International Standard 5400

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Leather — Determination of total silicon content — Reduced molybdosilicate spectrometric method

Cuir — Détermination de la teneur en silicium total — Méthode spectrométrique au molybdosilicate réduit

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

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 5400 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	Kenya	Sri Lanka
China	Korea, Rep. of	Tanzania
Egypt, Arab Rep. of	Mexico	Turkey
Ethiopia	New Zealand	United Kingdom
Germany, F.R.	Romania	USSR
Hungary	South Africa, Rep. of	
India	Spain	

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

France

Leather — Determination of total silicon content — Reduced molybdosilicate spectrometric method

1 Scope and field of application

This International Standard specifies a reduced molybdosilicate spectrometric method for the determination of the total silicon content of leather.

It is not possible to define the chemical identity of the silicon compounds present in leather and the results are expressed in terms of the oxide SiO_2. Organo-silanes are present in leather and they may be determined by this procedure. Organo-silanes are almost quantitatively extracted from leather by tetrahydrofuran or dichloromethane, and this forms the basis of a method for their determination (Elliott, JSLTC, 1964, **48**, 105; SLTC-Method SLC/16). A more reliable estimate of inorganic silicon-containing compounds will therefore be obtained if leathers suspected of containing organo-silanes are first extracted with dichloromethane.

2 References

ISO 835, *Laboratory glassware — Graduated pipettes* —

Part 1 : General requirements.

Part 2 : Pipettes for which no waiting time is specified.

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

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 Principle

Oxidation of a test portion in a Parr bomb under alkaline conditions to destroy organic matter and to solubilize the mineral matter. Formation of the molybdosilicate complex and spectrometric measurement at a wavelength of 630 nm.

4 Reagents

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

4.1 Concentrated sulfuric acid, ϱ_{20} = 1,83 g/ml, diluted 1 + 1 with water.

4.2 Sulfuric acid, standard solution, $c(1/2\ H_2SO_4)$ = 1,0 mol/l.

4.3 Glycerol.

4.4 Sodium sulfate, 100 g/l solution.

4.5 Ammonium molybdate, 100 g/l solution containing 2 ml of ammonium hydroxide, ϱ_{20} = 0,880 g/ml, per 100 ml of solution.

4.6 Tin(II) chloride, dihydrate, 10 g/l solution.

Dissolve 10 g of $SnCl_2.2H_2O$ in 12 ml of warm hydrochloric acid, ϱ_{20} = 1,19 g/ml and 5 ml water, then dilute to 1 000 ml.

Prepare this solution fresh.

4.7 Fusion mixture, consisting of

100 g of sodium peroxide;

10 g of potassium nitrate;

3 g of powdered sucrose.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Parr bomb, 22 ml.

5.2 Beaker, of capacity 250 ml.

5.3 One-mark volumetric flasks, of capacity 100 and 500 ml, complying with the requirements of ISO 1042.

5.4 Pipettes, complying with the requirements of ISO 835/1 and ISO 835/2.

5.5 Analytical balance, accurate to 0,001 g.

5.6 Spectrometer or photo-electric absorptiometer.

5.7 pH meter, fitted with glass and reference electrodes.

6 Sampling

6.1 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.

6.2 Other applications

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

7 Procedure

7.1 Prepare the test sample in accordance with ISO 4044.

7.2 Place 6 g of the fusion mixture (4.7) in the Parr bomb (5.1) followed by approximately 0,5 g of the sample, weighed to an accuracy of 0,001 g. Moisten the test portion with approximately 0,5 ml of the glycerol (4.3) and cover with 20 g of the fusion mixture (4.7). Seal the bomb. Place it in its protection tube and warm its base with a small gas flame for 20 min. Allow to cool.

Open the bomb and place it in the beaker (5.2), rinse the cover with water, collecting the rinsing water in the beaker. Add sufficient water to dissolve the contents of the bomb, remove it and rinse into the beaker. Transfer the beaker to a steam bath and leave for 2 h or until the hydrogen peroxide is decomposed. Allow to cool, neutralize with the sulfuric acid solution (4.1), adding a very slight excess. Transfer the solution quantitatively to the 500 ml one-mark volumetric flask (5.3), dilute to the mark and mix, to obtain the test solution.

Dilute a known volume V of the test solution containing, 0,05 to 0,2 mg of SiO_2 (usually 10 ml) with 40 ml of water and acidify to pH 1,8 with the sulfuric acid solution (4.2). Transfer quantitatively to the 100 ml one-mark volumetric flask (5.3), add 10 ml of the ammonium molybdate solution (4.5) and allow to stand for 30 min. Add 10 ml of sulphuric acid solution (4.1), 10 ml of the tin(II) chloride solution (4.6), dilute to the mark with water and mix. Allow to stand for 30 min.

Measure the blue coloration using the spectrometer or photoelectric absorptiometer (5.6) at 630 nm. The colour is stable for up to 1 h.

7.3 Prepare a calibration curve by fusing 0,1 g of pure silica, weighed to the nearest 0,001 g, with sodium carbonate and making up to volume. Develop the colour in aliquots of this solution by the method described in 7.2 for the treatment of the test solution.

8 Expression of results

8.1 Calculation

The total silicon content, expressed as a percentage by mass as SiO_2, is given by the formula

$$\frac{m_1}{m_0} \times \frac{50}{V}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of SiO_2 in the aliquot portion of the test solution, taken for the determination;

V is the volume, in millilitres, of the test solution taken for the determination;

50 is a factor taking into account the dilutions used in the procedure.

8.2 Repeatability

The results of duplicate determinations carried out by the same operator in the same laboratory shall not differ by more than 0,2 %, calculated on the original mass of leather.

8.3 Reproducibility

The results of two determinations carried out by different operators in different laboratories on the same sample shall not differ by more than 0,5 %, calculated on the original mass of leather.

9 Notes on procedure

9.1 Use of leather ash

The acid-insoluble residue obtained in the isolation of zirconium for analysis by hydrofluoric acid may be substituted for leather at the fusion stage. If followed, this fact should be stated in the test report.

9.2 Alternative bomb

A Wurzschmitt bomb may be used in lieu of the Parr Bomb, in which case the sample size should be reduced accordingly up to and including the preparation of the test solution.

10 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 on the sample as received and, if required, on the basis of a standard moisture content;

d) any unusual features noted during the determination;

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