

Revised

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

**ISO RECOMMENDATION
R 696**

**SURFACE ACTIVE AGENTS
MEASUREMENT OF FOAMING POWER**

**1st EDITION
March 1968**

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BRIEF HISTORY

The ISO Recommendation R 696, *Surface active agents – Measurement of foaming power*, was drawn up by Technical Committee ISO/TC 91, *Surface active agents*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee ISO/TC 91 began in 1960 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In May 1966, this Draft ISO Recommendation (No. 985) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

| | | |
|----------------|----------------|-----------------------|
| Argentina | Hungary | Romania |
| Austria | Ireland | South Africa, Rep. of |
| Belgium | Israel | Spain |
| Brazil | Italy | Sweden |
| Canada | Japan | Switzerland |
| Chile | Korea, Rep. of | Turkey |
| Czechoslovakia | Netherlands | U.A.R |
| France | New Zealand | United Kingdom |
| Germany | Poland | U.S.S.R. |
| Greece | Portugal | Yugoslavia |

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in March 1968, to accept it as an ISO RECOMMENDATION.

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SURFACE ACTIVE AGENTS

MEASUREMENT OF FOAMING POWER

1. SCOPE

This ISO Recommendation describes a method of measurement of the foaming power of a surface active agent.

2. FIELD OF APPLICATION

This method is applicable to all surface active agents. Measurement of the foaming power of solutions of readily hydrolysable agents, however, does not give reliable results, as the hydrolysis products collect in the films of liquid and affect the persistence of the foam.

NOTE. — The persistence of liquid films is very sensitive to the presence of particles of insoluble matter. This method of measurement of foaming power should be used only with the greatest of care, therefore, for measuring the foaming power of compositions based on surface active agents of which the solubility is rarely complete.

Foaming power is also very sensitive to small variations in composition. Consequently, the results obtained on formulated products should be interpreted with caution.

This method is not applicable for measurement of the foaming power of very dilute solutions of surface active agents, such as river waters containing surface active agents.

3. DEFINITIONS

3.1 *Foaming power.* Ability to produce foam.

NOTE. — In this ISO Recommendation, foaming power is characterized by the volume of foam obtained under specific experimental conditions. The decay of this volume of foam during the 5 minutes following its formation is also relevant.

3.2 *Foam.* A mass of small gas cells, separated by thin films of liquid and formed by the juxtaposition of bubbles, giving a gas dispersed in a liquid.

4. PRINCIPLE

Measurement of the volume of foam obtained after running 500 ml of a solution of a surface active agent, from a height of 450 mm, on to a liquid surface of the same solution.

5. APPARATUS

5.1 Test apparatus

5.1.1 Components of apparatus (see Fig. 1 and 2)

- 5.1.1.1 *Separating funnel* of 1 litre capacity, consisting of a pear-shaped bulb to the upper end of a tube about 200 mm long, having a tap at the lower end. The funnel carries a mark, 150 mm above the axis of the tap, which indicates the lower limit of discharge during the test. The lower end of the tube is cut off strictly perpendicular to its length, 40 mm below the axis of the tap. The tap is moulded, not blown, the hole through the key being of sufficient diameter (not less than 3 mm) to avoid undue obstruction of the flow of the liquid.
- 5.1.1.2 *Measuring cylinder* of 1 litre capacity, complying with ISO Recommendation R...*, with graduations at each 10 ml. The measuring cylinder stands in a water-bath fitted with a thermostat, the bath being sufficiently large to accommodate the measuring cylinder immersed up to half its height.
- 5.1.1.3 *Stand* comprising a vertical rod, sufficiently long to allow the separating funnel and the measuring cylinder to be held in place. To ensure that the assembly is centred and so maintained during the measurement, the separating funnel is held by means of two rings, one supporting the spherical part, the other of much smaller diameter placed as low as possible around the stem of the separating funnel, below the tap. The measuring cylinder is held in place by means of a screw-clamp with one movable jaw.
- 5.1.1.4 *Stainless steel metering tube*, 70 mm long, 1.9 ± 0.02 mm internal diameter and 0.3 mm wall thickness. The ends of the tube should be cut accurately at right angles to the axis of the tube, in a precision tool lathe.

The metering tube is a push fit in a steel mounting tube 5 to 10 mm long, of internal diameter equal to the external diameter of the metering tube, and of external diameter equal to that of the lower end of the glass tube of the separating funnel. The upper ends of the metering tube and of the mounting tube should be in the same plane. The mounting tube is fixed by means of a short length of thick rubber tube (vacuum tubing) so that the upper end of the mounting tube is in contact with the lower end of the glass tube.

5.1.2 Cleaning of apparatus

Perfect cleanliness of the apparatus is essential for the success of the test

Before the test, and, if possible, overnight, leave all glassware in contact with chromic-sulphuric acid mixture, prepared by slowly stirring concentrated sulphuric acid ($\rho_{20} = 1.83$ g/ml), into an equal volume of a saturated solution of potassium dichromate. Rinse the apparatus first in distilled water until free from acid and then with a small quantity of the solution under test.

Keep the mounting tube and metering tube assembly for 30 minutes in the vapour of an azeotropic mixture of ethanol and trichlorethylene, then rinse it with a small quantity of the solution under test.

Between each measurement, *of the same product*, simply rinse the apparatus with the solution under test. When it is required to remove the foam remaining in the measuring cylinder, no matter what method is employed to do so, follow by a rinse with the solution under test.

* At present in the form of a draft proposal.

5.2 Usual laboratory apparatus, including :

- 5.2.1 *Graduated measuring cylinder, 500 ml.*
- 5.2.2 *Graduated measuring cylinder, or pipette, 50 ml.*
- 5.2.3 *Volumetric flask, 1,000 ml.*
- 5.2.4 *Beaker.*

6. PREPARATION OF A SOLUTION

Prepare a solution of the material at the working strength.

The water used for dilution may be either distilled water saturated with air by bubbling, or hard water containing 300 parts per million of calcium carbonate.

Prepare the solution by pasting and then dissolution in the water chosen, previously warmed to 50°C. It is necessary to mix very gently to prevent the formation of foam. Keep the solution at $50 \pm 2^\circ\text{C}$ without stirring, until the test is made.

The age of the solution, at the time of the measurement, should be not less than 30 minutes nor greater than 2 hours.

Other conditions than those specified above (for example, hardness of the water, temperature) may be chosen provided that they are mentioned in the test report.

7. PROCEDURE

7.1 Assembly of apparatus

The apparatus should be assembled in a place free from draughts.

- 7.1.1 Adjust the thermostat of the water-bath to bring the temperature of the bath to $50 \pm 2^\circ\text{C}$.
- 7.1.2 Introduce 50 ml of the solution, prepared as described in Section 6, into the measuring cylinder, running it down the inside wall so that no foam is formed on its surface.
- 7.1.3 Place the measuring cylinder in the water-bath and hold it in place by means of the screw-clamp.
- 7.1.4 Mount the separating funnel with the metering tube attached and adjust the support, so that the axes of the measuring cylinder and the metering tube are co-incident, and also that the lower end of the metering tube is 450 mm above the level of the 50 ml of solution, previously introduced into the measuring cylinder.

7.2 Filling the apparatus

- 7.2.1 For the first measurement, introduce part of the test solution into the separating funnel up to the 150 mm mark. To do this, immerse the lower end of the metering tube in a portion of the test solution maintained at $50 \pm 2^\circ\text{C}$ in the beaker and aspirate the liquid by suitable means attached to the top of the bulb. This is the most certain way to avoid the formation of air-bubbles in the hole through the key of the tap. The beaker is kept below the separating funnel until the measurement is made.

To complete the filling, pour 500 ml of the test solution, maintained at $50 \pm 2^\circ\text{C}$, into the separating funnel from the 500 ml graduated cylinder, performing this operation gently to avoid the formation of foam. This may be achieved by the use of a special funnel with a curved stem, the end of which touches the interior wall of the separating funnel.

7.2.2 For subsequent measurements, empty the separating funnel down to a height of 1 to 2 cm above the tap. Place the small beaker full of the test solution, kept at $50 \pm 2^\circ\text{C}$ under the separating funnel as before. Fill the separating funnel with the test solution up to the 150 mm mark, then pour in 500 ml of the test solution, maintained at $50 \pm 2^\circ\text{C}$ as described above.

NOTE. — Filling to the 150 mm mark may also be achieved by not allowing the separating funnel to empty completely after the previous filling with the test solution. This simpler method gives less guarantee of freedom from air bubbles.

7.3 Measurement

Allow the solution to flow, without interruption, until the level falls to the 150 mm mark. Note *the time of efflux*. All measurements in which the time of efflux differs by more than 5 % from the arithmetic mean of previously observed efflux times should be neglected, an abnormally long time indicating the presence of an air-bubble in the metering tube or in the tap. Measure the volume of foam (*only the foam*) at 30 seconds, 3 minutes and 5 minutes after the efflux has been stopped.

If the upper level of the foam has a depression in the centre, record the reading as the arithmetic mean between the centre and the edges. *

Repeat the measurement ten times, preparing a fresh solution each time as described in section 6. Take the arithmetic mean of at least 8 results.

8. EXPRESSION OF RESULTS

Express the results in millilitres of foam formed 30 seconds, 3 minutes and 5 minutes after stopping the efflux. Then draw the corresponding curve.

9. TEST REPORT

The test report should give the method used and the results obtained. It should also mention

- the concentration of the test solution, expressed in grammes of surface active agent per litre,
- the temperature in degrees Celsius during the test, if it is different from that recommended, **
- the hardness of the water actually used, expressed in parts of calcium carbonate per million, if this differs from that recommended.

Finally, all operating details not covered by this ISO Recommendation, or any which are optional, as well as any other factors that may have affected the results, should be reported.

The report should include all details required for complete identification of the sample.

* It may be of assistance, in making this measurement, to use a white varnished slip carrying a longitudinal black varnished line. This slip is placed parallel to the axis of the receiving cylinder, and the lower level of the volume of foam to be measured is taken at the point where contrast between black and white is visible.

** Curves showing the relation between foaming power and temperature of different products can vary considerably in slope and also in general shape. A comparison of several surface active agents, according to their foaming power, cannot therefore be carried out unless this curve is prepared or at least three points upon it are given.

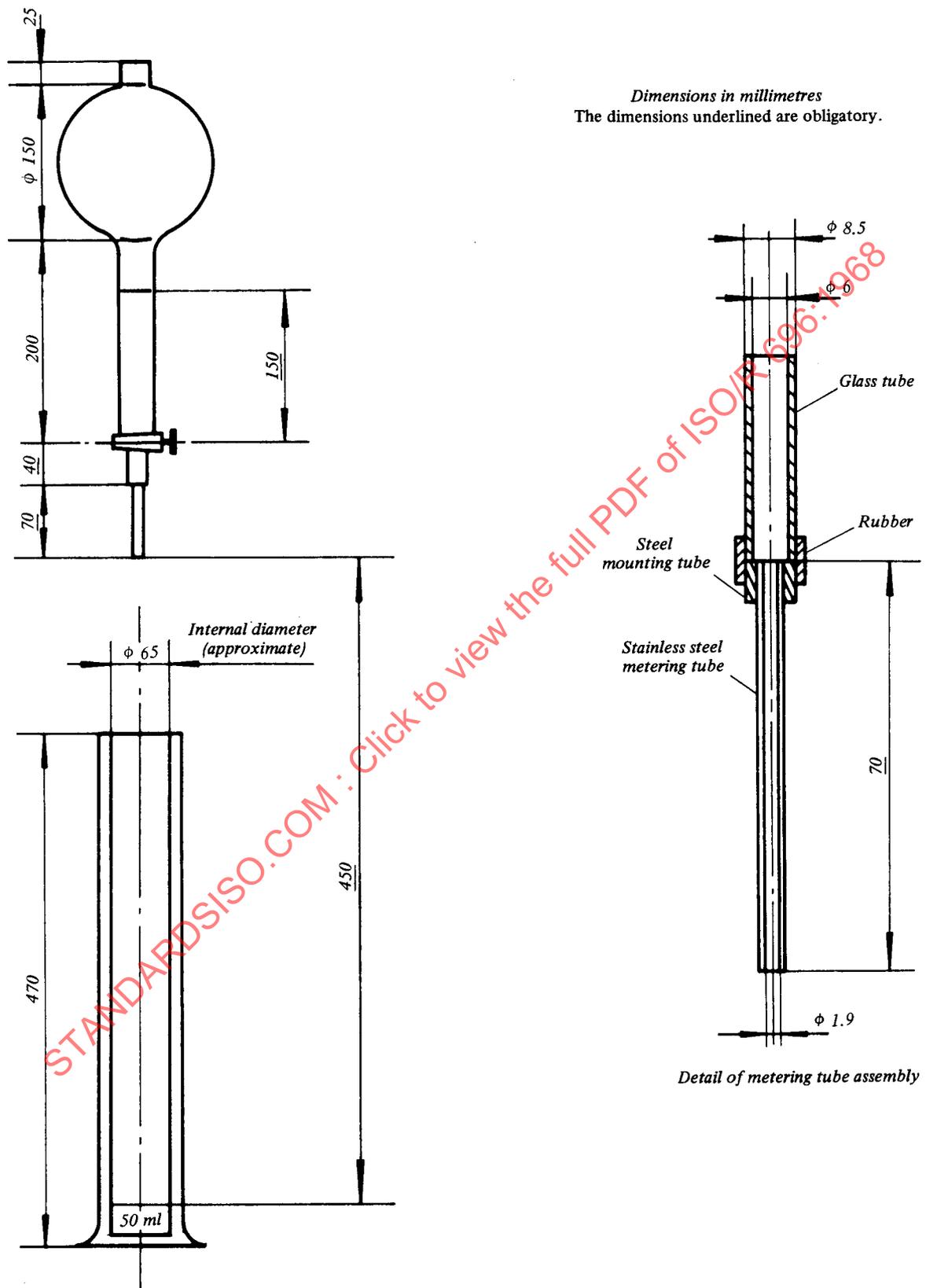


FIG. 1. — Test apparatus (not to scale).