooling, use 2 mL of water to wet the carbide. If there is still a small amount of carbon particles, nitric acid solution (4.8) can be dropped to wet the residue. Move the crucible to an adjustable electric heating plate or an adjustable electric furnace to dry on low heat. Move to a muffle furnace for ashing for 2 h. After cooling, add 2 mL of water along the crucible wall.

Draw 5 mL of hydrochloric acid solution (4.6) and add it dropwise into the crucible. Turn the crucible while adding until the solution overflows without bubbles. Then add all the remaining hydrochloric acid solution. Then add 5 mL of nitric acid solution (4.8). Turn the crucible. Use an adjustable electric heating plate or an adjustable electric stove to heat until the digestive juice reaches 2 mL ~ 3 mL (be careful not to splash). Remove. After cooling, use water to transfer the digestion solution to a 50 mL volumetric flask. Add a little water to rinse the crucible several times. The washing solution is merged into the volumetric flask. Dilute to the scale. Shake well. Use ash-free filter paper to filter for later use. At the same time, prepare a reagent blank solution.

7.1.1.2 Perchloric acid digestion

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

It is suitable for additive premixed feeds containing organic matter.

Do two tests in parallel. Weigh 1 g of specimen (accurate to 0.0001 g) in a polytetrafluoroethylene crucible. Add water to wet the sample. Add 10 mL of nitric acid (for samples containing more silicates, add 5 mL of hydrofluoric acid). Put it in a fume hood for 2 h. Add 5 mL of perchloric acid. Heat to digest on an adjustable electric stove below 250°C. Wait until the digestive solution emits white smoke. Remove. After cooling, use water to transfer to a 50.0 mL volumetric flask. Rinse the crucible several times with a little water. The washing solution is put into the volumetric flask and diluted to the scale. Shake well. Use ashless filter paper to filter for later use. At the same time, prepare the reagent blank solution.

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 (accurate to 0.0001 g) in a porcelain crucible. Add 2 mL of water to wet the specimen. Pipette 5 mL of hydrochloric acid solution (4.6). Add dropwise to the crucible. Turn the crucible while adding until the solution overflows without bubbles. Then add all the remaining hydrochloric acid solution. Then add 5 mL of nitric acid solution (4.8). Move the crucible to an adjustable electric furnace to heat and digest on low heat. Remove until the digestive solution reaches 2 mL ~ 3 mL (be careful not to splash out). After

cooling, use water to transfer to a 50.0 mL volumetric flask. Add a little water to rinse the crucible several times. The washing solution is put into the volumetric flask and diluted to the scale. Shake well. Use ashless filter paper to filter for later use. At the same time, prepare the reagent blank solution.

7.1.2 Standard curve drawing

Set the instrument to buckle background mode. Respectively pipette 0 mL, 1.0 mL, 2.0 mL, 4.0 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). Use water to set the volume constant to the scale. Shake well. Import into an atomic absorption spectrophotometer. Use water for zero adjustment. Measure the absorbance value at a wavelength of 283.3 nm. Draw the standard curve with the absorbance value as the vertical axis and the concentration as the horizontal axis.

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.

7.1.4 Test data processing

The content of lead in the specimen is calculated as mass fraction w. The value is expressed in milligrams per kilogram (mg/kg), calculated according to formula (1):

$$w = \frac{(\rho_1 - \rho_2) \times V}{m} \qquad \cdots\cdots (1)$$

Where,

ρ_1 - the mass concentration of lead in the specimen solution, in micrograms per milliliter (μg/mL);

ρ_2 - the mass concentration of lead in the blank reagent, in micrograms per milliliter (μg/mL);

V - the total volume of the specimen solution, in milliliters (mL);

m - the mass of the specimen, in grams (g).

Report the results as the arithmetic mean of the results of two parallel samples. Results shall be expressed to two decimal places.

7.1.5 Precision

Under repeatability conditions, the relative deviation between the obtained two independent measurement results shall meet the requirements of Table 1.

7.2.3 Standard curve drawing

Respectively and accurately pipette an appropriate volume of lead standard working solution (4.13). Prepare lead standard series solutions with concentrations of 0 μg/L, 10 μg/L, 20 μg/L, 30 μg/L, 40 μg/L, and 50 μg/L. Respectively pipette 10 μL into the graphite furnace. Add 5 μL of ammonium dihydrogen phosphate solution (4.9) and 5 μL of magnesium nitrate solution (4.10). Use nitric acid solution (4.7) for zero adjustment. Measure the absorbance value of the standard series solutions at a wavelength of 283.3 nm. Draw the standard curve with the absorbance value as the vertical axis and the concentration as the horizontal axis. If the concentration of the specimen solution exceeds the range of the standard curve, it can be diluted with nitric acid solution (4.7) to determine within the linear range.

NOTE: For instruments with automatic sampling, the automatic sampler can be used to complete the preparation of standard series solutions and the dilution of specimen solutions.

7.2.4 Determination of specimen solution

Under the same test conditions, inject 10 μL of specimen solution, 5 μL of ammonium dihydrogen phosphate solution (4.9) and 5 μL of magnesium nitrate solution (4.10) into the graphite furnace. Measure the absorbance value. Compare with the standard curve and quantify.

7.2.5 Test data processing

The content of lead in the specimen is calculated as mass fraction w. The value is expressed in milligrams per kilogram (mg/kg), calculated according to formula (2):

$$w = \frac{(\rho_1 - \rho_2) \times V}{m \times 1\ 000} \qquad \cdots\cdots(2)$$

Where,

ρ_1 - the mass concentration of lead in the specimen solution, in micrograms per liter (μg/L);

ρ_2 - the mass concentration of lead in the blank reagent, in micrograms per liter (μg/L);

V - the total volume of the sample solution, in milliliters (mL);

m - the specimen mass, in grams (g);

1000 - the unit conversion factor.

Report the results as the arithmetic mean of the results of two parallel samples. Results shall be expressed to two decimal places.

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------