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Determination of cadmium in feeds

饲料中镉的测定

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Determination of cadmium in feeds

1 Scope

This document describes flame atomic absorption spectrometry and graphite furnace atomic absorption spectrometry, for the determination of cadmium in feed.

This document applies to the determination of cadmium, in compound feeds, concentrated feeds, concentrate supplements, additive premixed feeds, feed additives, feed ingredients.

In this document, when the sampling amount is 5 g and the constant volume is 50 mL, the detection limit of flame atomic absorption spectrometry is 0.08 mg/kg AND the quantification limit is 0.20 mg/kg. When the sampling amount is 0.5 g and the constant volume is 25 mL, the detection limit of graphite furnace atomic absorption spectrometry is 0.002 mg/kg AND the quantification limit is 0.05 mg/kg.

2 Normative references

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, for dated references, only the version corresponding to the date applies to this document; for undated references, the latest version (including all amendments) is applicable to this document.

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

GB 6819-2004 Dissolved acetylene

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

3 Terms and definitions

There are no terms and definitions, that need to be defined in this document.

4 Principles

After dry ashing or wet digestion (microwave digestion), or hydrochloric acid dissolution, the specimen is introduced into the flame or graphite furnace atomizer of atomic absorption spectrophotometer, to measure the absorbance value at a wavelength of 228.8 nm. Within a certain concentration range, the cadmium concentration is proportional to its absorbance value; the standard curve is used for calibration and

quantitative.

5 Reagents or materials

Warning: When using various strong acids, it shall be carried out in a fume hood; when using perchloric acid for digestion, be careful not to burn it dry, to prevent explosion.

Unless otherwise specified, only analytical grade reagents are used.

5.1 Water: GB/T 6682, grade 1.

5.2 Nitric acid: Excellent grade pure.

5.3 Hydrochloric acid: Excellent grade pure.

5.4 Perchloric acid: Excellent grade pure.

5.5 Hydrochloric acid solution (6 mol/L): Dilute 500 mL of hydrochloric acid (5.3) to 1000 mL. Mix well.

5.6 Nitric acid solution: Dilute 10 mL of nitric acid (5.2) to 1000 mL. Mix well.

5.7 Nitric acid solution (6 mol/L): Dilute 43 mL of nitric acid (5.2) to 100 mL. Mix well.

5.8 Palladium nitrate solution (2 mg/mL): Weigh 0.20 g of palladium nitrate. Use water to dissolve it. Dilute it to 100 mL. Mix well.

5.9 Ammonium dihydrogen phosphate solution (10 mg/mL): Weigh 1.00 g of ammonium dihydrogen phosphate. Use water to dissolve it. Dilute it to 100 mL. Mix well.

5.10 Cadmium standard stock solution (1 mg/mL): Accurately weigh 1.0000 g of metal cadmium (certified reference material, purity 99.99%), into a 250 mL conical flask. Add 10 mL of nitric acid solution (5.7). Heat on an electric hot plate, to dissolve all. Then, transfer to a 1000 mL volumetric flask. Add water to the mark. Mix well. Store it in polyethylene bottles at 4 °C; the validity period is 6 months. OR otherwise purchase certified standard solutions.

5.11 Cadmium standard intermediate solution I (10 μg/mL): Accurately pipette 1 mL of cadmium standard stock solution (5.10), into a 100 mL volumetric flask. Use nitric acid solution (5.6), to dilute it to the mark. Mix well. The valid period is 1 month.

5.12 Cadmium standard intermediate solution II (100 μg/L): Accurately pipette 1 mL of cadmium standard intermediate solution I (5.11), into a 100 mL volumetric flask. Use nitric acid solution (5.6), to dilute it to the mark. Mix well. Prepare it before use.

8 Test procedures

8.1 Preparation of specimen solution

8.1.1 Dry ashing method

It is suitable for compound feed, concentrated feed, concentrate supplement, pre-mixed feed containing organic matter additives, feed additives, feed raw materials.

Do two tests in parallel. Weigh 5 g of the specimen (accurate to 0.01 g), in a porcelain crucible. Place it on an adjustable electric hot plate or an adjustable electric furnace, to slowly heat and carbonize it, until it is smokeless. Then transfer it to a 500 °C muffle furnace, for ashing for 5 hours, until the specimen turns white or grey-white, without carbon particles. If a small amount of carbon particles is found, nitric acid solution (5.7) can be added to wet the residue. Move the porcelain crucible to an adjustable electric hot plate or adjustable electric furnace, to dry on a small fire. Then move it into a muffle furnace, to continue ashing, until the specimen is white or grey-white, without carbon particles.

Take out the crucible. Cool to room temperature. Absorb 5 mL of hydrochloric acid solution (5.5). Add it dropwise to the porcelain crucible. Turn it while adding, until no bubbles overflow from the solution. Add the remaining hydrochloric acid solution. Then add 5 mL of nitric acid solution (5.7). Move the porcelain crucible to the adjustable electric heating plate or adjustable electric furnace. Slowly heat it, until the digestion solution reaches 2 mL ~ 3 mL (be careful to prevent splashing). Remove it. Cool it to room temperature. Transfer the digestion solution into a 50 mL volumetric flask. Use a small volume of water, to rinse the wall of the porcelain crucible several times. Incorporate it into the volumetric flask. Add water to the mark. Shake well. Filter it. Prepare for use. At the same time, do a blank test.

8.1.2 Wet digestion method

It is suitable for additive premixed feed which contain more organic matter, feed additives, feed raw materials.

Do two tests in parallel. Weigh 1 g of the specimen (accurate to 0.0001 g) into the digestion tube. Add a small amount of water to wet it. Add 10 mL of nitric acid (5.2). Put it in a fume hood. Let it stand for 2 hours. Then add 5 mL of perchloric acid (5.4). Use small fire, to heat it on an adjustable electric heating plate or an adjustable electric furnace, which is below 250 °C, for digestion, until the digestion solution emits white smoke. Remove it and cool it. Transfer the digestion solution into a 50 mL volumetric flask. Use a small volume of water, to rinse the digestion tube several times. Incorporate it into the volumetric flask. Add water to the mark. Shake well. Filter it. Prepare for use. At the same time, do a blank test.

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