



SLOVENSKI STANDARD

oSIST ISO 6495-1:2018

01-september-2018

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

Animal feeding stuffs -- Determination of water-soluble chlorides content -- Part 1:
Titrimetric method

iTeh STANDARD PREVIEW

(standards.iteh.ai)

Aliments des animaux -- Détermination de la teneur en chlorures solubles dans l'eau --
Partie 1: Méthode titrimétrique

SIST ISO 6495-1:2018

Ta slovenski standard je istoveten z: ISO 6495-1:2015

<https://standards.iteh.ai/catalog/standards/sist/d756669e-6475-4c56-b00a-221af04a42af/sist-iso-6495-1-2018>

ICS:

65.120

Krmila

Animal feeding stuffs

oSIST ISO 6495-1:2018

en

INTERNATIONAL STANDARD

**ISO
6495-1**

First edition
2015-06-15

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*

SIST ISO 6495-1:2018

<https://standards.iteh.ai/catalog/standards/sist/d756669e-6475-4c56-b00a-221af04a42af/sist-iso-6495-1-2018>



Reference number
ISO 6495-1:2015(E)

© ISO 2015

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ISO 6495-1:2018
<https://standards.iteh.ai/catalog/standards/sist/d756669e-6475-4c56-b00a-221af04a42af/sist-iso-6495-1-2018>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2015

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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
8.1 Preparation of test solution.....	4
8.1.1 General.....	4
8.1.2 Preparation of test solution of sample free from organic matter.....	4
8.1.3 Preparation of test solution of sample containing organic matter, excluding the products listed in 8.1.4.....	4
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.....	4
8.2 Titration.....	5
8.3 Blank test.....	5
9 Expression of results	6
10 Precision	6
10.1 Interlaboratory test.....	6
10.2 Repeatability.....	7
10.3 Reproducibility.....	7
11 Test report	7
Annex A (informative) Results of interlaboratory test	8
Bibliography	9

ISO 6495-1:2015(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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, $\geq 0,05$ %.

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 $\rho_{20}(\text{HNO}_3) = 1,38$ g/ml.

4.5 Dilute nitric acid, volume fraction $\rho(\text{HNO}_3) = 2$ %.

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

ISO 6495-1:2015(E)

4.6 Potassium chromate solution, mass concentration $\rho(\text{K}_2\text{CrO}_4) = 5 \%$ in water (4.1).

4.7 Ammonium iron(III) sulfate, saturated solution. Prepare from $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ 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 $[\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}]$ 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 $[\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ 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, $c(\text{NaCl}) = 0,1 \text{ mol/l}$. 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):

$$c_{\text{NaCl}} = \frac{m}{58,44} \quad (1)$$

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, $c_s = 0,1 \text{ mol/l}$.

The molarity of the standard volumetric solution should be known to 0,000 1 mol/l, and duplicate titrations shall agree within $\pm 0,1 \text{ ml}$.

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):

$$c_s = \frac{20 \times c_{\text{NaCl}}}{V_s} \quad (2)$$

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 $\pm 0,1$ 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):

$$c_t = \frac{20 \times c_s}{V_t} \quad (3)$$

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.

ISO 6495-1:2015(E)

6 Sampling

Sampling is not part of the method specified in this part of ISO 6495. A recommended sampling method is given in ISO 6497.

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 0,000 1 g, not more than 10 g of the test sample ([Clause 7](#)) containing not more than 3 g of chloride. 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 0,000 1 g, approximately 5 g of the test sample ([Clause 7](#)) containing not more than 3 g of chloride. 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 0,000 1 g, approximately 5 g of the test sample ([Clause 7](#)) containing not more than 3 g of chloride. 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](#)).