Using volatile liquid FC-40 as pressure transmitting medium for pressure gauge calibrations

H Kajikawa¹, H Iizumi and T Kobata
Research Institute for Engineering Measurement, National Metrology Institute of Japan (NMIJ), AIST, Tsukuba, Ibaraki, Japan

E-mail: kajikawa.hiroaki@aist.go.jp

Abstract. This study investigates the possibility of using a volatile liquid as the pressure transmitting medium for pressure gauge calibrations. A pressure balance used with volatile liquid FC-40 is calibrated against a reference pressure balance with dioctyl sebacate, which is commonly used for pressure calibrations. The boundary surface of the two liquids is monitored and adjusted to a fixed level during the calibrations. The metrological characteristics of the pressure balance with FC-40 are compared with those with dioctyl sebacate. The effective area with FC-40 is quite similar to that with dioctyl sebacate, while the piston fall rate becomes much larger with FC-40. After that, test pressure sensors were calibrated against the evaluated pressure balance in the calibration system filled with FC-40. Since FC-40 can be easily eliminated from pressure gauges and tubing, FC-40 would be a convenient pressure transmitting medium for the calibrations of pressure gauges that are used for high-pressure gas measurements and need oil-free treatment.

1. Introduction

Pressure measurements of pressurized gases are necessary in many industrial facilities, such as high-pressure gas cylinders, gas pipelines, chemical plants, and natural gas stations. The pressure range of use has been extended to higher pressures. Recently, high-pressure hydrogen is used in fuel-cell vehicles and hydrogen stations. The maximum filling pressure reaches as high as 70 MPa for fuel-cell vehicles, and 82 MPa for hydrogen stations in general. Pressure gauges used in such high-pressure devices and facilities need to be calibrated or inspected in an appropriate interval to ensure the reliability of the pressure measurement and the safety during use. The traceability of the data also needs to be secured.

To secure the traceability of pressure measurements in Japan, National Metrology Institute of Japan (NMIJ, AIST) establishes and maintains the national pressure standards from 1 Pa to 1 GPa. NMIJ use dioctyl sebacate as the pressure transmitting medium for the hydraulic pressure standard ranging from 1 MPa to 1 GPa, and nitrogen for the gas pressure standard from 1 Pa to 100 MPa. We have recently extended the gas pressure standard up to 100 MPa by using a liquid-lubricated pressure balance [1], and confirmed its international equivalence through the international comparison APMP.M.P-S6 [2].

Generally, pressure gauges used for gas pressure measurements are calibrated using a stable gas medium, such as nitrogen, dry air, or helium. At high pressures more than several MPa, however, it is
not easy to establish an appropriate calibration system using a pressurized gas, because of the high cost for high-pressure gas devices and safety management. In Japan, a system dealing with pressurized gas is, in some cases, subject to the regulation of the High Pressure Gas Safety Act, depending on the pressure range and the capability for pressurized gas production in the system.

Instead of using a gas as the pressure medium, pressure gauges can be calibrated using liquids, such as oils or water. After the calibration, the liquid inside the gauge is eliminated and the gauge is cleaned. But, this method cannot be applied to the gauges used with oxygen, hydrogen, or other combustible gases, because such gauges need oil-free and/or water-free treatments in all manufacturing and inspection processes.

In this study, we investigate the possibility of using volatile liquids as the pressure transmitting medium for pressure gauge calibrations. One of the candidate materials is Fluorinert FC-40 manufactured by 3M Company, which is a stable, fully fluorinated liquid and is commonly used in semiconductor manufacturing industries. It does not have any oil and water content. In this paper, pressure sensors filled with FC-40 are calibrated against the reference pressure balance with dioctyl sebacate. The boundary surface of the two pressure transmitting media is monitored through the transparent observation window in a pressure cell, and adjusted to a fixed level during the calibrations. The metrological characteristics of the pressure balance with FC-40 are also evaluated and compared with those with dioctyl sebacate. In this paper, the scheme of the experiment and experimental setup are explained in section 2. The metrological characteristics of the pressure balance are shown and discussed in section 3. The calibration results of test pressure sensors with FC-40 are also shown. A brief summary is given in section 4.

2. Experiments
Figure 1 shows the schematics for calibrating test pressure sensors with volatile liquid FC-40. Calibrations were conducted at pressures from 10 MPa to 100 MPa. In method (1), test pressure sensors were calibrated by pressure balance A through a pressure interface (Desgranges & Huot, model 43965), which is a pressure cell with a transparent observation window. The tubing in the test side was filled with FC-40, while the tubing in the reference side was filled with dioctyl sebacate. In method (2), pressure balance B was calibrated by pressure balance A through the pressure interface. The effective area of the pressure balance B and other characteristics were evaluated with FC-40. Then, test pressure sensors were calibrated against the pressure balance B in the calibration system which uses only FC-40. Pressure balance B was model PD-66 manufactured by Nagano Keiki Co. Ltd. The pressure balance has been used and characterized with dioctyl sebacate for about 10 years.

![Figure 1. Scheme of calibration experiments for pressure balance and pressure sensors used with FC-40. The two pressure media, dioctyl sebacate and FC-40, are contacted in the pressure interface.](image-url)
and form a clear boundary surface. The clear surface was also seen at higher pressures. During the measurements, the position of the boundary surface was always monitored by a CCD camera, and was adjusted within ±0.3 mm at the tip of the indicating cone located at the center in the pressure interface. It is worth to note that the boundary of the two liquids was kept stable during both the pressure increase and decrease processes.

Head differential pressure needs to be evaluated and corrected for precise pressure calibrations, but the density data of FC-40 at high pressures have not been provided; only the density data at atmospheric pressure is available [3]. We evaluated the density of FC-40 in-situ by precisely detecting head differential pressure generated by 1000 mm height difference. The measurement principle and typical procedures are explained in [4, 5].

![Diocyl Sebacate](image1)

**Figure 2.** Photo of boundary surface between diocyl sebacate and FC-40 in pressure interface.

3. Results and discussions

3.1. Metrological characteristics of pressure balance with FC-40

Metrologie characteristics of pressure balance B were evaluated with FC-40. The piston fall rate with FC-40 is larger than that with diocyl sebacate; the fall rate at 100 MPa is 0.35 mm/min with FC-40, and 0.22 mm/min with diocyl sebacate. The difference comes from the difference in viscosity. The viscosity of FC-40 under atmospheric pressure at 25 °C is 4.1 mPa·s [3], which is roughly one-fifth of that of diocyl sebacate.

Figure 3 shows the effective area evaluated from the calibration against the reference pressure balance in Method (2). The results are compared with the effective area which have been evaluated with diocyl sebacate. The two results are almost the same, showing no distinctive effects of the pressure medium, although the piston fall rate differs depending on the pressure medium.

The sensitivity and the short-time stability of the generated pressure were also evaluated. Then, totally, the pressure balance B with FC-40 showed similar performance to that with diocyl sebacate, and proved to be used as the standard device for pressure gauge calibrations.

![Effective area plot](image2)

**Figure 3.** Effective area of piston-cylinder in pressure balance B used with FC-40 (filled circles) and diocyl sebacate (open circles).
3.2. Calibration results of test pressure sensors

Figure 4 shows calibration results of test pressure sensor (DH Instruments, RPM4) by Method (1) and Method (2). The calibrations were conducted in pressure ascending order from 10 MPa to 100 MPa in steps of 10 MPa. The deviation of the test sensor's output from the standard pressure was plotted against the measurement pressure. The results by the two methods agree well with each other in all the pressure range. The two results are also compared with those calibrated using dioctyl sebacate directly by the reference pressure balance A. The maximum difference of the results was 0.6 kPa at 100 MPa, relatively 6 parts per million (ppm), which is close to the repeatability in the three runs and much smaller than the relative combined standard uncertainty, 22 ppm. We will also check the consistency of the results with those calibrated using gas medium such as nitrogen or helium.

![Figure 4. Calibration results for test pressure sensor (DH Instruments, RPM4) by Method (1) and Method (2). Results with dioctyl sebacate are also shown for comparison. Error bars represent the standard uncertainty of the calibration results with dioctyl sebacate.](image)

4. Summary

We used volatile liquid FC-40 as a pressure transmitting medium and conducted calibrations of pressure gauges using a reference pressure balance used with dioctyl sebacate. The boundary surface of the two liquids was monitored and adjusted to a fixed level during the calibrations. The metrological characteristics of the pressure balance with FC-40 were evaluated and compared with those with dioctyl sebacate. The effective area with FC-40 is quite similar to that with dioctyl sebacate, while the piston fall rate largely differ reflecting the difference in the viscosity between the two liquids. After that, test pressure sensors were successfully calibrated against the evaluated pressure balance in the calibration system filled with FC-40. Using a volatile material as a pressure transmitting medium enable us to calibrate pressure gauges with oil-free and water-free requirements efficiently and less costly.

[1] Iizumi H, Kajikawa H and Kobata T 2017 Measurement 102 106
[2] Kajikawa H, Olson D A, Iizumi H, Driver R G and Kojima M 2016 Metrologia 53 TS03002
[3] Product information of Fluorinert™ Electronic Liquid FC-40 (provided by 3M Electronics Markets Materials Division, 3M Company)
[4] Kobata T and Kajikawa H 2017 Abstracts in 6th CCM International Conference on Pressure and Vacuum Metrology, 5th International Conference IMEKO TC16 (Pereira, Colombia) p 35
[5] Kajikawa H and Kobata T 2017 Proc. SICE Annual Conference 2017 (Kanazawa) (SICE) p 1604