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Determination of chromium in feeds

饲料中铬的测定

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Determination of chromium in feeds

1 Scope

This Standard specifies two methods for the determination of chromium content in feed by atomic absorption spectrometer and spectrophotometer.

Method 1 of this Standard is atomic absorption spectrometry. It is applicable to feed raw materials (including leather powder for feed, hydrolyzed leather powder), trace element premixes, compound premixes, concentrates and compound feeds. The minimum detection limit of graphite furnace atomic absorption spectrometry is 0.005 $\mu\text{g/kg}$. The minimum detection limit of flame atomic absorption spectrometry is 150 $\mu\text{g/kg}$. Method 2 of this Standard is spectrophotometric method. It is applicable to the determination of chromium in feed raw materials (including hydrolyzed leather powder) and compound feed.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682, *Water for analytical laboratory use - Specification and test methods* (neq ISO 3696)

GB/T 14699.1, *Feeding stuffs - Sampling*

3 Method 1: Atomic absorption spectrometry

3.1 Principle

The sample is ashed at high temperature. After dissolving with acid, inject it into the atomic absorption spectrometer detector. In a certain concentration range, its absorption value is proportional to the chromium content. Compare with the standard series to quantify.

3.2 Reagents and solutions

Unless otherwise specified, the reagents used in Method 1 are guaranteed reagents; the

water is ultrapure water or water of corresponding purity, which meets the requirements of GB/T 6682 for grade one water.

3.2.1 Concentrated nitric acid.

3.2.2 Nitric acid solution: $V(\text{nitric acid}) + V(\text{water}) = 2 + 98$.

3.2.3 Nitric acid solution: $V(\text{nitric acid}) + V(\text{water}) = 20 + 80$.

3.2.4 Chromium standard solution

3.2.4.1 Chromium standard stock solution (100 mg/L): Weigh 0.2830 g of potassium dichromate that has been baked at 100°C~110°C to a constant amount. Use water to dissolve. Move into a 1000 mL volumetric flask. Dilute to the scale. This solution is equivalent to 0.1 mg of chromium per mL.

3.2.4.2 Chromium standard solution 1 (20 mg/L): Weigh 10.0 mL of chromium standard stock solution (3.2.4.1) in a 50 mL volumetric flask. Add nitric acid solution (3.2.2) to dilute to the scale. This solution is equivalent to 20 µg of chromium per mL.

3.2.4.3 Chromium standard solution 2 (2 mg/L): Weigh 1.0 mL of chromium standard stock solution (3.2.4.1) in a 50 mL volumetric flask. Add nitric acid solution (3.2.2) to dilute to the scale. This solution is equivalent to 2 µg of chromium per mL.

3.2.4.4 Chromium standard solution 3 (0.2 mg/L): Weigh 10.0 mL of chromium standard solution 2 (3.2.4.3) in a 100 mL volumetric flask. Add nitric acid solution (3.2.2) to dilute to the scale. This solution is equivalent to 0.2 µg of chromium per mL.

3.3 Instruments and equipment

All glassware and crucibles are immersed in nitric acid solution (3.2.3) for 24 h or longer. User pure water to rinse. Dry in the air.

3.3.1 Experimental sample grinder or mortar (chromium-free).

3.3.2 Ultrapure water device (Millipore).

3.3.3 Analytical balance: resolution is 0.0001 g.

3.3.4 Porcelain crucible: 60 mL.

3.3.5 Temperature-controlled electric hob: 600 W.

3.3.6 High temperature electric furnace (muffle furnace).

3.3.7 Volumetric flasks: 20 mL, 50 mL, 100 mL, 1000 mL.

3.3.8 Pipettes: 0.5 mL, 1.0 mL, 2.0 mL, 3.0 mL, 5.0 mL, 10.0 mL, 25.0 mL.

3.3.9 Short-necked funnel: 6 cm in diameter.

3.3.10 Filter paper: 11 cm, quantitative, fast.

3.3.11 Atomic absorption spectrometer.

3.4 Specimen preparation

According to GB/ T 14699.1, collect about 2 kg of representative feed raw materials (including leather powder for feed, hydrolyzed leather powder), mineral element premix, compound premix, concentrate and compound feed. Use quartering method to reduce it to about 250 g. Grind and pass through the 1 mm hole sieve. Mix well. Put in an airtight container. In order to prevent the specimen from deteriorating, it should be stored at low temperature for later use.

3.5 Analysis steps

3.5.1 Preparation of specimen solution

Weigh 0.1 g ~ 10.0 g of specimen (accurate to 0.0001 g). Place in the 60 mL porcelain crucible. Perform complete carbonization on the electric furnace. Place in the muffle furnace. Starting from room temperature, gradually increase the temperature to 600°C for 5 h, until the specimen is white or off-white without carbon particles.

Take out after cooling. Use 5 mL of nitric acid (3.2.3) to dissolve. Filter to a 50 mL volumetric flask. Use pure water to wash repeatedly the crucible and the filter paper. The washing solution is put into the volumetric flask. Then use pure water to set the volume constant. Mix it well as the specimen solution. At the same time, prepare the reagent blank solution.

3.5.2 Determination

3.5.2.1 Determination conditions

Adjust to the best state according to the performance of each instrument.

3.5.2.1.1 Flame method

Light source: Cr hollow cathode lamp.

Wavelength: 359.3 nm.

Lamp current: 7.5 mA.

Slit width: 1.30 nm.

Combustion head height: 7.5 mm.

Flame: air-acetylene.

The calculation result is the arithmetic mean value of two parallel samples of the same specimen, accurate to two decimal places.

3.7 Repeatability

The same analyst performs two measurements on the same specimen simultaneously or in rapid succession. The relative deviation of the obtained results:

- When the chromium content is less than 10 mg/kg, the relative deviation shall not exceed 20%.
- When the chromium content is greater than or equal to 10 mg/kg, the relative deviation shall not exceed 10%.

4 Method 2: Spectrophotometry

4.1 Principle

Decompose the sample by dry ashing. Chromium ions in it are oxidized to hexavalent chromium ions in alkaline potassium permanganate solution. Adjust the solution acidic again. Make hexavalent chromium ion (Cr^{6+}) and diphenylcarbazide $[(\text{C}_6\text{H}_5)_2(\text{NH})_4\text{CO}]$ to generate rose red chromium compound. Perform colorimetric measurement. Calculate the chromium content.

4.2 Reagents and solutions

Unless otherwise specified, the reagents used in this Method 2 are analytically pure; the water is distilled water or water of corresponding purity, which conforms to the provisions of GB/T 6682 for grade three water.

4.2.1 Sulfuric acid solution: $c(\text{H}_2\text{SO}_4) = 0.5 \text{ mol/L}$. Measure 28 mL of concentrated sulfuric acid. Add it into water slowly. Add water to dilute to 1000 mL.

4.2.2 Potassium permanganate solution: 20 g/L. Weigh 2 g of potassium permanganate. Dissolve in water. Add water to dilute to 100 mL.

4.2.3 Sulfuric acid solution: $V(\text{concentrated sulfuric acid}) + V(\text{distilled water}) = 1+6$. Measure 100 mL of concentrated sulfuric acid. Slowly add into 600 mL of water. Add 1 drop of 20 g/L potassium permanganate solution (4.2.2). Make the solution appear pink.

4.2.4 Sodium hydroxide solution: $c(\text{NaOH}) = 4 \text{ mol/L}$. Weigh 32 g of sodium hydroxide. Dissolve in water. Add water to dilute to 200 mL.

4.2.5 Diphenylcarbazide solution: 5 g/L. Weigh 0.5 g of diphenylcarbazide. Dissolve in 100 mL of acetone.

Weigh 1.0 g ~ 1.5 g of specimen (accurate to 0.0001 g). Place in a 60 mL porcelain crucible. After the carbonization in the temperature-controlled electric furnace is complete, place it in the muffle furnace. Starting from room temperature, gradually increase the temperature to 600°C for 5 h until the specimen is white or off-white without carbon particles.

Take out after cooling. Add 5 mL of sulfuric acid solution (4.2.1). boil slightly on the electric stove. Transfer all the contents into a 150 mL Erlenmeyer flask. Wash the crucible repeatedly with hot water 3 to 4 times. The washing solution is put into the Erlenmeyer flask. Add 1.5 mL of sodium hydroxide solution (4.2.4). Then add 2 drops of potassium permanganate solution (4.2.2). Add water to make the total volume in the flask about 60 mL ~ 70 mL. Shake well. The solution appears fuchsia. Heat to boil on the electric stove for 20 min (during the boiling process, if the fuchsia color fades, potassium permanganate solution should be added in time to keep the solution fuchsia). Then along the wall, add 3 mL of ethanol (4.2.6). Shake well. Filter it while it is hot. Place the filtrate in a 100 mL volumetric flask. Use a small amount of hot water to wash the triangular flask and filter paper 3 to 4 times. The washing solution is put into the volumetric flask. This filtrate shall be the specimen solution and reserved for future use.

4.5.2 Standard curve drawing

Pipette 0.0 mL, 5.0 mL, 10.0 mL, 15.0 mL, 20.0 mL, 25.0 mL, 30.0 mL of chromium standard solution (4.2.8). Respectively place them in 100 mL volumetric flasks. Add appropriate amount of water to dilute. Add 4 mL of sulfuric acid solution (4.2.3) and 2.0 mL of diphenylcarbazide solution (4.2.5) in sequence. Use water to dilute to the scale. Shake well. Place still for 30 min. Use a blank solution as reference. Use a 10 mm cuvette. Use the spectrophotometer to measure the absorbance at the wavelength of 540 nm. Draw a standard curve with the absorbance as the ordinate and the concentration of the chromium standard solution as the abscissa.

4.5.3 Specimen determination

In a 100 mL volumetric flask containing the specimen solution (4.5.1), add 4 mL of sulfuric acid solution (4.2.3) and 2.0 mL of diphenylcarbazide solution (4.2.5) sequentially. Use water to dilute to the scale. Shake well. Place still for 30 min. Measure the absorbance according to 4.5.2. Calculate the chromium content in the specimen solution.

4.6 Determination result

The chromium content in feeds, expressed as mass fraction milligrams per kilogram (mg/kg), is calculated according to formula (3):

$$X = \frac{c \times 100}{m} \dots\dots\dots (3)$$

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