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Development of A Cryocooler based Variable Temperature Setup for Calibration of Temperature Sensors

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Abstract:

Measurement of temperature is an important aspect of any system such as an experimental system, equipment or any process involving the temperature. The measurement and control of temperature is quite important for the proper functioning of the process, device, or the equipment. Especially, in the field of cryogenics, there is a need to measure temperatures of fluids entering or exiting systems such as a Joule Thompson (JT) valve, an expansion engine or a turbine to assess the performances and improve them. Measurement of temperatures in the cryogenic range is carried out by different transducers such as the Resistance Temperature Detectors (RTD)s (also known as Resistance Thermometers, such as Platinum resistance thermometer (PT500)), thermocouples, Cernox, Silicon diodes and Germanium sensors etc. The sensors produce an output proportional to the variation of some physical property with temperature. The temperature calibration of the above sensors is best done against a pre-calibrated temperature sensor.

In this work, we present the development of a variable temperature experimental setup using a two stage Gifford McMahon (GM) cryocooler, which provides the refrigeration to cover the temperature range from 4.2 K to 300 K needed for the temperature sensor calibration. The sensors to be calibrated are mounted within the holes of an Oxygen Free High Conductivity (OFHC) copper thermal block, which accommodates a maximum of eight temperature sensors. Its design is such that different types of temperature sensors such as Silicon / Germanium diodes, RTDs, Thermocouples, Cernox sensors, Carbon resistors etc. can be calibrated using a pre-calibrated Silicon diode temperature sensor. The sensor leads from the vacuum jacket of the experimental cryostat are connected to the measuring instruments which consist of a current source (Source meter), Data AcQuisition System (DAQ), a Temperature controller and a computer. An in-house developed LabVIEW based software is used for the temperature control of the copper lock and data collection. The results of calibration of typical temperature sensors along with their measurement accuracies are presented. This set up is in regular use for the calibration of various types of temperature sensors.

***Keywords* — Variable temperature set up, Temperature Sensors, Calibration, GM Cryocooler, Helium Compressor, Data Acquisition.**

I. INTRODUCTION

Temperature measurement is very important in a system. Whether it is an experimental set up, an equipment or a process undergoing variation in temperature, temperature measurement and control is needed for the proper execution of the process, the device or the equipment. Specifically, in cryogenics, we come across situations wherein we need to measure temperatures of the fluids flowing into or out of the systems involving a JT valve, heat exchanger, expansion engine etc. In such cases, reliable temperature measurements are essential to evaluate their performances and take the appropriate measures for their improvement.

Temperatures in the range from 4.2 K to 300 K can be measured by different sensors such as RTDs, Cernox sensors, Silicon / Germanium diodes etc. The sensor produces an output which is proportional to the variation of some property with temperature. In the case of a resistance thermometer, its electrical resistance varies with temperature, whereas in the case of a silicon diode, its forward voltage changes with temperature. The sensor calibration is best carried out by using a pre-calibrated temperature sensor, for the selected range of temperature. Further, the sensors should be calibrated at regular intervals, due to the environmental factors which affect their performances, such as thermal cycling, aging, continuous use etc.

Rubin [1] has presented a review on cryogenic thermometry and the progress made in this field since 1982. Ch. Balle et al [2] have discussed the development of industrial-type cryogenic thermometer with built –in heat interception needed for the accelerators in CERN. Ch. Balle et al [3-4] also developed the cryogenic thermometer calibration facility in CERN and carried out studies on the effect of thermal cycling on cryogenic thermometers.

Neutron irradiation tests on the calibrated cryogenic temperature sensors were carried out at low temperatures by Junquera et al [5]. Thermeau et al [6] have discussed details on the cryogenic thermometer calibration facility for the LHC. Ch. Ainslie[7] has discussed about the cryogenic thermometer database system specifically on the user requirements and implementation.

Different experimental arrangements have been reported in the literature for the calibration of temperature sensors. Szmyrka-Grzebyk et al [8], discuss the measuring systems for thermometer calibration in low temperature range. In this work, the authors discuss the calibration facility established in their laboratory. In the temperature range, between 13K and 273.16 K, capsule standard resistance thermometers (CPRSTs) have been calibrated at six fixed points. In the range between 2.5 K and 25 K, RhFe thermometers are calibrated by comparison with a standard thermometer. Various types of industrial platinum resistance thermometers as well as digital thermometers could be calibrated in the temperature range between 77 K up to 550°C.

Ricketson and Watkins [9] have studied the rhodium-iron ceramic sensor calibration for the temperature range from 1.2K to 300K. They discuss two types of calibration results namely (a) multi-point across the temperature range, and (b) three-point calibration at temperatures close to 4.2 K, 77 K, and 273 K. They also proposed a method for improving the interpolation accuracy for a three-point calibration when using a reference function.

Souri and Makinwa [10] proposed a new approach in which a temperature ramp is applied to the sensors under calibration, which is less time consuming. Further, unlike the conventional method which enables calibration only at a few selected temperatures, the new procedure facilitates the characterization of the sensor over its full operating range. By this, the authors calibrated platinum resistance thermometers up to 120 C.

Pertijs et al. [11] presented two calibration techniques for smart temperature sensors in the temperature range from -55 ˚C to 125 ˚C, based on voltage measurements instead of temperature measurements. This significantly reduced the time needed for calibration in the production of such sensors. Although the calibration techniques have been adopted for high temperature ranges, still the methodology will be quite suitable for cryogenic temperatures.

Pavese et al. [12] have reported the calibration setup realized at VNIIFTRI for cryogenic thermometers in the temperature range from 1.5K to 350 K. This system combined the possibility of thermometer calibration and fixed point measurements and used a normal liquid helium storage dewar with a cryogenic module of helium evaporation refrigerator.

The above works use either cryogenic fluids or oven for the calibration of temperature sensors and their availability is a must for conducting the experiments. On the other hand, Myung Su Kim et al. [13] have reported the development of a two stage GM cryocooler based system for calibrating temperature sensors in the range from 4.2K to 300 K. The same work is presented in more detail in another publication by Yeon Suk Choi [14].

From the practical point of view, the cryocooler based experimental setups are more convenient for operation, since the dependency on the cryogenic fluid is eliminated. Hence, in the present manuscript, we report the development of a cryocooler based variable temperature cryostat setup for the calibration of temperature sensors for the temperature range from 4.2 K to 300 K.

The latter has been used for the calibration of different temperature sensors such as Silicon diodes, RTDs, Cernox sensors, Zr-Nx (Zirconium nitride sensor) sensors etc. against a pre-calibrated silicon diode temperature sensor. In this system, the GM cryocooler serves as a heat sink (in place of cryogenic fluid) to maintain the thermal block with sensors at different preset temperatures within the vacuum jacket. This forms the variable temperature experimental cryostat for the temperature range from 4.2 K to 300 K. The latter also enables performing multiple experiments for the evaluation of accuracy and repeatability of the experimental results.

II. DETAILS OF THE TEMPERATURE SENSORS CALIBRATION SYSTEM

A. Experimental Setup

Figure 1 shows the schematic of the experimental setup for the calibration of temperature sensors at cryogenic temperatures. It consists of a copper block for mounting the sensors, an electric heater (cartridge type), and a two stage Gifford-McMahon (GM) cryocooler for producing the required refrigeration, a radiation shield, and a vacuum jacket.

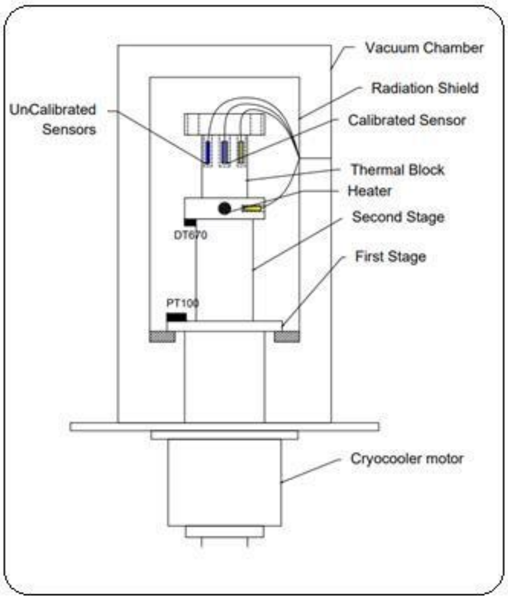


Fig. 1 Schematic of the temperature sensor calibration experimental setup.

The thermal block is made of OFHC copper to have enhanced thermal conductivity and is mounted directly on the second stage cold head of the GM cryocooler.

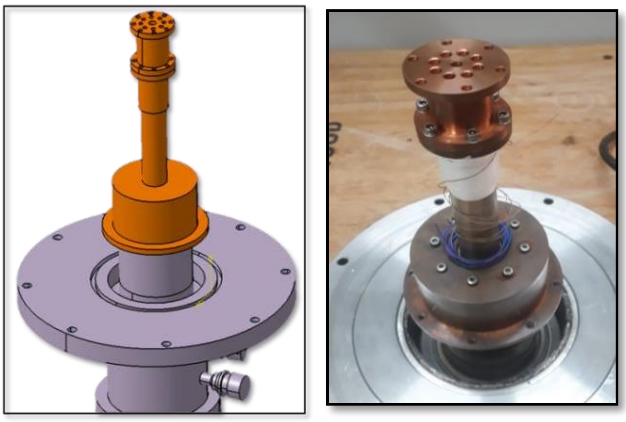
Figure 2 (a) and (b) show the three-dimensional view of copper block mounted on the second stage cold head and the photo of the actual system respectively.

Indium foil is used in between the copper block and the cold head to improve the heat transfer. The copper block is cooled by the second stage refrigeration power. A cartridge type heater is also mounted within the copper block to increase its temperature as desired.

The second stage cold head with copper block is insulated with 10 layers of Multi-Layer Insulation (MLI). The MLI consisted of alternate layers of double-sided aluminized Mylar film of 64 μm thickness along with a fiberglass spacer of 125 μm thickness.

This is further surrounded by the copper radiation shield which is thermally anchored to the first stage of the GM cryocooler.

The entire cryocooler from its room temperature flange including the first stage copper radiation shield is further insulated with 35 layers of MLI and housed inside the vacuum jacket. The latter is evacuated using a high vacuum pumping system, as discussed later.



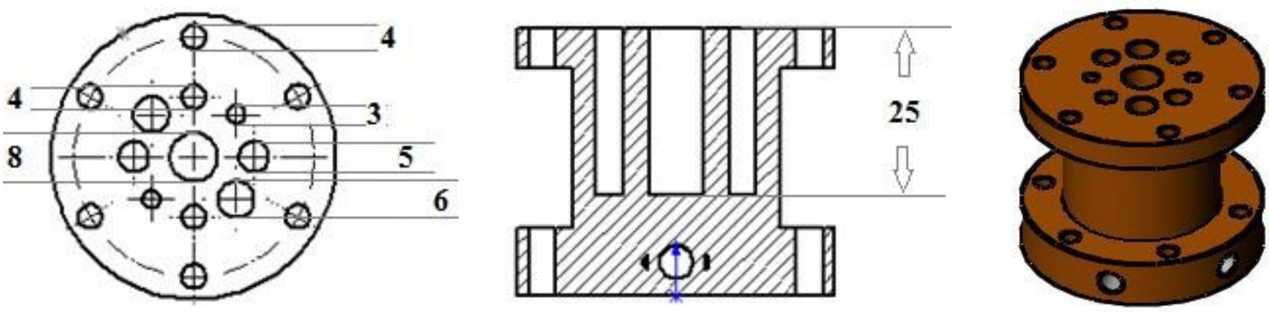
(a) (b)

Fig. 2 (a) 3D view and (b) Photo of isothermal block mounted on the 2nd stage cold head

B. Details of Thermal Block

The top view, front view and isometric views of the OFHC copper is shown in Figure 3. The copper block has 4 pairs of holes of diameters 3, 4, 5 and 6 mm drilled on its top surface to a depth of ~ 25 mm. The holes are positioned at equal distances from the centre of the copper block to ensure that they are at the same temperature (i.e. isothermally located).

The bottom part of the copper block has two 8mm horizontally drilled holes. They accommodate two cartridge heaters of resistance 50 ohms each.



(a) (b) (c)

Fig. 3 Detailed drawings of the OFHC copper thermal block. (a) top view and (b) front view. (c) Isometric view. The dimensions shown in the drawing are in mm. The cartridge heater hole size is 8 mm.

The copper block is maintained at different temperatures in the range from 4.2 K to 300 K using a temperature controller (model 335, make: Lakeshore), which supplies power to the cartridge heater in the presence of the refrigeration power of the GM cryocooler.

A calibrated silicon diode temperature sensor (model SI410C, make: Scientific Instruments, USA [15]) is used in the above set up. The calibration data of this sensor is given by the manufacturer as a table of forward voltage vs temperature for the calibration current of 10 μA.

This data is fitted using polynomial equation of appropriate degree in different ranges. These polynomial coefficients are obtained for obtaining temperature from forward voltage and vice versa. The error in temperature measurement of this silicon diode (as per the manufacturer) is given in Table 2.

This calibrated sensor along with the uncalibrated sensors are mounted in the holes designated for the sensors in the copper block. A maximum of 7 numbers of uncalibrated sensors can be accommodated in the thermal block along with the calibrated sensor.

Holes of larger diameters are used for mounting bigger sensors. The sensors are mounted such that there is minimum gap between their body and the thermal block.

The gap between the sensor body and the hole in the thermal block is filled with Apiezon N high vacuum grease to ensure good thermal contact of the sensor with the copper block and minimize the thermal losses. The central hole of diameter 8 mm in the copper block can be used for mounting a cartridge type heater.

Four wire method is used for the measurement of voltages from the sensors. Enamelled Manganin wires of size Standard Wire Gauge (SWG) 33 and 1 m length (prepared as twisted pairs) are used to connect the sensors from the thermal block to the vacuum feed through.

These wires are thermally anchored at two locations namely, a) at the bottom of the isothermal copper block and b) on the cryocooler zone between the first and second stages.

The cartridge heater of 50W and resistance of 50 Ohms is also connected using the twisted pair of enamelled copper wires up to the feed through point and thermally anchored to the first stage cold head zone.

From the feed through it is connected by normal electrical leads to the temperature controller. The latter provides the appropriate current to the heater to maintain the thermal block at the set temperature.

C. Estimation of Heat load to second stage cold head

The heat transfer to the second stage of the GM cryocooler arises due to different modes of heat transfer namely (a) solid conduction, (b) radiation, (c) gas conduction and (d) external heat loads.

For the estimation of above heat loads, it is assumed that the solid conduction heat transfer between the first and the second stage by the basic construction of the cryocooler is zero. This implies that only the heat loads due to the solid conduction by the electrical leads to the sensors, namely (a) the manganin wires connecting the sensors and (b) copper wires connecting the cartridge heater need to be evaluated. Since the thermal anchoring of the leads is done at the first stage of the cryocooler, the heat load from $35\ K\ to\ 4.2\ K$ only needs to be calculated.

Eight temperature sensors are connected using manganin wires in 4 wire configuration and one cartridge heater is connected by copper wires in 2 wire configurations. The length of each manganin wire of SWG 33 is ~ 50 cm. Thus, the total number of wires will be 32. The cartridge heater is connected by wire of same gauge but is made of copper.

The radiation heat load is estimated considering of the following. (a) The second stage cold head is insulated with 12 layers of multilayer insulation

(MLI) with an average emissivity value of 0.04. Both the second stage cold head at 4.2 K and the copper radiation shield at 35K are assumed to have the emissivity value of ~ 0.04 as given in [16]. Thus the radiation heat transfer occurs from copper radiation wall at 35K to the second stage cold head at 4.2 K.

The residual gas conduction heat transfer to the second stage cold head occurs due to the gas pressure in the vacuum jacket which is $\sim\cong 1.0\ x 10^{-3}\ Pa$.

Using the dimensional data of the cold head as input parameters and following the procedures outlined by Myung Su Kim et al. [13] and the reference texts [17-19], the heat loads by different modes have been estimated and they are given below. The total heat load to the second stage cold head is $\cong 67\ mW$. Of this, the heat transfer by solid conduction by the wires is $\cong 22\ mW$; the heat transfer by gas conduction is $\cong 4.3\ mW$. The radiation heat load is quite small and is $\cong 2.17\ x\ 10^{-3}\ mW$.

The above analysis shows that the heat transfer to the second stage cold head is quite small. This would mean that the calibration of temperature sensors is being performed under proper environmental conditions.

D. *Description of the experimental setup and calibration procedure*

Figure 4(a) shows the schematic of the experimental setup inclusive of the data acquisition system and Figure 4(b) shows the photo of the developed system: The two stage GM cryocooler is driven by water cooled helium compressor (Make: Sumitomo; Model F70H; 7.5 kW).

The vacuum jacket of the cryocooler is evacuated by a Turbo molecular pump (Make: Agilent Model V301) backed by the rotary pump. The temperature of the copper block housing the sensors is maintained at a preset value by the temperature controller (Make: Lakeshore Model; 335) along with the cartridge heater.

A source meter (Make: Keithley, model 2450) served as a constant current source to supply a current of 10 μA for all the temperature sensors which are connected in series. Since the source meter (Make: Keithley, model 2450) was used only to supply a constant current of 10 μA throughout the experiments on temperature sensors calibration, this instrument was not included in LabVIEW programming. A data acquisition unit (Make Keithley; model 6510 / 7700) is used for acquiring the measured voltages from all the sensors.

This instrument was interfaced using the USB port in the rear panel of the instrument. The Virtual Instruments (VI) library of National Instruments (NI) was used in developing the LabVIEW software for instrument control and data collection from the DAQ system.

The acquired data is stored in the computer. The experimental procedure for calibration is described briefly below.

After mounting the temperature sensors in the copper block, their leads are extended by using Manganin wires of ~ 1 m length in the 4 wire configuration. They are anchored onto the thermal posts. They are taken out through the feed through and connected to the measuring instruments.

The vacuum jacket is evacuated by the rotary pump till the pressure level reaches $\sim 1.0\ x\ 10^{-2}\ mbar$. Subsequently it is evacuated by the turbo molecular pumping system to reach the pressure level of $\sim 1.0\ x 10^{-5} mbar$.

The LabVIEW data acquisition program is started prior to the cryocooler cool down. When the second stage temperature reaches ~ 20 K, the valve connecting the vacuum jacket to the turbo molecular pump is closed so that the interspace vacuum is improved further by Cryopumping. After obtaining the lowest possible temperature of the cryocooler, two methods have been used for the calibration of temperature sensors.

In the first method, the temperature of the thermal block is selected to a preset value by the temperature controller. The thermal block is maintained at this temperature by the temperature controller which supplies the necessary current to the cartridge heater. When the temperature of the isothermal is stabilized; the data is acquired by the DAQ using the LabVIEW program.

Again, the next preset temperature is selected by the temperature controller and the procedure is continued for the entire temperature range. The number of experimental data points in this procedure is limited to ~ 100, due to the time constraints in experimentation.

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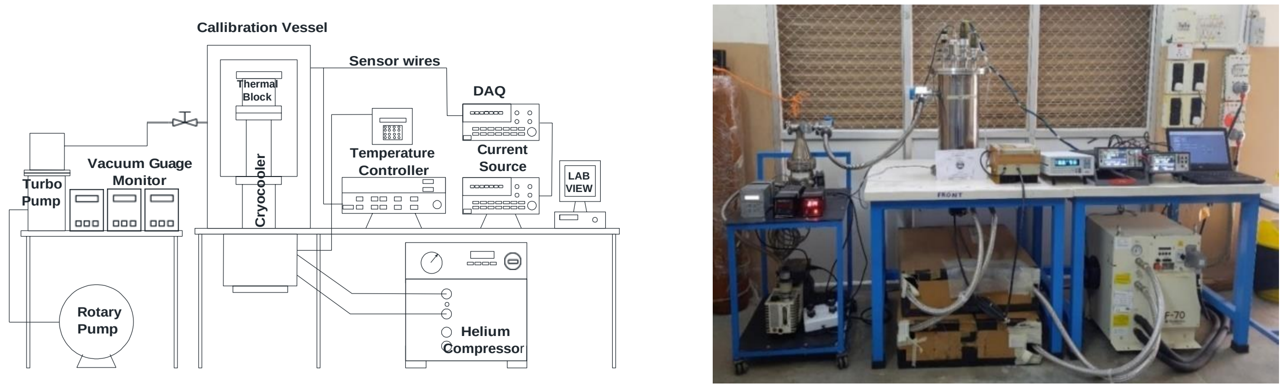


(a) (b)

Fig 4. (a) Schematic and (b) photo of the complete experimental set up for calibration

In the first method, the temperature of the thermal block is selected to a preset value by the temperature controller. The thermal block is maintained at this temperature by the temperature controller which supplies the necessary current to the cartridge heater. When the temperature of the isothermal is stabilized; the data is acquired by the DAQ using the LabVIEW program.

Again, the next preset temperature is selected by the temperature controller and the procedure is continued for the entire temperature range. The number of experimental data points in this procedure is limited to ~ 100, due to the time constraints in experimentation.

In the second method, the cryocooler is allowed to reach the lowest possible temperature and then switched off. The data acquisition by LabVIEW program is continued, so that the warmup data of all the sensors are continuously monitored at regular time intervals.

Due to the slow warmup of the thermal mass of the cold head, the system warmup occurs in quasi steady state and the acquired data is found to the quite satisfactory.

The latter method is less time consuming due to the continuous warm up of the thermal block mounted on the second stage cold head.

III EXPERIMENTAL RESULTS AND DISCUSSION

A. Cooldown Behaviour

In the following section, we discuss the experimental results of calibration of different temperature sensors. The cool down of the two stages of the GM cryocooler and the thermal block housing the sensors are shown in Figure 5. It is seen that the temperature of the first stage cold head of the cryocooler stabilizes much faster compared to that of the second stage cold head.

The cooldown of the thermal copper block follows that of the second stage cold head, and it is observed that the temperature of the thermal mass is slightly higher (~ 1 K) than that of the second stage cold head. The steady state cool down time is ≅ 90 minutes in the present experimental arrangement.

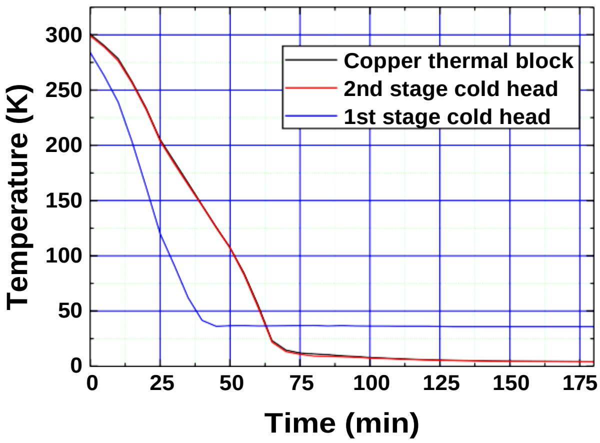


Fig 5 Typical Cool down of the two stages of the GM cryocooler and the copper thermal block

In the temperature sensor calibration experiments, we have used the pre-calibrated sensor SI410C procured from M/s Scientific Instruments, USA. This sensor is calibrated from 2 K to 450 K using a constant current of 10 μA, by the manufacturer. Figure 6 plots the calibration graph (forward voltage versus temperature) up to 300 K. As discussed earlier, all the uncalibrated sensors are connected in series with the calibrated sensor and supplied the same current.

The voltages from the uncalibrated sensors are monitored by the DAQ and recorded. These values and plotted against the temperature of the calibrated sensor. For all the sensors, polynomial data fitting has been used. The experiments are performed several times to verify that the repeatability of the measured data.

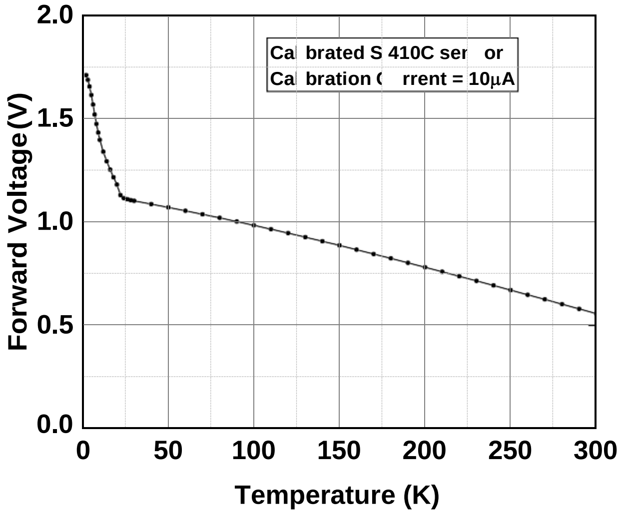


Fig.6 Forward voltage versus temperature of calibrated silicon diode sensor SI410C data from M/s Scientific Instruments, USA

B. Temperature Uniformity of the Copper Thermal Block

Before the discussion of temperature sensor calibration results, the temperature uniformity of the copper block has been examined by the following experiment. An uncalibrated silicon diode SI410B is mounted along with the calibrated sensor SI410C in two opposite holes of the copper block. The sensor calibration is performed to obtain the data of forward voltage versus temperature of the uncalibrated sensor.

Next, the same experiment is repeated by reversing the positons of the two sensors in the thermal block. The changes in the voltages (ΔV) measured at specific temperatures of the calibrated sensor can now be converted to changes in temperatures(ΔT) using the calibrated sensor data.

This indicates the temperature uniformity of the thermal block.

The experiments are repeated in different holes of the thermal block, to obtain the maximum deviation in the measured temperatures at specific values namely 4.2K, 10 K, 20 K, 77K and 300 K. The values of temperature uniformity of the copper thermal block are presented in Table 1.

Table 1. Temperature uniformity of the copper thermal block

Temperature (K)	**Measured $\Delta T(\mathrm{max})$ (K)**	**Temperature uniformity of the copper block (K)**
$42K$	$8mK$	$\pm 8\,mK$
$10\,K$	$8\,mK$	$\pm 8\,mK$
$20\,K$	$12\,mK$	$\pm 12\,mK$
$77\,K$	$15\,mK$	$\pm 15\,mK$
$300\,K$	$20\,mK$	$\pm 20\,mK$

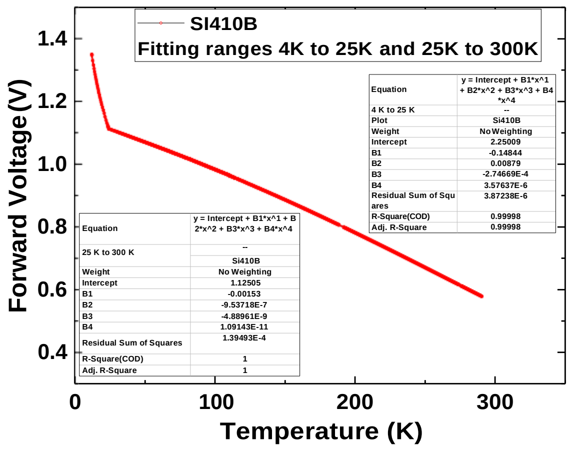
It is seen from Table 1 that the uniformity in temperature of the copper block is better at lower temperatures.

C. Calibration of Uncalibrated Temperature Sensors

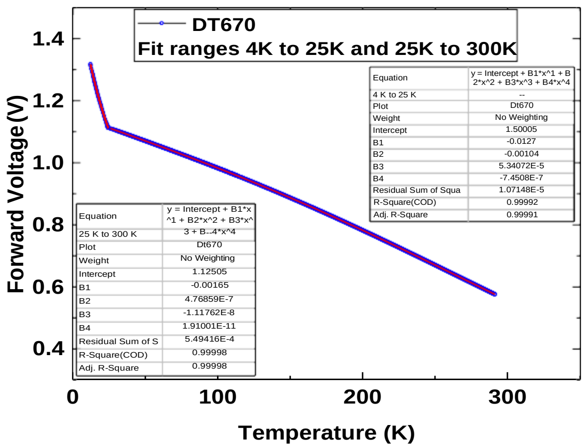
In the following, we discuss the calibration of different uncalibrated temperature sensors namely, the silicon diode sensors SI410B (from SI, USA), DT670 (from Lakeshore Cryotronics, USA), Platinum resistance thermometer PT500 and Cernox sensor (from Lakeshore Cryotronics, USA).

The figures 7(a) to (d) plot the data obtained from the measurements in appropriate units as a function of temperature. The number of measured data points are quite large and hence the number of data points displayed in the graph is regulated by the graphing software.

The least square fitting of the experimental data of different sensors can be done either by polynomial degree equations or any other suitable form of equation. For example, the platinum resistance sensor is usually fitted with CVD equation [20], but since we are dealing with temperatures down to 25K, the polynomial least square fitting was found to be more convenient and hence used in presenting our calibration results.



(a)



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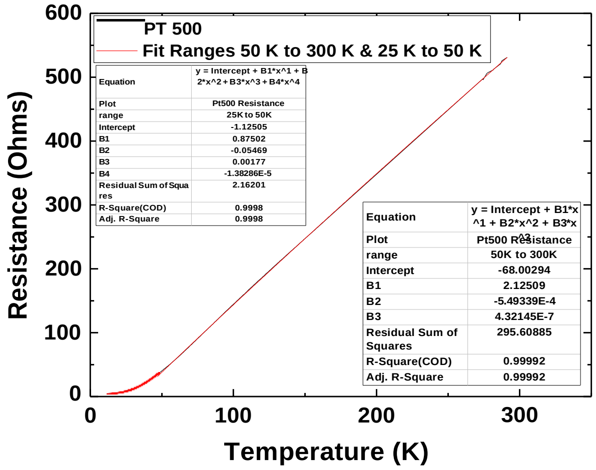
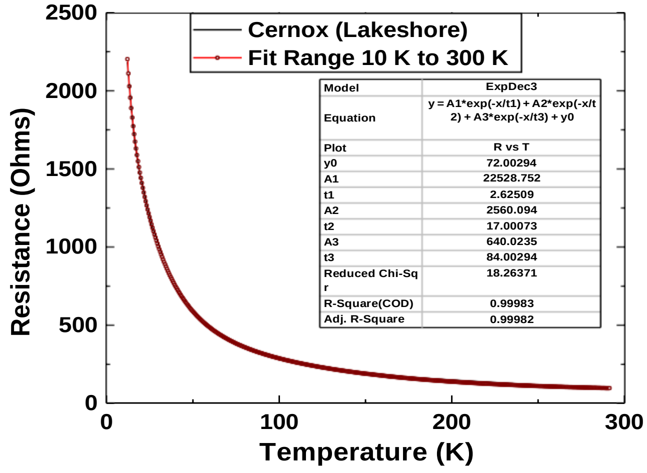


(c)



(d)

Fig.7. Calibration graphs for different temperature sensors along with fitting coefficients.
(a)Silicon diode SI410B, (b) Silicon diode DT670, (c) Platinum Resistance Pt500 and (d) Cernox sensor

For Cernox sensors, due to the large variation of resistances at cryogenic temperatures, fit equation with exponential functions have been used. The fit coefficients obtained from the above equations for different ranges are also presented along with the graphs. The calibration results of different temperature sensors with the present experimental system are found to be quite good.

D. Accuracy of Temperature Sensor Calibration in the Present Experimental Setup

As discussed above, the calibration of different temperature sensors has been performed using the pre-calibrated sensor from M/s Scientific Instruments, USA. The error with which a temperature sensor can be calibrated in an experimental setup is the net result of each step in the calibration process.

The temperature scale (ITS90) disseminated by the National Standards Laboratories is transferred to the secondary thermometers which are maintained in the industries, which are the vendors of temperature sensors. Hence, their secondary thermometers serve as standard for calibrating other sensors.

Thus, the error in the temperature sensor calibration with the present experimental set up is the sum of (a) the error of the calibrated sensor obtained from the supplier, (b) the temperature variation across identical positions of the copper thermal block in which the temperature sensors are mounted in the present set up and (c) the errors due to the measuring instruments which are used for the calibration.

In the present experimental setup, voltage measurements are done using by data acquisition system Keithley model 6510 and a constant current flow of 10 μA is supplied by the source meter Keithley model 2450. The errors involved in the measurement of voltage by DAQ is 0.0015% and that of current from source meter is 0.025%. By error analysis, the accuracy with which the temperatures can be measured using these units is ± 15 mK.

The overall accuracies of calibrating the temperature sensors in different temperature ranges using the present experimental set up is given in Table 2.

Table 2. Accuracy of calibration of temperature sensors in the present experimental setup

Temperature (K)	Accuracy of the Calibrated Si Diode	Temperature uniformity of copper thermal block	Accuracy with which temperatures are measured by measuring Instruments	Overall accuracy of Sensor Calibration of the experimental setup
4.2 K	± 12 mK	± 15 mK	± 15 mK	± 42 mK
10 K	± 12 mK	± 15 mK	± 15 mK	± 42 mK
20 K	± 14 mK	± 20 mK	± 15 mK	± 49 mK
77 K	± 22 mK	± 25 mK	± 15 mK	± 62 mK
300 K	± 32 mK	± 35 mK	± 15 mK	± 82 mK

IV CONCLUSION

In this work, we have presented the development of a variable temperature experimental setup using a two stage GM cycle based cryocooler, which provides the refrigeration to cover the temperature range from 4.2 K to 300 K needed for the temperature sensor calibration. The sensors to be calibrated are mounted within the holes of a thermal block made of OFHC copper along with a pre-calibrated sensor.

The design of the thermal block is such that different types of temperature sensors such as Silicon / Germanium diodes, RTDs, Cernox sensors, Carbon resistors etc. can be calibrated.

All the sensors are connected in series and energized with a constant current of 10 µA from the constant current source (source meter).

An in-house developed LabVIEW based Instrumentation software is used for the temperature control of the thermal block and data collection. The heat load entering the second stage of the cryocooler from its first stage is estimated. The results of calibration of typical temperature sensors such as silicon diode, platinum resistance thermometer PT500 and Cernox sensors are shown. Also the accuracies of temperature sensor calibration at different temperatures are presented.

The above experimental setup is used regularly for the calibration of different types of temperature sensors.

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