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Author(s): R. Cech, M. Craft, R. Fultz, R. Biddle, D. Bracken, B.
Cordova, and J. Wetzel

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Testing of the Small Sample (New Concept) Calorimeter Received from EG&G Mound Applied Technology*

Russell Cech and Mike Craft formerly of EG&G Mound Applied Technologies,
Richard Fultz of EG&G Mound Applied Technologies,
Rod Biddle, Dave Bracken, Becky Cordova, and Jerry Wetzel of NIS-5

Abstract

The Small Sample calorimeter, also known as the New Concepts calorimeter, has undergone testing in the transfer of the calorimeter operations to Los Alamos National Laboratory from EG&G Mound Applied Technology (Mound), Miamisburg, Ohio, in September 1996. The design of the calorimeter incorporated several new concepts, thus the name New Concepts. The normal water bath was replaced with a small self-contained bath and control that used a thermal electric cooling/heating device to supply the control for the bath temperature. This change replaces the large refrigeration unit that has been used in the past, thus reducing the weight and the power required to operate the system. The design was done to allow the complete calorimeter system to be contained in a single electronics rack. With the new electronics package, this change would allow the unit to use a short electronics rack with a laptop computer and make the complete system transportable. By reducing the amount of water in the bath, the control and size of the bath could also be reduced. By making the bath self-contained and sealed, there would be no need to replace water or supply de-ionized water for the system. This change would remove some of the concerns about using a water bath in certain situations. The water would be about 5 gal. or less depending on the size of the calorimeter. The present system is a 5 in. diameter sample chamber system which can accept most older material now in storage. It will not handle the new 3013 size container as built but could be easily designed for that size. There is also a new sensor design that takes less wire and can eliminate the constant current source used in past Mound calorimeter designs. With the new digital voltmeters, the complete system could be run from a single meter with the ability to monitor bath and room as well as the calorimeter operating voltages for electrical heater runs. A few problems, though minor, need to be corrected to make the system available which we will address later in this report. We would like to address the testing of the calorimeter as it was received first and then some of the modifications that we made along with some of the lessons learned from the testing.

System Description As Received

The sensor for the calorimeter is a resistance element design with a gradient configuration in which the reference winding is placed on the outside of the thermal gap material and the sample winding is placed on the inside next to the sample chamber. The gap material is made of an epoxy/glass microsphere combination to increase the thermal resistance while reducing the actual gap size. This difference allows the calorimeter to be contained in a single tube jacket, although the system, as received, had two jacket tubes to allow for a full-twin design test. The second

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jacket tube is not used in the present system. The sensor is the same nickel wire as used in the past, but it has only a single winding for the sample and a single winding for the reference. The system is not connected as a Wheatstone resistance bridge calorimeter, but as a delta resistance configuration making use of standard high precision resistance measurements techniques. This difference helps reduce construction time and removes the constant current source normally used in full twin-resistance bridge calorimeter design. Using the delta resistance measuring technique of the digital voltmeter, it is possible to equate change in resistance to wattage of the sample in the chamber. A single value is returned from the meter for this measurement. This makes use of the intelligence built into the digital voltmeter for making the measurement and it provides a fairly simple equilibration calorimeter system. The same technique can be used in doing a setpoint operation, also known as servo control, where the sample chamber is maintained at a constant internal temperature since the measuring cell has an internal electrical heater that can be used for temperature control. Present servo control techniques for calorimeters could easily implement this operating mode.

The calorimeter bath design was adopted to make use of readily available materials and to keep the cost of construction to a minimum. Standard stainless steel tubing, available off the shelf at tubing suppliers, was used as the jacket for the calorimeter. This kind of tubing was chosen to reduce the machining time as well as the cost of precision machining. The tubing was then welded to the top plate and bottom plates of stainless steel. The water jacket was formed of stainless steel sheeting around the tubing with a 1 in. gap between the tube and bath jacket at the closest point with a flat section large enough to accept the thermal electric cooling/heating plate. Both ends of the jacket are kept open so the sensor element can be easily slipped in the bath jacket. The bottom is then closed off with insulating material and the calorimeter wiring brought out through the bottom insulation. The thermal electrical cooling/heating plate is mounted directly to the bath with heat sink compound, and then a 1 in. layer of insulation applied over the entire outer bath area. A Hart precision temperature controller was used to operate the thermal electric cooling/heating plate. The internal heating element of the thermal electrical plate and the cooling caused by applying current to the cooling junctions of the plate is used for temperature control. This was done to prevent supplying extra power to the heater to overcome the over cooling of the cooling plate. By simply controlling the whether the plate was supplying cooling or heating, much less power was required to control the bath temperature. A water pump circulated the water in the bath forcing the return water directly onto the thermal plate to provide the best control and to provide maximum stirring of the bath. The bath has a seal plug in the top for filling and two temperature sensor feed-through seals, one for bath control and a second for computer sensing of the bath temperature. To fill the calorimeter in preparation for operation, 5.2 gal. of de-ionized water was needed.

Testing of System As Received

Russell Cech, formerly of EG&G Mound Applied Technology, came out to set up the calorimeter and make it operational since there was no documentation supplied with the system. After he had gotten the system operating, Mike Craft, also formerly of EG&G Mound Applied Technology, came out to help set up the electronics to allow us to start testing the system. After several days of allowing the calorimeter to come to equilibration, a series of tests was proposed to determine the capability of the calorimeter. The first was a series of baseline runs to determine

the stability of the system. The baseline runs had a stability of 0.002 ohms out of 0.649 delta ohms between the sample and reference windings. A standard isotopic set of samples was available equaling 0.199 W which was run to get a feel for the sensitivity of the system and to do a preliminary determination of the noise level. Figure 1 shows the complete run along with the noise level for both the baseline and the sample.

Fig. 1. New concept 0.199 W STDISO set sample.

The measurement of 0.0093 ohms change for 0.199366 W of sample calculates out to 0.0466 ohms per watt sensitivity with 0.0012 ohms peak to peak noise for the system as received. The noise is equivalent to about 0.025 W which defines the lower level of measurements the received system could detect. It was necessary next to determine the sensitivity curve for the system by running a series of wattages to see how linear the sensitivity was. The internal calorimeter heater was used to provide a series of runs from the 0.2 W level to 25 W which was the maximum power supply capability for the system. Figure 2 shows the sensitivity curve and at the lower end it was difficult to fit without using a third order equation.

Fig. 2. Heater Sensitivity Curve.

A series of runs was then made observing the sample resistance versus the reference resistance, rather than the delta resistance. This test allowed the two sensor windings to be plotted to see how they interacted. Using a maximum 30 W heater run, it was noted that the reference winding was seeing a resistance increase which was about half the sample resistance change and that both had a cyclic component, see Fig. 3.

Fig. 3. 30 W heater step function in New Concepts Calorimeter.

The cyclic component of the delta resistance, shown in Fig. 4, was due to the room temperature affecting the bath temperature which could be easily confirmed because the cycle was the same as the room temperature.

Fig. 4. Delta Resistance vs Bath Temperature Hart Controlled.

The increase in reference resistance was expected, but not at the level of change seen. This change is due to the thermal resistance between the reference winding and the water jacket. As was stated earlier, the jacket was made of off-the-shelf tubing which had not been honed to a good cylinder and the sensor required some clearance to be able to be inserted into the jacket. All of these added air gaps caused a thermal resistance which appears to be about one half that of the calorimeter thermal gap. Much of the noise level was connected directly to the room temperature affecting the bath temperature as can be seen in Fig. 5.

Fig. 5. Bath Temperature vs Room for Baseline with Aluminum Sleeve a Mass.

After running a near 30 W heater run, it was noted that the bath temperature shifted about 0.005°C . This change indicates that the bath controller has an upper limit of control that is less than 30 W and defines the range of the system as received. The as-received conditions indicate a low-level noise of 0.025 W which is at least 25 times what a normal calorimeter should be for any type of low level measurements. At a 5 W level this would be equivalent to 0.5%.

The system shows much promise and several modifications were proposed to increase the possibilities of the system. The first modification was to use one of the Hart controller baths to pump the water through the calorimeter jacket. This modification was an attempt to see if the room could be further isolated from the bath and to see if the noise level would decrease accordingly. Figure 6 shows the decrease in bath noise and room dependence by substituting the Hart bath system.

Fig. 6. Hart Controller vs Old Controller on New concept Calorimeter.

There was still some room influence, but much less. Then some extra insulation was added to the outer surface of the calorimeter to see if the room could be further isolated from the bath. This modification did not yield much decrease in noise level. Figure 7 shows the baseline noise level with the Hart bath and added insulation.

Fig. 7. Watts vs Bath Temperature for Baseline with Hart Controller.

This graph indicates that the wattage noise level was reduced to about .015 W. It was still not enough to make good low-level measurements. A redesign of the thermal electrical bath controller has been completed, but has not been tested yet. This new design is still the way to eliminate the compressor associated with the Hart bath controllers.

Lessons Learned

The following changes are proposed to make the calorimeter into a good mid-range calorimeter. First, the sensor was wound with only 167 ohms in an attempt to use normal four-terminal thermometry standards type measurements. As can be seen, this winding makes the sensitivity very low, and therefore, there is not much signal to work with. This winding was selected to stay within the range of normal high-precision resistance thermometer measuring circuitry used on digital voltmeters. If the sensor were wound with a resistance of just less than 2000 ohms, this measurement would be below the range change of the digital voltmeter and give 12 times the sensitivity. According to the digital voltmeter specifications, this range is acceptable. The test that must be made is to see if the noise increases with the signal level increase. A calorimeter presently available is being wired to allow the testing of this theory. Second, a better fit with the sensor and the jacket could reduce the thermal gap between the reference and the bath which would increase the delta resistance output. This goal could be accomplished by honing the jacket tube to give a good cylinder and making the clearance needed for the sensor to be inserted into the jacket smaller. Third, a newly designed thermal electric water bath controller would reduce the bath temperature noise. The final possibility is to use the second tube with a second sensor as the reference, which would completely remove the problem of the shifting reference resistance. Because the two sensors would be affected in the same time and way, they would produce the capability of decreasing the room to bath temperature effect. The present gradient sensor has the problem of the reference being affected by the bath change before the sample sensor. This sequence produces an out of phase signal which is not easily removed since the wattage of the sample has an effect on the amount of signal level difference.

Summary

The New Concepts calorimeter has many advantages over conventional large water bath calorimeters and with increased sensitivity and stability could produce a single rack mountable calorimeter system containing the complete electronics, bath control, and calorimeter. All of the tests done show great possibilities for future systems which could be constructed much more easily. The use of a laptop computer system is now possible with the new version of calorimeter software. The servo control, equilibration, prediction of equilibrium, and electrical heater calibration will be available under the same software system which would give the calorimeter more flexibility and usability.