INTERNATIONAL STANDARD 2916

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Wool — Determination of alkali content

Laine — Détermination de la teneur en alcali

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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 2916 was drawn up by Technical Committee ISO/TC 38, *Textiles,* and circulated to the Member Bodies in November 1972.

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

Australia	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Spain
Bulgaria	Israel	Sweden
Canada	Italy	Thailand
Czechoslovakia	Japan	Turkey
Denmark	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
Finland	Norway	U.S.S.R.
France	Poland	
Germany	Portugal	

No Member Body expressed disapproval of the document.

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Wool — Determination of alkali content

0 INTRODUCTION

This International Standard is based on the IWTO test method 21-69 drawn up by the International Wool Textile Organization.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the alkali content of wool.

The method is applicable to undyed wool in any form; for example, loose fibres, card sliver, tops, yarn or fabric. It can be used with dyed wool if the amount of dye extracted in the course of the test does not affect the determination of the end-point of the titration.

2 PRINCIPLE

Extraction of the alkali from a weighed quantity of wool by immersion in dilute boric acid solution. Determination of the amount of alkali extracted, by titration with a standard volumetric solution of hydrochloric acid.

3 REAGENTS

3.1 Boric acid solution.

Dissolve 10 g of boric acid (analytical reagent grade) in 1 l of distilled water.

3.2 Hydrochloric acid, N/20 solution.

Standardize this solution by titration with freshly standardized sodium hydroxide solution.

3.3 Methyl red — Methylene blue indicator :

a) A 1 g/l solution of methyl red in absolute ethanol.

b) 4 ml of a 10 g/l aqueous solution of methylene blue mixed with 96 ml of ethanol with warming.

4 APPARATUS

4.1 Conical flasks with glass stoppers, capacity 250 ml.

4.2 Erlenmeyer flasks, capacity 250 ml.

4.3 Microburette, 2 ml.

4.4 Pipettes, 50 and 100 ml.

4.5 Weighing bottle with lid.

4.6 Sintered-glass crucible, with pore diameter between 100 and 160 μm.

4.7 Analytical balance, accurate to 0,000 5 g.

4.8 Desiccator.

4.9 Oven for drying samples at 105 ± 2 °C.

4.10 Mechanical shaking device (optional).

5 SAMPLING AND PREPARATION OF TEST SPECIMENS

Take a representative sample of mass not less than 10 g from the material to be tested.

If the dichloromethane extract of the sample is greater than 1 %, degrease with dichloromethane as follows :

Extract the sample with dichloromethane for 1 h in a Soxhlet apparatus, at the minimum rate of 6 cycles per hour. Evaporate the dichloromethane from the cleaned sample and remove all vegetable matter and foreign substances from the wool. If the sample is yarn or cloth, dissect into short lengths of yarn (approximately 1 cm) and leave to attain equilibrium with the laboratory atmosphere. Felted materials that cannot be dissected into yarn must first be cut up into small pieces.

Take from the cleaned, or uncleaned, sample, as the case may be, at least two representative test specimens each of mass 2 ± 0,001 g and one representative test specimen of mass 1 ± 0,001 g for determining the dry mass.

6 TEST PROCEDURE

6.1 Determination of dry mass

Transfer the test specimen of mass 1 ± 0,001 g to the weighing bottle (4.5) and dry it in the drying oven (4.9) at 105 ± 2 °C.

Stopper the bottle, allow it to cool in the desiccator (4.8), remove and determine its mass. Repeat these drying and mass determination operations until constant mass has been attained.[1)]

Remove the test specimen, determine the mass of the weighing bottle and hence determine the dry mass of the test specimen. Calculate by proportion the dry mass of the other test specimens.

6.2 Determination of alkali content

Place each of the other test specimens in a conical flask (4.1) and add by pipette (4.4) 100 ml of boric acid solution (3.1). Stopper the flasks and either shake for 2 h on the mechanical shaker (4.10) or allow to stand overnight after first shaking by hand to wet out the wool.

Decant the liquid from the wool and filter through the sintered-glass crucible (4.6) in order to remove fibre fragments.

Transfer by pipette (4.4) 50 ml of the filtered liquid to an Erlenmeyer flask (4.2) and titrate with the N/20 hydrochloric acid solution (3.2), using 3 drops of methyl red and 3 drops of methylene blue solutions (3.3) as indicator.

Carry out a titration on each extract and obtain the mean volume of N/20 hydrochloric acid solution used in the two titrations.

7 EXPRESSION OF RESULTS

The mass of the alkali present in the wool, expressed as a percentage of the dry mass of the wool, is given by the formula :

$$\text{Alkali content} = \frac{T \times V \times k}{m} \%$$

where

T is the normality of the hydrochloric acid solution used;

V is the volume, in millilitres, of the hydrochloric acid solution used (mean of two titrations);

k is a constant;

m is the dry mass, in grams, of the dried test specimen.

The value of k depends on the form in which the alkali content is to be expressed; it is

5,3 if the alkali content is expressed as sodium carbonate;

4,0 if the alkali content is expressed as sodium hydroxide;

3,1 if the alkali content is expressed as sodium oxide.

8 TEST REPORT

The test report shall include the following particulars :

a) that the procedure was conducted in accordance with this International Standard;

b) the results for each test specimen obtained using the formula in clause 7 and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

1) Constant mass is reached when the mass of a sample does not change by more than 1 mg after repeated drying for at least 30 min.

ANNEX

ACCURACY OF THE METHOD

An interlaboratory trial was carried out by a Working Group using three wools of different alkali contents. Nine laboratories participated. The statistical evaluation of the resulting 3 × 9 × 2 values (each laboratory carried out a double test on each wool) produced the values summarized in the following table.

	Wool A	Wool B	Wool C
Mean alkali content of the tested wool, %	0,23	0,35	0,60
Estimated values for the interlaboratory component of variation s_L, % alkali content	0,019	0,018	0,023
Estimated values for the interlaboratory component of variation s_R, % alkali content	0,003 0	0,004 1	0,006 8

These data enable an estimation to be made of the confidence limits to be expected in a determination of alkali content according to the method specified.

A.1 CONFIDENCE LIMITS FOR THE RESULTS OF ONE LABORATORY

If the interlaboratory variance is not taken into consideration (comparison within one laboratory), the confidence limits, T_i, of the mean for the laboratory are calculated with 95 % statistical significance (5 % probability of error) using the formula :

$$T_i = \pm 2 \frac{s_R}{\sqrt{k}} \% \text{ alkali content}$$

where k is the number of tests from which the mean was calculated.

An estimated numerical value for s_R in relation to the mean alkali content, taken from the above table by approximate interpolation if necessary, is inserted in the above formula.

A.2 CONFIDENCE LIMITS TAKING INTO ACCOUNT THE VARIATION BETWEEN LABORATORIES

If the variance between laboratories is taken into account (comparison between different laboratories), then the confidence limits, T_L, of the mean are calculated with 95 % statistical significance (5 % probability of error) using the formula :

$$T_L = \pm 2 \sqrt{\frac{s_L^2}{i} + \frac{s_R^2}{i \times k}} \% \text{ alkali content}$$

in which the participating i laboratories each carried out k tests. When $i = 1$, the confidence limits for individual laboratories with interlaboratory variance taken into account are obtained.

Approximate numerical values for s_L and s_R are obtained from the above table.

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