SAND2016-2465C

OPTIMIZING THE PERFORMANCE OF THE COMPARE II LEAK CALIBRATION SYSTEM

Speaker: Timothy A. Moss
Sandia National Laboratories
P.O. Box 5800, MS0665
Phone: (505) 844-7356, Fax: (505) 844-4372
Email: tamoss@sandia.gov

Authors: Timothy A. Moss and Herb Case

Primary Physical Standards, Sandia National Laboratories, Albuquerque, NM, USA

Abstract

The Compare II leak calibration system at Sandia National Laboratories has been used for the secondary calibration of leaks for over 30 years. This system calibrates leaks by the direct comparison of an unknown leak with a standard reference leak using a commercial leak detector. Over the years the leak detector remained the same but other components were changed such as replacing the oil diffusion pump with a turbomolecular pump, a new temperature chamber for the unknown leak, and replacing a rotary vane pump with a molecular drag pump. Testing on this system was conducted for two reasons. First: determine the optimum operating conditions to enable this system to be used at peak efficiency. Second: this testing will help in determining the new system's requirements since this system is planned to be replaced with a newer system in the near future. Some of the tests included determining the time for the leak rate to stabilize when first installed on the system prior to calibration, and for the leak rate to stabilize when switching between the standard and unknown leak. Data for these and other tests conducted to optimize the performance of the Compare II leak calibration system are presented in this paper. The resulting improvements reduced calibration time from nine hours to five hours and increased the number of leaks calibrated per week from three to five, which significantly increased operational efficiency and reduced turnaround time for the customer.

1 Introduction

A program to systematically characterize the operation of the Compare II Leak Calibration System was undertaken with the goal of increasing the operational efficiency of the Compare II system resulting in reducing the turnaround time for customer's leaks. The Compare II system calibrates approximately 80 % of all leaks calibrated by the Primary Standards Lab at Sandia National Laboratories. In basic terms, the system calibrates leaks by comparing the output signal of the leak detector between the standard leak and the unknown leak, or the leak being calibrated. The output of the leak detector is linear over the selected range so, for example, if the unknown leak has an output ½ of the standard leak output the unknown leak rate is ½ the leak rate of the standard. The standard leak is calibrated by one of the fundamental leak calibration systems in the lab, described in more detail in ref's 1 and 2.

The Compare II system uses a Leybold-Heraeus Ultratest F leak detector approximately 30 years old. It was updated some years back from an oil diffusion pump to a Leybold-Heraeus Turbovac 151 turbomolecular pump backed by a Leybold-Heraeus Trivac D4A rotary vane pump. The other pump on the vacuum manifold was a rotary vane pump that was replaced by an Adixen Drytel 1025 Molecular Drag Pump station using a diaphragm pump as the backing pump, which makes this pump station oil free. See Figure 1 for a schematic and a picture of the system.

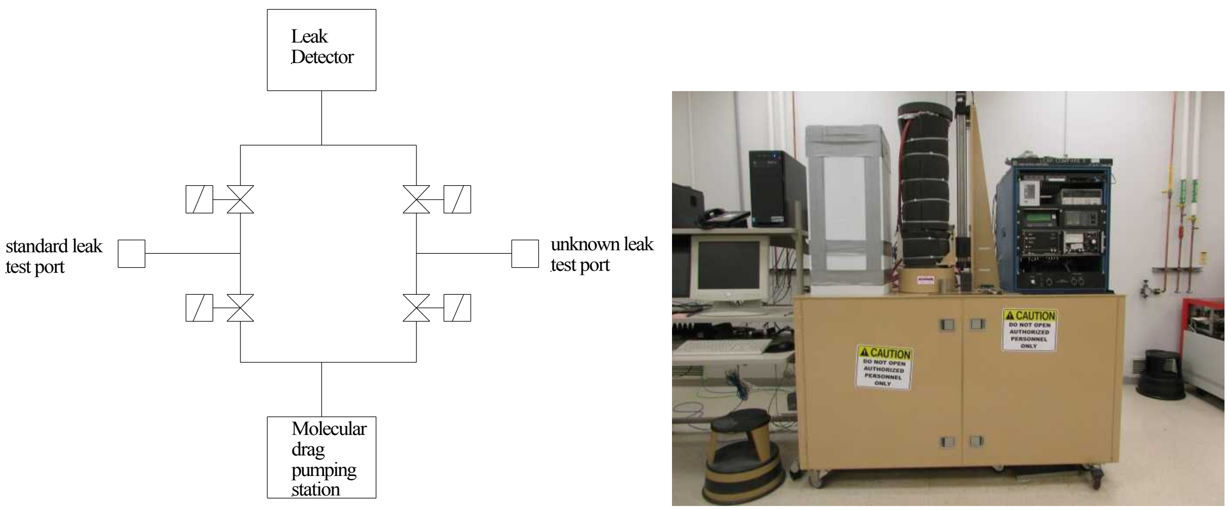

Figure 1: Leak Compare II system: the left is a schematic of the vacuum manifold; the right is a picture of the Leak Compare II.

Most of the leaks calibrated on this system are permeation leaks where the He gas permeates through a borosilicate glass tube. The permeation rate of the He gas through the glass tube has a large temperature coefficient, usually 3 to 4 %/°C. With this high temperature coefficient it is important to control the temperature of the leak during the calibration. To accomplish this two separate temperature chambers are used on Compare II. The temperature of the standard leak does not need to be varied during a calibration but it does need to be stable so its temperature chamber is a box with inside dimensions of 11”x 10.5”x 30” made from 2” thick rigid blue Styrofoam board. Three layers of bubble wrap bags are put around the leak inside the foam box. No means of artificially circulating the air is used inside the chamber. A calibrated stainless steel sheathed platinum resistance thermometer (PRT) probe is tied to the outside of the standard leak using Velcro strips with the tip of the probe, where the actual temperature is measured, placed approximately at the level of the leak element itself.

The Primary Standards Lab certifies leaks at a specified temperature, usually 26.67 °C. Because of the large temperature coefficient a linear equation is given to correct for the leaks’ usage at a different temperature over a small range, usually ±5 °C. During a calibration the leak rate of the unknown is measured at two temperatures, one at 26.67 - 5.0 °C and 26.67 + 5.0 °C in order to obtain its temperature coefficient. The temperature chamber for the unknown leak is actively controlled by using a ¼” thick copper cylinder, 7.625” ID and 29.75” high inside with a soldered ¼” copper plate endcap on top and spiral wound 3/8” copper tubing soldered on the outside. To change the unknown leak, the temperature chamber is raised and lowered using a 24 VDC stepper motor. The bottom plate is also made of ¼" copper plate with 3/8" copper tubing coiled and soldered on the bottom. The temperature of the chamber is controlled using a recirculating water bath to circulate water through the tubing. The temperature chamber is insulated with 1" thick black packing foam. A blower mounted near the middle on the bottom plate and pointing up is used to circulate the air inside the unknown temperature chamber. Just as for the standard leak, a calibrated stainless steel sheathed PRT probe is tied to the outside of the unknown leak using Velcro strips with the tip of the probe placed approximately at the level of the leak element itself.

2 Test Results

2.1 Temperature chambers:

Neither of the temperature chambers has been tested to determine the uniformity of the temperature distribution inside the chamber. A thermocouple (TC) tree was constructed using nine high accuracy type T thermocouples. The thermocouples were measured using a Fluke 1586 Super-DAQ having a combined accuracy of ±0.71 °C.   The nine thermocouples were arranged into three groups of three inside the chamber; three near the top, three in the middle, and three near the bottom. This arrangement was selected to best characterize the temperature distribution inside the chamber. The left side of Figure 2 shows a picture of the TC tree placed inside the standard leak's temperature chamber (shown inside the three layers of bubble wrap) and the right side shows a picture of the TC tree placed inside the unknown leak's temperature chamber.

Figure 2: On the left, the thermocouple tree used inside the standard leak temperature chamber. On the right, the thermocouple tree used inside the unknown leak temperature chamber.

The first test was to determine the temperature stability inside the standard temperature chamber and how long it took for the temperatures to stabilize when the standard leak was changed. The standard temperature chamber has no active temperature controls, but is set by the room temperature controlled to 23.5 ± 1.0 °C. Changing the standard leak requires the removal of the Styrofoam box and the three layers of bubble wrap. The temperature chamber was placed over the standard 10 minutes after the start of the test. The results of the test are shown in Figure 3.

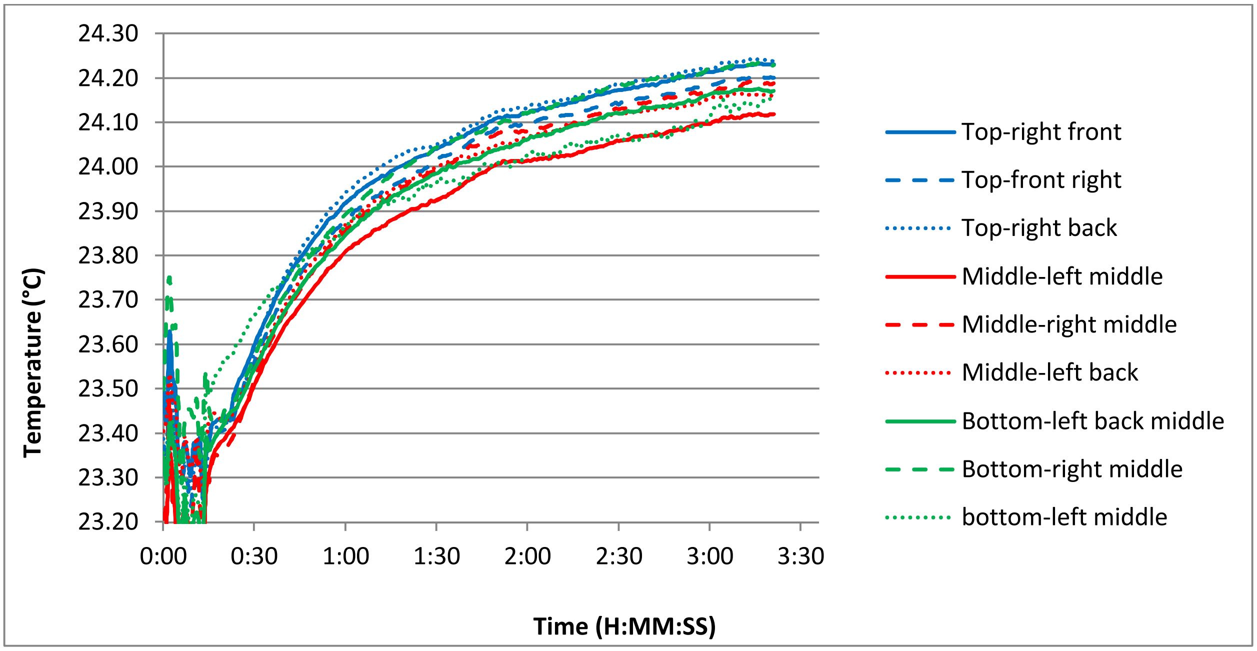

Figure 3: Temperature map of the standard leak showing the time to reach temperature stability after placing the temperature chamber over the standard leak.

The data shows it takes about 3½ hours for the standard leak to reach stable temperatures with a temperature difference of 0.15 °C between the lowest and highest temperatures inside the Styrofoam box.

The next test was to determine the temperature stability inside the standard temperature chamber during a normal calibration run. Data collection from the TC tree was started 1.5 hours after the start of a normal calibration run. The calibration run was started after both the standard and unknown leaks were placed on the system and stable temperatures established for over 24 hours. The data, shown in Figure 4, illustrates how stable the last part of the unknown's first temperature setting for the calibration is. As the temperature of the unknown is increased for the second calibration temperature, however, the air temperatures inside the standard's temperature chamber uniformly increases by 0.35 °C. Note how the TC's on the right side of the chamber, which is the side closest to the unknown chamber, has the warmest temperatures in its group. The temperature for the standard starts increasing when the temperature of the unknown leak starts increasing and both leaks took about 2.25 hours for the temperatures to stabilize, which is the same time it took for the temperature of the unknown leak to stabilize. As for the earlier test, there was a temperature difference of 0.15 °C between the lowest and highest temperatures inside the Styrofoam box.

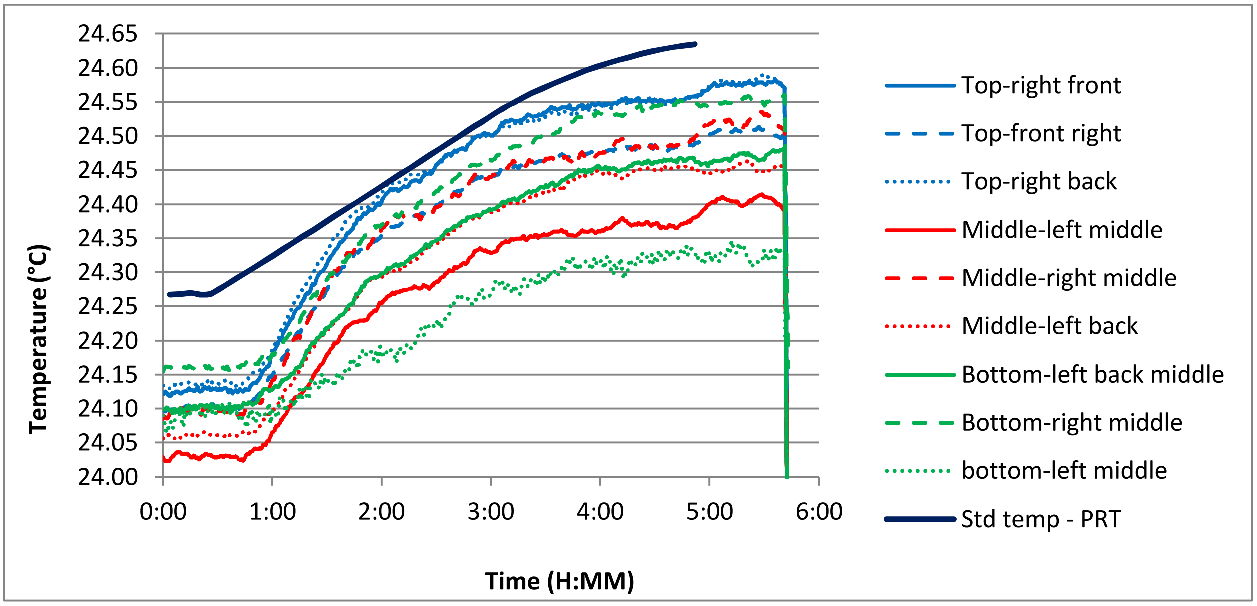

Figure 4: Chart showing the air temperature distribution inside the standard chamber during a normal calibration run. Note how the temperatures change as the temperature of the unknown is changed.

For the next test the TC tree was moved to the unknown temperature chamber. The test results from a normal calibration run are shown in Figure 5. The temperature of the leak was measured by the PRT normally used to measure the temperature of the unknown leak during a calibration. The calibration run was started after both leaks were stable at the desired temperature overnight. This data collection started the same time as the calibration run was started and ending long after the end of the calibration run, as shown by the lack of data from the PRT after 6 hours from the start of the test. Data from the PRT is collected by the calibration DAQ system and only when the leaks are actively being measured, thus there is no data from the PRT when the temperature of the unknown is being changed. Data from one of the TC's in the middle is not shown because it had shorted out at the terminal block. Notice after 7 hours into the test the temperatures changed by about 0.5 ºC and then returns about two hours later. This interesting anomaly has been seen several times but happens only when the temperature of the unknown chamber is held at one temperature for several hours and, fortunately, never during a calibration run. The reason for this anomaly is unknown.

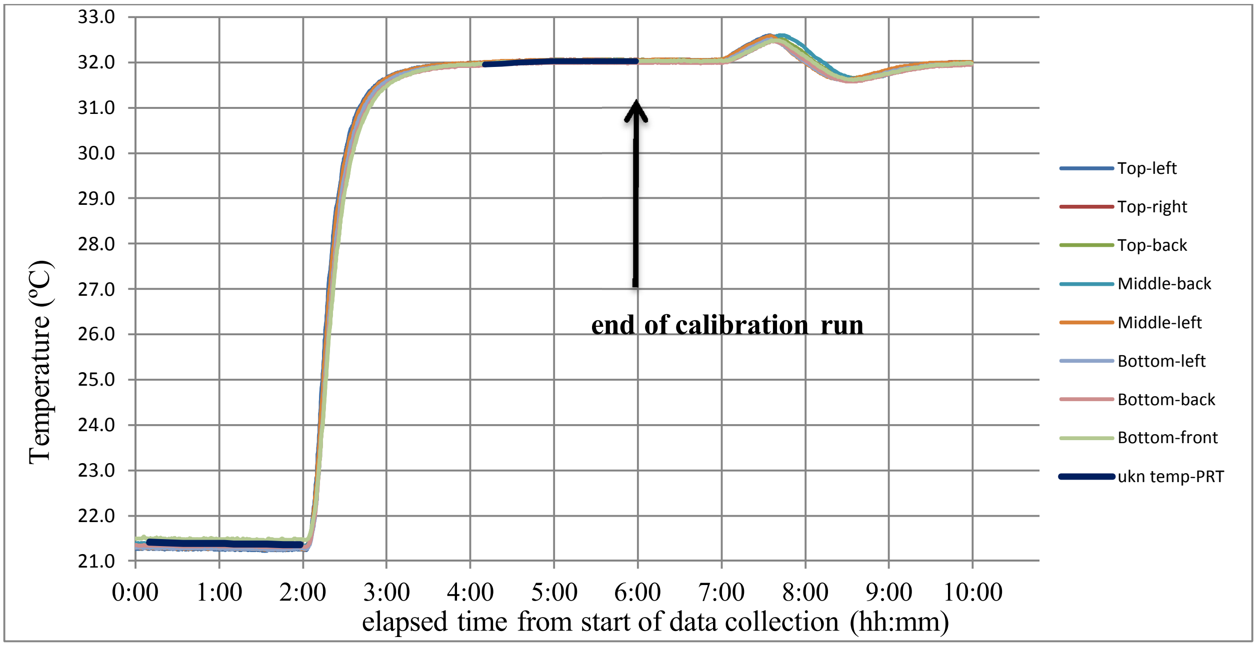

Figure 5: Temperature data from a normal calibration run for both the TC tree and the PRT inside the unknown temperature chamber. Data from the TC tree was collected after the end of the calibration run.

Figures 6 and 7 shows the temperature stability of the unknown temperature chamber at the lower and upper temperatures during a calibration. The temperatures at the lower temperature set point are more stable because the temperature was allowed to stabilize overnight before starting the calibration. The PRT temperatures appear to be more stable because the PRT is measured once every five minutes and the TC's are measured once every second. The maximum change for the upper temperature set point is 0.12 °C during the calibration. There is a maximum temperature difference measured by the TC tree of 0.22 °C inside the unknown temperature chamber, which shows the fan being used is adequately circulating the air and maintaining a stable temperature throughout the inside of the unknown chamber.

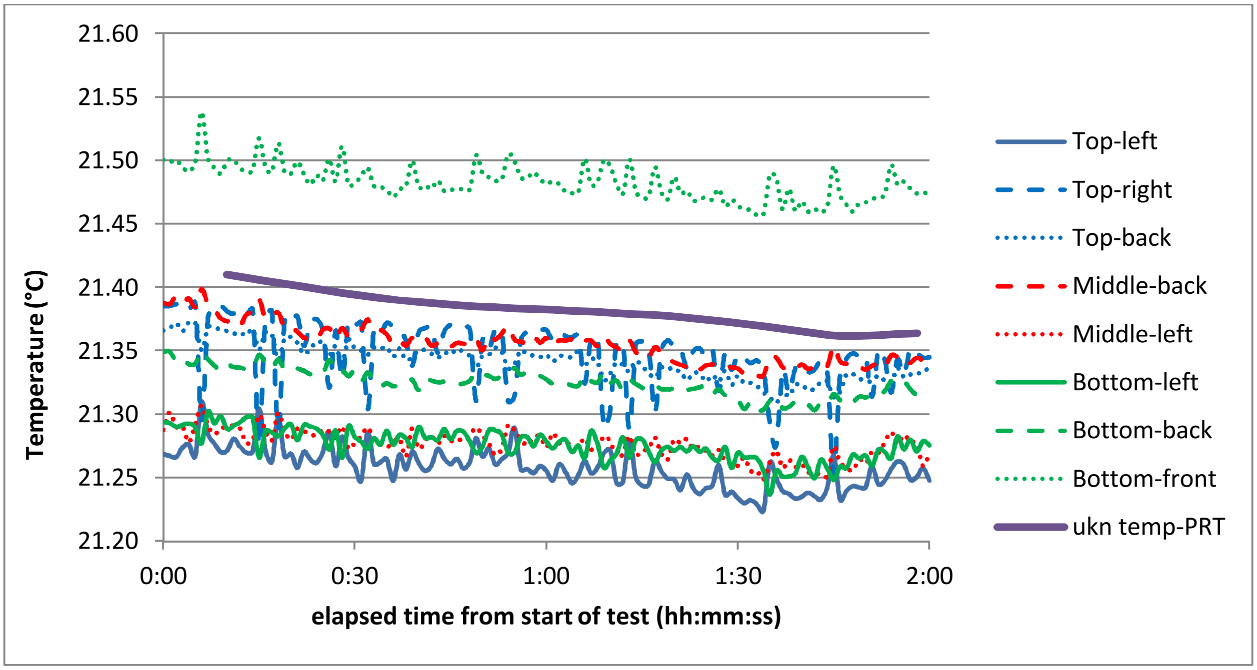

Figure 6: Temperature stability of the unknown leak at the lower temperature of a normal calibration run.

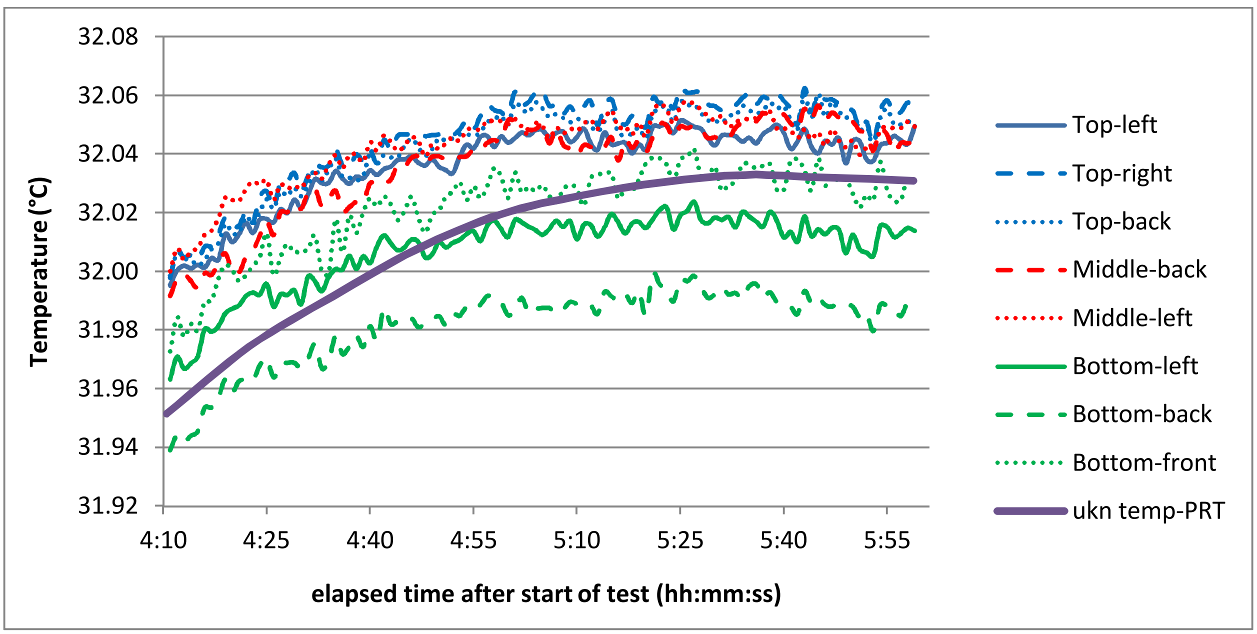

Figure 7: Temperature stability of the unknown leak at the upper temperature of a normal calibration run.

The TC temperatures from the TC tree shown in Figures 3 thru 7 have been corrected to account for the differences between the TC's when measuring the same temperature. This was done by tightly bundling all the TC's together using a rubber band, inserting the bundle into a packing foam block, put inside a plastic bubble wrap bag and placed inside a tightly controlled temperature chamber overnight. The next morning the TC's average temperature reading over a 154 minute time period was measured. TC 1 was randomly chosen to be the reference. The differences between the averages of the TC's from TC 1 were then used as the temperature correction in all the TC data.

2.2 Circulating fan:

The original circulating fan for the unknown temperature chamber was a 120 VAC piezoelectric fan with two blades. No data is available for this fan because it is no longer manufactured and was originally installed in the early 1980's. An internet search found a similar item rated for 4 CFM. Shortly after the start of this work one of the two fan blades stopped working and it was replaced with a 12 VDC fan with a rating of 2.8 CFM, which is roughly 3.5 air exchanges inside the chamber every minute, see Figure 8. The time needed to reach temperature stability when making a 10 degree change in temperature (standard temperature change during a calibration) with the original fan with two blades was 2 hours 35 minutes with a stability of 0.092 °C in a one hour time period. When only one of the blades worked properly the time went to 4 hours 30 minutes with a stability of 0.161 °C in a one hour time period. Using the DC blower the time to reach temperature stability was 2 hours 17 minutes with a stability of 0.058 °C in a 1 hour time period. A second DC blower, identical to the first, was added to more closely match the overall flowrate of the original piezoelectric fan. The time to reach temperature stability was reduced to 1 hour 5 minutes and with an increased stability of .015 °C in a 1 hour time period. Be aware of how much heat the blowers you select put out, which can significantly impact temperature stability. These DC blowers use 1.44 watts each.

Figure 8: On the left is a schematic of a one blade piezoelectric fan. On the right is a picture of a mini DC blower.

2.3 Reducing leak rate stabilization time when installing leak on vacuum system:

This series of tests were to determine the length of time needed to pump on a leak when first installed on the vacuum system in order to reach a stable leak rate from the leak itself. For this set of tests the leak temperatures were held constant. Leaks are to always have the outlet valve open. Permeation leaks take time to develop the permeation gradient through the glass membrane when not under a vacuum such as during shipping or between uses. The leaks being used as standards are under a vacuum, even when not on the Compare II system. The leak detector's turbomolecular pump has a He compression ratio of 20,000. The compression ratio is the ratio between the outlet pressure and the base pressure of the pump that is dependent on the molecular weight of the gases being pumped. The higher the compression ratio the more difficult it is for gases to backstream through the pump back into the high vacuum system. The vacuum pump on the other half of the vacuum manifold is an Adixen Drytel 1025 Molecular Drag Pumping Station, which has a He compression ratio of 250. The standard leak on the system had been installed on the system for many days. The unknown leak, a mid 10^{-10} cc/s @ STP leak that had been in air for several days, was installed in the unknown port and the test started immediately after sufficient vacuum was achieved. For this test the standard calibration was changed to run at one temperature only and complete 150 runs instead of the usual 10 runs at each temperature setting, which took a little over 30 hours to complete. The test data shown in Figure 9 shows the average voltage signal from the leak detector for the unknown leak over time. Notice how it takes almost 30 hours for the leak rate of the unknown to stabilize. The average leak rate signal at 29 hours is 15.6 % lower than it was at 4 hours.

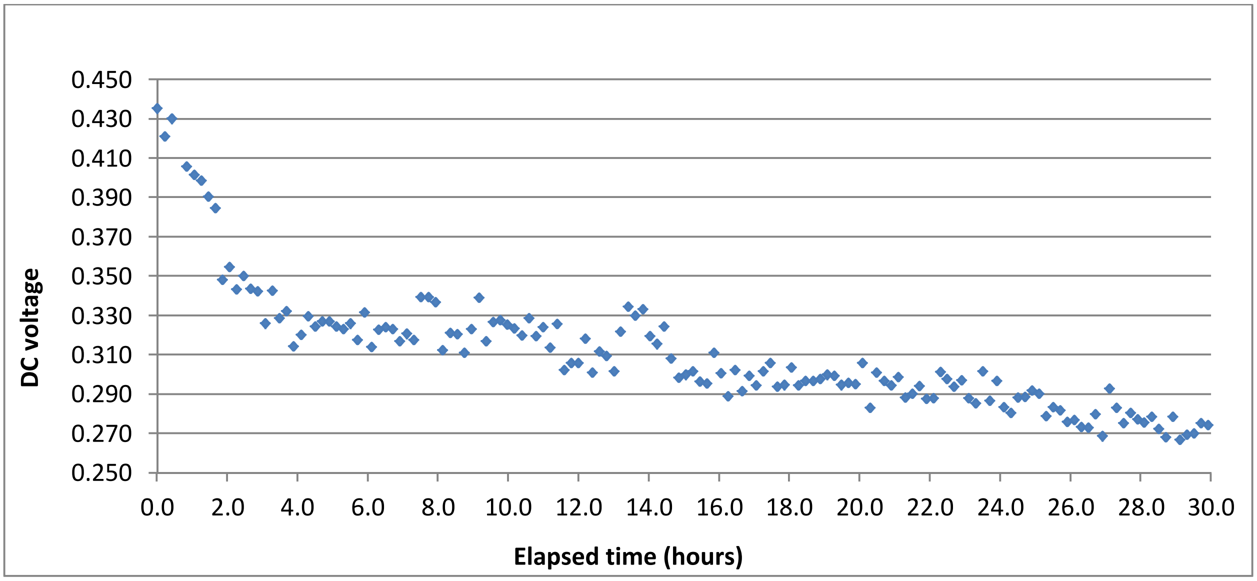

Figure 9: Test data showing the voltage signal of the unknown leak over time after initial installation on the Compare-II system using a molecular drag pump.

The above test was repeated using a low 10^{-9} cc/s @ STP leak in the unknown port under the same circumstances. This test showed it takes about 7.5 hours for the leak rate to stabilize. As expected, a leak with a higher leak rate took less time to reach stability.

The Adixen Drytel 1025 Molecular Drag Pump station with a He compression ratio of 230 was replaced with a Pfeiffer HiPace 80 turbo pump station with a He compression ratio of 13,000,000. The same test was repeated using another mid 10^{-10} leak under the same circumstances. The average voltage signal from the unknown leak is shown in Figure 10. This test shows the signal stabilizing in less than 2 hours. There was a 0.3 % change in the average signal between 1 hour and at 52 hours. This clearly demonstrates the advantages of using vacuum pumps with high He compression ratios.

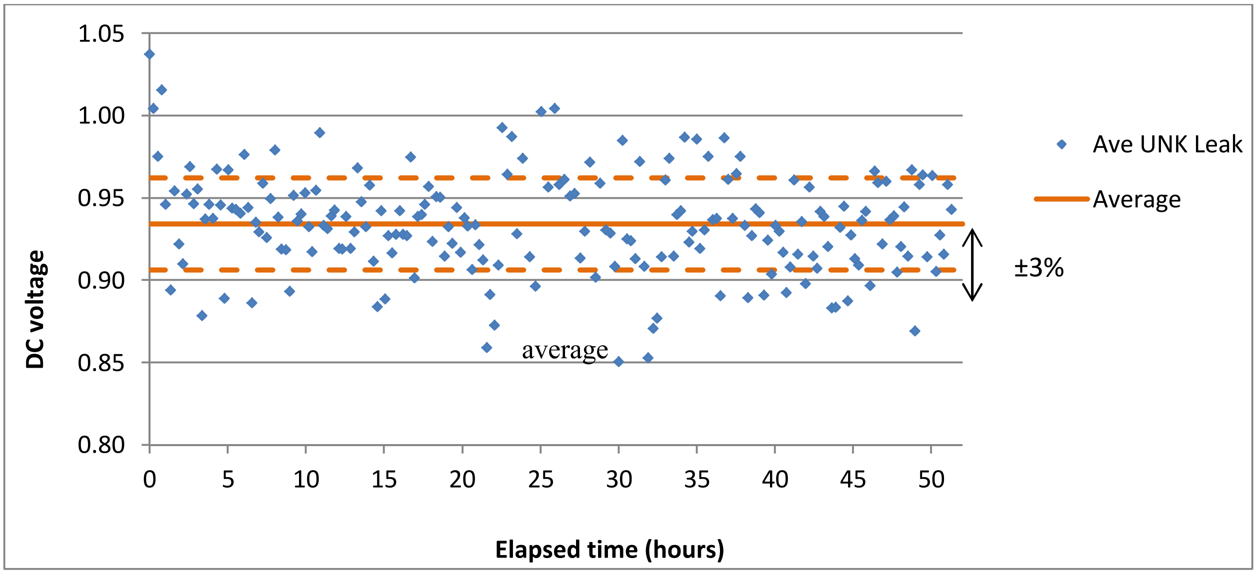

Figure 10: Test data showing the voltage signal from the unknown leak over time after initial installation on the Compare-II system using a turbomolecular pump.

2.4 Using two temperatures vs. three temperatures in the straight line fit for calibration:
Since the 1970's when leaks were first being calibrated, only two different temperatures were used, typically 10 °C apart with the center at 26.67 °C. A best fit straight line to the data is used to calculate the reported leak rate at the center point. If the leak is used at another temperature within 5 °C of 26.67 °C the leak rate is determined by using the best fit linear equation because of the large temperature coefficient for permeation leaks, which it typically 3.5 to 4.5 %/°C. It is known the temperature coefficient of a permeation leak is not linear with temperature, but with only a 10 °C difference it was assumed reasonable to use a straight line fit. To verify this three different leaks, an 10^{-7}, 10^{-8}, and 10^{-9} cc/s @ STP were calibrated using three temperatures with the same upper and lower temperatures as usual, but a third temperature of 26.67 °C added. For all three leaks, the calculated leak rate at 26.67 °C was between 0.44 % and 0.45 % lower when using three temperatures for the straight line fit compared to using only two temperatures, which is caused by using a straight line to approximate a non-linear function. This difference is not significant; however, it was decided to use three temperatures in the future based on the greater accuracy over the certified temperature range. Adding a third temperature will increase the time to complete a calibration but will not impact turnaround time since it will still be one leak per day on the system.

2.5 Zero stability:
The stability of the zero reading over time on the leak detector is important because the zero is measured only twice, once at the beginning and once at the end of each run with a typical run taking 12.5 minutes to complete. The test data showing the stability of the zero over time is shown in Figure 11. To reduce noise, each data point shown on the graph is a moving average of ten readings where each reading is taken once per second. To determine the unknown's leak rate the average of the two zero readings is subtracted from the average readings for each of the two leaks and then the unknown's voltage signal is compared to the standard's voltage signal. The data shows the zero can drift ± 0.02 volts during a 12 minute period, the time it takes to complete

a run. This could lead to a significant change in the calculated leak rate if the voltage signal of one of the leaks is small and the change occurs anywhere between the beginning and the end of a run. To minimize the effect of any zero drift during a run more zero readings were added into the valve sequence. The old run sequence, shown first, and the new sequence are shown below:

zero 1, std 1, ukn 1, sum 1, ukn 2, std 2, ukn 3, sum 2, ukn 4, std 3, zero 2
zero 1, std 1, ukn 1, sum 1, zero 2, ukn 2, std 2, sum 2, zero 3, std 3, ukn 3, sum 3, zero 4

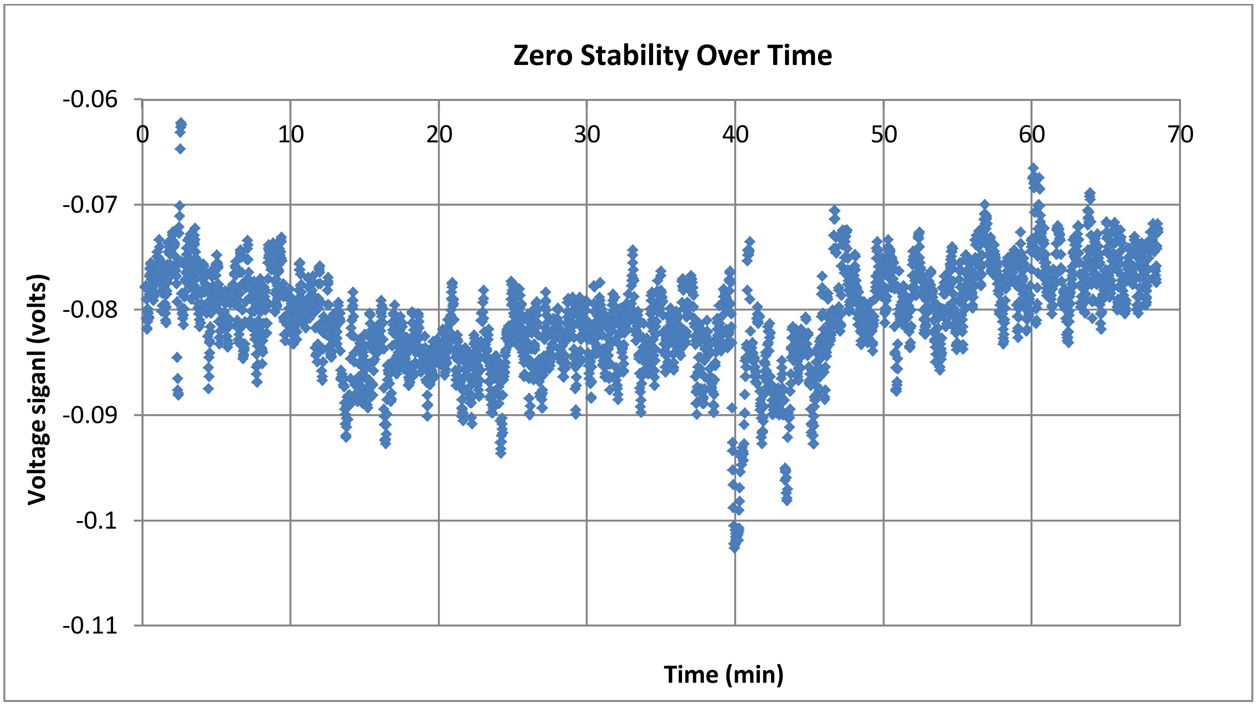

Figure 11: Chart of the moving average of ten readings over time

2.6 Leak detector sensitivity test:
A test was run to determine the capabilities of the leak detector when one leak is two orders of magnitude different than the other leak. This test, shown in Figure 12, was performed because there is increasing interest in calibrated leaks with a leak rate below 10^{-10} cc/s @ STP. Currently, the Primary Standards Lab can only fundamentally calibrate a leak as low as low 10^{-9} cc/s @ STP. A 2.5×10^{-10} cc/sec leak was placed on the standard port and a 2.8×10^{-8} cc/s leak was placed on the unknown port of Compare II. The calculated leak rate of the unknown leak was less than 2 % different then its calibrated value when using a standard leak much closer to its leak rate, however, the assigned uncertainty is much higher because of the low signal-to-noise ratio. This test shows the system is able to calibrate a leak when one leak is two orders of magnitude different than the other, but at a cost of a much greater uncertainty.

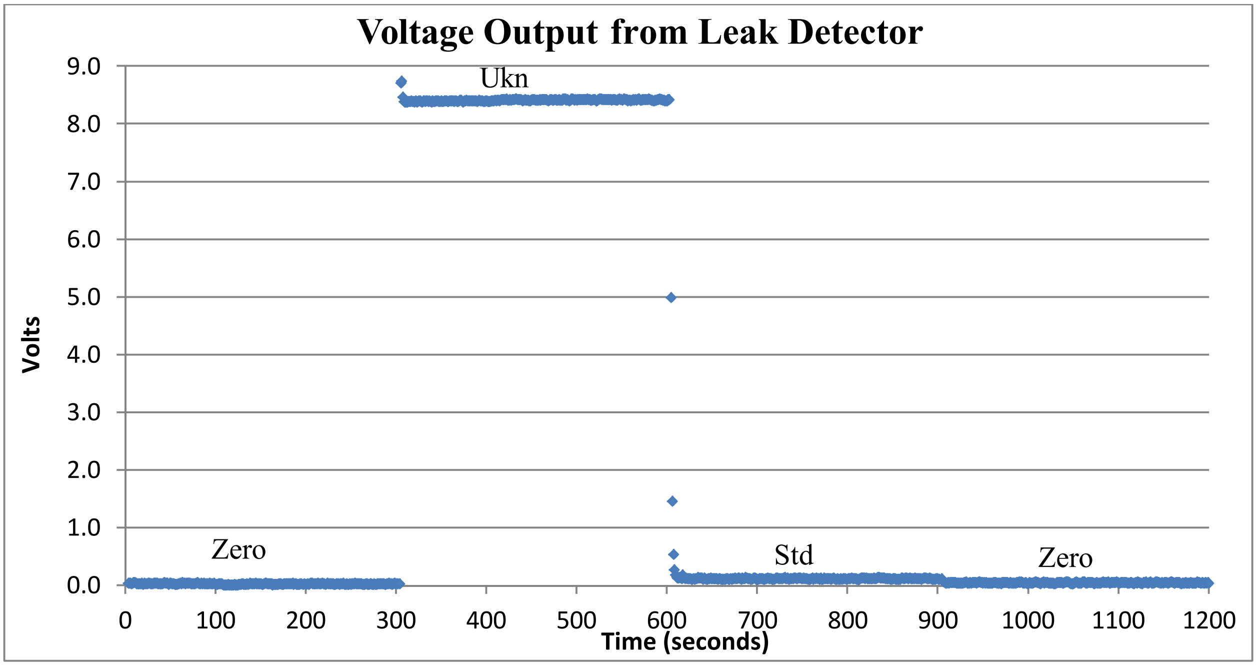

Figure 12: Chart showing the leak detector's output when the leak in the standard port is two orders of magnitude lower leak rate then the leak in the unknown port.

2.7 Optimizing the wait time to take readings after valve switching:
A series of tests were conducted to determine the optimum time for the leak rate to stabilize after being switched to the leak detector during a run. Since the 1980's the wait time to allow for the helium flow from the leak to the leak detector to stabilize has been 60 seconds. These tests used two 10^{-8} cc/s @ STP leaks. A calibration run was conducted following the old sequence and one with the new sequence (see zero stability tests above); both with the wait time of 60 seconds to verify changing the valve sequence did not change the calculated leak rate. More tests were run using the new valve sequence with wait times of 30, 20, 15, 10, and 5 seconds, shown in Figure 13. There was only a 0.6 % difference in calculated leak rate when using a wait time of 60 seconds vs 5 seconds. The only noticeable difference was the standard deviation of the readings from both leaks, which almost tripled going from a 60 sec to a 5 second wait time. The error in the readings remained fairly consistent with wait times between 60 and 15 seconds but increased with wait times less than 15 seconds. It was decided to use a wait time of 20 seconds for 10^{-8} and faster leaks and 30 seconds for slower leaks.

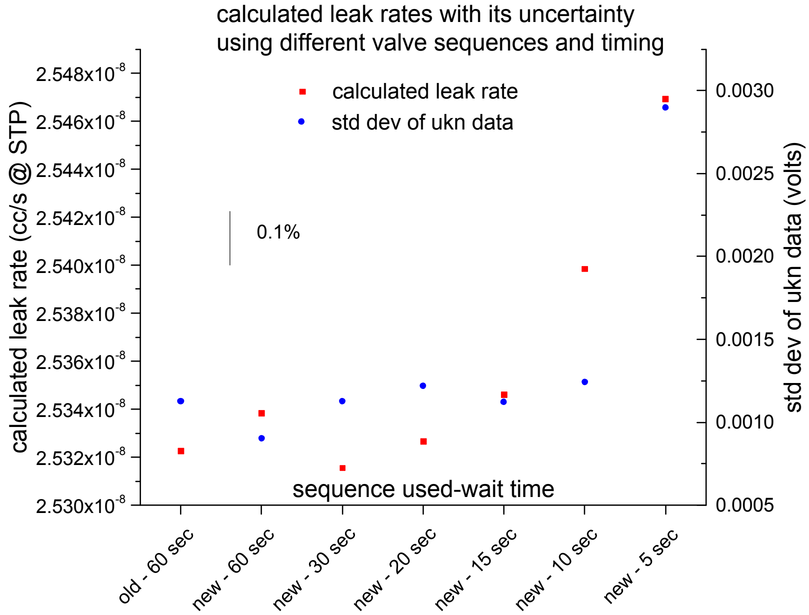

Figure 13: Plot showing how the calculated leak rate changes with changes in wait times after valve changes.

3 Conclusion:

Numerous tests were run and some equipment replaced to increase the operating efficiency of the Compare II system resulting in a faster calibration turnaround time for the customer. Replacing the molecular drag pump with a turbo pump reduced the time for the leak rate for slow leaks to stabilize from 30 hours to about 1 hour. Changes were made to the program to include more zero readings in a run increasing calculated leak rate stability. The wait time after a leak is valved to the leak detector was lowered from 60 to 20 seconds. Adding a third temperature during calibration increased the accuracy of the calculated leak rate within its specified temperature range. Demonstrated the ability of calibrating leaks when the difference between the standard and unknown are two orders of magnitude different in leak rate, however, it significantly increased the uncertainty. Replacing the 120 VAC fan inside the unknown temperature chamber with two 12 VDC blowers reduced the time to reach stable temperatures during a run. These changes decreased the time to complete a calibration from 9 hours to 5 hours without changing the uncertainty of the calculated leak rate. This work has increased the capability of calibrating leaks from two or three per week to five per week by increasing the operating efficiency of the system that significantly reduced turnaround time for the customer.

4 Acknowledgements:

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed martin Corporation, for the U.S. Department of Energy's national Nuclear Security Administration under contract DE-AC04-94AL85000.

[1]M.V. Iverson and J.L. Hartley, J. Vac. Sci. Tecnol. **20**, 982 (1982)
[2]D.R. Gerty, NCSLI Measure J. Meas. Sci., Vol.8 No. 3, 42 (2013)