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Determination of crude protein in feeds - Kjeldahl method

饲料中粗蛋白的测定 凯氏定氮法

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Determination of crude protein in feeds - Kjeldahl method

1 Scope

This Standard specifies the Kjeldahl method for the determination of crude protein in feeds.

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

2 Normative references

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

GB/T 601, Chemical reagent - Preparations of reference titration 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 Principle

The sample is digested by sulfuric acid under the action of the catalyst, and the nitrogen-containing compound is converted into ammonium sulfate. Add alkali to distill to make ammonia escape; absorb with boric acid; then, use hydrochloric acid reference titration solution for titration; measure the nitrogen content; multiply by 6.25, to calculate the crude protein content.

4 Reagents or materials

Unless otherwise stated, only use analytical reagents.

4.1 Water: GB/T 6682, grade 3.

4.2 Boric acid: chemical pure.

5.6 Azotometer: various types of semi-automatic and full-automatic azotometers manufactured according to the Kjeldahl principle.

6 Sample

Take representative feed samples according to GB/T 14699.1; use the quartering method to reduce the sampling. Prepare the samples according to GB/T 20195; crush them; pass them all through a 0.42 mm test sieve; mix them evenly; put them into airtight containers for later use.

7 Test steps

7.1 Semi-micro method (arbitration method)

7.1.1 Sample boiling and sterilization

7.1.1.1 Kjeldahl flask boiling and sterilization

Perform two tests in parallel. Weigh 0.5 g ~ 2 g of the sample (nitrogen content 5 mg ~ 80 mg, accurate to 0.000 1 g); place it in a Kjeldahl flask; add 6.4 g of mixed catalyst; mix well; add 12 mL of sulfuric acid and 2 glass beads; place the Kjeldahl flask on an electric furnace and start heating at about 200 °C; after the sample is coked and the foam disappears, increase the temperature to about 400 °C, until it turns transparent blue-green; then, continue heating for at least 2 hours. Take it out and cool to room temperature.

7.1.1.2 Boiling and sterilization of the boiling and sterilization tube

Perform two tests in parallel. Weigh 0.5 g ~ 2 g of the sample (nitrogen content 5 mg ~ 80 mg, accurate to 0.000 1 g); put it into the boiling and sterilization tube; add 2 pieces of Kjeldahl nitrogen catalyst tablets or 6.4 g of mixed catalyst, 12 mL of sulfuric acid; boil and sterilize on the stove at 420 °C for 1 h. Take it out and cool to room temperature.

7.1.2 Distillation of ammonia

After the boiled-and-sterilized solution of the sample is cooled, add 20 mL of water; transfer it to a 100 mL volumetric flask; after cooling, use water to dilute to the mark; shake well; use it as the sample decomposition solution. Immerse the end of the condenser tube of the semi-micro distillation device into the Erlenmeyer flask filled with 20 mL of boric acid absorption solution I (4.8) and 2 drops of mixed indicator (4.14). A few drops of methyl red indicator (4.12) and a few drops of sulfuric acid shall be added to the water in the steam generator; the liquid shall be kept orange-red during the distillation process, otherwise, sulfuric acid shall be added. Accurately pipette 10 mL ~ 20 mL of the sample decomposition solution into the reaction chamber of the distillation device; use a small amount of water to rinse the injection port; plug the inlet

glass plug; add 10 mL of sodium hydroxide solution (4.10); carefully lift the glass plug to let it flow into the reaction chamber; plug the glass stopper and seal it with water at the entrance to prevent air leakage. Distill for 4 min and lower the Erlenmeyer flask so that the end of the condenser tube is away from the absorption liquid level; then, distill for 1 min until the pH of the effluent is neutral. Use water to rinse the end of the condenser tube; when all the washing liquid flows into the Erlenmeyer flask, stop the distillation.

7.1.3 Titration

Immediately use 0.1 mol/L or 0.02 mol/L of hydrochloric acid standard titration solution (4.11) to titrate the absorption solution after distillation in 7.1.2. The solution changes from blue-green to gray-red as the titration end point.

7.2 Full volume method

7.2.1 Sample boiling and sterilization

Follow the steps in 7.1.1.

7.2.2 Distillation of ammonia

7.2.2.1 After the boiled-and-sterilized solution of the sample is cooled, add 60 mL ~ 100 mL of distilled water; shake well; cool down. Immerse the end of the condenser tube of the distillation device into the Erlenmeyer flask filled with 25 mL of boric acid absorption solution I (4.8) and 2 drops of mixed indicator (4.14). Then, carefully add 50 mL of sodium hydroxide solution (4.10) to the Kjeldahl flask; shake well and then heat and distill, until the distillate volume is about 100 mL. Lower the Erlenmeyer flask, so that the end of the condenser tube leaves the liquid surface; continue distillation for 1 min ~ 2 min until the pH value of the effluent is neutral. Use water to rinse the end of the condenser tube; when all the washing liquid flows into the Erlenmeyer flask, stop the distillation.

7.2.2.2 When using a semi-automatic Kjeldahl nitrogen analyzer, insert the boiling and sterilization tube with the boiled-and-sterilized solution into the distillation device; use 25 mL of boric acid absorption solution I (4.8) as the absorption solution; add 2 drops of the mixed indicator (4.14); immerse the end of the condenser tube of the distillation device in the Erlenmeyer flask containing the absorption liquid; then, add 50 mL of sodium hydroxide solution (4.10) to the boiling and sterilization tube for distillation, until the pH of the effluent is neutral. Distillation time is appropriate when the volume of the absorption liquid reaches about 100 mL. Lower the conical flask; use water to rinse the end of the condenser tube. All the washing liquid must flow into the conical flask.

7.2.2.3 When using an automatic Kjeldahl nitrogen analyzer, carry out the measurement according to the instrument operation manual.

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