

MEASURING PRESSURE AND VACUUM WITH LIGHT: A NEW PHOTONIC, QUANTUM-BASED, PRESSURE STANDARD

Jay H. Hendricks¹, Jacob E. Ricker², Jack A. Stone³, Patrick F. Egan⁴, Gregory E. Scace⁵, Gregory F. Strouse⁶, Douglas A. Olson⁷, Donavon Gerty⁸

¹NIST, Gaithersburg, MD USA, jay.hendricks@nist.gov

²NIST, Gaithersburg, MD USA, jacob.ricker@nist.gov

³NIST, Gaithersburg, MD USA, jack.stone@nist.gov

⁴NIST, Gaithersburg, MD USA, patrick.egan@nist.gov

⁵NIST, Gaithersburg, MD USA, gregory.scace@nist.gov

⁶NIST, Gaithersburg, MD USA, gregory.strouse@nist.gov

⁷NIST, Gaithersburg, MD USA, douglas.olson@nist.gov

⁸Sandia National Laboratories, Sandia National Labs, Albuquerque NM, drgerty@sandia.gov

Abstract – The future of pressure and vacuum measurement will rely on lasers and Fabry–Pérot optical cavities, and will be based on fundamental physics of light interacting with a gas. Light interacts with matter such that light travels at a slower speed and has shorter wavelength in gas than it does in vacuum. A photonic-based pressure standard represents a disruptive change in the way of realizing and disseminating the SI unit of pressure, the pascal. The underlying metrology behind the advance is the ultra-accurate determination of the refractive index of a gas by picometer optical interferometry, and when value for molar refractive index is calculated from *ab-initio* quantum chemistry calculations it provides a quantum-based primary pressure standard. The aim of the project is a new technique that will improve accuracy and enable the complete replacement of all mercury-based pressure standards. The National Institute of Standards and Technology (NIST) has now built a working prototype of a fixed length optical cavity (FLOC) with impressive preliminary results. NIST is also developing an even more accurate variable length optical cavity (VLOC) that will make simultaneous ultra-precise measurements of vacuum and gas cavity optical lengths. This paper covers the current status and early prototype results of the photonic-based pressure standard. The early results from the FLOC have exceeded first experimental expectations in that the photonic-based standard is fast, sensitive, accurate, and wide range. Early results demonstrate a pressure resolution of 0.1 mPa (7.5×10^{-7} Torr), outperforming the NIST ultrasonic interferometer manometer by 35X. The lowest pressure measured is 10X more sensitive (1 mPa vs. 10 mPa). Accuracy of the photonic based pressure standard varies between 0.02 parts in 100 (%) at medium vacuum (1 kPa) to 35 mPa/kPa (35 parts in 10^6) at atmospheric pressure (~100 kPa), with repeatability of 5 mPa/kPa (5 parts in 10^6) or better, indicating that the standard, once fully developed, will effectively replace mercury manometers for the barometric pressure range, providing improved functionality without the associated hazards of mercury. **Keywords:** Photonic, Pressure, Metrology, Refractive Index, Primary Standard.

1. INTRODUCTION

Pressure measurements play an important role in everyday life, from controlling feed-stock gas-flow in high technology

semiconductor manufacturing, to monitoring vertical separation minima of airplanes near airports. While pressure measurements are ubiquitous, the standard on which these measurements are based, the mercury manometer, is quite old and traces its early beginnings to 1643. The National Institute of Standards and Technology (NIST) has undertaken a 5 year Innovations in Measurement Science (IMS) program with the aim of replacing legacy mercury manometers with a new, photonically-based standard that relies on fundamental quantum chemistry calculations of helium's refractive index.

Measuring pressure optically represents a paradigm shift in the way the unit is realized. Pressure standards based on refractive index will significantly reduce measurement uncertainties with the added advantage of eliminating mercury manometers, which are expensive to operate and have hazards associated with mercury.

The story of barometric pressure measurements has been a story of the construction and use of mercury manometers. The first mercury manometer was constructed in 1643 by Evangelista Torricelli. Some of the earliest barometric measurements were made by taking the manometer up a mountain, observing the mercury column height along the way, and noting that the column was shorter at the top of the mountain. The pressure unit Torr (1 Torr equals 133.322 Pa) named in Torricelli's honor, is equal to 1 mm of mercury column height, and is still in common usage today.

Improvements in mercury manometers since Torricelli's day have been made by incrementally improving column height measurement accuracies. Today, mercury manometers are still used at 11 National Metrology Institutes, including NIST. The NIST 13 kPa, 160 kPa and 360 kPa Ultrasonic Interferometer Manometers (UIMs) use ultrasound pulses to determine column heights with a resolution of 10 nm or a pressure resolution of 3.6 mPa and provide the world's lowest pressure uncertainties [1-3]. However, further improvements in column height accuracy are very difficult to achieve because they are now at the limit of diffraction corrections. Furthermore, the manometers are large instruments (up to 3 m tall) and contain large quantities of hazardous mercury (up to 250 kg). The hazards associated with mercury spills have been the primary reason why mercury manometers are being phased out of use at many national and corporate metrology

programs, resulting in downgraded pressure-measurement capabilities.

In the search to develop a more compact and mercury-free way to realize pressure, NIST has developed an entirely new optical pressure standard that links the pascal to quantum calculations of helium's refractive index. The new photonic, quantum-based standard will have large impacts on industry, research and world metrology.

2. BASIC THEORY OF OPERATION

The basis of the optical technique is a Fabry–Pérot cavity, where two highly-reflecting mirrors face each other. The Fabry–Pérot technique for refractive index measurement is described in [4] and references therein. Here we employ a modification of the technique which utilizes two Fabry–Pérot cavities built on a single low-expansion glass spacer, with one of the cavities serving as a vacuum reference. Fig. 1 shows one of several possible geometries for a dual Fabry–Pérot device. A laser shines in the end of one cavity and the transmitted light is detected at the other end, after bouncing back and forth inside the cavity multiple times (depending on reflectance of the mirror coatings). When the laser light entering the cavity has a wavelength such that an integer number of half wavelengths exactly fits between the two mirrors, a resonance occurs along with a detected signal intensity maximum at the detector, which is used in a stabilization feedback loop to lock the laser into resonance with the cavity.

The Fabry–Pérot cavity can be used to determine pressure by measuring refractive index n or, equivalently, measuring the refractivity $n-1$. In a first approximation, the refractivity of a gas is simply proportional to the number density of molecules or to $P/(k_B T)$, where P is the pressure, k_B is the Boltzmann constant and T temperature such that:

$$n - 1 \propto P/(k_B T) \quad (1)$$

The proportionality is not exact and in practice it is necessary to modify (1) according to the Lorentz-Lorenz relation and to include virial coefficients to account for non-ideal gas behavior. Details will be discussed in a later publication. The refractive index or refractivity can be determined through frequency measurements of laser light when a laser is servo-locked to a Fabry–Pérot cavity filled with nitrogen (or other gas) whose pressure is to be determined. The light transmitted through a Fabry–Pérot cavity is a maximum (in resonance) when the round-trip length of the cavity ($2L$) is an integer number, m , of laser wavelengths such that:

$$2L = m\lambda \quad (2)$$

Equation (2) ignores small effects such as diffraction corrections and mirror phase shifts. The wavelength of light in the cavity, λ , depends on the speed of light in a vacuum, c , refractive index, n of the gas in the cavity, and on laser frequency (f) according to

$$\lambda = c/nf \quad (3)$$

Combining (1) and (2) and solving for frequency gives

$$f = mc/2nL \quad (4)$$

We can use frequency measurements to make measurements of the cavity length in vacuum or refractive index measurements of the gas in the cavity, and from (1) the

refractive index measurement can be related to pressure. Consequently all needed measurements are based on frequency metrology which provides stunning precision and accuracy. When the value for molar refractive index is calculated from *ab-initio* quantum chemistry calculations [5], this provides a quantum-based primary pressure standard.

2.1. Fixed Length Optical Cavity

In a fixed length optical cavity (FLOC), Fig. 1, one laser is locked to a reference Fabry–Pérot cavity maintained at vacuum while a second is locked to a cavity filled with gas. The frequency difference between the two lasers (the “beat” frequency) is measured to determine refractive index.

The lasers are servo-locked to the cavity resonance so that (4) is always satisfied. If pressure in the measurement cavity changes at constant temperature, causing n to change, the servo adjusts f so as to maintain resonance with the cavity. Equation (4) implies that a small frequency change, df , is proportional to a small change in refractive index, dn , or equivalently to $d(n-1)$, which is turn is proportional to pressure change, dP , such that:

$$df \propto dn = d(n - 1) \propto dP \quad (5)$$

When both cavities are evacuated, a zero pressure beat-frequency is noted, where the beat frequency is the difference between the frequencies of the two lasers. When the pressure in the gas cavity is increased, the laser lock cause the frequency of the measurement laser to change due to the refractive index of the gas changing the wavelength (or speed) of light in the gas cavity. The consequent change in beat frequency can be related to the change in pressure if the temperature and the molar index of refraction of the gas are known.

The lasers are red He-Ne lasers with a wavelength near 633 nm. They have a narrow frequency tuning range that is not sufficient to track the required changes in refractive index; consequently, the mode order m must be changed to keep the laser frequency within its tuning range, and this must be taken into account.

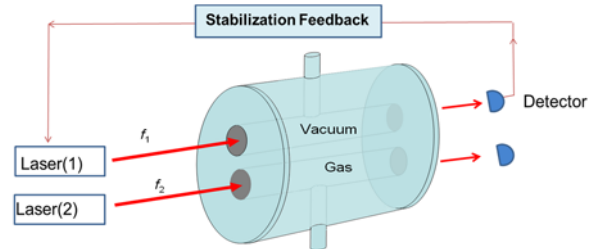


Fig. 1. A fixed length optical cavity realizes pressure by comparing the beat-frequency, f_1-f_2 , as the pressure in gas cavity is increased while the vacuum in the reference cavity is held constant. This standard is simple (has no moving parts) and requires that the temperature of the cavity and molar index of the gas in the cavity is known from either measurements (for example nitrogen) or from theory (helium).

2.2. Variable Length Optical Cavity

NIST is currently developing a variable length optical cavity (VLOC) shown in Fig. 2. In this device, measurements are made at constant pressure so as to avoid distortions that change as a function of pressure, a major source of uncertainty in the FLOC. This provides a path to the much lower uncertainties desired for a primary pressure standard. Four lasers are used with three interferometers in vacuum and the

central cavity filled with a gas such as helium. When one end plate (made of ultralow expansion glass) is moved, it changes the lengths of all four cavities. The change in length of the central gas filled interferometer is precisely measured by the three outer vacuum interferometers. However, the displacement measured by the central interferometer will appear to be different by virtue of the gas refractive index. Having three interferometers in vacuum allows precise measurement and control of angular tilts as the end-piece is translated, and is necessary for correcting Abbe errors. When the displacement is done at constant pressure and known temperature, the VLOC provides a method of measuring the refractive index of the gas in the central cavity. When the refractive index of the gas is known from first-principle *ab-initio* quantum chemistry calculation [5], we have a new quantum-based primary pressure standard.

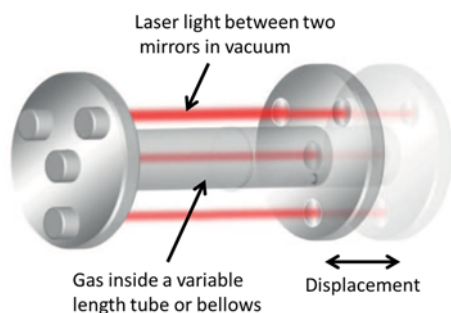


Fig. 2 Variable length optical cavity (VLOC), which will avoid uncertainties associated with pressure distortion.

3. PROTOTYPE FABRY-PÉROT INTERFEROMETER OPTICAL CAVITIES FOR PRESSURE MEASUREMENT

NIST has now built two prototype fixed length optical cavities (FLOC) shown in Fig. 3 and Fig. 4

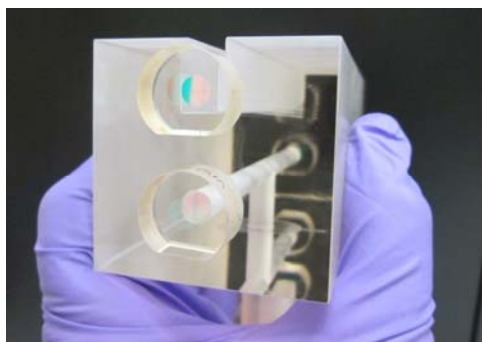


Fig. 3. Working prototype of a fixed length optical cavity (FLOC). A laser is locked to the top cavity which is filled with a gas, while a second laser is locked to the bottom cavity held at high vacuum.

In Fig. 3, the top cavity is filled with a gas to be measured, while the bottom, reference cavity, is held at vacuum. The cavity is 15 cm in overall length and is small enough to hold in your hand. To precisely hold and measure the temperature of the FLOC cavities, copper thermal shells are built so there is a 1 mm gap between the copper and ultralow expansion glass (Fabry-Pérot) cavity. Shown in Fig. 5 is the thermal chamber for the optical cavity design shown in Fig. 3. The temperature of the copper chamber is

measured with a calibrated platinum resistance thermometer (PRT) and was found to be stable to 0.2 mK or better.

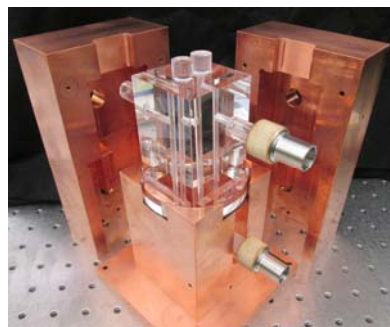


Fig. 4. Another prototype design of a FLOC where both vacuum and pressure cavities are self-contained in a solid spacer of ultralow expansion glass. The cavity is mounted by resting on an o-ring placed underneath the center mounting ring (at the cavity midpoint).



Fig. 5. The copper thermal shell/chamber (top lid removed) is built so there is a 1 mm gap between the ultralow expansion glass Fabry-Pérot cavity (center) and the chamber wall, and is designed to hold the temperature nearly constant while the cavity is filled with gas.

4. RESULTS AND DISCUSSION

First experimental results for the FLOC are shown in Fig. 6. The FLOC was run against an ionization gauge over pressures ranging from 1 mPa to 6 mPa to test the sensitivity of the optical technique. The FLOC demonstrates very high resolution (0.1 mPa), fast response (1 second integration time), and has sensitivity at very low pressures (1 mPa). Note that 0.1 mPa resolution is 35X more sensitive than the NIST mercury manometers which have a typical resolution of 3.6 mPa. Note that for the optical cavity, 1 kHz beat frequency is approximately equal to 1 mPa in pressure.

The photonic technique was also compared to a high accuracy capacitance diaphragm gauge (CDG) with the results being shown in Fig. 7. These results show continued resolution and speed (CDG set to 0.4 second) and linearity over pressure range of 0.5 Pa to 2.7 Pa. Note that the photonic technique is estimated to be 100X to 1000X faster at responding to pressure changes when compared to the mercury barometer.

The photonic technique was finally compared against the NIST mercury UIM primary pressure standard [1-3] with the results shown in Fig. 8 and Fig. 9. Note the $k=2$ uncertainty of the UIM approaches 5 mPa/kPa (5 parts in 10^6) at 100 kPa and the plotted results shows 35 mPa/kPa (35 parts in 10^6) difference between the FLOC and UIM when measuring pressure. The FLOC measures the refractivity of nitrogen gas, and pressure can be determined using a reference value for the molar refractivity [4] and known virial coefficients. The uncertainty in the reference value for nitrogen places a

~50 mPa/kPa uncertainty (50 parts in 10^6) in pressure determination using the FLOC (until further refinements are made) so the 35 parts in 10^6 difference we observe is not unexpected and is a very good “first attempt” measurement result. The difference ranges from 0.02 % to 35 parts in 10^6 . The photonic technique demonstrated this level of accuracy over multiple runs on different days. While the 0.02% value may seem large, this is a factor of 10 better than commercial instruments that have been calibrated such as CDGs.

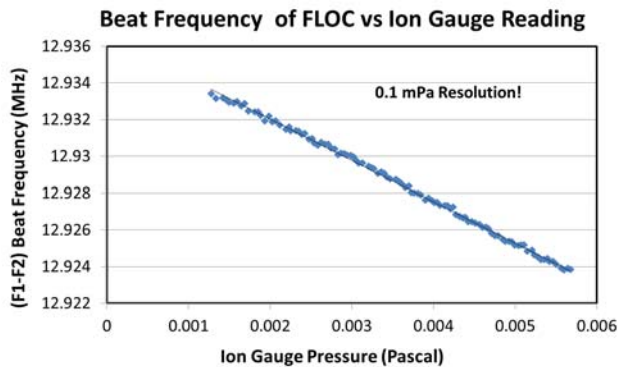


Fig 6. Fixed Length Optical Cavity (FLOC) was run for the first time using an ionization gauge to measure pressure. The photonic technique is high resolution (0.1 mPa), and fast (1 s integration).

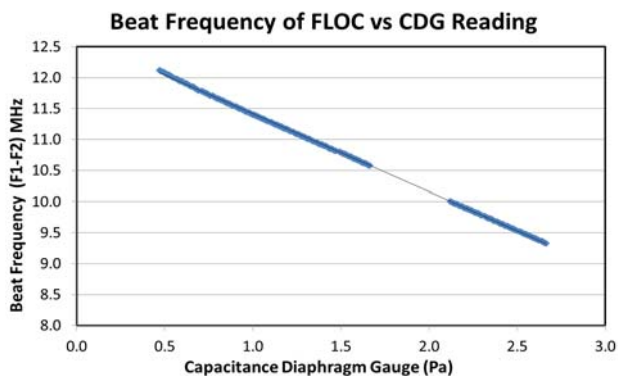


Fig. 7. Photonic technique vs a capacitance diaphragm gauge (CDG) shows continued resolution and speed (CDG set to 0.4 second) and linearity over pressure range of 0.5 Pa to 2.7 Pa.

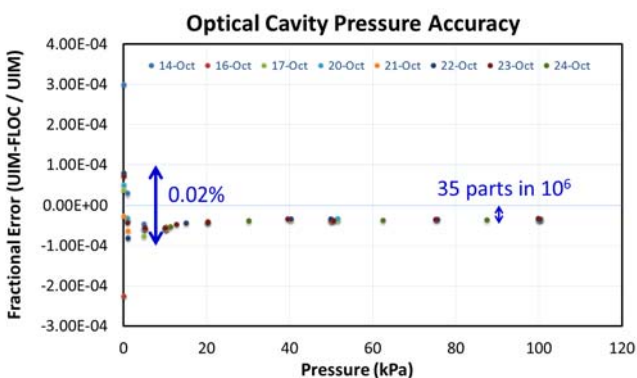


Fig. 8. Difference between FLOC and NIST UIM pressure readings over 8 different days of operation. At higher pressures a difference of 35 mPa/kPa (35 parts in 10^6) is observed between the UIM and the new optical technique.

Shown in Fig. 9 is the repeatability of the FLOC over multiple running days, indicating that it is very stable with 5 mPa/kPa (5 parts in 10^6) repeatability or better. This demonstrates that the FLOC can be calibrated against a UIM and serve as a

highly precise, wide range pressure transfer standard. Finally, the difference (accuracy) between the NIST UIM and the “uncalibrated” optical cavity results shown in Fig. 8 and Fig. 9 may be due to a consequence of outgassing, imprecise knowledge of the refractive index of nitrogen, and uncertainty in pressure-related cavity distortions, all of which are still under investigation.

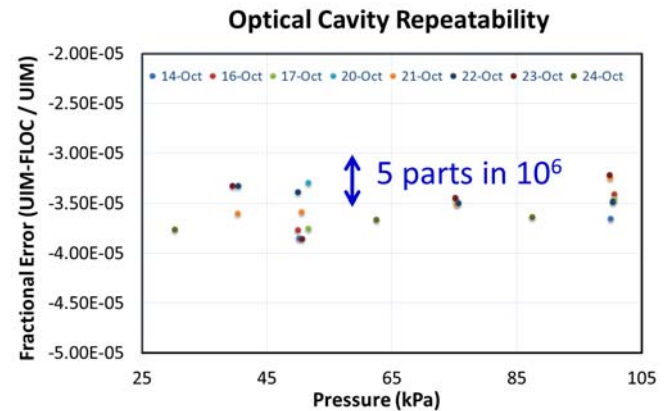


Fig. 9. Repeatability of the fixed length optical cavity is 5 mPa/kPa (5 parts in 10^6) over the range of 30 kPa to 100 kPa. This is comparable to the mercury manometer standards.

5. CONCLUSIONS

The preliminary results show proof of concept and are exciting because they demonstrate that the photonic technique has superior resolution to that of the NIST mercury barometer. In fact, the range to resolution ratio for the instrument is better than any previous single pressure measurement technique. The preliminary data show the optical technique has resolution of 0.1 mPa (7.5×10^{-7} Torr), an accuracy of 0.02% (or better) and repeatability at 100 kPa (~1 atmosphere) as low as 5 mPa/kPa (5 parts in 10^6). The optical device is exciting, not only because it creates new measurement infrastructure for NIST, but also because it can be more easily replicated and deployed wherever high-quality pressure standards are needed, such as in national laboratories and at advanced manufacturing facilities. Future work will focus on refining the photonic technique to better understand uncertainties associated with pressure related cavity deformations and development of the variable length optical cavity that will largely eliminate this issue.

REFERENCES

- [1] A.P. Miller, C.R. Tilford and J.H. Hendricks, “A low differential-pressure primary standard for the range 1 Pa to 13 kPa”, *Metrologia*, **42** (2005) S187-S192.
- [2] J.H. Hendricks, J.R. Ricker, J.H. Chow and D.A. Olson “Effect of dissolved Nitrogen gas on the density of di-2-ethylhexyl sebacate: Working fluid of the NIST oil UIM pressure standard” *Measure*, pp. 52-59. Vol.4 No.2 June 2009.
- [3] J.H. Hendricks, D.A. Olson, “1-15000 Pa Absolute mode comparisons between the NIST ultrasonic interferometer manometers and non-rotating force-balanced piston gauges” *Measurement* **43** (2010) 664-574.
- [4] P. Egan and J. Stone, “Absolute refractometry of dry gas to ± 3 parts in 10^9 ”, *Applied Optics*, vol. 50, no. 2, pp. 3076-3086, July 2011.
- [5] K. Piszczatowski, M. Puchalski, J. Komasa, B. Jeziorski, and K. Szalewicz “Frequency-dependent polarizability of helium including relativistic effects with nuclear recoil terms”, to be published, 2015.