

## New Method Of Evaluating Quality Characteristics Of Foam In Detergents

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### Abstract

Foam formation and foam stability are currently used as standard indicators for determining the quality of cosmetic hygienic cleaning agents and detergents. The indicators determine the limit values below which a product will not be considered standard in terms of foaming capacity and cleaning ability. These indicators do not characterize the foam quality which is an important consumer characteristic of foam cleaning agents. The existing methods are analyzed and their drawbacks revealed. A new method of evaluating the quality characteristics of foam is developed. It is proposed to construct a special device for determining the volume weight of foam in a wide range of temperatures to increase the reproducibility and accuracy of the value being determined (foam volume weight). The new method considers all of the drawbacks of the existing techniques.

**Keywords:** foam, method, detergents, foam density, foam formation (foaming), volume weight of foam.

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### INTRODUCTION

Detergents have become common attributes of our daily lives. Every day people use a lot of liquid soap, shower gels, bath foam, and other products not only for cleaning but also for deriving pleasure from taking a shower or a bath. Children cannot imagine taking a bath without fragrant air foam.

In terms of colloid chemistry, foam is a highly concentrated, disperse air-liquid system generated with the help of surfactants at the phase boundary. Air bubbles surrounded by films form a rigid frame, which makes the foam stable.

Foam can be produced as follows:

-by dispergation, by mixing or sparging gases in liquid;

-by condensation through changing the physical form of the solution (by increasing its temperature or reducing external pressure).

The structural features of foam are determined by the form, size, and packing of its bubbles. Disperse-phase foam bubbles can be spherical and multifaceted (polyhedral). In addition, ~~there is also a distinct~~ differentiate cellular foam structure formed in bubbles passing from spherical to polyhedral.

The films of interbubble liquid form so called Plateau triangles. Each polyhedron edge is the place, where liquid films converge that are known as bubble walls. The angles formed by these films are about 120°. The swells formed in film joints are called channels. They form triangles in cross section. Four channels converge in one point and form nodes. These channels and nodes penetrate the entire foam structure [3].

Foam has traditionally been used mainly to eliminate impurities from skin and hair. These impurities are crushed, dispergated, and sucked into the foam by capillary forces [5].

## **MATERIALS AND METHODS**

Foaming and foam stability are currently standard quality indicators of all detergents used for hygienic cleaning.

The techniques of determining the characteristics of foam are:

- GOST 22567.1-77 «Synthetic Cleansing Agents. Method of Determining Foam-Forming Ability»;
- measuring initial foam volume according to the method of the All-Russian Research Institute of Fats (ARRIF), determination of multiplicity factor;
- determining foam stability according to GOST 50588-2012 «Foaming agents for fire extinguishing. General technical requirements and test methods».

According to GOST 25567.1-77, the method of determining the foaming capacity essentially consists in determining the height of a foam column formed at the free fall of 200 cm<sup>3</sup> of the water solution of a test detergent from a height of 900 mm onto the surface of the same solution [1]. However, this technique does not allow measuring the density of foam.

According to GOST 50588-2012, the method of determining the multiplicity factor and stability of foam essentially consists in measuring the weight before and after filling a foam-collection vessel with foam and then calculating the foam's multiplicity factor and determining its stability [2].

The drawback of this procedure is the unavailability of a device for determining the foam volume weight during temperature measurement. The creation of foam with the help of a foam generator requires having large-volume samples for analysis, which makes this method unacceptable in analyzing Personal Care products.

The essence of measuring the initial foam volume by the ARRIF method consists in making 0.5 % soap solution that is poured into the device funnel. The funnel is closed with a cap and shaken for a minute (with about 180 shakes); then the cap is removed and the foam volume measured in the separatory funnel and its conical section.

The drawback of the device is that it does not allow determining the foam volume weight at different temperatures, which limits its scope of application. The position of the electric drive and gear set of the foam inductor in the upper part of the tripod affects the reproducibility of the measures because the device operation is attended not only by the back-and-forth motion of the funnel but also by its vibrations. The design of the tripod and the upper position of the drive affect the useful life of the device.

## **RESULTS AND DISCUSSION**

The current trends at the market for personal care products require producing foam with essentially new functions that are linked with its ability to influence organoleptic properties. First of all, these are the tactile perceptions of softness, tenderness, silkiness, density, flintiness with the application of detergents as well as visual images of desired shapes, objects, and airiness in bath mousse. In addition to rinse off detergents, other products gaining popularity among consumers are those able to generate leave-in foam (refreshing mouth foams, body care foams, and others).

To meet ever growing consumer demands, it is necessary to develop more new kinds of foam-forming cosmetic products. They must have not only cleansing properties at an elevated content of foam and its long-term stability but also dampening and softening properties, with additional new quality characteristics, such as foam density and flintiness and pleasant lightness from using. This essentially new foam is made using essentially new surfactants and special boosters and stabilizers.

Thus the current top priorities include not only the task of standardizing the foam quantity measurement procedure but also the determination of foam quality (volume weight) that must be measured and standardized.

Currently, when the developer elaborates complex foam products, he can align himself only with the visual appearance of foam that is a subjective indicator unable to objectively characterize the fulfilment of this goal. Any subjective, non-standard evaluation extends the development period and does not allow making products with consistently standard characteristics.

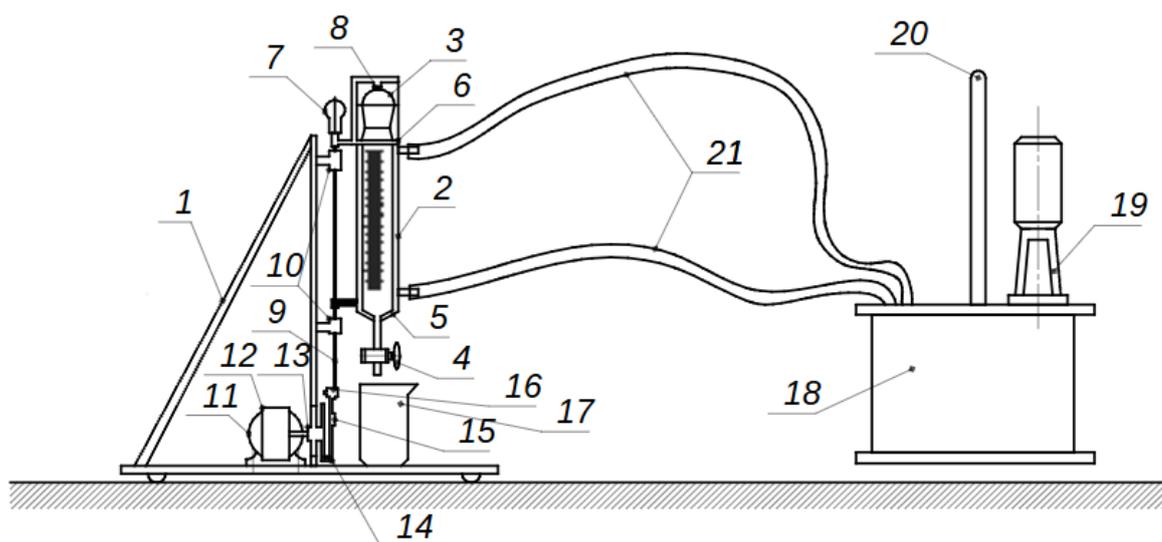
This article presents a new method developed to resolve the above specified issues; in addition, the resulting foams were exposed to microscopic examination.

Foam Volume Weight Determination Method

Considering all of the techniques' drawbacks exposed above, it is proposed to create a device for determining the volume weight of foam in a broad range of temperatures for increasing the reproducibility and accuracy of the quantity being defined.

The technical result is attained by using a separatory funnel with a shell for feeding heat water, which allows maintaining necessary temperature regimes for measurement. The design change in the position of the drive in the bottom part of the device with supporting plates will allow avoiding redundant vibration while using the device, which will extend its life time.

The foam volume weight, foam-forming ability, and foam stability can be measured at temperatures from 25 to 90°C, surfactant solution concentrations of 0.1 to 10 g/cm<sup>3</sup>, and water hardness from 0 to 7.14 mg•equiv/cm<sup>3</sup>.



1 is the tripod; 2 is the 500 cm<sup>3</sup> separatory funnel with double shell; 3 is the cap with a ground joint; 4 is the tap; 5 is the fixed tab; 6 is the mobile tab; 7 is the lock nut; 8 is the hold down spring; 9 is the rod; 10 are the guide bushes; 11 is the electric motor; 12 is the gearset; 13 is the gearset shaft bush; 14 is the crankgear; 15 is the piston rod; 16 is the bearing; 17 is the glass beaker; 18 is the thermostat; 19 is the pump; 20 is the temperature gage; 21 are the flexible connection hoses.

Figure 5. Foam volume weight measurement device

The system's operation principle is that a prepared sample is poured in the separatory funnel of standard diameter with a sensitivity of 1 mm and water chamber; the funnel is connected to the thermostat that ensures a preset measurement temperature. The test solution is conditioned for 5 to 10 minutes to bring the temperature to the preset level. The next step is one-minute shaking with the help of the electric drive (about 180 shakeups). Then the foam column in mm is measured as well as foam stability, and foam density.

The technique includes the following stages:

- preparation of 0.5 % work solution;
- collecting a sample of 50 cm<sup>3</sup>;
- weighing the sample on the scales within a steady-state accuracy;
- making the foam from the sample in the device shown in Figure 5;
- measuring the height of the foam column by the number of divisions in mm;
- conditioning for 5 minutes until the liquid separates from the foam that is discharged and weighed with a steady-state accuracy;
- measuring the height of the foam column by the number of divisions in mm after removing the separated liquid in which case the foam stability and density are calculated as the physico-chemical characteristic by formulae (1 and 2).

Foam stability and density calculation

Foam stability is calculated as

$$ST = \frac{H_5}{H_0}, \quad (1)$$

where ST of the foam stability;

H<sub>0</sub> is the initial foam column height, mm;

H<sub>5</sub> is the foam column height after 5 minutes, mm.

Foam density is calculated as

$$\rho = \frac{(m_1 - m_2)}{V}, \quad (2)$$

where  $\rho$  is the foam density, g/mm<sup>3</sup>;

m<sub>1</sub> is the work solution sample weight, g;

m<sub>2</sub> is the weight of the separated liquid upon conditioning, g;

V is the volume of the foam column upon the conditioning and removal of the released liquid, mm<sup>3</sup>, and it is calculated as

$$V = \pi \frac{d^2}{4} H_5, \quad (3)$$

where d is the measuring post diameter, mm.

If the foam column level has an uneven surface, the foam column height will be the arithmetic average of the minimal and maximal foam height measurement. The graduated cylinder is flushed with distilled water before each new measurement. The difference in diameter among tubes of specific units influences the height of the formed foam column. This is why, the adjustment coefficient for each unit must be preset that will help recalculate all the measured values to the values adequate to the foam column height accurately measured with the help of the unit with an internal tube diameter of 50 mm.

The adjustment coefficient is calculated as

$$K = \frac{D_1^2}{2500}, \quad (11)$$

where  $D_1$  is the actual internal diameter of the tested device, mm;  $2500 = (50)^2$  is the squared internal diameter of the tube of the standard device.

The test involved measuring the volume weight of the foam ( $V, \text{cm}^3$ ) of the model solutions of sodium laureth sulphate, cocamidopropyl betaine, glyceryl monostearate, alkyl betaine and sodium stearate.

The ultimate measurement result is the arithmetic average value of the results of three parallel measurements performed each time on a new portion of soap solution.

For the results of the tests using the foam volume weight determination technique see Table 3.

Table 3. Model surfactant solution test results

Surfactant	$H_0$ , mm	$H_5$ , mm	$\gamma$	$V$ , $\text{cm}^3$	$m_1$ , g	$m_2$ , g	$\rho \cdot 10^3$ , $\text{g}/\text{cm}^3$	Solution T, $^{\circ}\text{C}$
SodiumLaurethSulphate	143	135.9	0.95	266.7	48	35.43	47.13	40
CocamidopropylBetaine	85	77.4	0.91	151.9	46.6	39.54	46.48	40
Fatty acid monoglycerides (Glyceryl Monostearate)	120	96	0.8	188.4	46.4	38.79	40.39	80
AlkylBetaine	135	108	0.8	212	47.2	44.98	10.47	40
SodiumStearate	70	65.1	0.93	127.8	47	38.35	67.68	40

According to the results of examining the model solutions of surfactants, the character of the surfactants determines the capability of forming foam various in density and stability. As a rule, high foam density is attended by high foam stability over time.

This technique was applied to individual surfactants of various kinds; in practice, however, it is expedient to examine synergetic surfactant compounds containing not only basic foamers but also foam intensifiers and stabilizers. These compositions can be used to design new competitive products with enhanced consumer properties.

In addition, since this technique allows taking measurements in a broad range of temperatures, it provides ample opportunities for designing compounds and ensuring their enhanced consumer properties right in the course of use. For example, it is important to evaluate the foaming capacity of shampoo while washing the hair in warm water at  $40^{\circ}\text{C}$ , synthetic detergents in cold and hot water (at 20 to 25 and  $> 40^{\circ}\text{C}$ , respectively) and washing liquids in a wide range of temperature conditions (20 to  $90^{\circ}\text{C}$ ).

### Microscopic Examination of Foam

The produced foams were exposed to microscopic examination for determining the influence of the nature of surfactants on their character and properties at a definite moment in time. The examined samples of foam were obtained by determining the foam volume weight after determining the foam stability (in 5 minutes after the beginning of the test).

The results confirm the connection of the character and properties of foam with the character of surfactants. The main characteristics of the tested substances used as raw materials for making foam detergents are presented below.

#### Sodium Laureth Sulphate:

Chemical formula:  $\text{CH}_3(\text{CH}_2)_{10}\text{CH}_2(\text{OCH}_2\text{CH}_2)_n\text{OSO}_3\text{Na}$   $\text{C}_{12+2n}\text{H}_{25+4n}\text{NaO}_{4+n}\text{S}$

Molar weight: 420 g/mole.

Kind: anionic surfactant

INCI: Sodium Laureth Sulfate

CAS №: 68891-38-3

Structural formula:

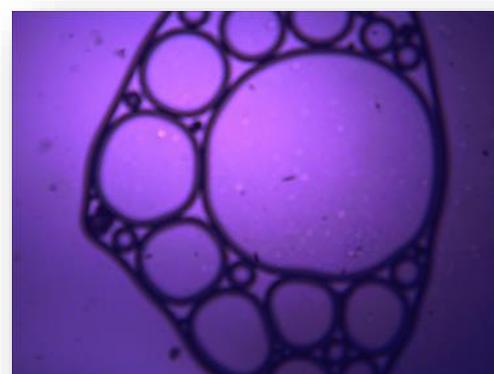
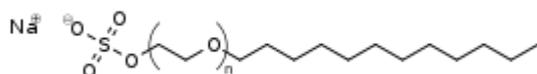


Figure 1. View under a Micromed 3 microscope with a magnification of 40x

The foam-forming capacity is 143; the foam stability is 0.95.

Function: main surfactant

#### Cocamidopropyl Betaine:

Chemical formula:  $\text{C}_{19}\text{H}_{38}\text{N}_2\text{O}_3$

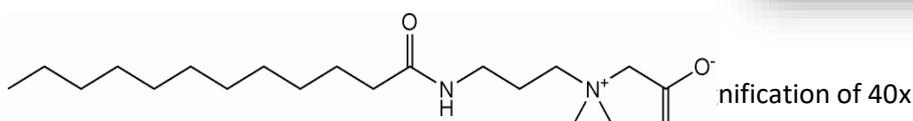
Molar weight: 342.288 g/mole

Kind: amphoteric surfactant

INCI: Cocamidopropyl Betaine

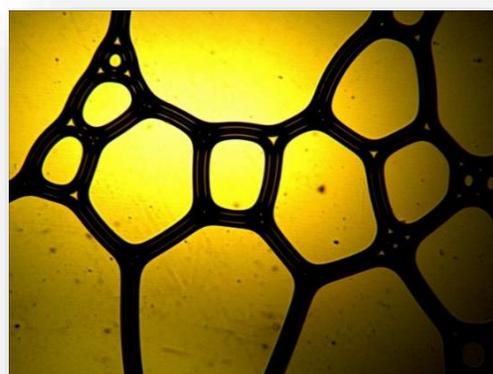
CAS №: 61789-40-0

Structural formula:



The foam-forming capacity is 85 mm; the foam stability is 0.95.

Function: co-surfactant



#### Fatty acid monoglycerides (Glyceryl Monostearate):

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Chemical formula:  $C_{21}H_{40}O_2(OH)_2$

Molar weight: 358.56 g/mole.

Kind: nonionic surfactant

INCI: Glyceryl Stearate

CAS № 123-94-4

Structural formula:

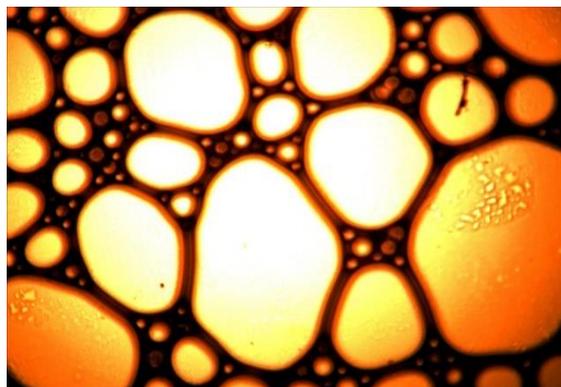
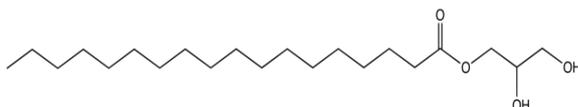


Figure 3. View under a Micromed 3 microscope with a magnification of 40x

The foam-forming capacity is 120 mm; the foam stability is 0.8.

Function: emulsifier

Alkyl Betaine:

Chemical formula:  $C_{14}H_{29}N^+(CH_3)_2CH_2COO^-$

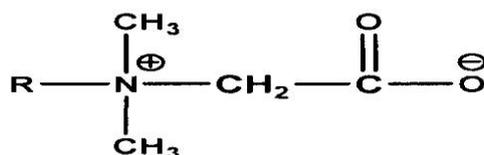
Molar weight: 282.499 g/mole.

Kind: amphoteric surfactant

INCI:Lauryl-Myristyl Betaine

CAS №:66455-29-6

Structural formula:



R-alkyl C<sub>12</sub> – C<sub>14</sub>

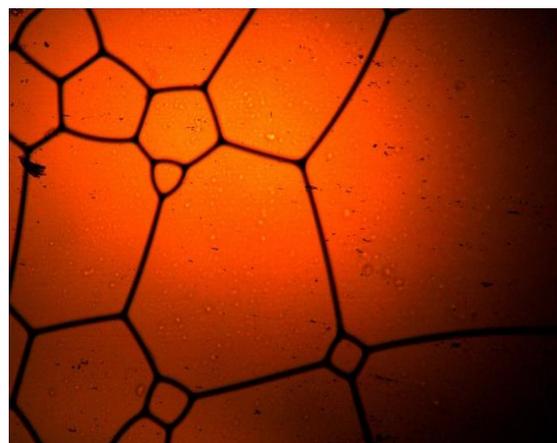


Figure 4. View under a Micromed 3 microscope with a magnification of 40x

The foam-forming capacity is 135 mm; the foam stability is 0.8.

Function:co-surfactant

Sodium Stearate

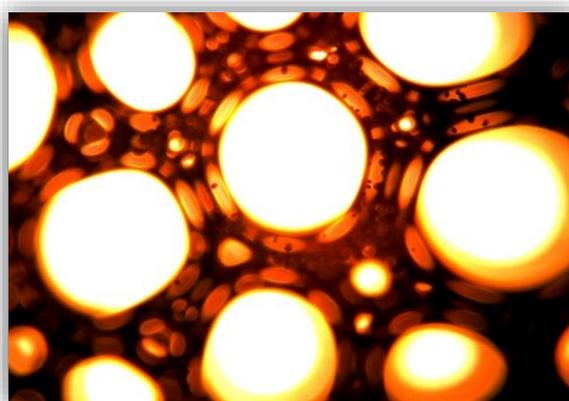
Chemical formula:  $C_{17}H_{35}COONa$ .

Molar weight: 306.46 g/mole.

Kind: anionic surfactant

INCI:Sodium Stearate

CAS#: 822-16-2



Structural formula:

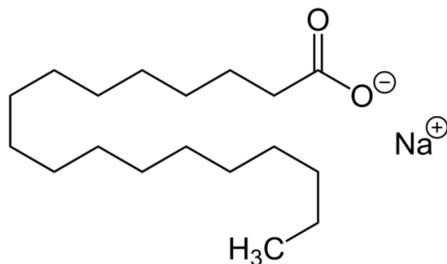


Figure 5. View under a Micromed 3 microscope with a magnification of 40x

The foam-forming capacity is 70 mm; the foam stability is 0.93.

Function: main surfactant, emulsifier, viscosifier.

The analysis of the above specified surfactant solution foams shows that they are all polydisperse, that is, the gas bubbles differ in size. The smaller is a gas bubble, the higher is the pressure inside. Therefore, the spontaneous diffusion of gas from small to big bubbles is observed over time; in this case, the small bubbles get smaller and big ones even bigger, which changes the foam stability, and it is said that the foam is aging. The greater are the differences in the bubble size (the higher is the polydispersion degree), the more unstable will the foam be; consequently, the lower is the polydispersion degree and the difference in the gas bubble size, the higher stability and density will the foam have.

In addition to the polydispersion degree, the foam destruction rate is influenced by the liquid film thickness; spontaneous runoff in the foam film makes it thinner and, ultimately, break. The thicker is the film, the stabler will the foam be.

In addition to basic properties, such as stability and density, the microscoping technique also allows evaluating the kind of foam, which includes its mono- or polydispersion, homogeneity, and aging character.

The integral application of the two techniques specified provides much broader opportunities for the directed development of detergent foam products with defined properties and will also help developers of new industrial raw materials roll out new competitive products widely sought after in various production sectors.

## CONCLUSION

1. The existing techniques of determining the characteristics of foam have been studied. None of the techniques allows determining foam density when analyzing Personal Care products.
2. A new method of determining the foam volume weight has been proposed that considers all of the drawbacks of the existing techniques.

3. The foam volume weight has been measured as  $V$  in  $\text{cm}^3$ , and the foam of the model solutions of sodium laureth sulphate, cocamidopropyl betaine, glyceryl monostearate, alkyl betaine, and sodium stearate was exposed to microscopic examination.
4. The results allow seeing the influence of the nature of surfactants on the quality characteristics of foam.
5. The proposed technique will allow obtaining in a wide range of temperatures reproducible and more accurate results of qualitatively determining the volume weight of foam of Personal Care products.
6. This technique is applicable in developing new raw materials with enhanced properties for making foam-washing products.
7. The new technique can be used for both, rinse off detergents and a broad range of leave-in foam-forming products of various actions and effects.
8. The proposed technique will simplify the development of new foam products with interesting consumption properties and, therefore, expand the existing range of such products and favour improvements in the output quality.

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