International Standard 5399

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Leather — Determination of water-soluble magnesium salts — EDTA titrimetric method

Cuir — Détermination de la teneur en sels de magnésium solubles dans l'eau — Méthode titrimétrique à l'EDTA

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5399 was developed by Technical Committee ISO/TC 120, *Leather*, and was circulated to the member bodies in May 1981.

It has been approved by the member bodies of the following countries :

Brazil	India	South Africa, Rep. of
China	Italy	Spain
Czechoslovakia	Kenya	Sri Lanka
Egypt, Arab Rep. of	Korea, Rep. of	Tanzania
Ethiopia	Mexico	Turkey
Germany, F. R.	New Zealand	United Kingdom
Hungary	Romania	USSR

The member body of the following country expressed disapproval of the document :

France

This International Standard is based on method IUC/9 of the International Union of Leather Technologists' and Chemists' Societies.

Printed in Switzerland

Leather — Determination of water-soluble magnesium salts — EDTA titrimetric method

1 Scope and field of application

This International Standard specifies an EDTA titrimetric method for the determination of water-soluble magnesium salts in leather.

It is applicable to all leather which contains magnesium salts.

2 References

ISO 385, *Laboratory glassware — Burettes.*

ISO 4098, *Leather — Determination of total water-soluble matter, water-soluble inorganic matter and water-soluble organic matter.*[1)]

ISO 4788, *Laboratory glassware — Graduated measuring cylinders.*

3 Definition

For the purposes of this International Standard, the following definition applies.

magnesium salts content of leather : The quantity of magnesium salts, calculated as magnesium sulfate ($MgSO_4 \cdot 7H_2O$), obtained by extraction with water under the specified conditions.

4 Principle

Aqueous extraction of a test portion, followed by evaporation and drying at 102 ± 2 °C, and sulfating and ashing of the residue at 800 °C, to yield the water-soluble inorganic matter. Treatment of the residue with hydrochloric acid, and titration with standard volumetric *di*sodium ethylenedinitrilotetraacetate solution. (See also 9.4.)

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Ammonium chloride, solid.

5.2 Sodium chloride, solid.

5.3 Ammonium hydroxide, 25 % (V/V) solution of ammonia, ϱ = 0,880 g/ml.

5.4 Ammonium hydroxide, 2 mol/l solution.

5.5 Sodium hydroxide, 2 mol/l solution.

5.6 Hydrochloric acid, 2 mol/l solution.

5.7 Ammonia/ammonium chloride buffer solution, of pH 10,5 (see 9.1).

5.8 ***Di*sodium ethylenedinitrilotetraacetate** dihydrate (Na_2EDTA), 0,01 mol/l standard volumetric solution (see 9.2).

5.9 Methyl orange, 2 g/l solution.

5.10 Suitable indicator, for example Eriochrome black T (see 9.3).

6 Apparatus

Ordinary laboratory apparatus and

6.1 Burette, with appropriate scale, complying with the requirements of ISO 385.

1) At present at the stage of draft.

6.2 Conical flask, of capacity 300 ml.

6.3 Measuring cylinders, of capacity 50 and 250 ml, complying with the requirements of ISO 4788.

6.4 Thermometer.

7 Procedure

Treat the residue in the crucible after the determination of the water-soluble ash in accordance with ISO 4098, with a little of the hydrochloric acid solution (5.6) dissolved by gentle warming (see 9.4.1). Transfer the solution into the conical flask (6.2). Rinse the crucible several times with a very little of the hydrochloric acid solution and water; neutralize the solution against the methyl orange solution (5.9) with either the ammonia (5.4) or the sodium hydroxide solution (5.5) and boil briefly. Dilute the solution in the flask with 150 ml of water, add 20 ml of the buffer solution (5.7), adjust to 50 °C by cooling or heating, and add the indicator (5.10) until the solution becomes a clear red.

Titrate with the Na_2EDTA solution (5.8) until the colour changes to pure blue (with no red tint).

8 Expression of results

8.1 Calculation

8.1.1 The magnesium salts content, expressed as a percentage by mass as MgO, is given by the formula

$$\frac{V}{m} \times 0{,}000\,403 \times 10 \times 100$$

$$= 0{,}403 \times \frac{V}{m}$$

where

V is the volume, in millilitres, of the standard volumetric Na_2EDTA solution (5.8) used for the titration;

m is the mass, in grams, of the test portion (see ISO 4098);

0,000 403 is the mass, in grams, of MgO corresponding to 1,00 ml of exactly 0,01 mol/l Na_2EDTA solution.

8.1.2 The magnesium salts content, expressed as a percentage by mass as $MgSO_4 \cdot 7H_2O$, is given by the formula

$$\frac{V}{m} \times 0{,}002\,465 \times 10 \times 100$$

$$= 2{,}465 \times \frac{V}{m}$$

where

V and m are as defined in 8.1.1;

0,002 465 is the mass, in grams, of $MgSO_4 \cdot 7H_2O$ corresponding to 1,00 ml of exactly 0,01 mol/l Na_2EDTA solution.

Take as the result the mean of two determinations, provided that the requirement for repeatability (see 8.2) is satisfied, and express it to one decimal place.

8.2 Repeatability

The results of duplicate determinations carried out by the same operator in the same laboratory shall not differ by more than 0,03 % (m/m) MgO or 0,2 % (m/m) $MgSO_4 \cdot 7H_2O$.

9 Notes on procedure

9.1 Preparation of ammonia/ammonium chloride buffer solution (5.7)

Add 54 g of the ammonium chloride (5.1) to 350 ml of the ammonium hydroxide solution (5.3) and make up with water to 1 litre.

9.2 Preparation and standardization of the Na_2EDTA solution (5.8)

9.2.1 Preparation

Dry the Na_2EDTA at 80 °C for several hours. Weigh accurately 3,722 g of the dried Na_2EDTA , dissolve in water in a 1 000 ml one-mark volumetric flask, make up to the mark and mix well.

The solution can be kept for a practically unlimited time in closed pyrex glass or polythene vessels, but should be restandardized before use.

9.2.2 Standardization

Weigh accurately approximately 0,16 g of chemically pure zinc powder, mix with 100 ml of the hydrochloric acid solution (5.6) and heat on a water-bath until completely dissolved. Transfer the solution to the 250 ml measuring cylinder (6.3), cool and make up to the highest graduation line. Pipette 25 ml of this solution into a conical flask, dilute with 50 ml of water and add 30 ml of the ammonium hydroxide solution (5.4). Add the indicator (5.10) (see 9.3) until clear red. Titrate with the Na_2EDTA solution (9.2.1) until the colour is converted to pure blue (without red tint).

9.2.3 Calculation of concentration

The concentration c of the standard volumetric Na_2EDTA solution, expressed in moles per litre, is given by the formula

$$c = \frac{m_1}{0{,}653\,8} \times \frac{10}{V_1}$$

where

m_1 is the mass, in milligrams, of zinc used for the standardization (9.2.2);

V_1 is the volume, in millilitres, of the Na_2EDTA solution (9.2.1), used for the titration;

0,653 8 is the mass, in milligrams, of zinc corresponding to 1,00 ml of exactly 0,01 mol/l Na_2EDTA solution.

9.3 Dilution of the Eriochrome black T indicator

Mix well together 1 part of Eriochrome black T with 300 parts of the solid sodium chloride (5.2). Add the indicator in its solid state to the solution. In this way, the diluted indicator will keep for an unlimited period.

9.4 Preliminary treatment of the test portion

If the sulphated ash of water-solubles obtained in accordance with SLC/5, 1966.5.4, is used for the determination, it is necessary first to eliminate any interfering phosphate and/or calcium ions, if qualitative tests have shown their presence. This can be done in accordance with 9.4.1 and 9.4.2.

9.4.1 Elimination of phosphate ions

Mix the ash in the crucible with 5 ml of 10 % (m/m) hydrochloric acid solution and dissolve under slight heat. Rinse the solution in a 250 ml glass beaker and dilute with water to approximately 50 ml. Then add 3 ml of 10 % (m/m) ammonium chloride solution, a few drops of concentrated nitric acid and a few drops of 10 % (m/m) iron(III) chloride solution. Make the solution alkaline with ammonia, and boil. The precipitate that forms should be quite brown in colour. If it is not, then acidify again with hydrochloric acid, add a few drops of iron(III) chloride solution and make alkaline with ammonia.

Repeat this procedure until the formation of a distinctly brown-coloured precipitate occurs.

Filter the solution while warm and wash out the beaker with hot water.

9.4.2 Elimination of calcium ions

Dissolve the ash in the crucible in accordance with 9.4.1 or use the filtrate after elimination of the phosphate ions in accordance with 9.4.1. Treat the solution of the ash or filtrate with 3 ml of 10 % (m/m) ammonium chloride solution, 7,5 ml of 10 % (m/m) ammonium hydroxide solution and 0,5 ml of 5 % (m/m) ammonium oxalate solution.

Heat the solution to boiling, transfer to a 100 ml measuring flask and adjust to volume; mix and filter through a fluted filter.

Pipette 50 ml of the filtrate into a 400 ml glass beaker, dilute with 100 ml of water, and determine the magnesium content in accordance with clause 7.

10 Test report

The test report shall contain the following particulars :

a) reference to this International Standard;

b) the results obtained and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or the International Standards to which reference is made, or regarded as optional.

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