International Standard 6495

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Animal feeding stuffs — Determination of water-soluble chlorides content

Aliments des animaux — Détermination de la teneur en chlorures solubles dans l'eau

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Foreword

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It has been approved by the member bodies of the following countries :

Australia	India	Romania
Canada	Israel	South Africa, Rep. of
Chile	Kenya	Spain
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Czechoslovakia	Malaysia	Turkey
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Ethiopia	Philippines	USSR
France	Poland	Yugoslavia
Hungary	Portugal	

No member body expressed disapproval of the document.

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Animal feeding stuffs — Determination of water-soluble chlorides content

1 Scope and field of application

This International Standard specifies a method for the determination of the water-soluble chlorides content, expressed as sodium chloride, of animal feeding stuffs.

It is applicable to all animal feeding stuffs.

2 Principle

Dissolution in water of the chlorides present in a test portion. Clarification of the solution if the product contains organic matter. Slight acidification with nitric acid and precipitation of the chlorides as silver chloride by means of standard volumetric silver nitrate solution. Titration of the excess silver nitrate with standard volumetric ammonium or potassium thiocyanate solution.

3 Reagents

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

3.1 Acetone.

3.2 *n*-**Hexane.**

3.3 Nitric acid, ϱ_{20} 1,38 g/ml.

3.4 Activated carbon, free from chlorides and not capable of adsorbing chlorides.

3.5 Ammonium iron(III) sulphate, saturated solution.

3.6 Carrez I solution.

Dissolve in water, 21,9 g of zinc acetate dihydrate [$Zn(CH_3COO)_2.2H_2O$] and add 3 ml of glacial acetic acid. Make up to 100 ml with water.

3.7 Carrez II solution.

Dissolve in water, 10,6 g of potassium hexacyanoferrate(II) [potassium ferrocyanide] trihydrate [$K_4Fe(CN)_6.3H_2O$]. Make up to 100 ml with water.

3.8 Ammonium or potassium thiocyanate, standard volumetric solution, $c(NH_4SCN)$ or $c(KSCN)$ = 0,1 mol/l [1].

3.9 Silver nitrate, standard volumetric solution, $c(AgNO_3)$ = 0,1 mol/l[1].

4 Apparatus

Usual laboratory equipment, and in particular

4.1 Rotary shaker, having a frequency of rotation of approximately 35 to 40 min^{-1}.

4.2 One-mark volumetric flasks, of capacities 200 and 500 ml.

4.3 Pipettes, of appropriate capacity.

4.4 Burettes.

4.5 Analytical balance.

1) Hitherto expressed as "0,1 N standard volumetric solution".

5 Procedure

5.1 Test portion and preparation of the test solution

According to the nature of the sample, take a test portion and prepare the test solution as specified in 5.1.1, 5.1.2 or 5.1.3.

5.1.1 Samples free from organic matter

Weigh, to the nearest 1 mg, a test portion of not more than 10 g presumed to contain not more than 3 g of chlorides. Place with 400 ml of water in a 500 ml volumetric flask (4.2) at approximately 20 °C.

Mix for 30 min in the rotary shaker (4.1), make up to the mark with water, mix and filter.

5.1.2 Samples containing organic matter (excluding the products listed in 5.1.3)

Weigh, to the nearest 1 mg, a test portion of approximately 5 g and place it with 1 g of the activated carbon (3.4) in a 500 ml volumetric flask (4.2). Add 400 ml of water at approximately 20 °C, and 5 ml of the Carrez I solution (3.6), stir, then add 5 ml of the Carrez II solution (3.7). Mix for 30 min in the rotary shaker (4.1), make up to the mark with water, mix and filter.

5.1.3 Cooked feeding stuffs, flax cakes and flour, products rich in flax flour and other products rich in mucilage or in colloidal substances (for example dextrinated starch)

Proceed as specified in 5.1.2 but do not filter. Decant (if necessary, centrifuge), remove 100 ml of the supernatant liquid and transfer to a 200 ml volumetric flask (4.2). Mix with acetone (3.1), make up to the mark with the same solvent, mix and filter.

5.2 Titration

By means of a pipette (4.3), transfer to a conical flask an aliquot portion of 25 to 100 ml of the filtrate (according to the expected chlorides content) obtained as specified in 5.1.1, 5.1.2 or 5.1.3. The aliquot portion shall not contain more than 150 mg of chlorine (Cl).

Make up, if necessary, to a volume of not less than 50 ml with water, add 5 ml of the nitric acid (3.3), 2 ml of the saturated ammonium iron(III) sulphate solution (3.5) and 2 drops of the ammonium or potassium thiocyanate solution (3.8) from a burette (4.4) filled to the zero mark (the remainder of the solution is to be used afterwards for titration of the excess silver nitrate).

Add from a burette (4.4), the silver nitrate solution (3.9) until an excess of 5 ml is obtained. Shake vigorously to coagulate the precipitate. [If necessary, 5 ml of *n*-hexane (3.2) may be added to assist coagulation.] Titrate the excess silver nitrate with the ammonium or potassium thiocyanate solution (3.8) until a reddish-brown, tint persisting for at least 30 s, develops.

5.3 Blank test

Carry out a blank test in parallel with the determination, using the same procedure and the same reagents, but omitting the test portion.

5.4 Number of determinations

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

6 Expression of results

6.1 Method of calculation and formulae

6.1.1 Samples treated in accordance with 5.1.1 and 5.1.2

The water-soluble chlorides content, expressed as sodium chloride as a percentage by mass, is equal to

$$\frac{5{,}845\,[(V_1 - V_1')\,c_1 - (V_2 - V_2')\,c_2]}{m} \times \frac{500}{V}$$

6.1.2 Cooked feeding stuffs, flax cakes and flour, products rich in flax flour and other products rich in mucilage or in colloidal substances

The water-soluble chlorides content, expressed as sodium chloride as a percentage by mass, is equal to

$$\frac{5{,}845\,[(V_1 - V_1')\,c_1 - (V_2 - V_2')\,c_2]}{m} \times \frac{1\,000}{V}$$

6.1.3 In the preceding formulae (6.1.1 and 6.1.2) :

c_1 is the exact concentration of the silver nitrate solution (3.9);

c_2 is the exact concentration of the ammonium or potassium thiocyanate solution (3.8);

V is the volume, in millitres, of the aliquot portion of filtrate taken (see 5.2);

V_1 is the volume, in millitres, of the silver nitrate solution added in the determination;

V_1' is the volume, in millitres, of the silver nitrate solution added in the blank test;

V_2 is the volume, in millilitres, of the ammonium or potassium thiocyanate solution used in the determination;

V_2' is the volume, in millilitres, of the ammonium or potassium thiocyanate solution used in the blank test;

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

6.1.4 Express the result to the nearest :

0,05 % (m/m) sodium chloride for sodium chloride contents less than 1 % (m/m);

0,1 % (m/m) sodium chloride for sodium chloride contents greater than or equal to 1 % (m/m).

6.2 Repeatability

The difference between the results of two determinations carried out in rapid succession by the same analyst shall not exceed :

0,05 (absolute value) for sodium chloride contents less than 1 % (m/m);

5 % (relative value) of the mean value for sodium chloride contents greater than 1 % (m/m).

7 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 result.

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

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