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# Calorimetry of Energy-Efficient Glow Discharge – Apparatus Design and Calibration

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### Introduction

This work aims to develop a "family" of low-powered calorimetrically-accurate glow discharge units, similar to that reported by Dardik, et al. at ICCF-10, and to use these to test a wide range of cathode materials, electrode coatings, gas types, gas pressures, and power input levels. We will describe the design and calibration of these units.

The strategy is to use a large number of very similar units so that the calorimetric response does not vary significantly for a given power level. The design is metal or sealed glass cylindrical tubes, charged with 0.4 to 50 torr mixtures of deuterium, hydrogen, argon, or helium gases. Units operate from 0.2 watt to >2 watt power input. The units have low mass (<400 grams) to enhance their sensitivity to excess heat, and they are designed to allow visual observation of the discharge, on-line spectroscopic analysis of the gas to follow any changes in composition, and replication of the geometry and thermal mass during numerous changes in electrode composition. The discharges are presently powered by several power supplies, including 1000 volts DC, 40 kHz, 1200 volt AC, and multiple-frequency/variable-waveform modulated DC. To allow accurate measurement of "true" power, all high-voltage power supplies are driven from 6-12 volt DC input and are enclosed in separate and redundant calorimeters. Calibration is by heat from resistors with known power input from regulated power supplies. Potential electrodes would incorporate nickel, copper, and palladium in thin film or nanoscopic form, although only a small number have been tested initially.

The glow discharge has distinct advantages over the electrochemical approach. It can be operated over a much wider range of temperature; hence, if excess heat is produced, the heat will be more widely useful. A wide range of elements can be applied at either of the two electrodes without regard to their compatibility with a water-based electrolyte. The energy input can be reduced so that excess energy signal will be more distinct, and the system can be scaled to essentially any desired size without deviating from the core design. Finally, the glow discharge area is a novel and potentially valuable area of study. Preliminary data using these two approaches show their basic feasibility in that they can be reliably calibrated. Thus far excess heat levels have been less than the known uncertainties in energy balances, which range from 5 to 30% depending on configuration.

## **Design of Apparatus**

General Layout

- 1. Glow Discharge Tube, gas pressurized with electrodes, encased in calorimeter sleeve and insulation (optional). Temperature measured and compared against ambient at equilibrium.
- 2. Driving power supply, high-voltage, miniaturized and encased in calorimeter sleeve, with temperature measurement.
- 3. Temperature controlled air-flow enclosure (optional).
- 4. Data capture system, logging temperature of ambient and active elements of calorimeter, and DC power input
- 5. Precision DC power supply, providing known voltage.



Figure 1: Layout of apparatus

### **Design of Glow Discharge Tube**

Devices have been built in 3 different configurations, with input powers ranging from 0.2 watt to >2 watt.

<u>Configuration 1:</u> Brass or other metal pipe approximately 40 mm in length and 30 mm in diameter serves as a cylindrical outer cathode, with an axial inner rod of 1-2 mm diameter, running the length of the cylinder, as the anode. The device is made vacuum-tight and includes a viewing window to monitor the progress of the glow. The anode and cathode are composed of a variety of materials.

<u>Configuration 2:</u> Similar to configuration 1, except the entire system is enclosed in a sealed glass tube enclosure. Rolled metal foil or metal mesh is inserted into the glass sleeve, forming a cylindrical outer cathode of approximately 12 mm diameter and 100 mm length. The anode is an axial inner rod of 1-2 mm diameter, running the length of the cylinder. The anode and cathode are composed of a variety of materials.

<u>Configuration 3:</u> A glass tube enclosure is built, encasing 2 opposing "hollow cup" electrodes of approximately 30 mm length and 10 mm inner diameter. Electrodes are made from tungsten with a variety of coatings. The opposing electrodes are linked by a glass tube surrounded by a wire coil; this coil is energized with RF high-frequency signal which ionizes the gas in a highly efficient manner. High voltage current of various types (DC, AC, and Modulated DC) is applied between the electrodes to further ionize the gas.



Figure 2: Opposing "cup" electrodes in sealed glass



Figure 3: Cutaway drawing of cup electrode in glass

### **Gas Handling**

In configuration 1 (brass tube), standard commercial parts are assembled and made sufficiently vacuum leak tight to allow a bleed-through system to maintain 1 to 50 Torr pressures of chosen gases or gas mixtures in a number of tubes simultaneously from a single manifold without significant levels of atmospheric or internal off-gas impurities to interfere. Configurations 2 and 3 use an initial fixed gas pressure in a sealed glass envelope which is then independent of a vacuum system or a gas source and thus can be more fully enclosed for more accurate calorimetry than is practicable in the first method.

### **Driver Power Supply**

The driving power for the glow discharge comes from DC high voltage (700-1200 volts) or variable waveform, multi-frequency modulated DC (700-1200 volts, with modulation of  $\pm$ 300 volts in frequencies from 5 to 5000 Hz). Also an AC high voltage unit (40 kHz, 1000 volts) is used to help ionize the gas with RF energy.

In all cases these driver power supplies are miniaturized and encapsulated in a small metal enclosure (on the order of a 10 cm long, 5 cm wide) which is used in online, continuous calorimetry in order to measure the true power delivered into the tube.

Input power to these driver power supplied comes from regulated DC power supplies with voltage and current measured to 3-figure accuracy. The total system is capable of 3 watts output but can be run stably down to less than 0.2 watts by reducing the input DC.

#### **Accurate Measurement of Input Power**

The calorimetry of the a low-power glow discharge system can be done with acceptable accuracy, as long as we understand some key issues related to input power:

- **Issue 1: In order to do calorimetry on a glow discharge system,** some method of measuring input electrical power must be employed.
- **Issue 2: Direct measurement of "driving" power is extremely difficult**, given the complex unpredictable waveforms of a glow discharge system. Dardik et al. (1) has reported using a 50khz-1mhz sampling data collection system to measure the current and voltage of a complex high voltage AC (or modulated DC) circuit. This approach is quite expensive and requires an unusual degree of effort, which many researchers may not be able to afford. Therefore, direct measurement of the AC/modulated DC power may be impractical.
- **Issue 3: Measure simple DC power into the power supply**. The solution therefore is to measure the DC power flowing into the driver power supply that creates the high-voltage AC (or modulated DC) power. For example, in our system, we might input approximately 6 volts DC, 150 milliamps (about .9 watts) of steady electric current into our oscillator/amplifier circuit, which converts this DC input into high-voltage modulated DC or AC. This approach, however, requires that we understand how much of that input power is being delivered to the glow tube, and how much is lost as waste heat in the electronic circuit.
- **Issue 4: Conversion efficiency of the power supply varies constantly**. For any given glow discharge, the amount of power lost as waste heat in the driver power supply can fluctuate from 30 to 90%. This conversion efficiency is based on gas pressure, temperature, power level, gas composition ( $D_2$ ,  $H_2$ , or Argon), surface conditions of the electrodes (metals in glow discharge will sputter, dynamically creating and destroying nanopores in the electrode surface and changing the ionization potential of the system) and many other factors. Essentially there is no way to predict or rely on any predicable power supply conversion factor.
- Solution: Real-time calorimetry of power supplies. In our system, we miniaturize all power supplies and perform calorimetry on them in parallel (usually with the same apparatus) as the glow tube itself. Therefore we can very easily measure the low-voltage DC power and current into the system, giving us an unambiguous energy input (Qi), then measure the proportion of this DC input power that is lost as waste heat in the power supply (Qp). The total energy into the glow tube (Qa) can easily be seen as Qi-Qp. In Figure 4 we see data from our "zero" control tubes; input power Qi can be compared to the sum of the two calorimetrically measured powers Qa and Qp, demonstrating an accuracy of better than 5% accuracy. If there were excess heat being produced in the tube, we would see it easily as excess in Qa.

Power In	Power Out, Calculated from Calorimeter					
Qi	Q <sub>p</sub>		Qa Tuk			% of DC
DC watts	Power Supply Loss		lube		lotal	Power
0.1378	0.1246	90.39%	0.0138	10.00%	0.138	100.39%
0.988	0.5854	59.25%	0.4142	41.92%	1.00	101.17%

Figure 4: Typical breakdown of power to supply or glow tube (control tube)

### **Calorimetry of Tube**

Calorimetry is based on measurement of equilibrium temperature of the device in air. We use two methods, based on screening speed versus accuracy of measurement:

<u>Calorimetry Method 1</u>: Both glow tube and power supply are enclosed in separate copper or aluminum enclosures, with the only connection being wires to conduct the low or high voltage currents. Thermocouples and/or thermistors (5K ohms, 2% precision) are attached to the outside of the metal enclosures, which measure the equilibrium temperature reached in the ambient air environment. Calibrating heaters are installed inside the metal enclosure (adjacent to the glow tube and/or power supply) and the system is calibrated for several given levels of power. In some cases a thermal fluid is poured into the enclosure to ensure even distribution of heat inside the unit. This method is very fast, for large-scale screening of systems, but provides at best 10% uncertainty (and at worst 30%) because of low temperature differences between the tube and ambient, variable air currents.

<u>Calorimetry Method 2:</u> The above metal enclosures are inserted into a thick insulating body (either Dewar or Styrofoam) with only a small portion of the metal exposed to the air. This insulated body is placed in a controlled laminar airflow box with temperature controlled  $\pm 0.1$  degrees C. The temperature of the exposed metal surface is measured by thermocouple or thermistor at equilibrium, compared to ambient. System is again calibrated for a number of input powers to an internal heater. This is similar in principle to the "double wall isoperibolic calorimetry" as described by Storms (2). Because the heat flux is concentrated into a smaller, more controllable point of exposed metal, and the airflow and temperature are more precisely controlled, this method provides much better accuracy, with uncertainty less than 5%.

Note that any number of other calorimetric approaches such as liquid isoperibolic, flow, or Seebeck, could be used with equal or better success to the above. Refer again to Storms (2) for discussion of relative merits.

Data is collected using a Keithley 2700 40-channel data collection device, which feeds data into MS Excel spreadsheet on any PC. This system is simple to use and robust.

#### **RF** Noise

Another common problem of the glow-discharge system is RF noise from the high-voltage signal or RF ionization signal. In our system this problem is avoided by enclosing both glow tube and power supply in copper or aluminum enclosures. These shield the external thermocouples or thermistors from RF noise, and also, as described above, serve as integral parts of the calorimeter. Finally the Keithley data collection system is quite effective at filtering stray RF noise.

#### **Time Constants**

Time constants vary from 30 to 45 minutes when exposed with no insulation to ambient air to 3 hours, encased in insulation, in thermally controlled enclosure. Time constants are defined as the time to reach thermal equilibrium for steady input power.

Our approach assumes the excess heat, when and if detected, will be of sufficiently long and steady nature to be observable with systems with relatively long time constants. Highly fluctuating excess power would be detectable but not easily quantified by these methods.

### **Results**

Figure 1 through 6 give the time history of two "Configuration 1" glow discharge tubes, during calibration with resistive heaters, during operation at 10, 20, and 5 Torr respectively, and finally the calorimetry of the power supplies feeding 40 khz AC to the two tubes. These temperature differences, while generally precise to ~0.2 deg C are accurate to ~0.5 deg C. The differences in the various thermistors are not significant when looking for >50% values of excess heat and are the primary cause of the uncertainty of up to 30% in these measurements. No apparent differences in thermal balances were noted between the above three gas pressures in the tubes, but there were obvious changes in the appearance of the discharges as pressure was changed. Higher pressures cause the discharge visual emissions to shrink toward the center electrode. Of the four tubes operated simultaneously, thermal balances varied from 87% to 111% where these percentages are the observed energies in the discharges divided by the power delivered to the tubes.

Similar runs were performed with Configuration 2 and 3 tubes, with similar results. No heat was seen in excess of known uncertainties.

### Discussion

The above results are the first set of measurements attempted by this approach and the absence of excess heat is not unexpected. We purposely tried a much simplified experiment from that described by Dardik, et.al. (1), but with a great number of changes to process variables to allow us to see a wide range of behaviors in the apparatus. In the first place, we tried placing the active metals (Pd) leaf wrapped around the center electrode, with none on the cylindrical electrode surface. Then we tested palladium powders and wire. Furthermore we used AC power, DC

power, and 3-frequency modulated DC power to drive the glow. Finally we used mixtures of argon and deuterium, in addition to pure deuterium, to test a variety of discharge modes.

Different configurations were shown to have different advantages:

Configuration 1 (gas bleed through system) allows the measurement of any product gases generated within the discharges to be captured and subjected to various measurements of composition on the fly. Even though this is less accurate, it is quicker to build, and the cell comes to equilibrium quickly, so that very large numbers of samples can be screened quickly.

Configurations 2 and 3 (sealed glass tubes) have important advantages. They do not need a continuously operating vacuum system or gas supply. They can employ much cleaner electrodes, more free of surface contamination. Their calorimetry is slightly more complex with a longer time to reach thermal equilibrium, but they are small enough to fit inside a small temperature-controlled chamber, which gives them greater accuracy. Additionally the sealed glass tube can be run for extended periods of time, allowing product gases to accumulate to higher concentrations.

### Conclusions

Methods have been developed for efficient screening of candidate materials on electrode surfaces of glow discharge tubes that will provide an environment conducive to nuclear reactions between deuterium and itself or other light elements. This method can detect excess heats ratios >1.2 with more than 95% certainty. It provides a valuable new platform for large-scale exploration of excess heat effects in the gas phase, using low power inputs in the 0-3 watt range. This method proves to be inexpensive, quick, accurate, and easy to perform once the basics are mastered.

Note: The authors are interested in testing electrode materials from other sources, especially those that have already been successful in a liquid (electrolytic) environment. Also we would be pleased to provide advice or equipment to other researchers. Interested parties please contact the authors.

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Fig. 10 Photo Of Sealed-Glass Type Glow Tube (Mounted On Vacuum Station)