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GB/T 6439-2023

Replacing GB/T 6439-2007

Determination of water-soluble chlorides in feeds

饲料中水溶性氯化物的测定

(ISO 6495-1:2015, Animal feeding stuffs - Determination of water-soluble chlorides
content - Part 1: Titrimetric method, MOD)

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Table of Contents

Foreword	3
1 Scope.....	5
2 Normative references	5
3 Terms and definitions.....	5
4 Principle	5
5 Reagents or materials.....	5
6 Instruments and equipment	8
7 Samples	9
8 Test procedure	9
9 Test data processing	10
10 Precision.....	11
11 Test report.....	12
Annex A (informative) A table of comparison of structure numbers between this document and ISO 6495-1:2015	13
Annex B (normative) Activated carbon verification tests.....	14
Annex C (informative) Results of interlaboratory test	15
Bibliography	16

Foreword

This document was drafted in accordance with the provisions of GB/T 1.1-2020 “*Directives for standardization - Part 1: Rules for the structure and drafting of standardizing documents*”.

This document replaces GB/T 6439-2007 “*Determination of water-soluble chlorides in feeds*”. Compared with GB/T 6439-2007, in addition to structural adjustments and editorial changes, the main technical changes are as follows:

- a) CLARIFY the scope of application and ADD the quantitative limit (see Clause 1 of this document, Clause 1 of the 2007 edition);
- b) MODIFY the concentration and preparation of nitric acid, sodium chloride standard solution, silver nitrate standard titration solution, and potassium thiocyanate standard titration solution (see Clause 5 of this document, Clause 4 of the 2007 edition);
- c) ADD the activated carbon verification step (see 5.4 of this document);
- d) MODIFY the weighed specimen mass and volume of filtrate taken (see 8.1.1 of this document, Clause 8 of the 2007 edition);
- e) MODIFY the test data processing (see Clause 9 of this document, Clause 9 of the 2007 edition).

This document has modified and adopted ISO 6495-1:2015 “*Animal feeding stuffs - Determination of water-soluble chlorides content - Part 1: Titrimetric method*”. Compared with ISO 6495-1:2015, the structure has been adjusted. The comparison of the structural number changes between the two documents is shown in Annex A.

The technical differences between this document and ISO 6495-1:2015 and their reasons are as follows:

- MODIFY the scope of application, to meet the needs of water-soluble chloride determination in feeds in China (see Clause 1 of this document);
- MODIFY the concentration of nitric acid solution to 20 %, to increase operability and facilitate the application of this document (see 5.5 of this document);
- ADD sodium chloride standard solution II (0.02 mol/L), to increase operability and facilitate the application of this document (see 5.11 of this document);
- ADD silver nitrate standard titration solution II (0.02 mol/L), to increase operability and facilitate the application of this document (see 5.13 of this document);

Determination of water-soluble chlorides in feeds

1 Scope

This document describes a titrimetric method for the determination of water-soluble chlorides content in feeds expressed as sodium chloride.

This document applies to the determination of water-soluble chlorides in compound feeds, concentrated feeds, concentrate supplements, additive premix feeds, and feed ingredients.

The limit of quantification of this document is 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.

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 20195 Animal feeding stuffs - Preparation of test samples (GB/T 20195-2006, ISO 6498:1998, IDT)

3 Terms and definitions

For the purpose of this document, there are no terms or definitions that need to be defined.

4 Principle

The chloride ions in a specimen are dissolved in water (if the specimen contains organic matter, use a precipitant to clarify the solution), acidified with nitric acid solution, and then added with silver nitrate standard solution to generate silver chloride precipitate from the chlorides. The excess silver nitrate is titrated with ammonium thiocyanate or potassium thiocyanate standard solution by Volhard's method.

5 Reagents or materials

Unless otherwise specified, only analytical reagents are used.

8.1.4 Preparation of specimen solution of cooked feeds, flax cakes and flour, products rich in flax flour, and products rich in mucilage or colloidal (such as gelatinized starch)

Accurately add 100 mL of water to a conical flask containing the specimen, add 1 g of activated carbon (5.4), and mix well. Accurately add 2.5 mL of precipitant I (5.8) and 2.5 mL of precipitant II (5.9) in sequence, and shake well after each addition. Oscillate at 200 r/min for 30 min and let stand for 30 min. Filter with filter paper (5.16), discard about 10 mL of the initial filtrate, and set aside the filtrate for later use (if it cannot be filtered, centrifuge at 7000 r/min for 10 min).

Accurately pipette 50 mL of the filtrate to a 100 mL volumetric flask, add acetone (5.2) and mix, dilute to the mark, mix well, and filter with filter paper (5.16).

NOTE: Cooked feeds include feeds and feed ingredients that have been processed by puffing, tableting, tempering, granulation, expansion, baking, etc., and starch gelatinized. Products rich in mucilage or colloidal substances (such as gelatinized starch) include feeds and feed ingredients that have been fermented or enzymatically hydrolyzed.

8.2 Titration

Accurately pipette an appropriate amount of filtrate (8.1.1, V_a , chloride content not exceeding 15 mg) into a 250 mL conical flask. If the volume of the solution is less than 50 mL, add water to 50 mL. Add 2.5 mL of nitric acid solution (5.5) and 2 mL of ammonium ferric sulfate saturated solution (5.7) in sequence, add 2 drops of potassium thiocyanate or ammonium thiocyanate standard titration solution II (5.15) from a burette filled with potassium thiocyanate or ammonium thiocyanate standard titration solution II (5.15) to the 0 mark, shake, accurately add 20 mL of (V_{s1}) silver nitrate standard titration solution II (5.13), add 5 mL of acetone (5.2) and 5 mL of n-hexane (5.3), and shake vigorously. Continue to titrate with potassium thiocyanate or ammonium thiocyanate standard titration solution II (5.15), shake until the solution turns reddish brown, and shake slowly while dripping until the solution turns reddish brown and can keep it without fading for 30 s, and record the volume V_{t1} of the potassium thiocyanate or ammonium thiocyanate standard titration solution II (5.15) consumed (including the 2 drops of potassium thiocyanate or ammonium thiocyanate standard titration solution added previously).

8.3 Blank test

Carry out the reagent blank determination simultaneously.

9 Test data processing

The content of water-soluble chloride (calculated as sodium chloride) is expressed as mass fraction w , and the value is expressed as percentage (%), calculated according to formula (4):

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