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DESCRIPTION OF THE STANDARDIZATION PROCEDURE OF STANDARD VOLUME MEASURES AND ANALYSIS OF THE STABILITY OF THEIR METROLOGICAL CHARACTERISTICS

Anna Sabadas [1, 2*], ORCID: 0000-0002-8064-6068,
Alexei Pianih [1, 2], ORCID: 0000-0002-1165-9690

[1]*Stefan cel Mare University of Suceava, 13 University Street, 720229 Suceava, Romania*
[2]*Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova*
*Corresponding author: Anna Sabadas, *any.sabadas@gmail.com*

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Abstract. The metrological characteristics of the measuring devices refer to the behavior of the measuring device in relation to the object under measurement, the environment and the human operator. Thus, having the same conditions in the laboratory and a well-trained and competent staff, the stability of the metrological characteristics of the standard volume measures were studied in order to demonstrate their stability over time and provide increased confidence to the subsequent beneficiaries. The measurements were carried out over a period of one year under the same conditions, their results demonstrating that the standard volume measures are able to ensure the traceability of the measurement unit. In this way to ensure the uniformity, accuracy and legality of the measurements performed in different fields - measurement of volume in commercial transactions, fuel supply, wine industry.

Keywords: *standardization, standard measures, volume, metrological characteristics, stability.*

Rezumat. Caracteristicile metrologice ale mijloacelor de măsurare se referă la modul de comportare a aparatului de măsurat în raport cu obiectul supus măsurării, cu mediul ambiant și cu operatorul uman. Astfel, având aceleași condiții în laborator și un personal bine instruit și competent, se studiază stabilitatea caracteristicilor metrologice ale măsurilor de volum etalon pentru a demonstra stabilitatea acestora în timp și a oferi o încredere sporită beneficiarilor ulteriori. Măsurările propriu-zise au fost efectuate pe perioada unui an în aceleași condiții, rezultatele acestora demonstrând că măsurile de volum etalon sunt capabile să asigure trasabilitatea unității de măsură. În acest fel, să se asigure uniformitatea, exactitatea și legalitatea măsurărilor efectuate în diferite domenii de măsurare la efectuarea măsurărilor volumului cu referire la tranzacțiile comerciale, alimentarea cu combustibili, industria vinicolă.

Cuvinte cheie: *etalonare, măsuri etalon, volum, caracteristici metrologice, stabilitate.*

1. Introduction

The measurement unit for volume is the cubic decimeter (dm^3) or the liter (L), which is defined as the volume occupied by 1 kg of distilled water at 4°C in vacuum at sea level and 4° latitude. A thousandth of a liter is a cubic centimeter (cm^3) or a milliliter (mL) [1].

The purpose of the article is to determine the metrological characteristics of standard volume measures and demonstrate the stability of the measurements over time. Being part of the national standard of the liquid volume unit, their study is very important. The volumetric method and the gravimetric method [2] can be used when calibrating standard volume measures, the latter providing a higher level of accuracy.

The national standard of the liquid volume measurement unit (ETN V) constitutes a basic element of the National System of Standards [4].

ETN V traceability [5] is ensured by the gravimetric method from ETN 09 -15 National Standard of mass. Subsequently, ETN transmits the volume measurement unit to other working standards and measuring instruments in the field according to the traceability scheme, in order to ensure the uniformity, accuracy and legality of the measurements performed throughout the territory of the Republic of Moldova. This is important to ensure correct measurements in different fields: measurement of volume in commercial transactions, fuel supply, wine industry.

2. Materials and Methods

The volume standard measures [6] are a component of the national standard and have the role of volume transfer standard. The standards allow the measurement of the volume of the liquid with an uncertainty of (0.0005 ÷ 0.04) dm^3, the measurement accuracy being dependent on the parameters of the environment. To reproduce the volume measurement unit, standard volume measures of 10 dm^3, 50 dm^3, 100 dm^3 are used. Each standard volume measure has a nominal value with an accuracy of 0.02%.

Metal volume measures of the first-order [1] are used to convey the unit of volume to metal volume measures of the second-order and technical volume measures (figure 1). The error of the first-order metal volume measurement at a temperature of 20.0 °C must not exceed 0.02% of the nominal value. First-order metal volume measures are of several types: M1p2; M1p5; M1p10; M1p20; M1p50; M1p100; M1p200; M1p500. In the standardization process, volume measures up to 200 dm^3 are used.

Figure 1. Classification of first-order metal volume measures.
(*done by authors).*

A volume measure (figure 2) consists of:

1. Adjustable legs;
2. Tap;
3. Casing;
4. Sight glass;
5. Maximum level.

The standardization of the volume measures is carried out in the following environmental conditions [3]:

- Ambient temperature: (20.0 ±5.0) °C.
- Air humidity: (30.0 ÷ 80.0) %.
- Atmospheric pressure: (840.0 ÷ 1060.0) hPa.
- The water temperature during standardization must not exceed ± 0.2 °C.

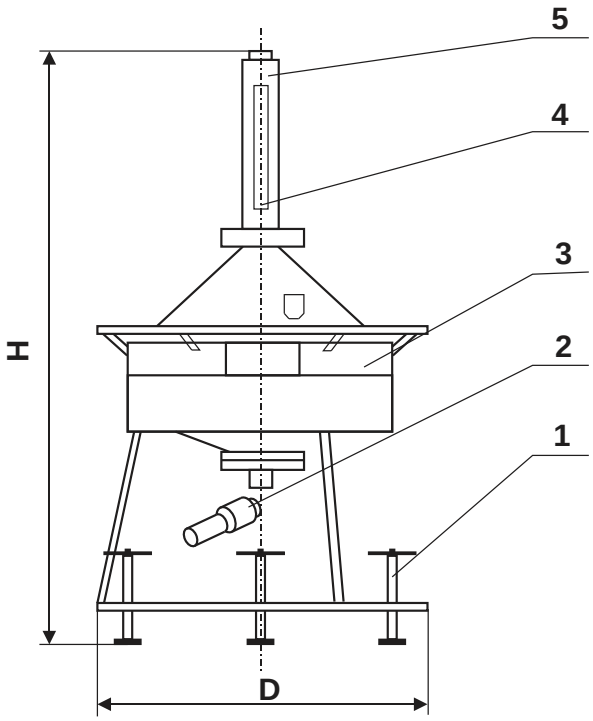

Figure 2. First-order volume measure [1]:
1. adjustable legs; 2. tap; 3. casing; 4. sight glass; 5. maximum level.

In case of non-compliance with the previously indicated environmental conditions, the measurements will be stopped and repeated only after removing the non-conformities.

The standardization operation of the first-order metal volume measure is carried out using measuring means and auxiliary devices, shown in table 1.

Table 1

List of standards, measuring instruments and auxiliary equipment [3].

No.	Name	Type	Technical characteristics
1.	Mass comparator	HRP 200.4 Y.KO	Measurement range: (0.0 ÷200000.0) g
2.	Weighing device with non-automatic operation	APP 25.3Y	Measurement range: (0.0 ÷ 25.0) kg
3.	Digital thermometer with platinum Pt100 resistance	TPM1	Temperature Measurement range: (-25.0 ÷ +250.0) °C

Continuation Table 1

4.	Mechanical chronometer	-	Measurement range: (0.0 ÷ 1800.0) s
5.	Digital thermohygrometer	ПИ-002 1/A	Temperature Measurement range: (5.0 ÷ 98.0) °C Humidity Measurement range: (5.0 ÷ 40.0) %
6.	Pressure, humidity, temperature indicator	PTU 303	Temperature Measurement range: (-20.0 ÷ 80.0) °C Humidity Measurement range: (0.0 ÷ 100.0) % Absolute pressure Measurement range: (500.0 ÷ 1100.0) hPa
7.	Micropipette	-	Measurement range: (2.0 ÷ 10.0) mL

The calibration procedure takes place in several stages:

1. Checking the exterior. Upon receipt of the MM, they undergo a visual examination and the compliance with the following requirements is established:
   - Lack of mechanical faults and traces of corrosion;
   - The interior of volume measure must be degreased and not contain elements that do not belong to the measure;
   - The presence of the identification plate: type, number, nominal value;
   - The presence of air bubbles.
2. Preparatory works for calibration. Before carrying out the standardization of the volume measures to be standardized, the following preliminary works must be carried out:
   - The volume measure to be standardized needs to be adapted to the environmental conditions in the laboratory and must be placed in the room at least 3 hours before the start of the measurement;
   - Check if the environmental conditions meet the requirements specified above;
   - Wet the walls, measure the volume to be standardized;
   - Check the flatness of the volume measures to be standardized;
   - Check the tightness of the standardized volume measures. The volume measure to be standardized is considered watertight if it does not leak water for 3 minutes. If the above condition is not met, the standardization is stopped.
   - After the entire volume has drained from the volume measure to be standardized, the tap is kept open for 30 seconds;
   - Wet the walls of the transfer vessel;
   - The tightness of the transfer vessel is checked. The transfer vessel is considered watertight if there are no water leaks for 3 minutes. If the above condition is not met, the stadardization is stopped.
   - After emptying the transfer vessel, it remains face down for 30 seconds.
   - Calibration of mass comparator and weighing device with non-automatic operation.
3. Description of the method used. The calibration of the volume measures is carried out by the gravimetric method, according to documents [2]. The gravimetric method consists in the indirect determination of the volume of the working liquid from the volume measure to be standardized, from the mass obtained by weighing the full volume measure and after emptying and converting it into volume at the reference temperature t_o =20.0 °C.

Measurements are performed by two methods: a) direct measurement, b) measurement through the transfer vessel.

Tap water is used for the measurements.

a) Direct measurement. The volume measure to be standardized is filled with water up to the nominal value. Insert the temperature transducer into the volume measure to be standardized and record the water temperature indications when they are stable and do not vary. The volume measure to be standardized is placed on the plate of the mass comparator, recording the indicated values (I_L). The water in the volume to be standardized is removed and after the entire volume has drained, the tap is kept open for 30 seconds. Then the valve is closed and the indicated values (I_E) are recorded. A minimum of 3 measurements are performed for each volume measurement.

After weighing the empty and full transfer vessel, the total mass is calculated according to the formula:

$$m_{tot} = (I_L) - (I_E), \quad (1)$$

where: I_L – the result of weighing the full vessel.

I_E – the result of weighing the empty vessel.

If direct measurement is not possible, the transfer vessel is used.

b) Measurement through the transfer vessel. The volume measure to be standardized is filled with water up to the nominal value. Insert the temperature transducer into the volume measure to be standardized and record the water temperature indications when they are stable and do not vary. The moistened transfer vessel is placed on the plate of the mass comparator, and the indicated values (I_E) are recorded. The water in the standardized volume measure is removed in the transfer vessel. The full transfer vessel is placed on the plate of the mass comparator, and the (I_L) values are recorded. A minimum of 3 measurements are performed for each volume measurement.

3. Results and Discussion

The uniformity and accuracy of the measurements in the Republic of Moldova are ensured by a complex of technical-organizational measures, which also includes the activity of transmitting the units of measurement from the national standards to the other standards and from them onwards, to the means of work measurement [7].

In the „Flows and Volumes" laboratory, which operates on the basis of SM EN ISO/IEC 17025 [8], measurements were made with standard volume measures in order to analyze and research the stability of their metrological characteristics. Thus, the measurement data [6] obtained on different days, but under the same conditions, will be presented below.

„The definitions given in the third edition of the International vocabulary of metrology—Basic and general concepts and associated terms (VIM) [9] for the two concepts on which this paper is focused are:

- **measurement uncertainty** non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

and

- **metrological traceability** property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty" [10].

The stability of the metrological characteristics of the volume measures were determined based on the concordance factor En according to SR EN ISO/CEI 17043:2010 [11], between the value obtained from the last standardization and the value obtained during the measurements is calculated according to the formula:

$$E_n = \frac{x_{lab} - x_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}}, \quad (2)$$

where : x_{lab} - the result reported by the participating laboratory;

x_{ref} - the reference value (obtained from reference laboratory determinations);

U_{lab} - the uncertainty estimated by the participating laboratory;

U_{ref} - the uncertainty associated with the reference value (estimated by the reference laboratory).

Thus for:

a) $|E_n| \leqslant 1.0$ shows „satisfactory" performance and does not generate any signal;

b) $|E_n| > 1.0$ shows „unsatisfactory" performance and generates action signals.

The measurement uncertainty was calculated based on the SM ISO/IEC Guid 98-3:2017 Uncertainty of measurement. Part 3: Guide to the expression of uncertainty of measurements [12].

Table 2

The values received as a result of the measurements performed for volume standard measurement, type, M1P-10 ГР-01, M1P-50 ГР-01, M1P-100 ГР-01

Standard volume measure type	Date of measurement	Nominal value, dm^3	Conventional volume, dm^3		Measurement uncertainty, dm^3		En
			X_m	X_{CE}	U_m	U_{CE}	
M1P-10 ГР-01	06.08.2018	10	10.013	10.013	0.00040	0.00019	0.74
	10.10.2018		10.013	10.013	0.00044	0.00019	0.88
M1P-50 ГР-01	08.08.2018	50	50.076	50.077	0.00117	0.00124	0.29
	08.10.2018		50.076	50.077	0.00099	0.00124	0.54
M1P-100 ГР-01	13.08.2018	100	100.126	100.128	0.00142	0.00196	0.65
	17.10.2018		100.126	100.128	0.00142	0.00196	0.65

The standard volume measures whose metrological characteristics have been analyzed are a component part of the national standard [13] of the unit of measurement of the volume of liquids, thus the results obtained confirm the arrangement in the means of measurement held by the National Institute of Metrology and ensure the traceability of the unit of volume measurement both nationally [14] and internationally [15].

Analyzing the data from the tables above, taking into account all the measurement, we can conclude that En factor is less than 1, what means that the standard [11] criteria is met.

4. Conclusions

Standard volume measures, as a component part of the national standard of the liquid volume measurement unit, contribute directly to ensuring the reproduction, preservation and transmission of the volume measurement unit, through the calibration process of standards and measuring instruments, in order to ensure uniformity, the accuracy and traceability of the measurements performed throughout the territory of the Republic of Moldova.

Analyzing the results of the measurements performed, it is observed that the concordance factor En between the value obtained from the last standardization and the value obtained during the measurements for the nominal value (10 dm^3, 50 dm^3 and 100 dm^3) is < 1.0, so according to SR EN ISO/CEI 17043:2010 it is confirmed that the standard volume measure can be used without any intervention. Therefore, it can ensure the traceability of the

volume measurement unit to other standard volume measures of another category. In this way the uniformity, accuracy and legality of the measurements performed in different fields where the volume is measured - for example wine industry, fuel industry is ensured.

Conflicts of Interest: The authors declare no conflict of interest.

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Submission of manuscripts: jes@meridian.utm.md