
**Surface active agents — Detergents —
Determination of cationic-active matter
content —**

**Part 2:
Cationic-active matter of low molecular
mass (between 200 and 500)**

*Agents de surface — Détergents — Détermination de la teneur en
matière active cationique —*

*Partie 2: Matière active cationique à faible masse moléculaire (entre
200 et 500)*



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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2871-2 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 2871-2:1990), of which it constitutes a minor revision.

ISO 2871 consists of the following parts, under the general title *Surface active agents — Detergents — Determination of cationic-active matter content*:

- *Part 1: High-molecular-mass cationic-active matter*
- *Part 2: Cationic-active matter of low molecular mass (between 200 and 500)*

Surface active agents — Detergents — Determination of cationic-active matter content —

Part 2: Cationic-active matter of low molecular mass (between 200 and 500)

1 Scope

This part of ISO 2871 specifies a method for the determination of low-molecular-mass cationic-active materials such as monoamines, amine oxides, quaternary ammonium compounds and alkylpyridinium salts which have a main chain of 10 to 22 carbon atoms and not more than 6 other carbon atoms in the cation.

The method is also suitable for other cationic-active materials.

The method is applicable to solids or to aqueous solutions of the active material when the relative molecular mass of the cationic-active matter is known or when it has been previously determined if its content is expressed as a percentage by mass. If more than one type of cationic-active material is present, an estimate of average relative molecular mass may be used.

The method is not applicable if anionic and/or amphoteric surface active agents are present.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

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

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 2271:1989, *Surface active agents — Detergents — Determination of anionic-active matter by manual or mechanical direct two-phase titration procedure*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

The cationic-active matter in a sample is titrated in a two-phase (aqueous chloroform) system against a standard anionic surface active agent in the presence of an indicator consisting of mixed anionic and cationic dyes. The cationic surface active agent present in the sample initially reacts with the anionic dye to form a salt which dissolves in the chloroform layer, imparting a blue colour to this layer. During the titration, the anionic surface active agent displaces the anionic dye and, at the end point, forms a salt with the cationic dye, imparting a greyish-pink colour to the chloroform layer.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of at least equivalent purity complying with the specifications for grade 3 of ISO 3696.

4.1 Chloroform, $\rho_{20} = 1,48$ g/ml, distilling between 59,5 °C and 61,5 °C.

4.2 Sodium lauryl sulfate (sodium dodecyl sulfate) $[\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}]$, standard volumetric solution, $c(\text{C}_{12}\text{H}_{25}\text{NaO}_4\text{S}) = 0,004$ mol/l.

While preparing the standard volumetric solution as described in 4.2.2, check the purity of the solid sodium lauryl sulfate used as described in 4.2.1.

4.2.1 Determination of purity of sodium lauryl sulfate.

Weigh, to the nearest 1 mg, $5 \text{ g} \pm 0,2 \text{ g}$ of the solid product into a 250 ml round-bottomed flask with ground-glass neck. Add exactly 25 ml of a standard volumetric sulfuric acid solution, $c(\text{H}_2\text{SO}_4) = 1$ mol/l, and reflux using a water condenser. During the first 5 min to 10 min, the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask.

In order to avoid excessive foaming, instead of refluxing, the solution may be left on a boiling water bath for 60 min.

After a further 10 min, the solution will become clear and foaming will cease. Reflux for a further 90 min.

Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water.

Add a few drops of ethanolic phenolphthalein solution (concentration 10 g/l), and titrate the solution with sodium hydroxide solution $c(\text{NaOH}) = 1$ mol/l.

Carry out a blank test by titrating 25 ml of the 1 mol/l sulfuric acid solution with the 1 mol/l sodium hydroxide solution.

Calculate the purity, τ , expressed as a percentage by mass, of the sodium lauryl sulfate using the formula

$$\frac{28,84(V_1 - V_0) c_0}{m_1}$$

where

V_0 is the volume, in millilitres, of the 1 mol/l sodium hydroxide solution used for the blank test;

V_1 is the volume, in millilitres, of the 1 mol/l sodium hydroxide solution used for the test portion of sodium lauryl sulfate taken;

c_0 is the exact concentration, expressed in moles of NaOH per litre, of the sodium hydroxide solution used;

m_1 is the mass, in grams, of the test portion of sodium lauryl sulfate taken.

4.2.2 Preparation of standard volumetric sodium lauryl sulfate solution.

Weigh, to the nearest 1 mg, between 1,14 g and 1,16 g of sodium lauryl sulfate and dissolve in 200 ml of water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask (5.3) fitted with a ground-glass stopper, and dilute to the mark with water.

Calculate the exact concentration c_2 , expressed in moles of $C_{12}H_{25}NaO_4S$ per litre, of the solution thus obtained, using the formula

$$\frac{m_2 \tau}{288,4 \times 100}$$

where

m_2 is the mass, in grams, of sodium lauryl sulfate used to prepare the solution;

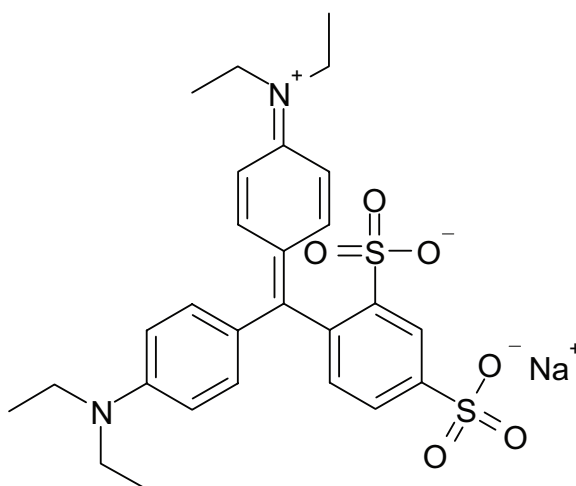
τ has the same meaning as in 4.2.1.

4.3 Mixed indicator solution¹⁾.

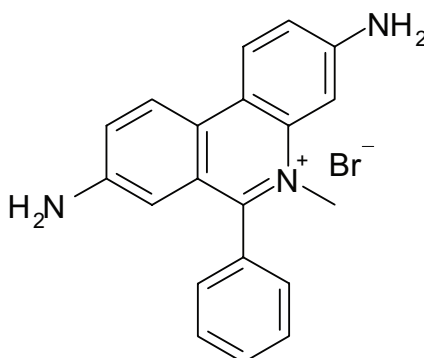
4.3.1 Stock solution.

This solution shall be prepared from acid blue 1 and dimidium bromide.

4.3.1.1 Acid blue 1²⁾ (Colour Index 42045) (Hydrogen [4-[4-(diethylamino)-2',4'-disulfonatobenzhydrylidene]cyclohexa-2,5-dien-1-ylidene]diethylammonium, sodium salt).



4.3.1.2 Dimidium bromide (3,8-diamino-5-methyl-6-phenylphenanthridinium bromide).



1) This mixed indicator is available commercially in the form of a basic solution, which should be acidified and diluted before use.

2) Acid blue 1, also known as disulfine blue VN 150, is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 2871 and does not constitute an endorsement by ISO of this product.

4.3.1.3 Preparation of the stock solution.

Weigh, to the nearest 1 mg, $0,5 \text{ g} \pm 0,005 \text{ g}$ of dimidium bromide (4.3.1.2) into a 50 ml beaker, and $0,25 \text{ g} \pm 0,005 \text{ g}$ of acid blue 1 (4.3.1.1) into a second 50 ml beaker.

Add between 20 ml and 30 ml of hot 10 % (volume fraction) ethanol to each beaker.

Stir until dissolved and transfer the solutions to a 250 ml one-mark volumetric flask. Rinse the beakers into the volumetric flask with the ethanol and dilute to the mark with the ethanol.

4.3.2 Acid solution.

Add 200 ml of water to 20 ml of the stock solution (4.3.1) in a 500 ml one-mark volumetric flask. Add 20 ml of approximately 245 g/l sulfuric acid solution, mix and dilute to the mark with water. Store in the dark.

5 Apparatus

Ordinary laboratory apparatus and the following:

5.1 Flask or measuring cylinder, 250 ml capacity, with ground-glass stopper.

5.2 Burette, 25 ml capacity, complying with the specifications for class A of ISO 385.

5.3 One-mark volumetric flask, 1 000 ml capacity, with ground-glass stopper, complying with class A of ISO 1042.

6 Sampling

The laboratory sample of the detergent shall be prepared and stored in accordance with instructions given in ISO 607.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,5 mg, a sufficient amount of the laboratory sample to contain between 0,002 mol and 0,003 mol of cationic-active matter.

Table 1, which has been calculated on the basis of a relative molecular mass of 360, may be used as a rough guide.

Table 1 — Guide to mass of test portion

Expected cationic-active % (mass fraction)	Mass of test portion g
10	10
20	5
100	1

Possible interferences:

- Low relative molecular mass sulfonates of toluene and xylene present as hydrotropes do not interfere when present in concentrations up to and including 15 % (mass fraction) with respect to the active material. At higher levels, their influence should be evaluated in each particular case.
- Non-ionic surface active agents, soap, urea and the salts of (ethylenedinitrilo)tetraacetic acid do not interfere.
- Typical inorganic components of detergent formulations, such as sodium chloride, sulfate, borate, tripolyphosphate, perborate, silicate, etc., do not interfere, but bleaching agents other than perborate shall be destroyed before the analysis, and the sample shall be completely soluble in water.

7.2 Determination

Dissolve the test portion (7.1) in water and transfer to the 1 000 ml one-mark volumetric flask (5.3). Dilute to the mark with water and mix well.

By means of a pipette, transfer 25 ml of this solution to the 250 ml flask or measuring cylinder (5.1).

Using measuring cylinders, add 10 ml of the mixed indicator solution (4.3), 15 ml of chloroform (4.1) and 25 ml of water. Mix well.

Fill the burette (5.2) with the sodium lauryl sulfate solution (4.2) and titrate; stopper the flask or measuring cylinder after each addition and shake well.

NOTE The lower (chloroform) layer will be coloured blue.

Continue the titration, drop by drop, with repeated vigorous shaking, until at the end point the blue colour is discharged and the lower (chloroform) layer turns greyish-pink.

8 Expression of results

8.1 Calculation

The content, expressed as a percentage by mass, of cationic-active matter is given by the formula

$$\frac{VcM_r \times 1000 \times 100}{25 \times 1000 m_0} = \frac{4VcM_r}{m_0}$$

where

V is the volume, in millilitres, of the sodium lauryl sulfate solution (4.2) used for the titration;

c is the actual concentration, expressed in moles of $C_{12}H_{25}NaO_4S$ per litre, of the sodium lauryl sulfate solution (4.2);

M_r is the mean relative molecular mass of the cationic-active matter;

m_0 is the mass, in grams, of the test portion (7.1)

8.2 Precision

8.2.1 Repeatability

1,6 % of the mean value.

In the normal and correct operation of the method, the difference between two individual results obtained within the shortest feasible time interval by the same operator using the same apparatus and identical test material will exceed the repeatability value on average not more than once in 20 cases.

8.2.2 Reproducibility

4 % of the mean value.

In the normal and correct operation of the method, the difference between individual results obtained by two different laboratories using identical test material will exceed the reproducibility value on average not more than once in 20 cases.

9 Test report

The test report shall include the following information:

- a) all the indications necessary for the complete identification of the sample;
- b) the method used (reference to this part of ISO 2871);
- c) the results and the way in which they have been expressed;
- d) any unusual features noted during the determination;
- e) any operation not specified in this part of ISO 2871 or in the International Standard to which reference is made, as well as any operation regarded as optional.

