H-13-33

International Standard 6837

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

Surface active agents — Water dispersing power in dry cleaning solvents

Agents de surface — Pouvoir de dispersion d'eau dans les solvants de nettoyage à sec

First edition — 1982-12-01

UDC 661.185 : 620.1 : 541.182.023 **Ref. No. ISO 6837-1982 (E)**

Descriptors : surfactants, tests, determination, emulsifying power, water, solvents, detergents.

Price based on 5 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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 6837 was developed by Technical Committee ISO/TC 91, *Surface active agents,* and was circulated to the member bodies in November 1981.

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

Australia	Hungary	Romania
Austria	Ireland	South Africa, Rep. of
Belgium	Japan	Spain
China	Korea Rep. of	Switzerland
Egypt, Arab Rep. of	Mexico	USSR
France	Netherlands	
Germany, F.R.	Poland	

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

INTERNATIONAL STANDARD ISO 6837-1982 (E)

Surface active agents — Water dispersing power in dry cleaning solvents

0 Introduction

Dry cleaning detergents, and related auxiliary products used for prespotting and prebrushing, may solubilize or emulsify greater or lesser amounts of water in organic solvents.

Water dispersing power is defined as the ability of a dry cleaning detergent to solubilize or emulsify water in dry cleaning solvents.

The purpose of the method described in this International Standard is to determine the water dispersing power in a simple and easy manner.

The method is not suitable for accurate measurement of the solubilizing power of surface active agents.

There is no value in knowing the exact rate of solubilization, as the action is quite different in the presence of fabrics. In the latter case, there are three effects which occur at different rates, i.e.:

a) relatively fast absorption of water by the textiles;

b) relatively slow solubilization of water in the solvent containing the cleaning detergent;

c) desorption of water from the textile due to its solubilization in the solvent containing the cleaning detergent.

Because of the larger active surface area, the rate at which effect c) occurs is greater than that at which effect b) occurs.

Furthermore, the method uses considerably greater quantities of solvent in order to reduce as much as possible the error due to azeotropic evaporation of the solvent.

The value of the water solubilizing power and of the water emulsifying power depends, to a large extent, on the procedure, and also on the concentration of surface active agents in the organic solvent, on the nature of the solvent, on the method of preparation of the mixture and on the temperature of the mixture.

In accordance with its definition, the water solubilizing power represents the maximum quantity of water which may be solubilized in the solvent by the surface active agent.

Similarly, the water emulsifying power represents the maximum quantity of water which may be emulsified in the solvent by the surface active agent. These properties, together with other properties, enable a dry cleaning detergent, a prespotting product or prebrushing product to be characterized.

1 Scope and field of application

This International Standard specifies a method for the determination of the water solubilizing power and the water emulsifying power of a dry cleaning detergent or similar auxiliary product with a usual concentration of 3 to 5 g/l in cleaning vats.

The method is applicable to other concentrations of auxiliary products in the solvent.

The method described in this International Standard is not recommended for higher concentrations of auxiliary products containing the surface active agent in the solvent and water, for example as is the case in chemical cleaning processes using brushing or spraying.

2 References

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*[1]

ISO 565, *Test sieves — Woven metal wire cloth, perforated plates and electroformed sheet — Nominal sizes of openings.*[2]

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

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 2456, *Surface active agents — Water used as a solvent for tests.*[3]

ISO 3819, *Laboratory glassware — Beakers.*[3]

ISO 4788, *Laboratory glassware — Graduated measuring cylinders.*

ISO 4797, *Laboratory glassware — Flasks with conical ground joints.*

1) At present at the stage of draft. (Partial revision of ISO/R 385-1964.)

2) At present at the stage of draft. (Revision of ISO 565-1972.)

3) At present at the stage of draft.

3 Definitions

3.1 dry cleaning detergent: Product designed to increase (intensify) and amplify the cleaning power of organic solvents by introducing water into the organic medium, thereby extending the cleaning power of the system to hydrophilic soil.

3.2 water dispersing power: The effectiveness of a dry cleaning detergent to bring about a homogeneous dispersion of water in organic solvents, either by solubilization or by emulsification.

3.3 water solubilizing power: Maximal amount of water which the dry cleaning detergent is able to solubilize in an organic solvent.

3.4 water emulsifying power: Maximal amount of water which the dry cleaning detergent is able to emulsify in order to form a W/O emulsion that is sufficiently stable.

NOTE — The emulsion is sufficiently stable for the intended purpose if it persists for at least 15 min.

3.5 spotting agent: Product used to eliminate stains which have not been removed by dry cleaning or washing.

3.6 prespotting product: Product containing in most cases surface active agent, used for the preparatory treatment of specific stains on textiles, furs and leather articles.

3.7 prebrushing agent: Preparation containing surface active agents which is used, either concentrated or mixed with water and/or solvents,[1] for the preparatory treatment of stained or heavily soiled areas on textiles, furs and leather articles. Application by brushing or spraying before cleaning in machine. Prespotting agents must be completely removable from the articles treated by rinsing in the following cleaning bath.

4 Principle

Preparation of several premixes with different water contents, containing relatively constant amounts of the detergent and solvent. Preparation, from these premixes, of dry cleaning solutions with the desired final concentration of detergent.

Determination of the water solubilizing power of the detergent by visual assessment of the optical density of the solutions, and, after adding water to those solutions that are still clear, determination of the solubilized water content for a given concentration of detergent.

Determination of the water emulsifying power of the detergent by observation of phase separation in the solutions, and, after adding water to those solutions in which the phases are considered not to have separated, determination of the emulsified water content for a given concentration of the detergent.

5 Reagents

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

5.2 Dry cleaning solvent.

5.3 Sudan III [Cerol scarlet] C.I. 26110.

6 Apparatus

Ordinary laboratory equipment, and in particular:

6.1 Conical flasks, of capacities 100 and 500 ml, with conical ground joints, complying with the requirements of ISO 4797.

6.2 Beakers, of capacities 150 and 400 ml, low form, complying with the requirements of ISO 3819.

6.3 One-mark pipettes, of capacities 5 and 10 ml, complying with the requirements of ISO 648.

6.4 Burette, of capacity 50 ml, complying with the requirements of ISO 385/1.

6.5 Graduated measuring cylinders, of capacities 100 and 500 ml, complying with the requirements of ISO 4788.

6.6 Magnetic stirrer, with a bar of length 30 mm, controlled at a rotational frequency of about 700 min^{-1}.

6.7 Sieving disc, of aperture size 630 µm, complying with the requirements of ISO 565, or other apertures of slightly lower dimension.

6.8 Illuminating box (see the figure), constructed of plywood or fibreboard, with ventilation holes on the upper and lower faces.

The interior of the box shall be painted matt black. A 100 W pearl glass lamp shall be fixed at the bottom of the box. The box shall be covered with two panes of glass of thickness approximately 3 mm. Between the two panes shall be placed a black card with a hole of diameter 45 mm in its centre, and covered with tracing paper of grammage 80 to 85 g/m^2.

6.9 Black card, of dimensions 100 mm × 200 mm, and thickness 0,3 to 0,5 mm, serving as a cover for protection against the light.

7 Sampling

Prepare and store the laboratory sample of dry cleaning detergent in accordance with the specifications of ISO 607.

1) The legislation of some countries forbids the use of mixes of prebrushing products with solvents, in particular with the cleaning solvent itself (perchloroethylene).

8 Procedure

8.1 Test portions

Weigh, to the nearest 0,01 g, 10 g of the laboratory sample into each of a series of 100 ml conical flasks (6.1).

8.2 Preparation of test solutions

Carry out all operations at a constant temperature of 23 ± 2 °C.

Add to the test portions (8.1), using a pipette (6.3), 5 ml of the dry cleaning solvent (5.2) [coloured for easier determination of the water emulsifying power by adding 0,025 g of the Sudan III (5.3) per litre of solvent].

Using the burette (6.4), add, to the nearest 0,05 ml, 1 ml of the water (5.1) to one of the above mixtures of surface active agent and solvent. Similarly add 3 - 6 - 9 - 12 - 15 ml - etc. of water, respectively, to the other mixtures.

After stoppering the conical flasks, stir for 3 min using the magnetic stirrer (6.6) at such a speed that there is no turbulence above the stirrer bar.

By means of a 100 ml measuring cylinder (6.5), add 55 ml of the solvent (5.2) (coloured for determination of water emulsifying power) to each of the above mixtures, stopper the conical flasks and stir for 3 min.

Immediately, using one of the pipettes (6.3), take 10 ml of each of the above mixtures (to give a final surface active agent concentration of 5 g/l) or 5 ml (to give a final concentration of 3 g/l) and transfer to a series of 400 ml beakers (6.2). Then, using the 500 ml measuring cylinder (6.5), add the volumes of colourless solvent (5.2) required to obtain the final desired concentration indicated in the table.

8.3 Determination of water solubilizing power

Stir the test solution (8.2) using a glass stirrer and rapidly pour, while still at 23 ± 2 °C, into a series of 150 ml beakers (6.2) until a liquid height of 5 cm is obtained.

Place the beakers successively over the hole in the black card of the illuminating box (6.8).

Immerse the sieving disc (6.7) in the test solution in the beaker and illuminate with the 100 W pearl glass lamp.

Observe, from a distance of 40 to 50 cm and vertically above the beaker, whether the mesh of the sieve can be distinguished, when the mesh is no longer clearly visible, there is an emulsion of water in the solvent.

From the premix solution in which the sieve mesh can still be clearly seen, prepare other test solutions as indicated in 8.2 by adding increasing quantities of water in successive 0,5 ml portions (equivalent to 0,25 g of water per litre for concentrations of 5 g of surface active agent per litre of solvent or to 0,15 g of water per litre for concentrations of 3 g/l).

Carry out the same observations as above until the mesh of the sieve can no longer be distinguished.

Note the concentration of water in the last solution in which the mesh of the sieve was still visible.

8.4 Determination of the water emulsifying power

Transfer the test solutions (8.2), coloured with the Sudan III (5.3), into a series of 500 ml conical flasks (6.1) and stopper.

Stir for 3 min using the magnetic stirrer (6.6) and transfer slowly the emulsion formed into a 500 ml measuring cylinder and allow to stand. After 15 min, observe at a temperature of 23 ± 2 °C, whether phase separation has occurred.

NOTE — Phase separation is more easily observed because of the red colour of the solvent. In the case of halogenated compounds, when all the water added to the solvent can no longer be emulsified, a thin upper layer is formed, usually consisting of a white emulsion which has separated from the red emulsion of the solvent.

It is also possible that instead of an emulsion being formed, flocculation occurs. In such cases, the water emulsifying power corresponds to the maximal amount of water which can be added without causing flocculation.

It is possible, for phase separation (emulsification or flocculation) to be observed at lower water concentrations than the water solubilizing power. In such cases, the water emulsifying power is equal to the solubilizing power.

Premix composition			Volume of solvent to be added, ml	
Cleaning detergent	Water	Solvent	5 ml sample	10 ml sample
g	ml	ml	Final concentration 3 g/l	Final concentration 5 g/l
10	0 to 1,5	60	235	280
10	1,5 to 3,5	60	230	280
10	3,5 to 7,5	60	225	270
10	7,5 to 12,5	60	210	250
10	12,5 to 17,5	60	200	240
10	17,5 to 22,5	60	185	220
10	22,5 to 27,5	60	175	210
10	27,5 to 35	60	165	200

From the premix solution which showed no phase separation, prepare other test solutions as indicated in 8.2 and 8.3.

Carry out the same observations as above, after allowing to stand for 15 min, until phase separation occurs.

Note the concentration of water in the last solution in which there was no phase separation.

9 Expression of results

9.1 Method of calculation and formulae

The water solubilizing power, S, expressed in grams of water per litre of solvent for a cleaning detergent concentration, is given by the formula

$$\frac{V \cdot c}{10}$$

where

V is the maximum volume of water in the premix solution, expressed in millilitres, determined in 8.3 or 8.4;

c is the concentration, in grams per litre, of the cleaning detergent submitted to the test.

The water emulsifying power, E, expressed in grams of water per litre of solvent for a cleaning detergent concentration, is given by the same formula.

9.2 Precision

9.2.1 Repeatability

The difference between two results obtained on the same sample, using identical apparatus in two different laboratories, shall not exceed 0,5 in the case of solubilizing power and 1 in the case of emulsifying power.

9.2.2 Reproducibility

The difference betweem two results obtained on the same sample, using identical apparatus in two different laboratories, shall not exceed 0,5 in the case of solubilizing power and 1 in the case of emulsifying power.

10 Test report

The test report shall include the following information:

a) all information necessary for the complete identification of the sample;

b) the reference of the method used (reference to this International Standard);

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

1) solubilizing power,

2) emulsifying power;

d) any operating details not specified in this International Standard, or regarded as optional, as well as any incidents which may have influenced the results.

Dimensions in millimetres

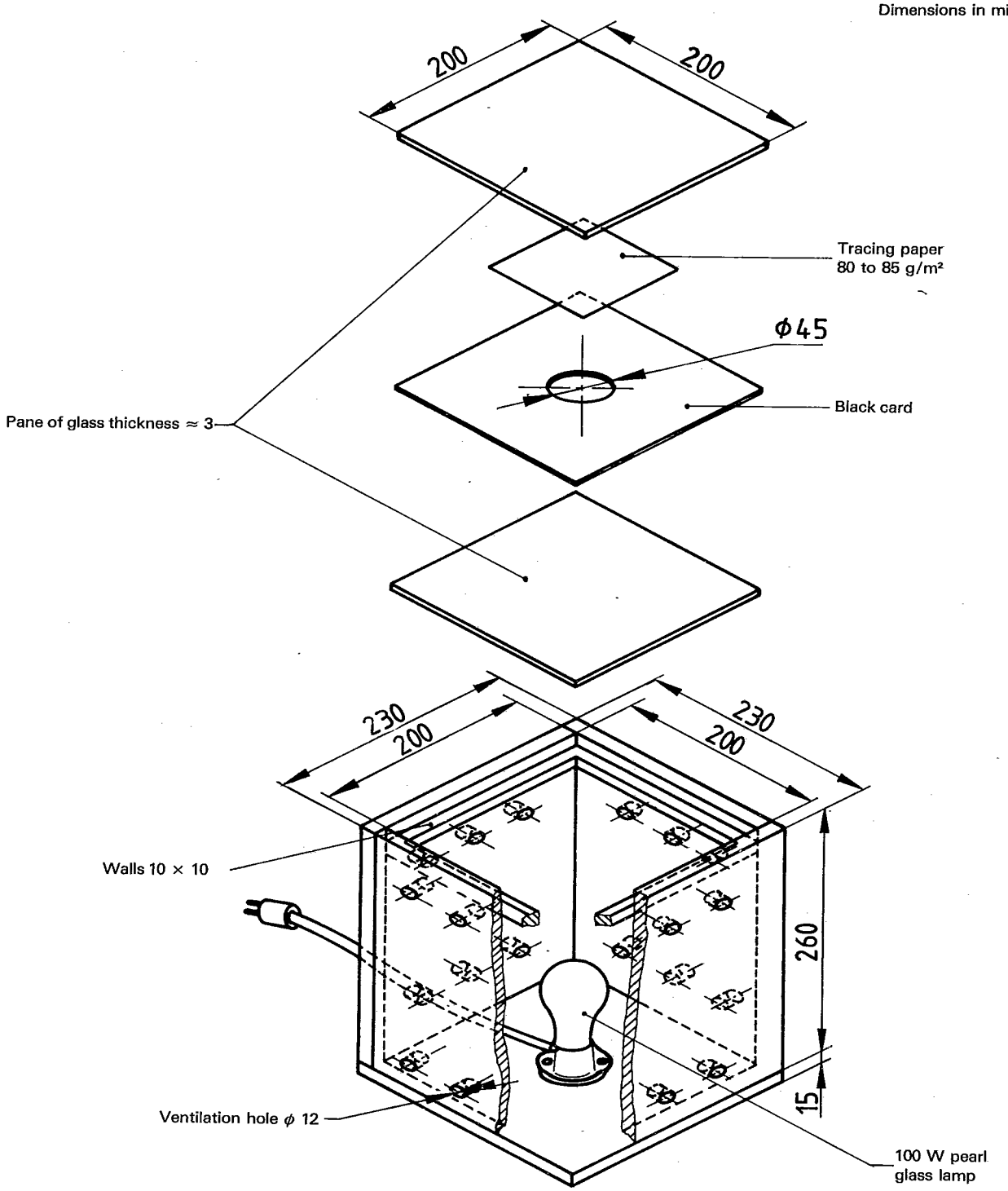

Figure — Illuminating box