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Determination of calcium in feeds

饲料中钙的测定

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Determination of calcium in feeds

1 Scope

This Standard specifies the potassium permanganate method and disodium edetate complexometric titration method for the determination of calcium content in feeds.

This Standard applies to the determination of calcium in feed raw materials, compound feeds, concentrated feeds, concentrate supplements and additive premixed feeds.

The detection limit of this Standard method is 0.015%. The limit of quantitation is 0.05%.

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 601, *Chemical reagent - Preparations of standard volumetric solutions*

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

GB/T 14699.1, *Feeding stuffs - Sampling*

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

3 Potassium permanganate method

3.1 Principle

Destroy the organic matter in the specimen. Calcium turns into water-soluble ions. Use ammonium oxalate to perform quantitative precipitation. Use potassium permanganate method to indirectly determine the calcium content.

3.2 Reagents or materials

Unless otherwise specified, analytically-pure reagents and grade 3 water specified in GB/T 6682 are used in this Standard.

3.2.1 Concentrated nitric acid.

3.2.2 Perchloric acid: 70%~72%.

3.5 Analysis steps

3.5.1 Specimen extraction

3.5.1.1 Dry method

Weigh 0.5g~5g of the specimen into the crucible, accurate to 0.0001g. Be careful with charring on the electric stove. Then put it into a high-temperature furnace. Burn at 550°C for 3h. Add 10mL of hydrochloric acid solution (3.2.3) and a few drops of concentrated nitric acid (3.2.1) into the crucible. Boil carefully. Transfer this solution into a 100mL volumetric flask. Cool to room temperature. Use water to dilute to the scale mark. Shake well. It shall be the specimen decomposition solution.

3.5.1.2 Wet method

Weigh 0.5g~5g of the sample into a 250mL Kjeldahl flask, accurate to 0.0002g. Add 10mL of concentrated nitric acid (3.2.1). Boil over low heat until the yellow smoke of nitrogen dioxide escapes. Add 10mL of perchloric acid (3.2.2) after cooling. Boil carefully until the solution is colorless. Do not evaporate. Add 50mL of water after cooling. Boil to expel nitrogen dioxide. Transfer to a 100mL volumetric flask after cooling. Use water to set volume constant to the scale mark. Shake well. IT shall be the specimen decomposition solution.

WARNING -- If the solution turns black during heating and boiling, remove it immediately. Add perchloric acid after cooling. Boil carefully until the solution is colorless. After adding perchloric acid, the solution must not evaporate to dryness. It may explode if evaporated to dryness.

3.5.2 Determination

Accurately pipette 10mL~20mL of the specimen decomposition solution (calcium content is about 20mg) into a 200mL beaker. Add 100mL of water, 2 drops of methyl red indicator (3.2.9). Add ammonia solution (3.2.5) dropwise until the solution turns orange. If the drop is excessive, add hydrochloric acid solution (3.2.3) to adjust to orange. Add 2 more drops to make it pink (pH is 2.5~3.0). Boil carefully. Slowly heat 10mL of ammonium oxalate solution (3.2.7). Keep stirring. If the solution turns orange, add hydrochloric acid solution (3.2.3) to make it red. Boil for 2min~3min. Leave overnight to age the precipitate (or heat on a water bath for 2h).

Use quantitative filter paper to filter. Wash the precipitate with ammonia solution (3.2.6) for 6~8 times until there is no oxalate ion. Add a few milliliters of filtrate and a few drops of sulfuric acid solution (3.2.4). Heat to 80°C. Add 1 drop of potassium permanganate standard solution (3.2.8). It is reddish and does not fade in 30s.

Transfer the precipitate and filter paper to the original beaker. Add 10mL of sulfuric acid solution (3.2.4), 50mL of water. Heat to 75°C~80°C. Use potassium permanganate standard solution (3.2.8) to titrate. When the solution is pink and does not fade in 30s,

When the calcium content is less than 1%, the absolute difference of two independent measurement results obtained under repeatability conditions shall not be greater than 18% of the arithmetic mean value of these two determinations.

4 Disodium ethylenediaminetetraacetic acid complexometric titration method

4.1 Principle

Destroy the organic matter in the specimen. Calcium turns into water-soluble ions. Use triethanolamine, ethylenediamine, hydroxylamine hydrochloride and starch solution to eliminate the influence of interfering ions. Calcein is the indicator in alkaline solution. Use disodium edetate standard titration solution to complexly titrate calcium. Calcium content can be quickly determined.

4.2 Reagents or materials

Unless otherwise specified, analytically-pure reagents AND grade 3 water specified in GB/T 6682 are used in this Standard.

4.2.1 Hydroxylamine hydrochloride.

4.2.2 Triethanolamine.

4.2.3 Ethylenediamine.

4.2.4 Hydrochloric acid solution (1+3).

4.2.5 Potassium hydroxide solution (200g/L): Weigh 20g of potassium hydroxide and dissolve it in 100mL of water.

4.2.6 Starch solution (10g/L): Weigh 1g of soluble starch in a 200mL beaker. Add 5mL of water to wet. Add 95mL of boiling water and stir. Boil. Cool for use (prepare when required).

4.2.7 Malachite green solution (1g/L).

4.2.8 Calcein-methyl thymol blue indicator: Finely grind and mix 0.10g of calcein, 0.10g of methylthymol blue, 0.03g of thymolphthalein, 5g of potassium chloride. Store in a grinding bottle for later use.

4.2.9 Calcium standard solution (0.0010g/mL): Weigh 2.4974g of reference calcium carbonate that have been dried at 105°C~110°C for 3h. Dissolve in 40mL of hydrochloric acid solution (11.4). Heat to remove carbon dioxide. Cool. Use water to transfer to a 1000mL volumetric flask. Set volume to the scale mark.

4.2.10 Disodium ethylenediaminetetraacetic acid (EDTA) standard titration solution: Weigh 3.8g of EDTA in a 200mL beaker. Add 200mL of water. Heat to dissolve and cool. Transfer to a 1000mL volumetric flask. Use water to set volume to the scale mark.

4.2.10.1 Calibration of EDTA standard titration solution: Accurately pipette 10.0mL of calcium standard solution (4.2.9). Titrate according to the specimen determination method.

4.2.10.2 The titer of EDTA titration solution to calcium is calculated by formula (2):

$$T = \frac{\rho \times V}{V_0} \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (2)$$

Where,

T - The titer of EDTA standard titration solution to calcium, in grams per milliliter (g/mL);

ρ - The mass concentration of the calcium standard solution, in grams per milliliter (g/mL);

V - The volume of the calcium standard solution taken, in milliliters (mL);

V_0 - The consumption volume of EDTA standard titration solution, in milliliters (mL).

The results obtained shall be expressed to 0.0001g/mL.

4.3 Instruments and equipment

Instruments and equipment are the same as 3.3.

4.4 Analysis steps

4.4.1 Specimen extraction

Specimen extraction is the same as 3.5.1.

4.4.2 Determination

Accurately pipette 5mL~25mL of specimen decomposition solution (calcium content is 2mg~25mg). Add 50mL of water. Add 10mL of starch solution (4.2.6), 2mL of triethanolamine (4.2.2), 1mL of ethylenediamine (4.2.3), 1 drop of malachite green solution (4.2.7). Add potassium hydroxide solution (4.2.5) dropwise until colorless. Excess by 10mL. Add 0.1g of hydroxylamine hydrochloride (4.2.1) (shake well after adding each reagent). Add a little calcein-methyl thymol blue indicator (4.2.8). Immediately titrate with disodium ethylenediaminetetraacetic acid (EDTA) standard titration solution (4.2.10) under the black background, until the green fluorescence disappears, and purple appears. When it does not turn back for 30s, it shall be the end

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