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Krma - Določevanje v vodi topnih kloridov - 1. del: Titrimetrijska metoda

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

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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

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 34, *Food Products*, Subcommittee SC 10, *Animal feeding stuffs*.

This first edition of ISO 6495-1 cancels and replaces ISO 6495:1999, which has been technically revised.

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

— *Part 1: Titrimetric method*

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

Part 1: Titrimetric method

1 Scope

This part of ISO 6495 specifies a method for the determination of water-soluble chloride content, expressed as sodium chloride, of animal feeding stuffs.

This method is applicable to animal feeding stuffs containing water-soluble chloride content, expressed as sodium chloride, ≥0,05 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable to its application. 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 6497, *Animal feeding stuffs — Sampling*

ISO 6498, *Animal feeding stuffs — Guidelines for sample preparation*

3 Principle

The chloride 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 solution of silver nitrate. The excess silver nitrate is titrated with a standard volumetric solution of ammonium thiocyanate or potassium thiocyanate, 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 $\rho_{20}(HNO_3)$ = 1,38 g/ml.

4.5 Dilute nitric acid, volume fraction $\rho(HNO_3)$ = 2 %.

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

4.6 Potassium chromate solution, mass concentration $\rho(K_2CrO_4)$ = 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 chloride and not capable of adsorbing chloride.

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 (4.1).

4.11 Sodium chloride, standard volumetric solution, c(NaCl) = 0,1 mol/l. The molarity of the standard volumetric solution shall be known to 0,000 1 mol/l.

Put about 20 g of finely pulverized sodium chloride in a thin layer on watch glass. Dry at about 250 °C for 1 h to 2 h. Let it cool in a desiccator, accurately weigh 5,8 g, recorded to 0,000 1 g (m), and dissolve in water (4.1). Dilute to the mark in a 1 000 ml volumetric flask with water (4.1) and mix well.

The concentration of standard sodium chloride solution can be calculated using Formula (1):

$$c_{\mathrm{NaCl}} = \frac{m}{58,44} \tag{1}$$

where

c_{NaCl} 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 should be known to 0,000 1 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 °C 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 1 000 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 using Formula (2):

$$c_s = \frac{20 \times c_{NaCl}}{V_s} \tag{2}$$

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 0,000 1 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 1 000 ml volumetric flask and mix well.

Pipette 20 ml standard silver nitrate (4.12) into a 200 ml conical flask. Add 10 ml of dilute 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 using Formula (3):

$$c_t = \frac{20 \times c_s}{V_t} \tag{3}$$

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 1 000 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 0,000 1 g.

5.6 Filter paper, of appropriate porosity.