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Animal feeding stuffs — Determination of water-soluble chlorides content —

Part 1: Titrimetric method

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

Partie 1: Méthode titrimétrique

(Revision of ISO 6495:1999)

ICS 65.120

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Contents

Page

Foreword iv
1 Scope 1
2 Normative references 1
3 Principle 1
4 Reagents 1
5 Apparatus 3
6 Sampling 4
7 Preparation of test sample 4
8 Procedure 4
9 Expression of results 5
10 Precision 6
11 Test report 7
Annex A (informative) **Results of interlaboratory test** 8
Bibliography 9

Foreword

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ISO 6495-1 was prepared by Technical Committee ISO/TC 34, *Food stuffs*, Subcommittee SC 10, *Animal feeding stuffs*.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

ISO 6495 consists of the following parts, under the general title *Animal feeding stuffs — Determination of water-soluble chlorides content*:

— *Part 1: Titrimetric method;*

— *Part 2: Potentiometric method.*

Animal feeding stuffs — Determination of water-soluble chlorides content —

Part 1: Titrimetric method

1 Scope

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

This method is applicable to animal feeding stuffs containing water-soluble chlorides content, expressed as sodium chloride, ≥ 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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

3 Principle

The chlorides present in a test portion are dissolved in water. The solution is clarified if the product contains organic matter. It is then slightly acidified with nitric acid and the chlorides are precipitated as silver chloride by means of standard volumetric silver nitrate solution. The excess silver nitrate is titrated with a standard volumetric ammonium thiocyanate or potassium thiocyanate solution, by Volhard's method.

4 Reagents

Use only reagents of recognized analytical grade.

4.1 Water, complying with at least grade 3 in accordance with ISO 3696.

4.2 Acetone.

4.3 n-Hexane.

4.4 Nitric acid, mass concentration ρ_{20} (HNO_3) = 1,38 g/ml.

4.5 Dilute nitric acid, volume fraction φ(HNO_3) = 2 %.

Dilute 20 ml nitric acid (4.4) to 1000 ml with water (4.1).

4.6 Potassium chromate solution, mass concentration ρ (potassium chromate solution) = 5 % in water (4.1).

4.7 Ammonium iron(III) sulfate, saturated solution. Prepare from $NH_4Fe(SO_4)_2 \cdot 12H_2O$ dissolved in water (4.1) until saturation. Approximately 125 g ammonium iron(III) sulfate per 100 ml water is required.

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

4.9 Carrez I solution.

Dissolve 10,6 g of potassium hexacyanoferrate(II) trihydrate [$K_4Fe(CN)_6 \cdot 3H_2O$] in water (4.1). Dilute to 100 ml with water (4.1).

4.10 Carrez II solution.

Dissolve 21,9 g of zinc acetate dihydrate [$Zn(CH_3COO)_2 \cdot 2H_2O$] in water (4.1),and add 3 ml of glacial acetic acid. Dilute to 100 ml with water.

4.11 Sodium chloride, standard volumetric solution, c(NaCl) = 0,1 mol/l. The molarity of the standard volumetric solution shall be known to 0,0001 mol/l.

Put about 20 g of finely pulverized sodium chloride in a thin layer on a watch glass. Dry at about 250 ºC for 1 h to 2 h. Let it cool in a desiccator, accurately weigh 5,8 g, recorded to 0,0001 g (m), and dissolve in water (4.1), dilute to the mark in a 1000 ml volumetric flask with water (4.1) and mix well.

The concentration of standard sodium chloride solution can be calculated as follows:

$$c_{NaCl} = \frac{m}{58{,}44}$$

where

$c_{NaC\,l}$ is the concentration of standard sodium chloride solution, in mol/l;

m is the mass of sodium chloride, in grams.

4.12 Silver nitrate, standard volumetric solution, c_s = 0,1 mol/l.

The molarity of the standard volumetric solution shall be known to 0,0001 mol/l, and duplicate titrations shall agree within ± 0,1 ml.

Put about 20 g of finely pulverized silver nitrate in a thin layer on watch glass. Dry at about 80 ºC for 2 h to 3 h. Let it cool in a desiccator, weigh about 17,0 g and dissolve in water (4.1), dilute to the mark in a 1000 ml volumetric flask with water (4.1) and mix well.

Pipette 20 ml of standard sodium chloride solution (4.11) into a 200 ml conical flask. Add 1 ml of potassium chromate solution (4.6) and titrate while shaking vigorously with standard silver nitrate (4.12) until a reddish brown colour persists for at least 30 s.

The concentration of standard silver nitrate solution can be calculated as follows:

$$c_s = \frac{20 \times c_{NaCl}}{V_s}$$

where

c_s is the concentration of standard silver nitrate solution (4.12), in mol/l;

c_{NaCL} is the concentration of standard sodium chloride solution (4.11), in mol/l;

V_s is the volume of standard silver nitrate solution (4.12) used for titration, in ml.

4.13 Ammonium thiocyanate or potassium thiocyanate, standard volumetric solution, c_t = 0,1 mol/l.

The molarity of standard volumetric solution shall be known to 0,0001 mol/l, and duplicate titrations shall agree within ± 0,1 ml.

Weigh 7,6 g of ammonium thiocyanate, or 9,7 g of potassium thiocyanate and dissolve in water (4.1). Dilute to the mark in a 1000 ml volumetric flask and mix well.

Pipette 20 ml standard silver nitrate (4.12) into a 200 ml conical flask. Add 10 ml of boiled nitric acid (4.5) and 2 ml of saturated ammonium iron(III)sulfate solution (4.7). Titrate while shaking vigorously with standard ammonium, or potassium thiocyanate solution until a reddish brown colour persists for at least 30 s.

The concentration of standard ammonium or potassium thiocyanate solution can be calculated as follows:

$$c_t = \frac{20 \times c_s}{V_t}$$

where

c_t is the concentration of standard ammonium or potassium thiocyanate solution (4.13), in mol/l;

c_s is the concentration of standard silver nitrate;

V_t is the volume of standard ammonium or potassium thiocyanate solution (4.13) used for titration, in ml.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Rotary shaker, operating at a rotation frequency of approximately 35 min^{-1} to 40 min^{-1}

5.2 Volumetric flasks, class A of capacities 200 ml, 500 ml, and 1000 ml

5.3 Pipettes, class A of appropriate capacities

5.4 Burettes, class A of appropriate capacities

5.5 Analytical balance, capable of weighing to 0,0001 g

5.6 Filter paper, of appropriate porosity

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 6497[1].

It is important that the laboratory received a sample which is truly representative and has not been damaged or changed during transport or storage.

7 Preparation of test sample

Prepare the test sample in accordance with ISO 6498.

If solid, grind a representative part, 100 g to 150 g of the laboratory sample (usually 500 g) so that it passes completely through a sieve with 1 mm apertures. Mix thoroughly.

8 Procedure

8.1 Preparation of test solution

8.1.1 General

If the test sample is free from organic matter, proceed in accordance with 8.1.2.

If the test sample contains organic matter, proceed in accordance with 8.1.3, unless the test sample concerns cooked feeding stuffs, flax cakes and flour, products rich in flax flour, and other products rich in mucilage or in colloidal substances (e.g. dextrinated starch). In this case proceed in accordance with 8.1.4.

8.1.2 Preparation of test solution of sample free from organic matter

Accurately weigh, to 0.0001 g, not more than 10 g of the test sample (7) containing not more than 3 g of chlorides. Transfer to a 500 ml volumetric flask (5.2) and add about 400 ml of water (4.1) at a temperature of approximately 20 °C.

Mix for 30 min in the rotary shaker (5.1), dilute to the mark with water (4.1), then mix and filter through the filter paper (5.6)

Proceed in accordance with 8.2.

8.1.3 Preparation of test solution of sample containing organic matter, excluding the products listed in 8.1.4

Accurately weigh, to 0.0001 g, approximately 5 g of the test sample (7) containing not more than 3 g of chlorides. Transfer to a 500 ml volumetric flask (5.2). Add 1 g of activated carbon (4.8), about 400 ml of water (4.1) at a temperature of approximately 20 °C, and 5 ml of Carrez I solution (4.9). Then mix and add 5 ml of Carrez II solution (4.10).

Mix for 30 min on the rotary shaker (5.1). Dilute to the mark with water (4.1), then mix and filter through the filter paper (5.6).

Proceed in accordance with 8.2.

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

Accurately weigh, to 0.0001 g, approximately 5 g of the test sample (7) containing not more than 3 g of chlorides. Transfer to a 500 ml volumetric flask (5.2). Add 1 g of activated carbon (4.8), about 400 ml of water (4.1) at a temperature of approximately 20 °C, and 5 ml of Carrez I solution (4.9). Then mix and add 5 ml of Carrez II solution (4.10).

Mix for 30 min on the rotary shaker (5.1). Dilute to the mark with water (4.1), then mix.

Decant (if necessary, centrifuge). By means of a pipette (5.3), transfer 100 ml of the supernatant to a 200 ml volumetric flask (5.2).

Dilute to the mark with acetone (4.2), then mix, bring the volume to the mark with acetone or water, remix and filter through the filter paper (5.6).

Proceed in accordance with 8.2.

8.2 Titration

By means of a pipette (5.3), transfer to a conical flask an aliquot portion of between 25 ml and 100 ml (V_a) of the filtrate. The aliquot portion shall not contain more than 150 mg of chloride.

Dilute, if necessary, to a volume of not more than 100 ml with water. Add 5 ml of nitric acid (4.4), 2 ml of saturated ammonium iron(III) sulfate solution (4.7) and 2 drops of standard ammonium or potassium thiocyanate solution (4.13) from a burette (5.4) filled to the zero mark.

Add standard silver nitrate solution (4.12) from another burette (5.4), while shaking until the reddish brown tint disappears, then add an excess of 5 ml standard silver nitrate solution (total volume of $AgNO_3$ = V_{s1}). Shake vigorously to coagulate the precipitate. If necessary, 5 ml of n-hexane (4.3) may be added to assist coagulation.

Titrate the excess standard silver nitrate solution (4.12) with standard ammonium or potassium thiocyanate solution (4.13) from the burette until a reddish brown tint persists for at least 30 s (total volume including 2 drops = V_{t1}).

8.3 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.

9 Expression of results

Calculate the water-soluble chlorides content, expressed as sodium chloride, by the equation:

$$w_{wc} = \frac{M\left[\left(V_{s1} - V_{s0}\right)c_s - \left(V_{t1} - V_{t0}\right)c_t\right]}{w} \times \frac{V_i}{V_a} \times F \times 100\%$$

where

w_{wc} is the water-soluble chlorides content, expressed as sodium chloride, as mass fraction of the test sample, in %;

M is the molar mass of sodium chloride (M = 58,44 g/mol);