International Standard 6490/1

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Animal feeding stuffs — Determination of calcium content — Part 1 : Titrimetric method

Aliments des animaux — Détermination de la teneur en calcium — Partie 1 : Méthode titrimétrique

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Animal feeding stuffs — Determination of calcium content — Part 1 : Titrimetric method

1 Scope and field of application

This part of ISO 6490 specifies a titrimetric method for the determination of the calcium content of animal feeding stuffs.

The method is applicable to all animal feeding stuffs having calcium contents greater than 1 g/kg.

2 References

ISO 6490/2, *Animal feeding stuffs — Determination of calcium content — Part 2: Atomic absorption spectrometric method.*

ISO 6497, *Animal feeding stuffs — Sampling.* [1)]

ISO 6498, *Animal feeding stuffs — Preparation of test samples.*

3 Principle

Ashing of a test portion, treatment of the ash with hydrochloric acid and precipitation of the calcium as calcium oxalate. Dissolution of the precipitate in sulfuric acid and titration with standard volumetric potassium permanganate solution of the oxalic acid formed.

4 Reagents

All reagents shall be of recognized analytical quality and the water used shall be distilled water or water of at least equivalent purity.

4.1 Hydrochloric acid, approximately 30 % (m/m) (ϱ_{20} = 1,15 g/ml).

4.2 Nitric acid, concentrated (ϱ_{20} = 1,40 g/ml).

4.3 Sulfuric acid, approximately 20 % (m/m) (ϱ_{20} = 1,13 g/ml).

4.4 Ammonia solution, approximately 33 % (m/m) (ϱ_{20} = 0,89 g/ml).

4.5 Ammonium oxalate, cold saturated solution.

4.6 Citric acid monohydrate, 300 g/l solution.

4.7 Ammonium chloride, 50 g/l solution.

4.8 Bromocresol green, 0,4 g/l solution.

4.9 Potassium permanganate, standard volumetric solution, c (1/5 $KMnO_4$) = 0,1 mol/l.

5 Apparatus

Usual laboratory apparatus, and in particular

5.1 Electric muffle furnace, with air circulation, capable of being maintained at 550 ± 20 °C.

5.2 Incineration dish, made of platinum, silica or porcelain.

5.3 Sintered glass filter crucible, of porosity grade P 16 (pore size 10 to 16 µm).

5.4 Boiling water-bath.

5.5 Beakers, of capacity 250 ml.

5.6 Volumetric flask, of capacity 250 ml.

5.7 Analytical balance.

6 Sampling

Take the laboratory sample as specified in ISO 6497.

7 Procedure

7.1 Preparation of the test sample

Prepare the test sample in accordance with ISO 6498.

7.2 Test portion

Weigh, to the nearest 1 mg, approximately 5 g of the test sample (or more if necessary), into the incineration dish (5.2).

1) At present at the stage of draft.

7.3 Determination

7.3.1 Ash the test portion in the electric muffle furnace (5.1), maintained at 550 ± 20 °C, until all organic matter has been destroyed (usually 4 h is sufficient). If some organic matter remains (black particles), add a few drops of nitric acid (4.2), dry on a hotplate and ash again in the muffle furnace at 550 ± 20 °C for 30 min. Repeat this until all organic matter has been destroyed. Transfer the ash to a 250 ml beaker (5.5).

7.3.2 Add 40 ml of the hydrochloric acid (4.1), 60 ml of water, and a few drops of the nitric acid (4.2). Bring to the boil and boil for 30 min. Cool and transfer the solution to a 250 ml one-mark volumetric flask (5.6). Rinse, dilute to the mark with water, mix and filter to give the test solution.

7.3.3 By means of a pipette, transfer an aliquot portion of the test solution (7.3.2), containing 10 to 40 mg of calcium, according to the expected calcium content, to a 250 ml beaker (5.5). Add 1 ml of the citric acid solution (4.6) and 5 ml of the ammonium chloride solution (4.7). Dilute to approximately 100 ml with water. Bring to the boil, add 10 drops of the bromocresol green solution (4.8) and 30 ml of a warm solution of the ammonium oxalate (4.5). If a precipitate forms, dissolve it by adding a few drops of the hydrochloric acid (4.1).

Neutralize very slowly with the ammonia solution (4.4), stirring continuously, until a pH of 4,4 to 4,6 is reached (i.e. when the indicator changes colour). Place the beaker on a boiling water-bath (5.4) and leave for 30 min to allow the precipitate which has formed to settle. Remove the beaker from the water bath. Leave for 1 h and filter through the filter crucible (5.3).

Wash the beaker and the crucible with water until the excess ammonium oxalate is completely removed as shown by the absence of chloride in the washing water.

Place the crucible in a 250 ml beaker (5.5) or wide-mouth flask. Add 80 ml of the sulfuric acid (4.3) and heat to 70 to 80 °C to dissolve the precipitate.

7.3.4 Titrate the hot solution with the standard volumetric potassium permanganate solution (4.9), until a pink colour, persisting for 1 min, is obtained.

7.4 Number of determinations

Carry out two determinations on test portions taken from the same test sample.

8 Expression of results

8.1 Method of calculation and formula

The calcium content, expressed in grams per kilogram of sample, is equal to

$$\frac{20{,}04 \times V \times c}{m} \times \frac{250}{V'}$$

where

V is the volume, in millilitres, of the standard volumetric potassium permanganate solution used for the titration;

c is the exact concentration, in moles per litre, of the standard volumetric potassium permanganate solution;

m is the mass, in grams, of the test portion;

V' is the volume, in millilitres, of the aliquot portion taken in 7.3.3.

Take as the result the arithmetic mean of the values obtained in the two determinations (see 7.4), provided that the requirement for repeatability (see 8.2) is satisfied.

Report the result to the nearest 1 g/kg.

8.2 Repeatability

The difference between the values obtained in the two determinations, carried out simultaneously or in rapid succession by the same analyst, shall not exceed

1 g/kg (absolute) for calcium contents less than 50 g/kg;

2 % (relative) of the mean for calcium contents of 50 g/kg or more.

9 Notes on procedure

9.1 For the determination of very low calcium contents use the method specified in ISO 6490/2.

9.2 If the sample consists exclusively of mineral substances, dissolve it in hydrochloric acid without ashing.

For products such as aluminocalcium phosphates, which are difficult to dissolve in acids, mix the test portion in a platinum dish with five times its mass of a mixture comprising equal parts of potassium carbonate and sodium carbonate. Heat carefully until the mixture is completely molten. After cooling, dissolve in the hydrochloric acid.

9.3 If the magnesium content of the sample is likely to exceed the calcium content, or in cases of doubt, precipitate the calcium oxalate a second time.

10 Test report

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

The test report shall include all the details required for the complete identification of the sample.