

Surface active agents — Determination of wetting power by immersion

The European Standard EN 1772:2000 has the status of a
British Standard

ICS 71.100.40

National foreword

This British Standard is the official English language version of EN 1772:2000. It supersedes BS EN 1772:1995 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/34, Methods of test for surface active agents, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 13 and a back cover.

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English version

Surface active agents - Determination of wetting power by immersion (ISO 8022:1990 modified)

Agents de surface - Détermination du pouvoir mouillant par immersion (ISO 8022:1990 modifié)

Grenzflächenaktive Stoffe - Bestimmung des Tauchnetzvermögens (ISO 8022:1990 modifiziert)

This European Standard was approved by CEN on 18 February 2000.

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Contents

	Page
Foreword	3
Introduction	4
1 Scope.....	5
2 Normative references	5
3 Term and definition.....	5
4 Principle	5
5 Reagents and products	6
6 Apparatus.....	6
7 Sampling	9
8 Procedure.....	9
8.1 Test portion.....	9
8.2 Preparation of the surface active agent solution.....	9
8.3 Preparation of the discs of cotton control cloth.....	9
8.4 Cleaning the apparatus	9
8.5 Filling the measurement beaker	10
8.6 Determination (see figure 3).....	10
8.7 Calibration of the cloth	10
9 Expression of results.....	10
10 Test report.....	11
Annex A (informative) Commercially available control cloths.....	12
Bibliography	13

Foreword

The text of the International Standard from Technical Committee ISO/TC 91 "Surface active agents" of the International Organization for Standardization (ISO) has been taken over as a European Standard by Technical Committee CEN/TC 276 "Surface active agents", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2000, and conflicting national standards shall be withdrawn at the latest by September 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

This European Standard replaces EN 1772:1995.

Annex A is informative.

Endorsement notice

The text of the International Standard ISO 8022:1990 has been approved by CEN as a European Standard with the following modifications as given below.

Punch of diameter 30 mm, carefully degreased using a volatile solvent.

NOTE The following solvents may be used:

- acetone;
- hexane;
- hexane/acetone mixture, 50 % (V/V);
- ethanol;
- acetone/ethanol mixture, 50 % (V/V);
- distilled water (in some cases).

The standard product has been modified: sodium di-*n*-hexylsulfosuccinate and sodium di-*n*-heptylsulfosuccinate have been replaced by sodium bis (2-ethylhexyl) sulfosuccinate.

Introduction

In many textile operations, for example softening or washing textiles, as well as in the rinsing or the cleaning of hard surfaces - in short in all processes in which a phase (air, oil or soil) has to be replaced by a liquid phase (aqueous or organic) - it is useful to know the wetting agents used. It is also important to know after how long complete wetting is obtained.

1 Scope

This European standard specifies a method for determining the wetting power of a surface active agent in solution by immersion of a disc of raw cotton cloth in the solution. The method is applicable to all surface active agents, whatever their ionic character, used as wetting agents in neutral, slightly acid or slightly basic baths for textile applications. The method is not applicable to mercerizing assistants (baths highly basic) or to carbonizing assistants (baths highly acid).

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 20139, *Textiles - Standard atmospheres for conditioning and testing (ISO 139:1973)*.

ISO 607, *Surface active agents and detergents - Methods of sample division*.

ISO 2456, *Surface active agents - Water used as a solvent for tests - Specification and test methods*.

ISO 3819, *Laboratory glassware - Beakers*.

3 Term and definition

For the purposes of this standard, the following term and definition apply:

3.1

wetting power (by immersion)

degree of ability of a solution of surface active agent to displace the air trapped in a cloth when the cloth is steeped in the solution

NOTE The wetting power of a surface active agent can be evaluated by examination of plots of wetting time of discs of raw cotton cloth immersed in solutions of surface active agents or solutions of standard wetting agents of known concentration, against concentration.

4 Principle

Immersion, while held in a gripper, of a cotton disc of known nature and characteristics, in a solution of surface active agent of known concentration; maintenance of complete submersion in the solution, by means of the specially designed gripper, of the cotton disc, which tends to float to the surface due to air trapped in the cloth. After displacement of air and penetration of the solution into the cloth, the cotton disc starts to sink. Determination of the wetting time by measuring the interval between the moment of immersion of the cotton disc and the moment when it begins to sink.

Determination of the wetting time using a standard solution at five different concentrations, and then using the surface active agent solution under test, also at five different concentrations.

After plotting the two "wetting time/concentration" curves, determination of the wetting power of the surface active agent under test by comparison of the position of its curve with the standard curve.

5 Reagents and products

5.1 Distilled water, or water of equivalent purity, complying with the specifications of ISO 2456.

NOTE Other grades of water can be used provided details are noted in the test report.

5.2 Sodium bis (2-ethylhexyl) sulfosuccinate, of recognized analytical grade (purity $\geq 98\%$).

5.3 Raw cotton control cloth, of known nature and characteristics, conditioned in the standard temperate atmosphere specified in EN 20139, i.e. a relative humidity of 65 % and a temperature of 20 °C. (Various types of commercially available control cloths are described in annex A).

6 Apparatus

Ordinary laboratory apparatus and, in particular:

6.1 Beaker, low form, of capacity 1 000 ml, complying with the specifications of ISO 3819.

6.2 Cloth-immersion gripper, made of stainless-steel wire of about 2 mm diameter and whose dimensions are given in figure 1 (see also the photo, figure 2, which shows an example of a gripper constructed in accordance with figure 1, with three support arms projecting at right angles from the gripper body). These arms can be mounted on a sliding collar as shown in figure 1. It is important that the design of the gripper is such that, when a raw cotton disc held in the gripper is immersed in 700 ml of test solution in the 1 000 ml beaker (6.1), the cotton disc is held about 40 mm below the surface of the solution. It is also important that the gripper tips only open about 6 mm so that the cotton disc remains nearly vertical in the solution.

6.3 Punch, of diameter 30 mm, carefully degreased using a volatile solvent.

NOTE The following solvents can be used:

- acetone;
- hexane;
- hexane/acetone mixture, 50 % (V/V);
- ethanol;
- acetone/ethanol mixture, 50 % (V/V);
- distilled water (in some cases).

6.4 Stopwatch, accurate to 0,1 s.

Dimensions in millimetres

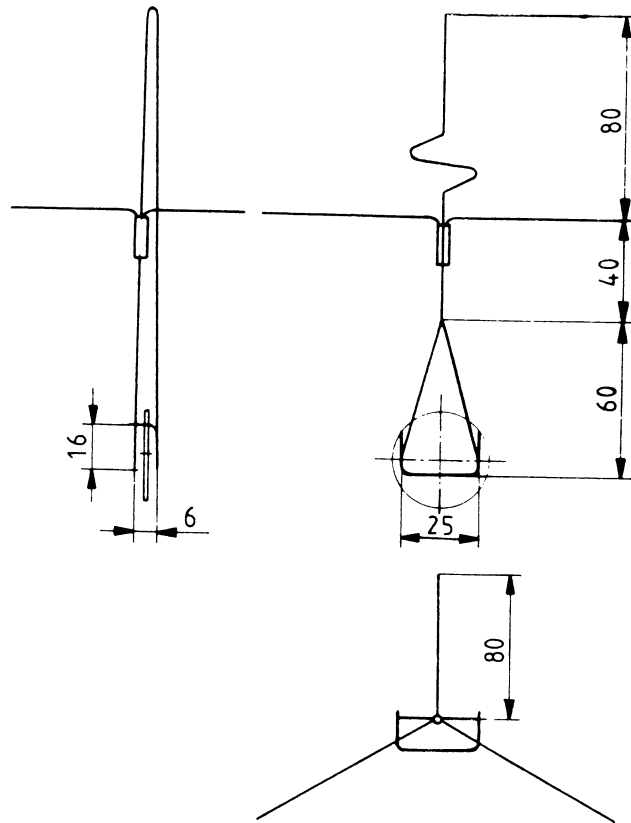


Figure 1 – Cloth-immersion gripper

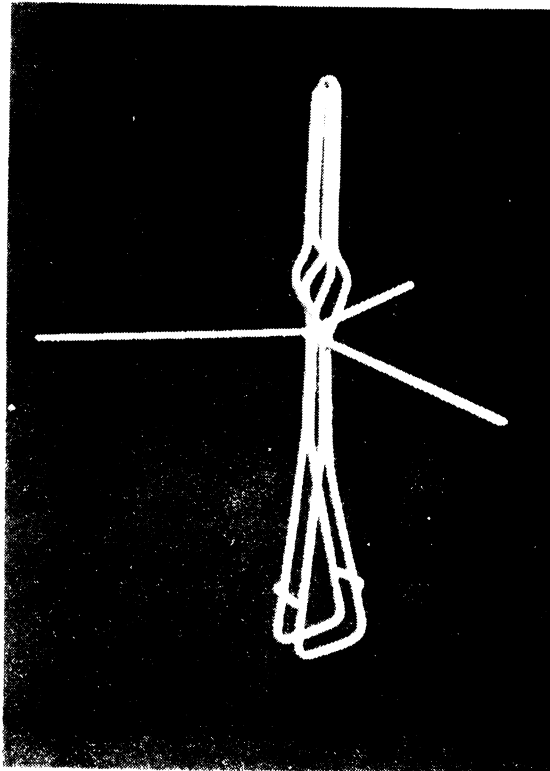


Figure 2 – Photo showing example of gripper constructed in accordance with the requirements specified in 6.2

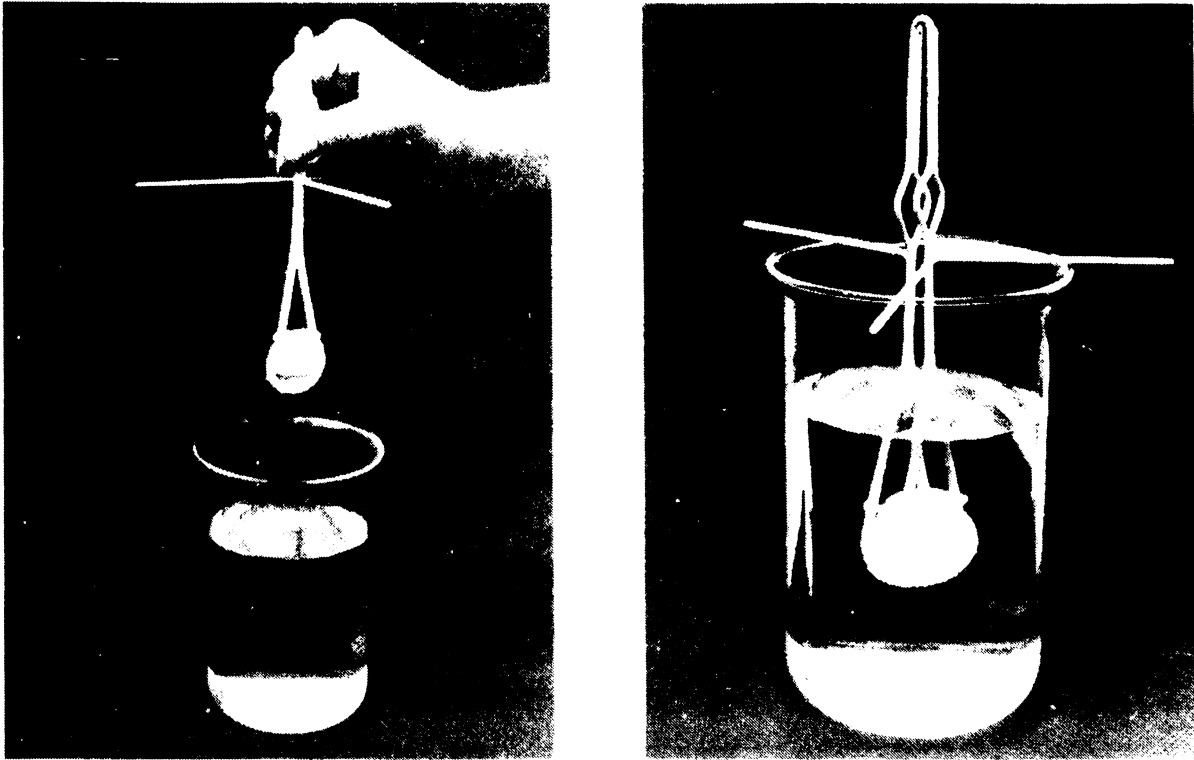
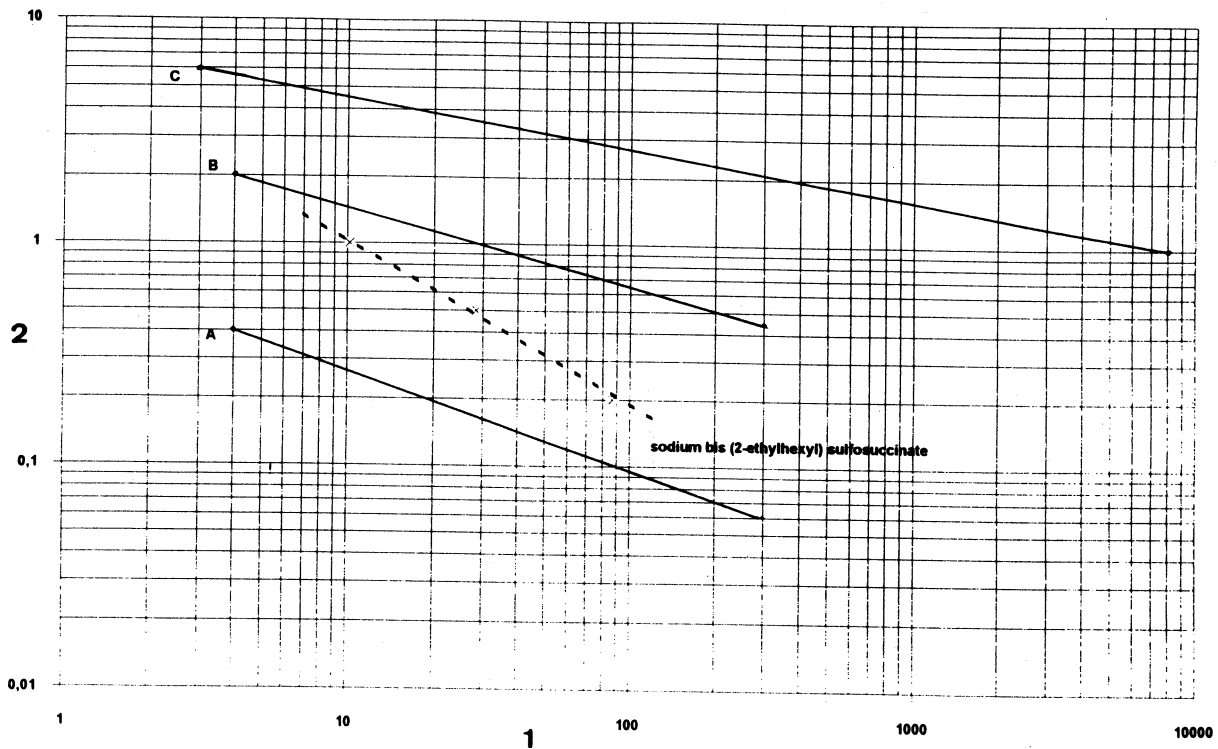


Figure 3 – Illustration of procedure



Key

- 1 Wetting time in seconds
- 2 Concentration in grams per litre

Figure 4 – “Wetting time/concentration” curves for surface active agents A, B and C, with standard curve for comparison

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7 Sampling

The surface active agent laboratory sample shall be prepared and stored in accordance with ISO 607.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,05 g, 5 g of the laboratory sample into a 100 ml beaker.

8.2 Preparation of the surface active agent solution

Dissolve the test portion (8.1) in water (5.1), if necessary after first making a paste of the surface active agent with water warmed to 40 °C, then diluting with water at about 20 °C. Transfer quantitatively to a 1 000 ml volumetric flask, make up to the mark with water and mix.

Take 200 ml of the solution thus obtained, transfer to a 1 000 ml volumetric flask, make up to the mark with water and mix.

If the Krafft-temperature of the surface active agent is higher than 40 °C, make the paste and carry out the dissolution at a temperature at least equal to the Krafft temperature.

Keep the solution at (20 ± 2) °C until the beginning of the test.

Prepare the solution not less than 15 min, but not more than 2 h, before the measurement.

NOTE Conditions other than those given above (concerning the hardness or pH of the water, temperature, possible assistants) can be chosen provided that they are noted in the test report.

8.3 Preparation of the discs of cotton control cloth

Using the punch (6.3), cut out discs of 30 mm diameter from the raw cotton cloth (5.3). It is very important to avoid touching the cloth with the fingers as the presence of fatty materials or perspiration on the surface of the cloth can affect the results.

8.4 Cleaning the apparatus

The success of the tests depends, to a certain extent, on the cleanness of the apparatus.

Before the test, and if possible overnight, leave a chromic/sulfuric acid mixture¹⁾, prepared by gently stirring sulfuric acid ($\rho_{20} = 1,84$ g/ml) into an equal volume of a saturated solution of potassium dichromate, to stand in the beaker (6.1). Then rinse the beaker with water (5.1) until all traces of acid have disappeared, finally rinsing with a small quantity of the solution under test.

Clean the gripper (6.2) using a volatile solvent, dry, then rinse with a small quantity of the solution under test.

For a particular surface active agent under test, the apparatus is only rinsed between measurements with the solution at a new concentration.

¹⁾ Other cleaning solutions can be used provided that they are noted in the test report.

8.5 Filling the measurement beaker

Using a measuring cylinder, introduce 700 ml of the test solution prepared in 8.2 into the measurement beaker (6.1).

During these operations, in order to avoid the formation of undesirable foam, it is recommended that the test solution be allowed to flow down the internal walls of the vessels. If necessary, remove any foam formed on the surface of the solution in the measurement beaker with a filter paper.

8.6 Determination (see figure 3)

Measure, to the nearest 1 °C, the temperature of the solution.

Clamp a raw cotton disc (8.3) in the gripper (6.2). Immerse the disc in the solution, starting the stopwatch (6.4) at the moment when the lower part of the disc touches the solution. Rest the support arms on the rim of the beaker and allow the gripper to open.

Stop the stopwatch when the disc begins to sink of its own accord.

In the case of solutions at high temperatures, it is important to wait at least 15 min after stabilization of the temperature before carrying out the measurement.

If the wetting time for the first test solution (concentration 1 g/l) is not about 300 s, adjust the mass of the test portion correspondingly and repeat the procedure until the test solution concentration has been found which gives a wetting time of about 300 s.

When this test solution concentration has been found, repeat the measurement nine times on this solution, but taking a fresh cotton disc for each measurement.

Calculate the arithmetic mean of the 10 measurements and record this as the wetting time for that particular test solution concentration.

Prepare, in the same way as for the first test solution, four further solutions of increasing concentration and determine the wetting time for each, using the procedure specified above. Ensure, by carrying out preliminary measurements if necessary, that the solution of highest concentration has a wetting time of $5 \text{ s} \pm 1 \text{ s}$. If this wetting time cannot be attained, use a saturated solution as the solution of highest concentration.

8.7 Calibration of the cloth

Determine, using the same procedure and at the same temperature as for the surface active agent under test, the wetting times for five solutions of the sodium bis (2-ethylhexyl) sulfosuccinate standard (5.2).

The first (least concentrated) solution of sodium bis (2-ethylhexyl) sulfosuccinate (5.2) shall be 0,2 g/l.

The calibration of the cloth is necessary only if a new batch of cotton control cloth is used, or if it is desired to compare the results obtained from two different control cloths.

9 Expression of results

On log-log paper, plot wetting time against concentration for the surface active agent under test and for the sodium bis (2-ethylhexyl) sulfosuccinate standard (see figure 4 for an example).

A comparison of the positions of the curves gives a reasonable estimate of the wetting power of the surface active agent under test.

10 Test report

The test report shall contain the following information:

- a) all details necessary for complete identification of the sample;
- b) the nature and characteristics of the raw cotton cloth from which the discs were cut (see annex A);
- c) the method used (a reference to this European Standard);
- d) the nature of the water used and, if appropriate, the assistants used;
- e) the exact temperature of the determination;
- f) the curves representing the results;
- g) details of any operations not specified in this European Standard or in the international standards to which reference is made, and details of any operations regarded as optional, as well as details of any incidents which can have influenced the results.

Annex A (informative)

Commercially available control cloths

A.1 German cloth

Unbleached cotton cloth:

- mass per unit area determined in accordance with ISO 3801: 494 g/m²;
- number of threads in warp determined in accordance with ISO 7211-2: 11 threads per centimetre;
- number of threads in weft determined in accordance with ISO 7211-2: 11 threads per centimetre;
- warp threads: 50 tex Z 330 x 4S 330; R 200 tex;
- weft threads: 50 tex Z 330 x 4S 330; R 200 tex.

A.2 French cloth

Unbleached cotton cloth pure superior American, without finish or charge:

- mass per unit area determined in accordance with ISO 3801: 300 g/m²;
- number of threads in warp determined in accordance with ISO 7211-2: 18 threads per centimetre;
- number of threads in weft determined in accordance with ISO 7211-2: 13 threads per centimetre;
- warp threads: 42 tex Z 550 x 2S 500; R 84,5 tex;
- weft threads: 42 tex Z 550 x 2S 500; R 84,5 tex.

Bibliography

- [1] ISO 3801, *Textiles - Woven fabrics - Determination of mass per unit length and mass per unit area.*
- [2] ISO 7211-2, *Textiles - Woven fabrics - Construction - Methods of analysis - Part 2: Determination of number of threads per unit length.*

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