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AN IMPROVED ZERO GRADIENT CALORIMETER FOR THE INVESTIGATION OF COLD FUSION PHENOMENA

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ABSTRACT

A second generation null balance calorimeter has been constructed for measuring anomalous heat in electrolytic cells. This calorimeter is similar in concept to an isothermal calorimeter except that it is operated with zero temperature differential. The calorimeter accuracy is 4 milliwatts when operated at a total power of 12 watts. Calibration is performed in situ by operating the cells under test reversed or at zero current.

APPARATUS

Introduction

The first version of this calorimeter was hurriedly assembled after the initial announcement of Fleischmann and Pons [1]. Work with this design suggested interesting phenomena which were just beyond its sensitivity and stability. After a years operation, experience has guided an improved design.

Calorimeter

Thermo Electric Devices (TED) (Melcor Corp., Trenton, NJ) are used as in the earlier design [2] to pump and measure heat flow. The new design is built from machined parts around a 2 liter stainless steel dewar, Figure 1. A puck shaped plug of aluminum fills the mouth of the dewar and is sealed to it with an "O" ring. The dewar is mounted over a copper plate with the gap between the puck and the plate filled with two sets of three TEDs arranged symmetrically to provide uniform heat distribution. A high power set is driven at constant current in the Peltier mode and pumps heat from the dewar. The second set, with fewer thermoelectric junctions, is operated in the Seebeck mode and measures the temperature difference between the puck and the plate. The plate is mounted through another set of six high power, 30 watt, TEDs to a water cooled base plate.

A spool shaped aluminum block with thick walls to evenly distribute the heat is bolted to the puck. The spool contains a cavity to hold the test cell. Several sets of heaters are contained in holes drilled in the spool wall. Milled slots in the puck allow the routing of wiring from the thermal stabilization entry pipe to terminal strips for connection to a cell.

The arrangement of the temperature servos has been improved over the earlier design. A high power, high speed servo amplifier uses the puck-plate seebeck devices as error

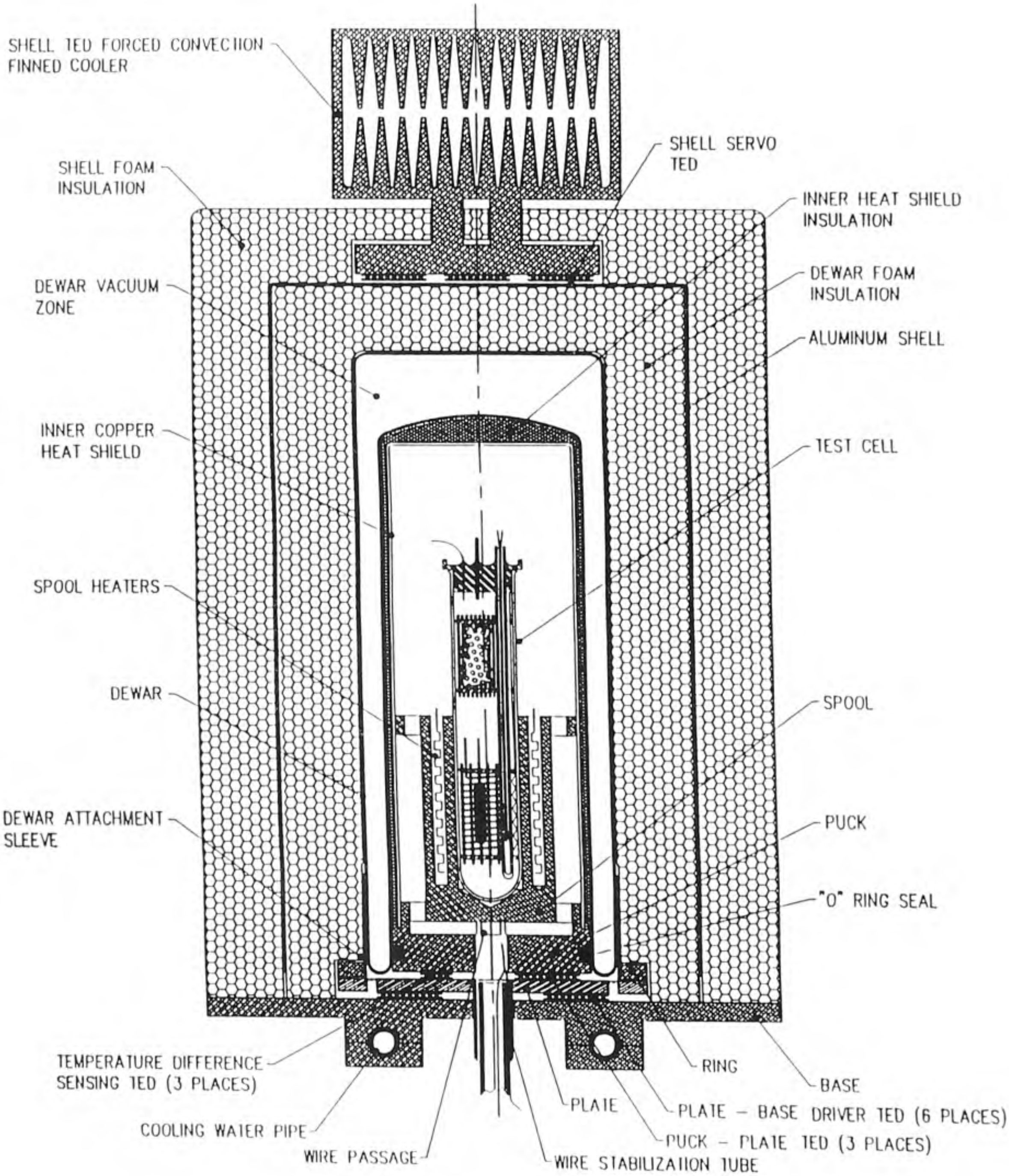

Figure 1. The new calorimeter. The mouth of a stainless steel dewar is filled with the aluminum puck. Two sets of thermoelectric devices (TED) couple the puck to the plate. One set pumps heat from the dewar to the plate. The second set measures the temperature difference between the puck and the plate. The plate makes thermal contact with the outside of the dewar. A high power set of six thermal electric devices uses the puck-plate sensor TEDs to hold the plate to the same temperature as the puck. This holds the inside and outside of the dewar to the same temperature. A copper heat shield inside the dewar reduces thermal gradients which cause radiation losses. A servo TED driven outer heat shell provides a second level of radiation loss protection. A heavy spool shaped aluminum block holds the test cell. Heaters in the spool balance the internal heat load to the refrigeration provided by the puck-plate TEDs. The heater power required to hold the dewar inside to the set temperature plus the measured cell power are balanced against the refrigerator calibration. Any difference is a measure of anomalous heat.

detectors and drives the plate-base TED's to hold the plate and connected outer wall of the dewar to the same temperature as the puck and inner wall. The purpose of this servo is to hold the outer wall of the dewar to the same temperature as the inside of the dewar without concern as to what the temperature might be. To the extent that this is done, there will be no heat lost from the dewar except that due to transient thermal gradients which cause radiation losses. To further reduce thermal gradients, the spool is connected to a heavy copper heat shield which is insulated from the inner wall of the dewar.

The plate cooling water is pumped through a 30 gallon garbage can which exhausts heat to the environment. This provides a large thermal mass so that the relatively fast plate-base servo can easily track changes.

A slower response servo is controlled by a temperature sensor mounted in the puck. This servo drives the spool heaters to hold the experiment to the desired temperature. If the experiment is set up so that the cell power is less than the refrigerator power, then the spool heaters will always require some power and control will be possible. The system fails if the cell power rises above the anticipated maximum value whence it is no longer possible to maintain a balance.

The outer wall of the dewar is insulated with foam, which is in turn covered by an aluminum shell heat shield. This shield is driven by another set of TEDs which are connected to an air cooled heat sink. A servo amplifier drives the TEDs to hold the shell heat shield at the same temperature as the inside of the dewar providing a second level reduction of radiation.

Electrical leads and gas tubes necessary for operating an experiment are brought out through a double walled copper pipe thermally connected to the plate, Figure 2. The arrangement provides good thermal contact between the leads and the pipe. By making this pipe relatively long, the leads will be brought to the uniform temperature of the experiment by the time that they reach the puck-plate interface. To the extent that both sides of the interface are at the same temperature, there will not be any heat transfer through the leads. In practice, the thermal conduction of the puck-plate TEDs is much greater than the lead conduction and so is the primary concern of the design.

Great care is taken to assure that the energy supplied to the inside of the dewar is accurately measured. All sensitive electronics is located in a temperature controlled case. The dewar is treated as a multi-terminal black box. Each lead is provided with a temperature controlled shunt. A separate potential lead measures the voltage as each lead transits the puck-plate (see Figure 2.) interface to assure that only the lead heat loss that occurs in the calorimeter is included in the energy balance.

Heat supplied to the dewar is determined by computing the algebraic sum of the volt-ampere product of the various leads. Because all leads contain a shunt, which is one more than needed, a consistency check requires that the currents add to zero.

Experiments are performed by installing a test cell in the spool, Figure 1. Any heat released by the cell will result in an unbalance in the heater servo since the puck will attempt to become warmer than the set point. The servomechanism acts to prevent this by reducing the power applied to the spool heaters. Anomalous heat is indicated when the total power supplied to the cell and heaters is less than the heat removed by the refrigerator.

Access to the test space is provided by lifting off the dewar, the attached foam shells, and the convection cooler. This exposes the spool which can then be approached from all sides for experimental changes. Machined surfaces and bolts inserted from underneath allow clamping the plate to the ring to provide good heat transfer between the plate and the dewar outer wall. A milled groove in the plate reduces the effect of the temperature gradient through the plate.

Instrumentation

Closed loop control of the experiment is performed by analog techniques. A 16 bit high speed digital system collects data under the control of a 80286 based computer. An extended version of this paper contains some details relating to system accuracy.

CALIBRATION

For calibration it is first necessary to determine the refrigeration constant at the selected calorimeter power and temperature. A first level calibration can be made by setting a temperature and noting the power in the balancing heater. With a closed loop time constant of 13 minutes, the system settles to 0.01% in 2 hours. By integrating the difference between the balance power and the determined calorimeter constant over time, the energy drift of the calorimeter can be measured.

Figure 3 was made with a Pt-Pd cell in the calorimeter operating with the Pd electrode as the anode so as to not introduce anomalous heat. The 3.39 cm sq electrode was operated at 60 ma per sq cm. The refrigerator was operating at 8 watts. The maximum indicated drift rate of 50 joules in 5 hours during the first part of the run represents 3 mw. At ten hours and at each hour thereafter, an impulse was added through a second heater which was not included in the computed power balance. Pulses were 5, 10, 20, 40, and 80 joules. Since the computer does not know the pulses have been inserted, the heat is detected as a positive net energy balance. With heaters only, the calorimeter noise is 10 milliwatts rms. Operating cells are noisy, 15 milliwatts rms at 50 ma per sq cm and 40 mw rms at 500 ma per sq cm are typical measurements made with 1 sq cm cathodes. The impulse sensitivity is a function of the calorimeter time constant. We estimate that a practical sensitivity is close to 20 joules (1 sigma) at a high current operating point.

While standard calibration curves have been performed, we believe that a dynamic test is a better measure of the calorimeter under operating conditions. We have performed such tests both with and without an operating cell in the calorimeter. For this test, the calorimeter is operated with two heaters in the active volume. One is connected to the balance servo which works to hold the inside of the calorimeter at constant temperature. The second heater is driven by the computer from a random number generator so that the heat applied varies over a range, in the following case of one watt. This is an

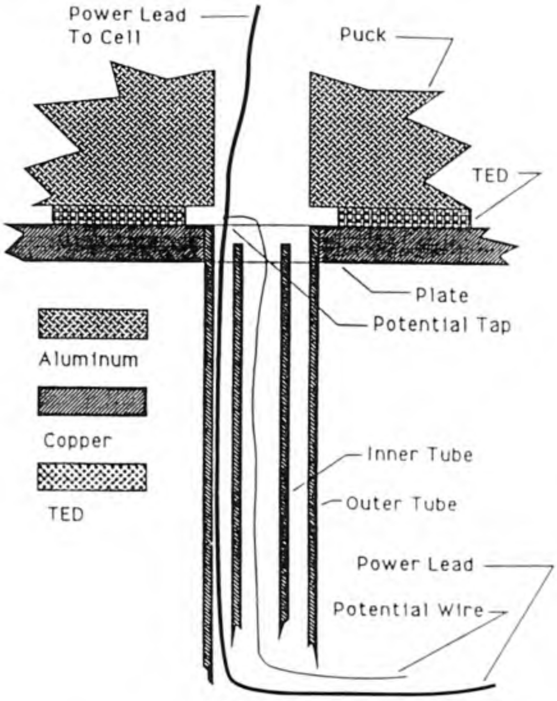

Figure 2. Lead entry detail. Power leads entering the dewar are pressed between two copper pipes so that they make good contact with the outer pipe. The outer pipe is in contact with the plate. This results in the power leads reaching the dewar temperature by the time they reach the puck-plate interface. The lead heat load is carried by the plate. Potential leads measure the power leads as they cross the puck-plate interface to insure that only the lead loss in the dewar is included in the energy balance.

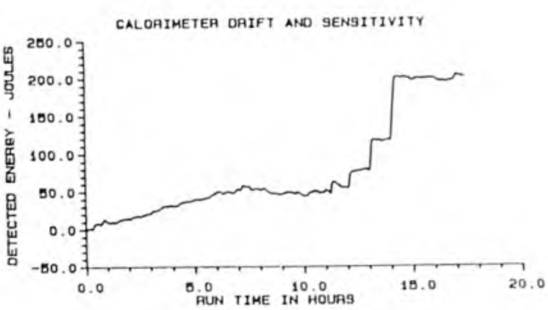

Figure 3. Calorimeter drift and sensitivity. The first ten hours show typical drift for the calorimeter operating at 8 watts total power. At hour 10 and at each hour following, calibration pulses of 5, 10, 20, 40, and 80 joules were inserted by a separate heater not in the computed energy balance.

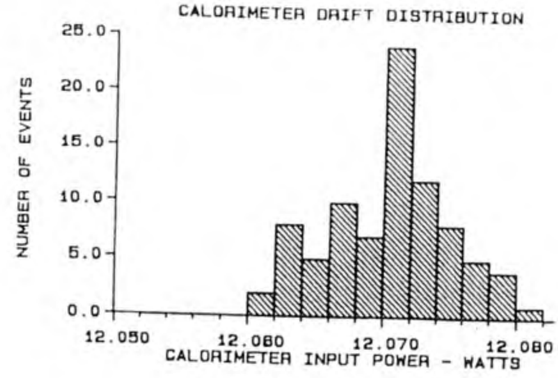

Figure 4. Calorimeter random drift test. In addition to the balance heater, the calorimeter is fitted with a heater driven by a random number generator. Every three hours the random value is changed. After two hours, the total power input is averaged over the third hour to determine a power value. The curve contains 86 three hour measurements, with a mean of 12.0784 watts and a sigma of 0.0044 watts.

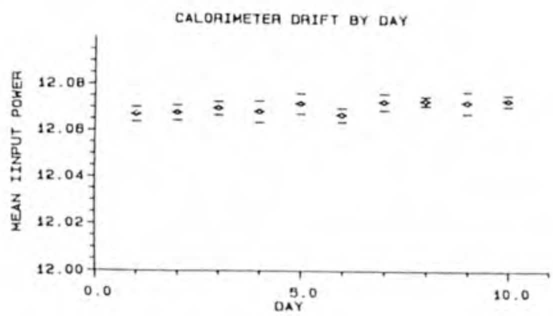

Figure 5. Calorimeter drift by day. The data of Figure 4. plotted by day. The error bars are one sigma..

optimistically high range for anticipated anomalous heat.

For the test of Figure 4, every three hours the random number generator selects a new heat value. After two hours, the two heater powers are averaged over the next hour to give a power value. A perfect calorimeter would always come to the same total power value. The distribution shown in Figure 4 represents 86 points or nearly 11 days of operation. The measured mean was 12.0785 watts with a sigma of .0044 watts. Figure 5 shows the mean and 1 sigma error by day over the 10 full days of the run. The daily variation is somewhat less (3 mw one sigma typical) than the error of Figure 4 and would be even less were it not for a curious periodic component which is under investigation.

DISCUSSION

Measurements indicate that heat removed from the dewar is a constant if the puck-plate Peltier TEDs are operated at constant current and constant temperature. With this design, both sides of the TEDs are held to the same temperature. We have not identified any aging effects, but have mechanically damaged the TEDs on several occasions requiring re-calibration.

The long time constants of these servo loops were a challenge. Because the conventional measurements are so lengthy with long time constants, we attempted a computer model. Because of the limitations of personal computer SPICE simulators, we were not able to generate a model which approached the real calorimeter. After a number of "seat of the pants" attempts gave rather poor results, a long run of random data was processed by the FFT to derive the transfer function. The resulting 13 micro-hertz corner frequency demonstrates the difficulty of generating a Bode plot by a conventional frequency scan.

CONCLUSIONS

We are able to detect a change of 4 milliwatts (1 sigma) from a 12 watt operating point for a passive experiment. An experimental cell generates noise and requires a broad range of operating point with time. This is a greater challenge for calibration. A definitive calibration over the calorimeter range has not yet been accomplished but preliminary data indicates an accuracy in the 10 milliwatt range.

ACKNOWLEDGEMENTS

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REFERENCES

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