International Standard 5397

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

Leather — Determination of nitrogen content and "hide substance" — Titrimetric method

Cuir — Détermination de la teneur en azote et de la «substance dermique» — Méthode titrimétrique

First edition — 1984-03-15

UDC 675 : 543.846 **Ref. No. ISO 5397-1984 (E)**

Descriptors : leather, tanning, tests, determination of content, nitrogen, volumetric analysis.

Price based on 3 pages

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 5397 was developed by Technical Committee ISO/TC 120, *Leather*, and was circulated to the member bodies in November 1980.

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

Australia	India	Romania
Brazil	Korea, Rep. of	South Africa, Rep. of
Canada	Mexico	Spain
Czechoslovakia	New Zealand	Turkey
Egypt, Arab Rep. of	Poland	United Kingdom
Hungary	Portugal	USSR

The member body of the following country expressed disapproval of the document on technical grounds:

France

This International Standard is based on Method IUC/10 of the International Union of Leather Technologists' and Chemists' Societies.

Printed in Switzerland

Leather — Determination of nitrogen content and "hide substance" — Titrimetric method

0 Introduction

The determination of "hide substance" from nitrogen content is based on the fact that, according to Schroder and Passler, the grease- and ash-free dry substance of the pelts of various animals has a somewhat different, but practically constant, nitrogen content for certain types of animal.

1 Scope and field of application

This International Standard specifies a titrimetric method for the determination of the nitrogen content and of the "hide substance" of leather.

The method is applicable to all types of leather in all types of tannage.

NOTE — Other nitrogenous substances (e.g. certain fixatives, synthetic tannins, cationic fats and dyestuffs) falsify the value for the "hide substance". If these materials are present, it is not possible to obtain an accurate result for the "hide substance".

2 References

ISO 385, *Laboratory glassware — Burettes.*

ISO 2418, *Leather — Laboratory samples — Location and identification.*

ISO 2588, *Leather — Sampling — Number of items for a gross sample.*

ISO 4044, *Leather — Preparation of chemical test samples.*

3 Definition

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

hide substance : The nitrogenous substances content, calculated from the nitrogen content by multiplication by the factor 5,62.

4 Principle

Decomposition of a test portion by the Kjeldahl method. Distillation of the ammonia liberated, by one of the usual methods. Determination of the nitrogen content by titration of the ammonia with sulfuric or hydrochloric acid, using phenolphthalein as indicator.

5 Reagents

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

5.1 Fuming sulfuric acid, [7 % (m/m) of free SO_3], or **sulfuric acid,** 98 % (m/m).

5.2 Catalyst mixture.

Any suitable catalyst mixture which considerably shortens the digestion may be used. The following are examples of such mixtures :

a) 100 mg of $CuSO_4$ anhydrous
6 to 8 g of K_2SO_4 anhydrous

b) 10 g of selenium
25 g of $CuSO_4$ anhydrous
350 g of K_2SO_4 anhydrous

Prepare a stock of catalyst (preferably by mixing in a ball mill) and use it in the ratio of approximately 5 g of catalyst to 3 g of test portion.

5.3 Boric acid, borate-free saturated solution in water, containing, if possible, a suitable indicator, for example, in 1 litre, 2 ml of the following mixed indicator solution :

0,06 % (m/m) methyl red and 0,04 % (m/m) methylene blue in 96 % (V/V) ethanol.

5.4 Sodium hydroxide, 35 % (m/m) solution.

5.5 Sulfuric acid, standard volumetric solution, $c(1/2\ H_2SO_4)$ = 0,5 mol/l, or **hydrochloric acid,** standard volumetric solution, $c(HCl)$ = 0,5 mol/l.

5.6 Phenolphthalein, 10 g/l indicator solution in 50 % (V/V) ethanol.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Kjeldahl flask, of suitable capacity — 230 to 300 ml if an external source of steam is to be used for the distillation.

6.2 Suitable distillation apparatus.

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

7 Sampling

7.1 Whole pieces of leather

In the absence of any other agreement on sampling between the interested parties, use the procedure specified in ISO 2588 for sampling from a lot.

Take samples from the pieces as specified in ISO 2418.

7.2 Other applications

Carry out the sampling as required by the relevant specification or contract.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, 3 g of the sample of ground leather (2 g in the case of chrome leather), prepared in accordance with ISO 4044, in a small weighing bottle and transfer it quantitatively to the Kjeldahl flask (6.1) by lightly tapping the bottom of the bottle (see 10.2).

8.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same reagents as used for the determination.

8.3 Determination

To the test portion (8.1) in the Kjeldahl flask, add 30 ml of the sulfuric acid (5.1) and approximately 5 g of the catalyst mixture (5.2). Then heat to boiling, first with a low and later with a higher flame, until 1 h after all the carbon has been oxidized.

If an external source of steam is to be used, allow the digest to cool, and dilute with approximately 50 ml of water. Allow to cool, and transfer to the distillation flask (see 6.2). Rinse the Kjeldahl flask twice each time with water, add a few drops of the phenolphthalein solution (5.6), make alkaline with an excess (approximately 70 ml) of the sodium hydroxide solution (5.4) and steam distil. Alternatively, if the digestion has been carried out in a 700 ml Kjeldahl flask, allow to cool, dilute with 250 ml of water, add a few fragments of anti-bump material and a few drops of the phenolphthalein solution, and make alkaline with an excess (approximately 70 ml) of the sodium hydroxide solution. Connect the flask with a vertical condenser by means of a tube bent twice, preferably including a spray trap.

Distil the ammonia with water vapour into the receiver, containing 100 ml of the saturated boric acid plus indicator solution (5.3). Dip the cooling tube into the boric acid solution. The distilled ammonia colours the indicator green.

Distil until 150 to 200 ml of distillate is obtained. Before ending the distillation, lower the receiver so that the cooling tube no longer dips in. Distil for approximately 3 min more, and rinse the end of the condenser with water.

Titrate the ammonia with the sulfuric or hydrochloric acid solution (5.5) to pH 4,6. When using the indicator referred to in 5.3, titrate to the first constant pale pink colour.

Carry out the determination in duplicate.

9 Expression of results

9.1 Calculation

9.1.1 The nitrogen content N, expressed as a percentage by mass, is given by the formula

$$N = \frac{V}{m} \times 0{,}7$$

where

V is the volume, in millilitres, of the standard volumetric sulfuric or hydrochloric acid solution (5.5) used for the titration, corrected for the blank test (8.2);

m is the mass, in grams, of the test portion (8.1).

Take as the result the mean of two determinations, provided that the requirement for repeatability (see 9.2) is satisfied, and express it to one decimal place.

9.1.2 The "hide substance" H, expressed as a percentage by mass, is given by the formula

$$H = N \times 5{,}62$$

where N is as defined in 9.1.1.

9.2 Repeatability

The results of duplicate determinations carried out by the same operator in the same laboratory shall not differ by more than 0,1 %, calculated on the original mass of leather.

10 Notes on procedure

10.1 With the prescribed mass of test portion and using the acid specified in 5.5 for the titration, the end-point is very clear and sharp.

10.2 The determination can also be carried out with 0,5 g of leather weighed to 0,000 2 g and 15 to 20 ml of the concentrated or fuming sulfuric acid (5.1) and 2,5 g of the catalyst (5.2). Titration of the ammonia is then carried out with acid of one-fifth the concentration specified in 5.5.

11 Test report

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the sample;

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

d) any unusual features noted during the determination;

e) 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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