INTERNATIONAL STANDARD 1063

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

Surface active agents — Determination of stability in hard water

Agents de surface — Détermination de la stabilité à l'eau dure

First edition — 1974-12-15

UDC 661.185

Ref. No. ISO 1063-1974 (E)

Descriptors : surfactants, chemical tests, stability, hardness of water.

Price based on 3 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 1063 was drawn up by Technical Committee ISO/TC 91, *Surface active agents*. It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

This International Standard cancels and replaces ISO Recommendation R 1063-1969, which had been approved by the Member Bodies of the following countries :

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

No Member Body had expressed disapproval of the Recommendation.

Printed in Switzerland

Surface active agents — Determination of stability in hard water

0 INTRODUCTION

A knowledge of the stability of surface active agents in hard water is of great importance for all the applications of these substances, since the precipitates obtained with hard water can reduce their effectiveness in practice.

Systematic investigations have shown that, in many tests, there is no essential difference between calcium hardness and magnesium hardness, so that these tests are carried out with an aqueous solution of calcium chloride of known hardness.

If, in certain cases, it is necessary to use other ions that give rise to hardness in water, this fact shall be mentioned in the test report.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for assessing the stability in hard water of surface active agents which are readily soluble at ambient temperature or slightly higher temperatures.

This method is applicable to surface active agents soluble in water at 20 °C. It can be extended to those which are soluble at approximately 50 °C.

2 REFERENCES

ISO/R 835, *Graduated pipettes (excluding blowout pipettes).*

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

3 TERMINOLOGY

3.1 The **hardness of water** is due to the presence of soluble alkaline earth compounds and especially of calcium salts; it is expressed in milligram equivalents of calcium(II) ions per litre[1].

3.2 The **stability of a surface active agent in hard water** is determined by the solubility of the compounds formed by ion exchange between the surface active agent and the calcium ions, or by the modification of the colloidal state by ionic forces, salt effect, etc.

4 PRINCIPLE

Mixing of a solution of the surface active agent in differing concentrations with hard water solutions of differing known calcium hardness.

After leaving the solutions to stand in specified conditions, observation of their appearance, i.e. clearness, opalescence, cloudiness or precipitation.

5 REAGENTS

The reagents shall be of recognized analytical quality. Distilled water or water of at least equivalent purity shall be used.

5.1 Hard water solutions, prepared according to ISO 2174.

— Solution S_1, of calcium hardness 6 meq of calcium(II) ions per litre.

— Solution S_2, of calcium hardness 9 meq of calcium(II) ions per litre.

— Solution S_3, of calcium hardness 12 meq of calcium(II) ions per litre.

6 APPARATUS

Ordinary laboratory apparatus, and in particular :

6.1 Fifteen test tubes, for example 30 mm in diameter and 200 mm long, graduated at 50 ml.

Experience has shown that flat-bottomed test tubes are preferable, as they make it easier to observe cloudiness or precipitates.

6.2 Pipette, 5 ml, graduated at every 0,05 ml, complying with ISO/R 835.

6.3 Thermostatically controlled water bath for measurements carried out above 20 °C.

1) See ISO 2174, which gives, in an appendix, a conversion table indicating the correlation between the various units of measurement of the hardness of water.

7 PROCEDURE

7.1 Preparation of test solution

Prepare a stock solution of 50 g of the surface active agent in 1 000 ml of water at 20 °C. If the products are not readily soluble at 20 °C, prepare the solution at 50 °C. This temperature shall be stated in the test report.

7.2 Determination

Using the pipette (6.2), transfer 5,0 ml of the test solution (7.1) to one of the test tubes (6.1) and add the hard water solution S_1 (5.1) to give a volume of 50 ml.

Formation of foam may be troublesome and should be avoided when mixing the test solution with the hard water solution. To achieve this, close the test tube containing the mixture with the hand or a bung, turn it slowly upside down and bring it back slowly to the original position. This operation should take 1 s; repeat it ten times.

Leave the test tube to stand for more than 1 h but less than 2 h at 20 ± 2 °C and examine at this temperature for precipitates, cloudiness or opalescence[1]. If it appears that the solubility of the calcium slats increases with temperature, carry out the test at 50 ± 3 °C and make the observation at this temperature.

Carry out the determination similarly with 2,5 ml, 1,2 ml, 0,6 ml and 0,3 ml of the test solution (7.1).

Proceed similarly with identical volumes of the test solution (7.1) and with the hard water solutions S_2 and S_3.

7.3 Scoring

Allot to the result of each determination a score number in accordance with the numbering system in table 1.

TABLE 1 – **Score number corresponding to the appearance of the liquid**

Appearance of the liquid	Score number (unit value)
Clear	5
Opalescent	4
Cloudy	3
Slight precipitate	2
Heavy precipitate	1

In case of doubt between the score numbers (for example, cloudiness and slight precipitate), choose the less favourable score number.

A liquid which is not clear, but through which objects can be seen, is regarded as opalescent.

A liquid which is not clear, and through which objects cannot be seen, is regarded as cloudy.

NOTE – Although solutions in hard water of certain commercial surface active agents containing insoluble inorganic products are perfectly transparent, small quantities of crystals may appear at the bottom of the test tube or on the surface of the liquid. These crystals are entirely different from the colloidal precipitates arising from hardness of the water and it is necessary to centrifuge the solution, before testing, until it is clear.

8 EXPRESSION OF RESULTS

8.1 Mean stability

In general, a surface active agent is given a **single** stability figure which represents the "mean stability".

Add the 15 unit values obtained as described in 7.3 to obtain the total value, and calculate the mean stability, as shown in table 2.

TABLE 2 – **Mean stability**

Sum of the 15 unit values	Mean stability
15 to 18	"one"
19 to 37	"two"
38 to 56	"three"
57 to 74	"four"
75	"five"

8.2 Differential stability

In some cases, however, it can be useful to note the stability of a surface active agent in hard water in terms of water hardness.

For each of the three solutions S_1, S_2 and S_3, and in increasing order of hardness, add the five unit values obtained and determine the partial stability for each solution as shown in table 3.

TABLE 3 – **Differential stability**

Sum of the five partial values for each sample of hard water	Partial stability	
5 or 6	one	$= \bar{1}$
7 to 12	two	$= \bar{2}$
13 to 18	three	$= \bar{3}$
19 to 24	four	$= \bar{4}$
25	five	$= \bar{5}$

Three figures are thus obtained, expressing the stability for each of the three hardnesses, 6 meq/l, 9 meq/l and 12 meq/l, and characterizing the "differential stability". The latter is, for example, $\bar{1}11$ for the worst stability and $\bar{5}\bar{5}\bar{5}$ for the best stability in hard water.

1) In the case of a solution which changes with time (soap solution for example), the age of the solution, to the nearest 5 min, shall be stated in the test report.

9 TEST REPORT

The test report shall indicate the results obtained as follows :

— mean stability at t °C : **one** . . . **five**,

or

— differential stability at t °C : $\overline{111}$ . . . $\overline{555}$.

It shall also state the temperature of measurement and mention any operation not included in this International Standard, or regarded as optional, as well as any circumstances which may have influenced the results.

The test report shall give all details required for complete identification of the sample.

This page intentionally left blank

This page intentionally left blank

This page intentionally left blank