



DRAFT INTERNATIONAL STANDARD ISO/DIS 6495-1

ISO/TC 34/SC 10

Secretariat: NEN

Voting begins on:
2007-08-24

Voting terminates on:
2008-01-24

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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)

ITeH STANDARD PREVIEW
(standards.iteh.ai)

ICS 65.120

[ISO/PWI 6495-1](#)

[https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cca483d0f1d5/iso-pwi-6495-1)

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

Pour accélérer la distribution, le présent document est distribué tel qu'il est parvenu du secrétariat du comité. Le travail de rédaction et de composition de texte sera effectué au Secrétariat central de l'ISO au stade de publication.

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO/PWI 6495-1](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cca483d0fdb/iso-pwi-6495-1)

<https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cca483d0fdb/iso-pwi-6495-1>

Copyright notice

This ISO document is a Draft International Standard and is copyright-protected by ISO. Except as permitted under the applicable laws of the user's country, neither this ISO draft nor any extract from it may be reproduced, stored in a retrieval system or transmitted in any form or by any means, electronic, photocopying, recording or otherwise, without prior written permission being secured.

Requests for permission to reproduce should be addressed to 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

Reproduction may be subject to royalty payments or a licensing agreement.

Violators may be prosecuted.

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

iTech STANDARD PREVIEW
(standards.itech.ai)

ISO/PWI 6495-1
<https://standards.itech.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cea483d0f1db/iso-pwi-6495-1>

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

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.*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO/PWI 6495-1](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cea483d0fdb/iso-pwi-6495-1)

[https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-
cea483d0fdb/iso-pwi-6495-1](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cea483d0fdb/iso-pwi-6495-1)

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, $\geq 0,05\%$

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*

<https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cca483d0fdb/iso-pwi-6495-1>

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 $\varphi(\text{HNO}_3)$ = 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 $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ 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 chlorides and not capable of adsorbing chlorides.

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.

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

Put about 20 g of finely pulverized sodium chloride in a thin layer on a 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 0,0001 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:

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

<https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-cea483d0f1db/iso-pwi-6495-1>
 ISO/PWI 6495-1

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 shall be known to 0,0001 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 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:

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

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 $\pm 0,1$ 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 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:

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

where

[ISO/PWI 6495-1](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-4837b5111100/iso-6495-1)

[https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-4837b5111100/iso-6495-1)

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.

[ISO/PWI 6495-1](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-6ea483d0f1db/iso-pwi-6495-1)

[https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-6ea483d0f1db/iso-pwi-6495-1)

[6ea483d0f1db/iso-pwi-6495-1](https://standards.iteh.ai/catalog/standards/sist/5d81c7c1-8401-4e3e-912b-6ea483d0f1db/iso-pwi-6495-1)

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 $\text{AgNO}_3 = V_{s1}$). 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:

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

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);