INTERNATIONAL STANDARD 3072

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Wool — Determination of solubility in alkali

Laine — Détermination de la solubilité en milieu alcalin

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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 3072 was drawn up by Technical Committee ISO/TC 38, *Textiles,* and circulated to the Member Bodies in October 1974.

It has been approved by the Member Bodies of the following countries :

Australia	Hungary	Romania
Belgium	India	South Africa, Rep. of
Bulgaria	Iran	Spain
Canada	Ireland	Sweden
Chile	Israel	Turkey
Czechoslovakia	Japan	United Kingdom
Denmark	Netherlands	U.S.A.
Finland	New Zealand	U.S.S.R.
Germany	Poland	Yugoslavia

No Member Body expressed disapproval of the document.

This International Standard is based on Test Method IWTO-4-60, drawn up by the International Wool Textile Organization (IWTO).

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Wool — Determination of solubility in alkali

0 INTRODUCTION

The solubility of wool in alkali provides a useful index of the extent of the change in its chemical properties brought about by certain agencies. Treatment with acids, oxidizing or reducing agents and exposure to heat or light cause an increase in the solubility, whereas treatment with alkalis or cross-linking agents causes the solubility to decrease. The change in solubility is thus a measure of the severity of the treatment.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the solubility of wool in alkali.

The method is applicable to wool textiles in any form, namely loose fibre, sliver, roving, yarn or cloth. The test is most useful when an untreated control sample is available and when the nature of the treatment of the sample under test is known, i.e. as a method of control. When the sample has been treated by two agencies having opposite effects on the solubility, the interpretation of the results, even when an untreated control sample is available, is difficult and may be misleading.

2 REFERENCES

ISO 1130, *Textile fibres — Some methods of sampling for testing.*

ISO 3071, *Wool — Determination of the pH value of the aqueous extract.*

ISO 3073, *Wool — Determination of acid content.*

3 PRINCIPLE

Immersion of the wool in sodium hydroxide solution under specified conditions of time, temperature and volume. Determination of the loss in mass as the difference in the mass of the dry sample before and after treatment.

4 REAGENTS

4.1 Sodium hydroxide, 0,1 N solution.

4.2 Acetic acid solution containing 10 ml of glacial acetic acid per litre.

4.3 Dichloromethane

WARNING. Dichloromethane is toxic; the room in which extractions are made shall be adequately ventilated.

5 APPARATUS

5.1 Soxhlet extraction apparatus.

5.2 Water-bath, thermostatically controlled at 65 ± 0,5 °C. To ensure uniform temperature, the water must be stirred.

5.3 Stoppered flasks with a working capacity of 100 ml, of the same shape and wall thickness.

5.4 Sintered-glass filtering crucibles, 30 ml capacity, porosity 1. If possible, these crucibles should have ground glass stoppers. If ground glass stoppers are not available, the crucibles shall be enclosed in weighing bottles for determination of their masses.

5.5 Filter-flask, filter-pump, and adaptor to enable the crucibles to be fitted to the filter-flask.

5.6 Ventilated oven for drying samples at 105 ± 3 °C.

5.7 Stoppered weighing bottles for obtaining the masses of test specimens.

5.8 Analytical balance, accurate to 0,000 2 g.

5.9 Desiccator.

6 SAMPLING

Take a sample representative of the bulk and sufficient to provide wool (free of fat and vegetable matter) for the following test specimens :

— one test specimen of mass approximately 1 g for determining dry mass (see 7.3);

— two test specimens each of mass approximately 1 g for determining the solubility in alkali (see 7.4);

— two test specimens each of mass approximately 2 g for determining the acid content (only required when the sample contains acid — see 7.5).

Useful information on sampling is given in ISO 1130.

7 PROCEDURE

7.1 Preparation of sample

Extract the sample in the Soxhlet extraction apparatus (5.1) with dichloromethane (4.3) for 1 h at a minimum rate of six cycles per hour. Allow the dichloromethane to evaporate and then remove all vegetable and other obvious foreign matter. Disintegrate the test sample into short lengths of approximately 1 cm and allow it to come to equilibrium with the laboratory atmosphere.

7.2 Determination of masses of the test specimens

Determine the masses of the test specimens described in clause 6 to an accuracy of 0,000 2 g.

7.3 Determination of dry mass

Place the test specimen in a weighing bottle (5.7) and dry in the ventilated oven (5.6) at 105 ± 3 °C. Stopper the bottle, cool it in the desiccator (5.9) and determine its mass. Repeat these drying and weighing operations until constant mass[1] has been attained. Remove the test specimen, determine the mass of the weighing bottle and hence calculate the dry mass of the test specimen. Calculate by proportion the dry masses of the other test specimens.

NOTE — Ensure that each test specimen is treated in such a way that all have the same moisture content.

7.4 Determination of solubility in alkali

Measure 100 ml of the sodium hydroxide solution (4.1) into a flask (5.3), stopper loosely, and fix the flask in the water-bath (5.2) by any suitable means so that the level of the water outside the flask is at least 5 cm higher than the level of the solution inside. This procedure is essential for precise control of temperature.

When the temperature of the sodium hydroxide solution reaches 65 ± 0,5 °C, carefully introduce one test specimen of known mass into the flask, replace the stopper and shake the flask gently to ensure complete wetting of the test specimen. Again shake the flask gently after 15, 30 and 45 min, the time of shaking not to exceed 5 s on each occasion.

After 1 h carefully transfer the contents of the flask to a glass filtering crucible (5.4) of known mass and drain the crucible by suction. Wash any fibrous material remaining in the flask into the crucible with distilled or de-ionized water. Wash the residue in the crucible six times with water, draining completely after each wash, and release the suction.

Fill the crucible twice successively with the acetic acid solution (4.2). Allow to stand for 1 min and drain the crucible by suction. Finally, wash the residue six times with water, draining completely after each wash. Dry the crucible and contents at 105 ± 3 °C, cool in the desiccator and determine the mass.

Repeat these operations until constant mass has been attained.

7.5 Determination of acid content

Determine the pH of the aqueous extract by the method given in ISO 3071. If an aqueous extract of the material has a pH value less than 4,0, determine the acid content by the method given in ISO 3073.

8 EXPRESSION OF RESULTS

8.1 Samples not containing acid

The solubility in alkali, S, as the loss in mass of the test specimen expressed as a percentage of its calculated dry mass, is given by the formula :

$$S = \frac{m_1 - m_2}{m_1} \times 100$$

where

m_1 is the dry mass of the test specimen (determined as in 7.3);

m_2 is the dry mass of the residue (determined as in 7.4).

8.2 Samples containing acid

The corrected solubility in alkali, S', as the loss in mass of the test specimen expressed as a percentage of its calculated dry, acid-free mass, is given by the formula :

$$S' = (S - s)\frac{100}{100 - s}$$

where

S is the uncorrected alkali solubility (calculated as in 8.1);

s is the percentage of acid (determined as in 7.5).

9 TEST REPORT

The test report shall include the following information :

a) reference to this International Standard;

b) the individual results and their mean, each to three significant figures;

c) any departure from the method described above, for example, owing to insufficient material being available.

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1) Constant mass may be deemed to have been attained when the change in mass of a specimen which has been re-dried for at least 30 min does not exceed 0,000 5 g.