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Surface active agents — Measurement of foaming power — Modified Ross-Miles method

*Agents de surface — Mesurage du pouvoir moussant — Méthode de
Ross-Miles modifiée*

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Descriptors : surfactants, tests, measuring, foaming power.

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 696 was drawn up by Technical Committee ISO/TC 91, *Surface active agents*. It was submitted directly to the ISO Council, in accordance with clause 6.11 of the Directives for the technical work of ISO.

This International Standard cancels and replaces ISO Recommendation R 696-1968, which had been approved by the Member Bodies of the following countries :

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

No Member Body had disapproved the Recommendation.

Surface active agents — Measurement of foaming power — Modified Ross-Miles method

0 INTRODUCTION

The method specified in this International Standard makes it possible to check one of the characteristics of surface active agents which it is important to take into account in order to assess their potential use.

The precisely defined conditions specified for this method do not necessarily correspond to practical conditions of use. Consequently, the results obtained do not necessarily provide information regarding the behaviour of products under actual conditions of application. The use of this method, and the assessment of the results obtained, must therefore depend upon the aims sought after. In particular, application of the method to products having a low foaming power may lead to results which have no practical interest.

1 SCOPE

This International Standard specifies a method of measurement of the foaming power of a surface active agent.

2 FIELD OF APPLICATION

The 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 solution 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.

The 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 REFERENCES

ISO 607, *Surface active agents — Detergents — Methods of sample division.*¹⁾

ISO/R 862, *Surface active agents — Glossary.*

ISO/R 1042, *One-mark volumetric flasks.*

ISO 2174, *Surface active agents — Preparation of water with known calcium hardness.*

ISO . . . , *Graduated measuring cylinders.*²⁾

4 DEFINITIONS

4.1 foaming power : The ability to produce foam.³⁾

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

4.2 foam : A mass of gas cells separated by thin films of liquid and formed by the juxtaposition of bubbles, giving a gas dispersed in a liquid.³⁾

5 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.

6 APPARATUS

Ordinary laboratory apparatus, and

6.1 Test apparatus

6.1.1 Components of apparatus (see figures 1 and 2)

6.1.1.1 Separating funnel of 1 l capacity, consisting of a spherical bulb joined to the upper end of a tube about 200 mm long, having a tap at the lower end. The separating funnel carries a mark, 150 mm above the axis of the tap,

1) In preparation. (Revision of ISO/R 607.)

2) In preparation.

3) Definition taken from ISO/R 862.

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.

6.1.1.2 Measuring cylinder of 1 l capacity, complying with ISO . . . , 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.

6.1.1.3 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 a ring supporting the spherical part, and a clamp placed as low as possible around the stem of the separating funnel, near the tap. The measuring cylinder is held in place by means of a screw-clamp with one movable jaw.

6.1.1.4 Metering tube, stainless steel, 70 mm long, $1,9 \pm 0,02$ mm internal diameter and 0,3 mm wall thickness. The ends of the tube shall be cut accurately at right angles to the axis of the tube, on 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 low end of the glass tube of the separating funnel. The upper ends of the metering tube and of the mounting tube shall 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.

6.1.2 *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 (ρ_{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 min in the vapour of an azeotropic mixture of ethanol and trichlorethylene, then rinse it with a small quantity of the solution under test.

Between successive measurements, *on 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.

6.2 Graduated measuring cylinder, capacity 500 ml.

6.3 Graduated measuring cylinder, or **pipette**, capacity 50 ml.

6.4 Volumetric flask, capacity 1 000 ml, complying with ISO/R 1042.

7 SAMPLING

The laboratory sample of surface active agent shall be prepared and stored by the procedures specified in ISO 607.

8 PREPARATION OF THE TEST SOLUTION

Prepare, from the laboratory sample, a solution of the material at its working strength.

The water used for dilution may be either distilled water saturated with air by bubbling, or hard water containing 6 meq of calcium(II) ions per litre (see ISO 2174).

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 ± 2 °C without stirring, until the test is made.

The age of the solution, at the time of the measurement, shall be not less than 30 min and not greater than 2 h.

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.

9 PROCEDURE

9.1 *Assembly of apparatus*

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

Adjust the thermostat of the water bath to bring the temperature of the bath to 50 ± 2 °C.

Introduce 50 ml of the solution, prepared as described in clause 8, into the measuring cylinder (6.1.1.2), running it down the inside wall so that no foam is formed on the surface.

Place the measuring cylinder in the water bath and hold it in place by means of the screw-clamp.

Mount the separating funnel (6.1.1.1) with the metering tube (6.1.1.4) attached and adjust the support (6.1.1.3), so that the axes of the measuring cylinder and the metering tube coincide, and also that the lower end of the metering tube is 450 mm above the level of the 50 ml of solution in the measuring cylinder.

9.2 Filling the apparatus

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 a small beaker and aspirate the liquid by suitable means attached to the top of the separating funnel. This is the most certain way to avoid the formation of air-bubbles in the hole through the key of the tap. Keep the small beaker 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 measuring cylinder (6.2), 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.

For subsequent measurements, empty the separating funnel down to a height of 10 to 20 mm above the tap. Place the 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.

9.3 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 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) 30 s, 3 min and 5 min 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.¹⁾

Carry out ten measurements, preparing a fresh solution each time as described in clause 8. Take the arithmetic mean of at least eight results.

10 EXPRESSION OF RESULTS

Express the results in millilitres of foam formed 30 s, 3 min and 5 min after stopping the efflux. If necessary draw the corresponding curve.

11 TEST REPORT

The test report shall contain the following information :

- a) all information necessary for complete identification of the sample;
- b) the concentration of the test solution, expressed in grams of surface active agent per litre;
- c) the temperature, in degrees Celsius, during the test, if it is different from that recommended²⁾;
- d) the hardness of the water actually used, expressed in milligram equivalents of calcium(II) ions per litre, if this differs from that recommended;
- e) the results, and the method of expression used;
- f) the reference of the method used;
- g) all operating details not covered by this International Standard, or any which are optional, as well as any other factors that may have affected the results.

1) 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.

2) 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.

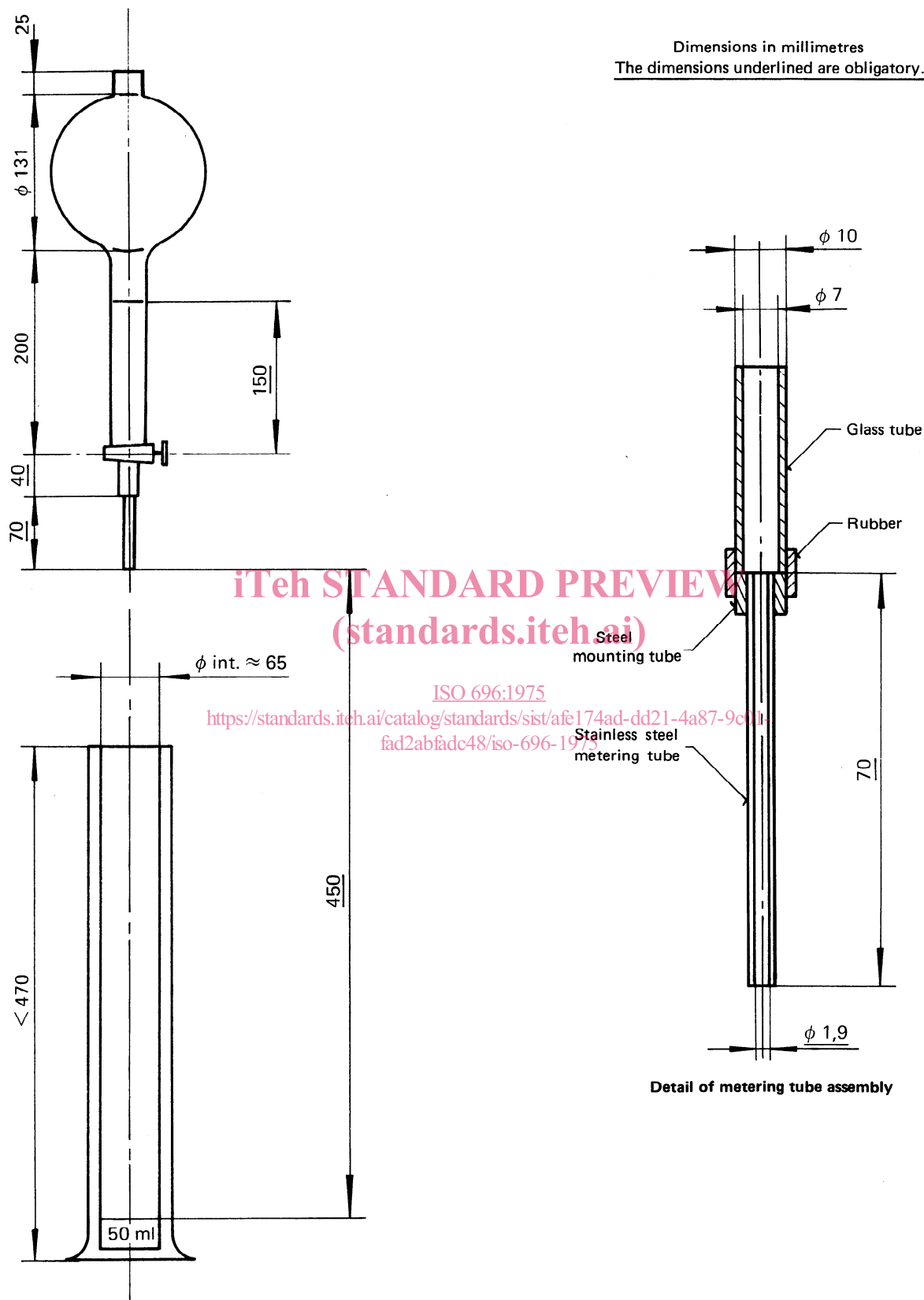


FIGURE 1 — Test apparatus (6.1) (not to scale)

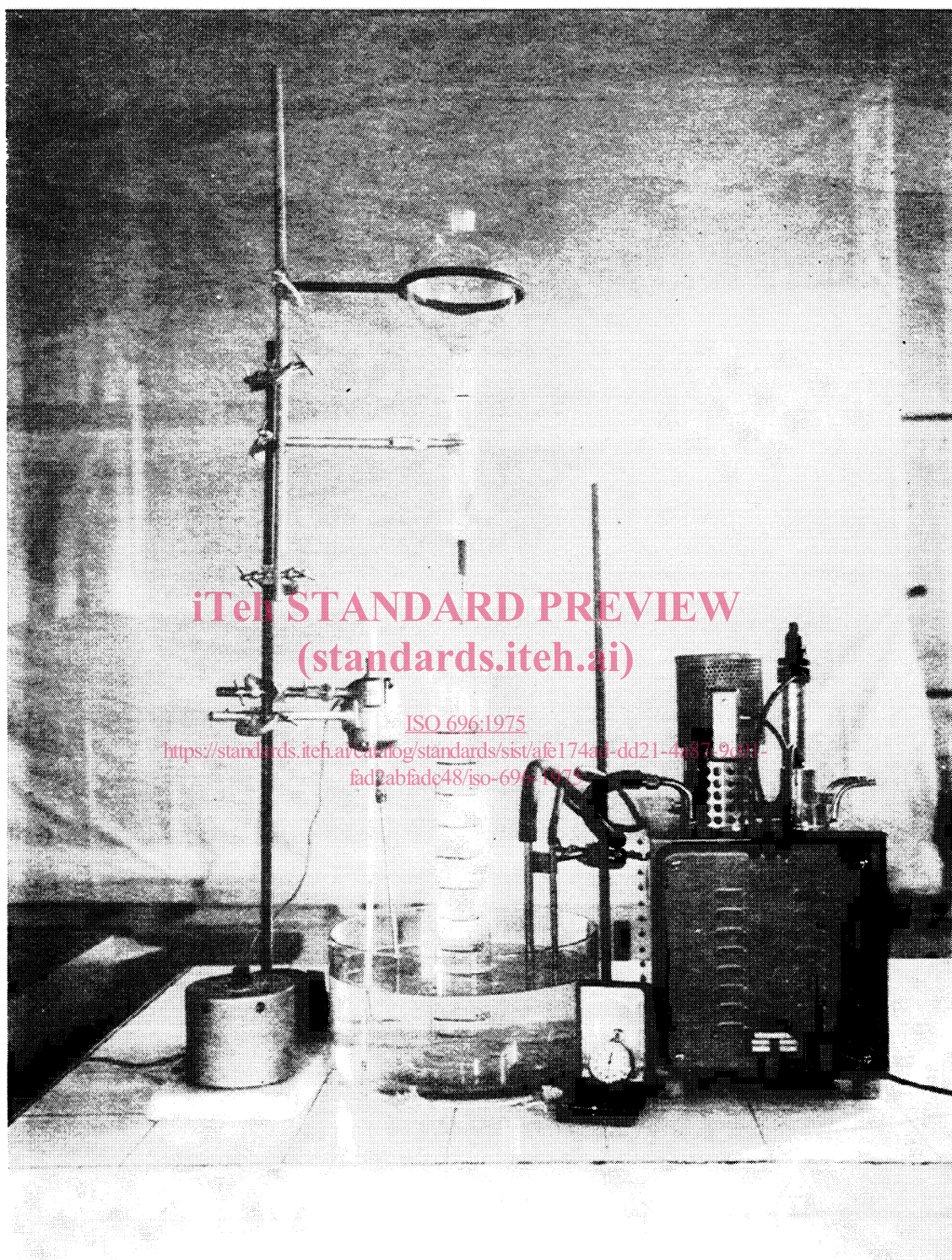


FIGURE 2 — Test apparatus — Photograph showing example of assembly

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