LASER INTERFEROMETRIC LIQUID MANOMETERS - THE BASIS OF THE STATE PRIMARY ABSOLUTE PRESSURE STANDARD OF RUSSIA

I. V. Sadkovskaya, A. I. Eikhvald

Abstract: In 2011 Laser Interferometric Oil and Mercury Manometers (LIOM and LIMM) for measuring gas pressure in the range 10-1 -103 Pa (LIOM) and 102 -1,3·105 (LIMM) were developed in D. I. Mendeleyev Institute for Metrology. Hydrostatic operating principle in combination with the precision optical method for measuring liquid column height allowed these manometers to become highest accuracy absolute pressure measurement devices in Russia and a basis for the State Primary Absolute Pressure Standard, approved in 2012. This new standard participated in the Key Comparison CCM.P-K4.2012 in the range from 1 Pa to 10 kPa, in which metrological characteristics of LIOM and LIMM were confirmed. Continuous research carried out in VNIIM in the field of liquid manometry provides further improvement of the standard and refinement of its measurement uncertainty.

Keywords: pressure standard, Laser Interferometric Oil and Mercury Manometers, laser interferometric piezometer, measurement uncertainty

Introduction

For gas pressures at present there are two types of primary standards in use: mercury-based U-tube manometers and piston gauges give the most accurate realization of pressure around 100 kPa and may be extended to lower pressures of about 0.1 Pa with oil-based U-tube manometers, or to higher pressures up to about 1000 MPa using piston gauges. Static and continuous expansion systems extend the scale down to 10^{-6} Pa. These systems, however, need traceability to U-tube manometers or piston gauges.

In 2011 Laser Interferometric Oil and Mercury Manometers (LIOM and LIMM) for measuring gas pressure in the ranges 10^{-1}-10^{3} Pa (LIOM) and 10^{2}-$1{,}3\cdot10^{5}$ (LIMM) were developed in D. I. Mendeleyev Institute for Metrology (VNIIM). Liquid manometers became a basis for the State Primary Absolute Pressure Standard of Russia, approved in 2012.

Further improvement of primary standard requires metrological and scientific research in the field of liquid manometry.

1. Laser Interferometric Liquid Manometers developed in VNIIM

1.1 LIOM

Figure 1 shows the operating principle of the LIOM.

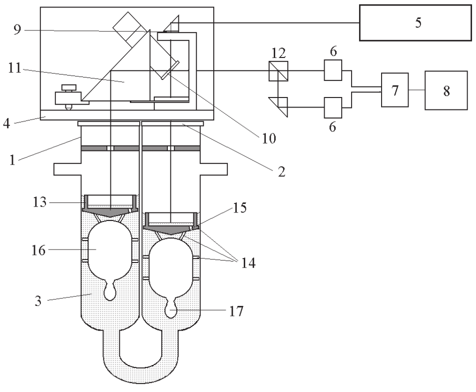

Fig. 1. Operating principle of the LIOM

The U-tube manometer 1, made of stainless steel and closed with flat optical windows 2, contains about 200 ml of the oil 3 (VM-1) for diffusion vacuum pumps. The free surfaces of the oil are serving as mirrors of the Michelson's interferometer 4, which is illuminated by frequency-stabilized He-Ne laser 5. The fringes are detected with two photo-diodes 6 and counted with interface scheme 7 and computer 8.

After evacuation the both limbs (measurement and comparison) of LIOM to residual pressure about $P_0 = 10^{-4}$ Pa, the pressure change in measurement limb causes the shift of interferometric fringes and can be evaluated as

$$P - P_0 = \rho \cdot g \cdot \frac{N \cdot \lambda}{2} \qquad (1)$$

where P and P_0 are the gas pressure values in the measurement and comparison limbs of the manometer, ρ is the density of the oil, g is the acceleration of gravity, N is the number of counted fringes and λ is the wavelength of laser.

1.1.1 Features of the optical system

The laser beam passes through the complementary polarizer 9, which is oriented at 45º to the plane of the drawing. Passing the half mirror 10 and the left limb, it reflects twice in the prism 11 and so receives the polarization near to circular. The second beam remains linearly polarized. Thus the polarization beam splitter 12 and photo-diodes 6 in the output of the interferometer form two interference signals with phase difference about 90º, that is necessary for reversible fringe counting.

The very low reflectivity of the oil surfaces and polarization losses provide the intensity of light returned to the laser low enough not to disturb its stabilization system.

1.1.2 Wave-damping system

The design of special floats is shown in Figure 1. The float consists of two parts. The upper part of the float is the teflon cup 13 with external radius, that is 1 mm less than radius of the manometer tube, and three pins 14, that stabilize its coaxial position with the tube. The cup bottom is flat inside and is cone-shaped from below to let air bubbles exit free when oil is being degassed. The cup has three small holes 15 near walls providing the penetration of the oil inside. The lower part of the float represents a glass sealed cylinder 16 with three glass pins on the top bearing the teflon cup, and six pins directed radial to stabilize its vertical position inside a tube of the manometer. The size of the parts is chosen to ensure the necessary thickness of the oil layer (about 1 mm) in the float. The immersion depth of the float is easily adjusted by changing the ballast glass drop 17, soldered to the bottom part of the glass cylinder.

The investigations have shown such a construction of the floats to reduce fluctuations of free oil surface about two orders of magnitude (from 2-3 to 0.1-0.2 fringes) thus permitting interferometric measurements in a wide range of pressure [1].

1.1.3 The uncertainties of LIOM

The main sources of uncertainty in pressure measurements for the LIOM (Equation 1) are well-known. The greatest contribution to the measurement uncertainty is the uncertainty of the liquid density. The contribution of laser wavelength and acceleration of gravity to the systematic uncertainty is less than 1 ppm.

1.1.3.1 The oil density

The oil density ρ at 20.0° C (ρ = 859.72 kg/m^3) is measured in VNIIM by the standard of density with uncertainty of about 46 ppm. But there is a problem to correct this value for dissolved air and compressibility of the oil.

The density correction associated with the working liquid compressibility of LIOM functioning as a component of State Primary Pressure Standard GET 101-2011 was defined by the use of specially developed interferometric low pressure liquid piezometer. The design of the piezometer, principle of its operation, as well as the results of the oil compressibility measurements will be presented below in a separate section.

1.1.3.2 Capillary uncertainty

The capillary uncertainty of pressure measurement is determined by possible fluctuations of the meniscus curvature radii rather than by their values themselves. We evaluated the rate of these fluctuations by means of direct measurements of the curvature radius using a Michelson interferometer [2]. For this purpose, a manometer tube was inserted into one arm of the interferometer, while a flat mirror was placed into the other one. The interferometer was lit by a parallel He-Ne laser beam. The interference pattern localized near the oil surface in a float, which geometry was similar to Newton rings, was recorded by means of a digital video camera with a PC. As a result of processing the sampling of ten shots taken with different positions of the meniscus, the average value of the meniscus curvature radius was R = 1.40 m with the dispersion δR = 0.04 m. And thus following estimate was obtained for the capillary uncertainty component of pressure measurement:

$$\delta P = \frac{2\sigma}{R^2} \cdot \delta R = 0{,}001\ Pa \qquad (2)$$

where σ is the surface tension of oil.

A relatively small value of the meniscus curvature fluctuations is probably explained by the fact that the floats isolate the meniscus from the manometer tubes, which results in the contact angle fluctuations staying insignificant during the float displacement.

Results of this research show, that there is no need to increase the tube diameters up to more than 40 mm for reducing the capillary uncertainty, as it has been done before usually. So, the device can be made compact enough to reduce mechanical and temperature instabilities of the interferometer.

1.1.3.3 Temperature instability and zero-drift.

To diminish temperature instability the U-tube manometer is placed in a thermostat - water bath, inside of which there is a spiral of copper tube surrounding the manometer body. Water from the external liquid thermostat circulates through the tube.

The uniformity of the temperature is of great importance because it determines the drift of zero point. The special test in which the pressure didn't alter showed that it was about 0.5-1.0 λ per hour. That is well enough because the mean time of the measurement is about 1 – 3 minutes. Another experiment in which the measured pressure was returned to zero gave the similar results.

1.1.3.4 Total uncertainty

The total uncertainty for LIOM is estimated to be $3.6 \cdot 10^{-3}$ Pa + $0.5 \cdot 10^{-4} \cdot p$ in the range 10^{-1} - 10^{3} Pa.

1.2 LIMM

The scheme of Laser Interferometric Mercury Manometer (LIMM) is similar to LIOM' one in the main. The manometer tubes are made of stainless steel and have an inner diameter 50 mm. The homodyne interferometer with frequency stabilized He-Ne laser, optical scheme and fringe counting device are the same applied earlier to the Laser Interferometric Oil Manometer.

1.2.1 Wave-damping system

The idea to suppress surface waves and to stabilize interference by creating thin layer of mercury and using cat's eye device [3] has been realized now in the original construction of floats as shown on Fig.2.

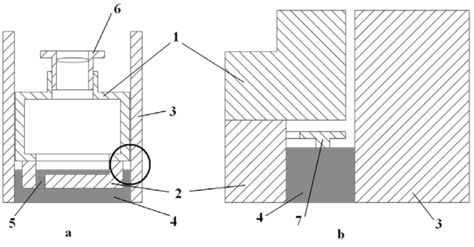

Fig. 2. The construction of the float (a) and its enlarged fragment in the circle (b)

The float stabilizer 1 is a hollow cylinder (49,8 mm in diameter) made of caprolon (polyamide-6). The stainless steel bowl 2 is fixed to it with screws. The gap between it and the wall of the tube 3 is 3 mm. The mercury 4 penetrates into the bowl through the small hole 5 on the bottom and creates a layer with the depth about 2 mm. The lens holder 6 is fixed to the stabilizer with screw- thread to adjust the cat's eye.

The mercury surface tension affects on the float's plunging and depends noticeably on the impurity (mercury oxide). It was found that when the mercury moved down the impurity deposited on the wall of the tube and the surface became cleaner (and vice versa). That could detune the cat's eyes and disturb the fringe counting. To eliminate this effect the fine caprolon guard ring 7 was placed into the gap between the float and the wall. So the state of the surface remains constant during the measurement.

1.2.2 The uncertainties of LIMM

The uncertainty budget of LIMM was obtained on the basis of theoretical analysis and experimental manometer studies [4]. The total uncertainty for LIMM is estimated to be $5.2 \cdot 10^{-2}$ Pa + $4.9 \cdot 10^{-6} \cdot p$ in the range $1 \cdot 10^{2}$-$1.3 \cdot 10^{5}$ Pa.

2. Further research in the field of liquid manometry

In the higher part of the LIOM range the oil's density uncertainty gives the main contribution to the standard's uncertainty. The oil's density is measured at normal conditions (atmospheric pressure), but the real density value at the working pressure is less due to the liquid compressibility.

Laser interferometric piezometer was developed in VNIIM to investigate the liquids compressibility at the working pressures of LIOM.

2.1 The scheme of piezometer

Schematic picture of the piezometer is shown on Fig. 3.

The steel cylindrical vessel 1 filled with the test liquid 2 is mounted in a vacuum chamber 3. The flat mirror 4 is installed under the oil surface at the depth of 2-3 mm. The laser beam from He-Ne laser 5 is directed by the mirror 6 to the center of the oil surface where it is split in two. One of the beams is reflected from the oil surface while the other is reflected from the mirror 4. Then these two beams are directed with the help of the mirror 6 to the screen 8. The resulting interference pattern is recorded by the web camera 9 on the computer 10.

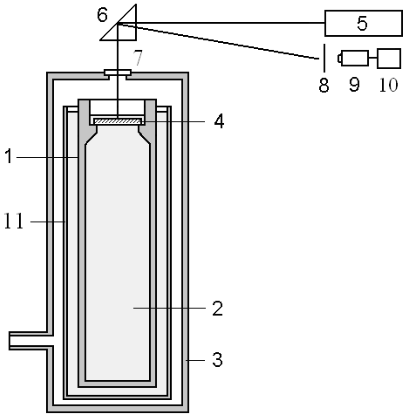

Fig. 3. Schematic picture of the piezometer

For thermal insulation from the ambient air, the cylindrical vessel 1 was placed in a vessel with oil 11 (Fig. 3). Experience has shown that it is completely eliminate all thermal effects connected with the pumping (inlet) of the air in the chamber of the piezometer. In particular, at the end of the measurement cycle, the interference pattern returns to its initial state with an uncertainty of no more than 0.5 fringe.

2.2 Measurement equation and uncertainties

The measurement equation has the following form:

$$\beta=-(1/V_L)\cdot(\Delta V/\Delta p)=$$
$$=(S\cdot N\cdot\lambda)/(2\cdot n_L\cdot V_L\cdot\Delta p)+\beta_V \qquad (3)$$

where S – the cross-sectional area of the vessel, N – the number of counted fringes, λ – the laser wavelength, n_L – the refractive index of the liquid, V_L – the volume of the liquid, Δp – the pressure change, β_V – compressibility of steel vessel.

The number of counted fringes N makes the main contribution to the total uncertainty of measurement due to low resolution ($\delta N = 1$). The other uncertainty components type B – u_B (k=1) are given in Table 1.

The total standard measurement uncertainty was $u_C = 1.9 \cdot 10^{-2}$.

2.3 Measurement results, discussion and conclusions

In accordance with (3) and taking into account the values of the input variables (table. 1) the value of the compressibility of the oil VM-1 β was obtained, which was $(5,2 \pm 0,1)\cdot 10^{-10}$ Pa^{-1}. This means that when the pressure changes by 105 Pa, the density of the oil Δρ/ρ changes by $5.2\cdot 10^{-5}$.

Table 1. Uncertainty components of measurement

Input value	The value of the input variable	u_B
λ	0.632991 mkm	$8\cdot 10^{-6}$
n_L	1.4738	$1\cdot 10^{-4}$
S	$8.04\cdot 10^{-4}$ m^2	$6{,}3\cdot 10^{-3}$
Δp	90 kPa	$6\cdot 10^{-5}$
V_L	$2.730\cdot 10^{-4}$ m^3	$5\cdot 10^{-4}$
β_V	$6\cdot 10^{-12}$ Pa^{-1}	negligible due to the smallness of the input value

According to the results of KOOMET project 724/RU-a/17 “Study of liquids of manometers – primary pressure standards”, obtained in PTB, the working liquid density changes due to liquid outgassing is $6.2\cdot 10^{-5}$. This means that the effect of degassing and pressure reduction on the oil density almost compensate each other, their combined effect leads to a slight change in density, which is ~ $1\cdot 10^{-5}$.

The result of these research allows to conclude that there is no need to make an adjustment to the oil density value, associated with the effects of degassing and compressibility.

3 Participation in the Key Comparison CCM.P-K4.2012

In 2013 Laser Interferometric Oil and Mercury Manometers participated in the Key Comparison in Absolute Pressure from 1 Pa to 10 kPa [5] and confirmed their uncertainties. In 2018 two new lines were added to the International CMC tables (Calibration and Measurement Capabilities) in the field of pressure.

Conclusion

The creation of LIOM and LIMM allowed Russia to participate for the first time in the largest key comparison and to take a place among the leading countries in the field of absolute pressure metrology. Liquid manometers can be used also for measuring gauge-mode pressures. In 2015 in PTB, Germany, the international European project started to create laser interferometric oil micromanometer – LIOM’s analogue.

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Information about the Authors:

Irina Vladimirovna Sadkovskaya

St Petersburg University, Faculty of Mathematics and Mechanics (1980), Candidate of Technical Sciences (2007). Scientific interests: pressure and vacuum metrology. Place of work: D.I. Mendeleyev Institute for Metrology, research department of pressure and vacuum standards. Russia, St. Petersburg, 190005 Moskovsky pr., 19

Web address: *www.vniim.ru*
e-mail addres: I.V.Sadkovskaya@vniim.ru

Aleksey Igorevitch Eikhvald

St Petersburg University, Faculty of Physics (1971), Candidate of Physical and Mathematical sciences (1978). Scientific interests: optics, pressure and vacuum metrology. Place of work: St Petersburg University, Department of General physics. Russia, St. Petersburg, 199034,

Universitetskaya emb., 7-9
Web address: *http://spbu.ru*
e-mail addres: eich47@mail.ru