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Professional paper

Method performance verification and measurement uncertainty evaluation for determination of total active matter content in shampoos

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Abstract

The aim of our study was to perform in-use method verification of the standard ISO 6842:1989(E) method for determination of total active matter (TAM) content in shampoos. The method was verified for accuracy and precision (method precision, repeatability and intermediate precision). To assure quality, measurement uncertainty was also estimated, through evaluation of the possible sources of uncertainty, using the Nordtest approach.

The obtained results for the accuracy, repeatability (method precision) and intermediate precision confirmed that the standard method could be successfully applied when compared to data for repeatability and intermediate precision provided in ISO 6842:1989(E) method. Measurement uncertainty was estimated for each measurement. The verified method was successfully applied for routine determination of total active matter content in shampoos.

Key words: Method performance verification, gravimetric determination, total active matter, measurement uncertainty

Introduction

The availability and the application of appropriate test methods in the laboratories is one of the most important factors for obtaining reliable test results. The laboratories can develop their own methods, they can choose methods published in international, regional or national standards or they can use methods published in relevant scientific literature. Among the most commonly applied methods are those provided by ISO/IEC 17025 standard which has long been recognized as the "golden standard" for testing and calibration of laboratory quality assurance (Đukić et al., 2023; Kotsiuba, 2022).

The methods provided by ISO/IEC 17025 standard have already been validated by previous collaborative studies and found to be fit for purpose as defined by the scope of the method. If such method is chosen, the laboratory does not have to perform revalidation but needs to conduct method performance verification (Đukić et al., 2023; NATA, Tech. note 17, 2012). The verification must be done to show that under actual conditions of use in the

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individual laboratories the (validated) method is adequate (fit for use, obtaining the same outcomes as defined in the validation data provided in the standard method). This may be achieved by carrying out the system suitability tests (e.g., resolution in a chromatographic method), controlling sensitivity at the reporting threshold, controlling the completeness of a reaction step (e.g., extraction, hydrolysis reaction) before the actual determination can be performed, verifying the precision of the method etc. (PA/PH/OMCL (13) 82 2R, 2020).

In addition to performance verification studies, another important point to express reliability of the analytical results is the measurement uncertainty (MU) estimation, since the result of the measurement without the values for measurement uncertainty is only an estimate of the value of measurement. The MU expresses, quantitatively, the value of the accuracy of the measurement result (Ellison and Williams, 2012; JCGM, 2008). Thus, MU is a parameter related to a measurement, not the method, representing a range of values that can be attributed to the analysis result with a certain level of confidence. Uncertainty sources such as sampling, weighing, environmental conditions, equipment and instruments, purity of reagents, and reference standards may contribute MU (Couto and Lourenço, 2023). Knowledge of MU is necessary for the effective comparison of measurements and for the comparison of the obtained results with the specification limits (Couto and Lourenço, 2023). It should be readily available and reported together with result as X±U, where U is the expanded uncertainty (ISO/IEC, 1999; ISO 17025, 2017; King, 2001; Mueller, 2002, Populaire and Gimènez, 2006).

Shampoos are cosmetic products intended for effective scalp and hair cleansing that contain different ingredients with distinct but equally important functions in the formulation (Azadbakht et al., 2018; Thompson et al., 2023). The surfactants, as one of the most important ingredients in a shampoo, carry out not only the cleansing mechanisms, but contribute to foaming capabilities, the solubilization of active agents and components, viscosity moderation, and suspension of additives (Myers, 2020; Thompson et al., 2022). The determination of surfactant content in shampoos is essential for ensuring product quality and performance. Depending on the type of surfactant and the level of sensitivity required, various methods can be applied such as gravimetric determination, titration, cloud point determination, spectroscopy, and chromatography. The choice of method should be based on factors such as cost, sample preparation complexity, and the availability of equipment and expertise (Beneito-Cambra et al., 2013; Prieto-Blanco et al., 2018). Gravimetric techniques measure the mass of the analyte of interest or its derivative to determine its quantity. They typically include sample preparation, precipitation of the analyte and drying or calcination to obtain pure substance for accurate mass measurement. Gravimetry is considered as one of the most accurate analytical techniques, since the effects of measurement uncertainty are minimized (Mao, 2024).

This study aims to present the method performance verification of a standard ISO 6842:1989(E) method intentended for determination of surface active agents – sulphated ethoxylated alcohols and alkylphenols-determination of total active matter (TAM) content in shampoos and to estimate the uncertainty of measurement related to the determination.

Material and methods

Chemicals, reagents and reference materials

Ethanol (96% *v/v*) and dichloromethane (p.a) were obtained from Alkaloid AD, Skopje. Sodium sulfate - anhydrous, acetone (50% *v/v*, aqueous solution), and potassium chromate (100 g/L, used as an indicator) were purchased from Merck (Darmstadt, Germany). The standard volumetric solution of $AgNO_3$ (0.1 mol/L) was purchased from Fisher Chemical. The commercially available baby shampoo used for determination of method precision was obtained from local markets. Hair shampoo from the proficiency testing scheme MIFF 1–2019, was used as a reference material for verification of method accuracy and estimation of measurement uncertainty.

Instrumentation

The rotary evaporator was LABO ROTA 300 (Gemini B.V., the Netherlands) and the samples were dried using BINDER ED 115/E2 drying chamber (Binder, Germany). Samples were incubated at a constant temperature using NB9 water bath (Vincent Leermiddelen Scientific, Belgium).

Sample preparation

The principle of the ISO 6842:1989(E) standard method is based on the different solubility of the sample components in 96% (*v/v*) ethanol. The total active matter dissolves in 96% (*v/v*) ethanol, while most of the inorganic ingredients are insoluble. Samples were kept at ambient temperature until analysis.

Accurately weighted sample (3-5 g) of the was homogenized thoroughly by mixing (required quantity to achieve 0.5-1.0 g dry residue after ethanol extraction). The homogenized sample was transferred into a conical flask and anhydrous sodium sulfate and ethanol (96%, *v/v*) were added. The reaction mixture was boiled under reflux for 30 min. The obtained content was filtered into a dried round-bottom flask. The undissolved residue was washed with dichloromethane and filtered in the flask. Ethanol was removed via rotary evaporation. The flask's contents were dried, and the residue was weighed. Afterwards, the residue was dissolved in acetone solution, followed by the addition of potassium chromate, and titrated with $AgNO_3$ to achieve a permanent brown color. The mass of the residue was corrected for the NaCl content (ISO 6842:1989).

The content of total active matter content (TAMC) was calculated by the formula:

$$TSAM = \frac{m_1 - 0{,}0585\ c\ (V_1 - V_0)}{m_0}\ x\ 100 \quad (1)$$

Where: m_0 is the mass of the sample portion (g), $m_1$is the mass of the residue obtained (g), c is actual concentration of the silver nitrate solution, (mol L^{-1} $AgN0_3$), V_0 is the volume of $AgN0_3$ used for the blank test (mL), V_1 is the volume of $AgN0_3$ used for the determination of any sodium chloride present (mL), 0,0585 is the mass of sodium chloride (g) corresponding to 1.00 mL of $AgNO_3$ solution, c($AgNO_3$) = 0.1 mol/L.

Method performance verification

The method performance verification was done according to ISO 5725, by assessing method accuracy and precision (ISO 5725-6:1994).

The precision was assessed by determination of TAMC content in commercially available shampoo. Repeatability was evaluated in 5 different shampoo solutions on the same day, by the same analyst and using the same equipment, while the reproducibility (intermediate precision) was evaluated on three different days. Accuracy (trueness) was investigated using hair shampoo from the proficiency testing scheme (PTS) MIFF 1–2019 with assigned value of 7.46% TAMC.

Estimation of measurement uncertainty (MU)

Measurement uncertainty was estimated using the Nordtest approach or "top-down" approach (Bich, 2016; NT TR 537 4 Ed., 2017).

The combined standard uncertainty, u_c, was calculated using the following formula:

$$u_c = \sqrt{u(R_w)^2 + u(bias)^2} \quad (2)$$

The u_c, consists of two components: the precision component (within-lab reproducibility or intermediate precision), *u(Rw)* and the bias component (method and laboratory bias), *u(bias)* (NT TR 537 4 Ed., 2017).

The uncertainty as a result of bias was estimated using the data obtained from the participation in a proficiency testing (PT), using the following formula:

$$u(bias) = \sqrt{RMS_{bias}^2 + u(Cref)^2} \quad (3)$$

The first component in the upper formula, the root mean square of the bias, RMS_{bias}, is calculated by the following formula:

$$RMS_{bias} = \sqrt{\frac{\sum(bias_i)^2}{n}} \quad (4)$$

$Bias_i$, is the result from an individual bias determination and n is number of bias determinations carried out.

$$bias_i = \frac{V_i - V_{Ri}}{V_{Ri}} * 100\ (\%\ rel) \quad (5)$$

Where V_{Ri} is the assigned (nominal) value, and V_i is the mean value from the analyte determination performed by the laboratory.

The *u(Cref)* component represents the uncertainty of the assigned (nominal) value, and it can be calculated as:

$$u(Cref) = \sqrt{\frac{\sum(Cref_i)^2}{n}} \quad (6)$$

Where $u(Cref_i)$ is the standard uncertainty of the *i-th* assigned value. In the case of interlaboratory comparisons (PT) where the consensus value of the participants is used as the assigned (nominal) value, a reliable uncertainty cannot be found. The best estimate in that case would be the standard deviation of the average value obtained with the PT after elimination of outliers. Therefore, the following formula is used:

$$u(Cref) = \frac{S_R}{\sqrt{n}} \quad (7)$$

Where S_R is the mean value of the between laboratory RSD, and n is the number of participants in the PT.

The MU should normally be expressed as *U*, the expanded measurement uncertainty, with a stated confidence level and a coverage factor, *k.* In most cases *k* = 2, providing a level of confidence of approximately 95 % (Bich et al., 2016).

The expanded measurement uncertainty, *U*, can be calculated using the formula:

$$U = k * u_c \quad (8)$$

All values were expressed as percentages.

Results and discussion

In this study, the verification of the standard ISO 6842:1989(E) method for determination of total active matter (TAM) content, in shampoos is presented. Prior application on real samples, the standard method was verified for precision (repeatability) and accuracy. Results were calculated from the weight difference between the sample and/or the recipient that contains it before and after the sample preparation step (Populaire and Gimènez, 2006).

Precision -The precision of a measurement system, related to repeatability and reproducibility, is the degree to which repeated measurements under unchanged conditions show the same results (Plant and Hanish, 2020). Method precision (repeatability) was assessed by determination of

Table 1. Results obtained from verification of method's repeatability

N°	m empty flask, (g)	m sample, (g)	m flask with sample (g)	Residue mass (g)	TAM content (%)
1.	134.8472	2.4547	135.2797	0.4324	17.62
2.	128.3902	2.0323	128.7493	0.3591	17.67
3.	131.9370	2.1740	132.3243	0.3873	17.81
4.	129.8219	2.2732	130.2240	0.4021	17.69
5.	128.6163	2.7864	129.1128	0.4965	17.82
				Average	17.72%
				SD	0.090
				RSD (%)	0.51

Table 2. Results from verification of method's intermediate precision (laboratory reproducibility)

N°*	m empty flask, (g)	m sample, (g)	m flask with sample (g)	Residue mass (g)	TAM content (%)
1.	134.8468	2.5467	135.3013	0.4545	17.85
2.	130.8034	2.4618	131.2414	0.4380	17.79
3.	128.6147	2.5762	129.0622	0.4475	17.37
				Average	17.67%
				SD	0.26
				RSD(%)	1.47

*Analysis was performed on three different days

TSAM content in 5 baby shampoo sample solutions on the same day, by the same analyst and using the same equipment. Repeatability was expressed as the standard deviation (SD) and relative standard deviation (RSD), obtained by the 5 replicate measurements. The results are presented in Table 1.

The values for SD, and RSD were 0.090 and 0.51% respectively.

Intermediate precision - The intermediate precision of the analytical procedure (laboratory reproducibility) was assessed analyzing the TAM content in baby shampoo sample solutions on three consecutive days. The precision was expressed as the RSD of series of measurements. The results obtained from the verification of intermediate precision are shown in Table 2.

The obtained results for the repeatability and the laboratory reproducibility are in accordance with the criteria provided in the ISO 6842:1989(E) standard, indicating the method is precise.

Method accuracy - accuracy expresses the closeness of agreement between a measured quantity value and the quantity's true value (Guzel and Canli, 2020). Method accuracy was determined by the deviation of the average value obtained in our laboratory from the reference value (the assigned value of the reference material obtained with the PT scheme MIFF 1- 2019 (hair shampoo)). The results are presented in Table 3.

The EU Cosmetics Regulation (EC) No 1223/2009 (where shampoos are listed) does not specify exact acceptable recovery limits for the analysis of cosmetic products. ISO 6842:1989(E) specifies a method for determining the TSAM content, which comprises surfactants commonly used in shampoos. However, the standard does not explicitly define acceptable recovery limits for this analysis. In the absence of specific recovery limits within ISO 6842:1989(E), laboratories typically adhere to general analytical guidelines or regulatory standards to establish acceptable recovery ranges. Therefore, recovery rates between 80% and 120% are commonly considered acceptable (EMA/CHMP/ICH/82072/2006). The recovery values obtained in our study were in accordance with the criteria provided in EMA/CHMP/ICH/82072/2006, confirming method's accuracy.

The Nordtest approach used to calculate the measurement uncertainty uses the values obtained from method verification, namely the with-in laboratory reproducibility (intermediate precision) and the method and

Table 3. Results obtained from verification of method's accuracy

N°	PT determination Assigned value for TAM content (%)	PT determination Obtained value for TAM content (%)	Recovery (%)
1.	7.46	8.74	117.16
2.	7.46	7.99	107.10
3.	7.46	7.70	103.22
	Average	8.14	109.16
	SD	0.54	7.19
	RSD(%), *u(Rw)*	5.38	6.59

laboratory bias component, *u(bias)*. The latter can be calculated using certified referent materials (CRM), using data from participation in PT or can be calculated using recovery tests (NT TR 537 4 Ed., 2017). In the next section, the step by step calculation of the MU for a result obtained after TAM content determination in a commercially available shampoo is presented.

Step 1: Uncertainty component from the uncertainty of the assigned value:

The uncertainty, *U* of the assigned value was given by the PT provider (U (k=2) = 0.841%) and was calculated according to the procedure described in ISO 13528 (ISO 13528, 2015).

Step 2: Quantification/Calculation of the u(bias) components:

$$RMS_{bias} = \sqrt{\frac{\sum(bias_i)^2}{n}} = \sqrt{\frac{(0.6833)^2}{7.46}} * 100 = 9.16\ (\%\ rel.) \quad (9)$$

$$u(Cref) = \sqrt{\frac{\sum(Cref_i)^2}{n}} = \sqrt{\frac{(0.4205)^2}{7.46}} * 100 = 5.64\ (\%\ rel) \quad (10)$$

In cases where the PT provider gives the value for U, the U/2 should be used as u($Cref_i$) (NT TR 537, 2017).

The *u(bias)* component:

$$u(bias) = \sqrt{RMS_{bias}^2 + u(Cref)^2} = \sqrt{9.16^2 + 5.64^2} = 10.76\ (\%\ rel) \quad (11)$$

Step 3: The combined measurement uncertainty was calculated as follows:

$$u_c = \sqrt{u(R_w)^2 + u(bias)^2} = \sqrt{6.59^2 + 10.76^2} = 12.61\ (\%\ rel.) \quad (12)$$

The total uncertainty, known as standard combined uncertainty u_c, is an estimated standard deviation equal to the positive square root of the total variance obtained by combining all the uncertainty components (Farrance and Frenkel, 2012).

For most purposes in analytical chemistry, expanded uncertainty *U* should be used – provides an interval within which the value of the measurand is believed to lie with a higher level of confidence. *U* is obtained by multiplying u_c, the standard combined uncertainty, by a coverage factor, *k=2* (Bich et al., 2016).

The expanded uncertainty *U* equals:

$$U = k * u_c = 2*12.61\% = 25.2\% \quad (13)$$

It should be noted that one disadvantage of the presented approach using data from PT is that the laboratory result is based on one determination which results in an increased uncertainty compared to a mean value (NT TR 537 4 Ed., 2017).

After the verification and MU calculation the method was applied for determination of the TAM content in different products obtained by the State Sanitary and Health Inspectorate. Each result was expressed together with *U*, according to the requirements of ISO 17025:2017. The results are presented in Table 4.

The TAM content in all investigated samples was in compliance with the national legislation (Official Gazete of SFRJ N^0 26/83).

Conclusion

In this study, the verification of the performance of the standard ISO 6842:1989(E) method for gravimetric determination of the TAM content in shampoos is presented. The results obtained from the verification of method's repeatability, intermediate precision and accuracy showed that it can be successfully applied for routine analysis of TAM content in commercially available shampoos. Further, the results obtained from the verification of the with-in laboratory reproducibility (intermediate precision) and the results from accuracy

Table 4. Content of TAM content (%) in different shampoos

No.	Product code	Product characteristics	Country of origin	TAM content (%)	U (k=2) (%)	Result ± U (%)
1	Product 1	Herbal Shampoo	Slovenia	17.76	4.48	17.76 ± 4.48
2	Product 2	Anti-Dandruff Shampoo	Serbia	19.27	4.86	19.27 ±4.86
3	Product 3	Shampoo	Turkey	19.08	4.81	19.08 ± 4.81
4	Product 4	Anti-Dandruff Shampoo	Turkey	21.13	5.32	21.13 ± 5.32
5	Product 5	Shower Gel for Body and Hair	Serbia	8.20	2.07	8.20 ± 2.07
6	Product 6	Sensitive Hair Shampoo	Austria	28.94	7.29	28.94 ± 7.29
7	Product 7	Hair Shampoo	Slovenia	8.86	2.23	8.86 ± 2.23
8	Product 8	Baby Hair Shampoo	Turkey	12.06	3.04	12.06 ± 3.04
9	Product 9	Hair Shampoo	Italy	12.07	3.04	12.07 ± 3.04
10	Product 10	antidandruff Shampoo	Turkey	28.94	7.29	28.94 ± 7.29
11	Product 11	Hair Shampoo	Serbia	8.20	2.07	8.20±2.07
12	Product 12	Hair Shampoo	Italy	14.02	3.53	14.02 ± 3.53
13	Product 13	Hair Shampoo	Russia	14.86	3.74	14.86 ± 3.74
14	Product 14	Volume Hair Shampoo	Romania	15.91	4.01	15.91 ± 4.01
15	Product 15	Anti-Dandruff Shampoo	Switzerland	6.75	1.70	6.75 ± 1.70
16	Product 16	Hair Shampoo	Turkey	12.18	3.07	12.18 ± 3.07
17	Product 17	Hair Shampoo	Turkey	13.82	3.48	13.82 ± 3.48
18	Product 18	Shampoo for Oil Hair	Germany	15.68	3.95	15.68 ± 3.95
19	Product 19	Herbal Hair Shampoo	Bulgaria	9.80	2.47	9.80 ± 2.47
20	Product 20	Hair Shampoo	China	9.10	2.29	9.10 ± 2.29
21	Product 21	Hair Shampoo	Italy	20.67	5.21	20.67 ± 5.21
22	Product 22	Baby Hair Shampoo	Italy	15.20	3.83	15.20 ± 3.83
23	Product 23	Baby Hair Shampoo	R.N. Macedonia	12.80	3.23	12.80 ± 3.23
24	Product 24	Hair Shampoo	Italy	13.29	3.35	13.29 ± 3.35
25	Product 25	Baby Hair Shampoo	Serbia	14.76	3.72	14.76 ± 3.72
26	Product 26	Hair Shampoo	Turkey	10.25	2.58	10.25 ± 2.58
27	Product 27	Hair Shampoo	Italy	18.18	4.58	18.18 ± 2.58
28	Product 28	Baby Hair Shampoo	Austria	37.84	9.54	37.84 ± 9.54
29	Product 29	Anti-Hair Loss Shampoo	R.N. Macedonia	12.01	3.03	12.01 ± 3.03
30	Product 30	Hair Shampoo	German	16.13	4.06	16.13 ± 4.06
31	Product 31	Hair Shampoo	Bulgaria	11.42	2.88	11.42 ± 2.88
32	Product 32	Herbal Hair Shampoo	Serbia	4.61	1.16	4.61 ± 1.16
33	Product 33	Hair Shampoo	R.N. Macedonia	11.11	2.80	11.11 ± 2.80
34	Product 34	Baby Hair Shampoo	Germany	15.62	3.94	15.62 ± 3.94

verification using PT data, were used for the estimation of method and laboratory bias. Based on these two results, MU was determined. The approach for MU determination presented in this article refers to the given laboratory. However, MU calculated as presented in the paper can be used to assess the maximum TAM content in shampoos and to confirm that the information provided on the label is in accordance with national or international regulations.

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Резиме

Верификација на перформансите и процена на мерната неодреденост на метод за одредување на содржината на вкупни површински активни материи во шампони

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Клучни зборови: верификација на метод, гравиметриско определување, вкупни суфрактанти, мерна неодреденост

Целта на нашето истражување беше да извршиме верификација на стандардниот метод ISO 6842:1989 (E) за одредување на содржината на вкупни сурфактанти во шампони. Верификацијата ги опфати параметрите точност и прецизност (прецизност на методот, рипитабилност и интермедиерна прецизност). За да се осигура квалитетот на аналитичкиот резултат, беше проценета мерната неодреденост, преку процена на можните извори на грешка, со примена на Nordtest пристапот за одредување на мерна неодреденост.

Добиените резултати за точноста и прецизност на методот (рипитабилност и интермедиерна прецизност) беа задоволителни споредено со вредностите дадени во стандардниот метод. Мерната неодреденост беше пресметана за секое одредување. Верификуваниот метод беше успешно применет за рутинско одредување на содржината на вкупни површинско активни материи во шампони.