H-14-06

International Standard ISO 6839

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

Anionic surface active agents — Determination of solubility in water

Agents de surface anioniques — Détermination de la solubilité dans l'eau

First edition — 1982-12-01

UDC 661.185 : 620.1 : 541.8 **Ref. No. ISO 6839-1982 (E)**

Descriptors : surfactants, anionic surfactants, tests, determination, solubility.

Price based on 3 pages

ISO 6839-1982 (E)

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 6839 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in February 1982.

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

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

No member body expressed disapproval of the document.

Printed in Switzerland

INTERNATIONAL STANDARD ISO 6839-1982 (E)

Anionic surface active agents — Determination of solubility in water

0 Introduction

The method described in this International Standard is one of the simplest of the methods which can be used for this purpose; it is sufficiently accurate and is suitable for a number of practical uses.

1 Scope and field of application

This International Standard specifies a method of establishing the curve representing the solubility of an anionic surface active agent in water as a function of temperature, and, consequently, of allowing evaluation of its solubility at a given temperature.

The method is applicable both to pure surface active agents and to technical products or formulations of liquid anionic surface active agents, provided that the solutions of these products are optically clear and are not very strongly coloured.

NOTE — The determination of solubility may be carried out without restriction in the temperature range from 0 to 90 °C; at temperatures lower than 0 °C, the determination is possible provided that the solution does not freeze.

The solubility curve obtained in the case of pure products may possibly allow the Krafft temperature to be determined.

2 Reference

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

3 Principle

Preliminary determination on an aqueous solution of known anionic surface active agent concentration of the temperatures at which the solution changes, on heating, from being cloudy to clear and, on cooling, from being clear to cloudy.

Placing in a bath, controlled at a temperature within the range established in the preliminary determination, of two solutions of the same concentration, one being colder and cloudy and the other being warmer and clear, and noting the appearance of the two solutions at temperature equilibrium.

Repetition of the test, varying the temperature of the bath within the range established by the preliminary determination, until the clear solution remains clear and the cloudy solution remains cloudy, or the solutions change very slowly from being cloudy to clear or vice versa.

From the surface active agent concentrations and the limiting temperatures of solubility, plotting the solubility curve.

4 Reagent

During the determination, use only distilled water or water of equivalent purity.

5 Apparatus

Usual laboratory equipment, and

5.1 Test tubes, made of borosilicate glass, of diameter 20 mm and length 200 mm.

5.2 Precision thermometers, complying with the requirements of ISO 653.

5.3 Thermostatically controlled water bath, capable of being controlled at −5 °C to + 90 °C, to within ± 0,1 °C, with a transparent cell.

6 Sampling

The laboratory sample of anionic surface active agent shall be prepared and stored in accordance with the instructions given in ISO 607.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, the quantity of laboratory sample corresponding to one of the surface active agent concentrations to be studied [concentrations usually between 1 and 50 % (*m/m*)] then prepare approximately 100 ml of solution.

If the solution contains dispersed impurities, it is advisable to filter it after heating to a temperature higher than that at which it is cloudy.

This procedure shall not involve any alteration of the surface active agent concentration.

7.2 Preliminary determination

Transfer approximately 10 ml of the test solution (7.1) to a test tube (5.1) and heat it directly over a flame until the solution becomes clear.

Allow to cool slowly in the ambient environment until it becomes cloudy, then slowly raise the temperature again, stirring the solution with a thermometer (5.2), and, as soon as the solution becomes clear, note the temperature (t_1).

Allow to cool slowly, stirring the solution with the thermometer, and, as soon as the solution becomes cloudy, note the temperature (t_2).

The temperature range thus determined is generally of the order of 10 °C.

7.3 Determination of the limiting temperature of solubility

Set the thermostatically controlled bath (5.3) at a temperature within the range defined by the preliminary determination (7.2) and keep it constant to within 0,1 °C.

Fill two test tubes (5.1) with the solution (7.1), stopper them, adjust their temperatures so that the solution in one becomes clear and the solution in the other becomes cloudy, and then place them in the thermostatically controlled bath.

When the temperatures of the two solutions are equal to that of the water bath, note whether the two solutions are clear or cloudy.

If both solutions are clear, lower the temperature of the water bath by a few degrees Celsius if both solutions are cloudy, raise it to a slightly higher temperature and repeat the above test.

Carry out a third test, or more if necessary, controlling the bath at a temperature deduced from the previous tests, until the change in appearance of the solutions (cloudy to clear or clear to cloudy) occurs very slowly or the clear solution remains clear and the cloudy solution remains cloudy. A maximum observation period of 2 to 3 h may be adopted.

Note the temperature at which this occurs to the nearest 0,1 °C, as the limiting temperature of solubility. If the appearance of the solutions remains unchanged, take the temperature immediately below that at which the two solutions remained cloudy.

NOTE — The speed with which the limpidity and cloudiness are reversed is a function of the temperatures t_1 and t_2 determined in 7.2 in relation to that of the bath.

7.4 Plotting the solubility curve

Repeat the above procedures (7.1, 7.2 and 7.3), using different quantities of test portion so as to cover the range of concentrations to be studied. Plot the solubility curve as a function of the concentrations and corresponding limiting temperatures of solubility.

This curve makes it possible to

— deduce the solubility of the surface active agent at a given temperature;

— determine, if required, the Krafft temperature.

8 Expression of results

8.1 Method of calculation

Express the solubility of the anionic surface active agent in water, for a given temperature, as a percentage by mass.

8.2 Precision

Comparative analyses on samples of three different surface active agents, carried out in eight laboratories, have provided the information given in the table.

9 Test report

The test report shall include the following information :

a) all the information necessary for the complete identification of the sample and details of the treatment of the test portion (and, if required, the temperature to which the solution was heated before filtration);

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

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

— temperature (t_1) at which the solution becomes cloudy for the concentration being studied;

— temperature (t_2) at which the solution becomes clear for the concentration being studied;

— limiting temperature of solubility for the concentration being studied;

d) if necessary, the solubility curve (and the Krafft temperature, if required);

e) details of any operations not specified in this International Standard or regarded as optional, together with details of any incidents likely to have affected the results.

Table

Surface active agent	Concentration	Mean observed limiting temperature of solubility	Standard deviation of reproducibility
	% (*m*/*m*)	°C	°C
Sodium laurate, purity 96,4 %	2,5	24	2
	5	27,7	1,7
	10	31,7	2
	20	38,6	1,6
	30	42,7	1,6
Sodium lauryl sulphate, purity 99 %	2,5	14	1,2
	5	15,3	1,6
	10	17	1,2
	20	19,8	1,3
	30	22,1	1,4
Sodium alkane sulphonate, purity 98 %	40	13,5	1,5
	41	17,7	1
	42	23	2,5
	43	34,7	3
	45	57,5	3,5