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An assessment of claims of 'excess heat' in 'cold fusion' calorimetry

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Abstract

Claims of 'excess heat' from measurements of the heat of electrolysis at several watts of power are largely based on use of poorly characterized, isoperibol, heat-conduction calorimeters with single-point temperature sensors. This paper describes construction, testing, and calibration of a calorimeter of similar design. Heat-conduction calorimeters with single-point temperature sensing and inadequate mixing are subject to large systematic errors resulting from non-uniform heat distribution within the system. Confirmation of electric-heater calibration by a chemical reaction with a well-known enthalpy change is a minimum requirement to insure accuracy. Improper or incomplete calibration is a probable cause for many claims of 'excess heat' in 'cold fusion' experiments. © 1997 Elsevier Science B.V.

Keywords: Calibration; Calorimetry; Cold fusion; Excess heat

1. Introduction

Claims of nuclear products accompanying excess heat in 'cold fusion' electrolysis experiments have largely been discredited [1–3], but little work has been done to test the claims of 'excess heat'. Continuing claims of excess heat and the untested and poorly characterized calorimeter designs used to generate these claims became the reason for us to reluctantly enter this arena in an effort to determine the validity of observations of 'excess heat'. In the first paper [4] in this series, we used a commercially available, isoperibol, differential heat-conduction calorimeter to demonstrate that many claims of 'excess heat', particularly at low current densities, were the result of assuming 100% Faradaic efficiency when in reality Faradaic efficiencies were much less. Because of the non-linear response at heat rates > 60 mW, experiments in that calorimeter were restricted to relatively low currents and current densities. For work at higher power, EPRI provided a small grant to construct a calorimeter which would fit in our neutron detectors and could accurately measure heat rates up to several watts. Because of its potential utility in other applications, development of this calorimeter has continued even though EPRI withheld further support after being informed of the results of the work done at low power. Obtaining accurate heat rates from the high-power calorimeter proved to be a challenge because of subtle, but large systematic errors associated with mixing and single-point temperature sensing in the calorimeter.

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The governing equation for heat conduction calorimetry is [5]

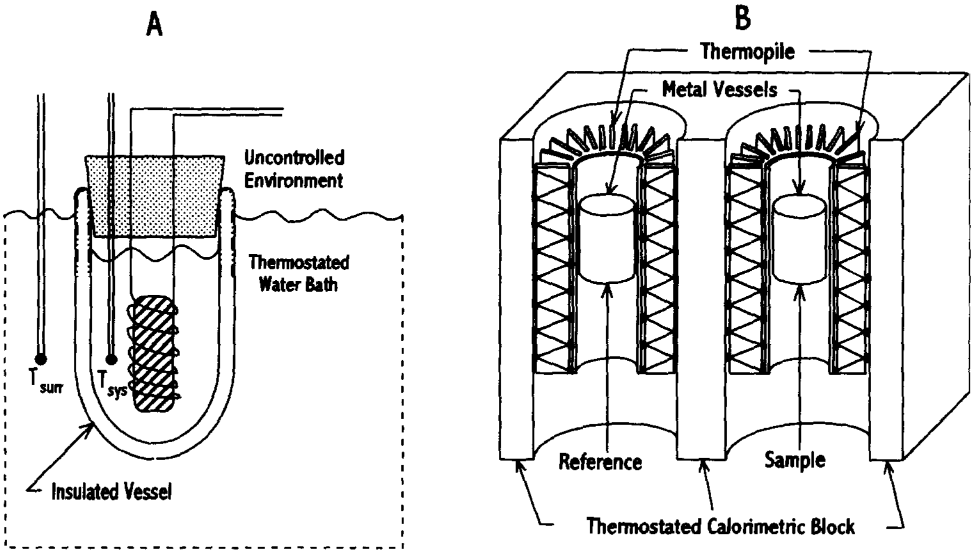
$$\Phi(t) = G\{\theta(t) + \tau[(\mathrm{d}\theta(t)/\mathrm{d}t]\} \qquad (1)$$

where $\Phi(t)$ is the rate of heat accumulation by the calorimetric cell and exchange with the surroundings as a function of time (t), $\theta(t)$ the temperature difference between the vessel and surroundings as a function of time, τ the calorimeter time constant, and G the thermal conductance between the calorimeter and the surroundings. Under conditions in which the time constant (τ) is very small or the derivative of the temperature difference $(\mathrm{d}\theta/\mathrm{d}t)$ is negligible [5] and the surroundings are at constant temperature, Eq. (1) reduces to

$$\Phi(t) \equiv G\theta(t) = G[T_{\mathrm{system}}(t) - T_{\mathrm{surroundings}}] \qquad (2)$$

where $\Phi(t)$ is the rate of heat exchange with the surroundings as a function of time, $T_{\mathrm{system}}(t)$ the average temperature of the surface of the calorimeter vessel, and $T_{\mathrm{surroundings}}$ the average temperature of the materials surrounding the calorimeter vessel. Determination of $\Phi(t)$ thus only requires the measurement of $\theta(t)$ and G, but only if the calorimeter is designed so that Eqs. (1) and (2) accurately describe the thermal behavior of the calorimetric system.

The design criteria for obtaining accurate results with heat-conduction calorimeters were recorded by Calvet et al. [6] over fifty years ago. The thermal conductivity of all heat paths between the cell and its surroundings must be constant or G will vary. The θ measured must accurately represent the actual temperature difference between the calorimeter vessel and the surroundings. The heat-conduction paths must be the same during calibration and measurement. As can be seen in Fig. 1B, commercially available heat-conduction calorimeters that have been proven to perform accurately meet these conditions. These have vessels constructed of a good thermal conductor with a major part of the surface area in contact with a thermopile which also serves as the constant thermal path to the heat sink. Even in these calorimeters, the calibration constant determined by electrical calibration may depend (to a few percent) both on the location of the heater and the contents of the calorimetric vessel [7,8].



Schematic of Typical "Cold Fusion" Calorimeter

Schematic of Typical Commercially Available Heat-Conduction Calorimeter

Fig. 1. A comparison of typical heat-conduction calorimeter designs used in 'cold fusion' studies and in proven, commercially available instruments.

In 'cold fusion' type calorimeters (Fig. 1A), on which most claims of 'excess heat' [9–14] are based, several complications are immediately apparent. An evacuated Dewar flask or similar insulated vessel provides a low thermal conductivity from the reaction vessel to the environment and, thereby, increases the sensitivity by decreasing G. However, the time constant, τ, is proportional to G^{-1}, and thus a vessel with a small value of G must take a very long time to reach the steady state, thus, invalidating Eq. (2) under most conditions [5]. Events occurring in less than 6τ must be evaluated with help of Eq. (1), and not Eq. (2). Part of the proper calibration procedure is to verify that the calorimeter obeys Eqs. (1) and (2) under the conditions it will be operated. Furthermore, Eq. (1) does not apply to systems with multiple, disparate time constants. For example, a Dewar flask [14] or thickly insulated vessel [12] has a time constant associated with each thermal path to the surroundings, e.g. through the top, through electrical leads, through the glass, through the silver on the glass, and through radiation, that differ significantly, but all apply to thermal paths that make a significant contribution to heat exchange with the surroundings. For example, heat transfer by radiation and conduction can be comparable, but have different time constants in the Dewar flasks. Fleischmann et al. [14] neglected conductive heat transfer and assumed only radiative heat transfer in their data analysis, but calculations (Table 1) demonstrate that conduction of heat through the glass and the silver can be significant.

The typical design shown in Fig. 1A also raises the following question: Are the effective thermal conductivities of all heat transfer paths to the environment constant? Because the thermal conductivity through the vessel walls is so much smaller than through the electrical leads, etc., that go through the top, heat exchange with the surroundings through the lid may be significant compared with, or even greater than, the heat exchange with the bath through the walls.

In calorimeters similar to the design in Fig. 1A, another untested assumption is that the calorimeter and surrounding temperatures are accurately represented by the temperatures measured at single points in each. Any thermal gradients present in the solution or the surroundings could greatly influence the results. This systematic error may also be the basis for claims of 'excess heat' in flow calorimeters such as those of McKubre [15] and Patterson (as described in [16,17]). Heat-rate measurement in these flow calorimeters also depends on measurement of θ, the accuracy of which could easily suffer from the presence of temperature gradients and unaccounted-for thermal paths to the surroundings. Such isoperibol, temperature-rise, flow

Table 1
Relative importance of heat transfer by conductance and radiation in evacuated Dewar flasks

Glass thickness/ cm	Silver thickness/ cm	Glass, heat rate by conduction [a]/ mW	Silver, heat rate by conduction [a]/ mW	Glass, heat rate by radiation [b]/ mW	Ratio of heat rate by conduction to heat rate by radiation [c]/ %
Assumptions: (1) Dewar has 10 cm circumference; (2) Height of unsilvered glass is 8 cm; (3) ΔT of 1 K at 300 K; and (4) 5 cm from solution to top of Dewar. See Fig. 1A.					
0.1	0.001	2.343	8.54	50.36	22
0.01	0.0001	0.2343	0.854	50.36	2
0.1	0.0001	2.343	0.854	50.36	6
0.01	0.001	0.2343	8.54	50.36	17
Assumptions: (1) Dewar has 10 cm circumference; (2) Height of unsilvered glass is 4 cm; (3) ΔT of 1 K at 300 K; and (4) 3 cm from solution to top of Dewar.					
0.1	0.001	3.905	14.23	27.47	66
0.01	0.0001	0.3905	1.423	27.47	7
0.1	0.0001	3.905	1.423	27.47	19
0.01	0.001	0.3905	14.23	27.47	53

[a] Calculated with heat rate = λ(area)(ΔT)/(length), where area = circumference × thickness of material and λ is the thermal conductivity.
[b] Calculated with Stefan's law, heat rate = R(area) = $e\sigma(T_1^4 - T_2^4)$(area), where area = unsilvered area of Dewar.
[c] Sum of conduction heat rates for glass and silver layers, radiation from unsilvered glass only because heat rate from silvered glass is only 2% of unsilvered.

calorimeters have not proven to be generally useful because of such problems [18].

A minimum requirement to establish the accuracy of a calorimeter is verification of electrical calibration with a known chemical reaction [19], a requirement 'cold fusion' workers have largely chosen to ignore. In heat-conduction calorimeters with single-point temperature measurement, it is also necessary to show that the calibration constant is independent of the location of the calibration heater and temperature sensors in the vessel [20]. With the 'cold fusion' type heat-conduction calorimeter constructed in this study, when stirring was inadequate, we were able to repeatedly produce apparent 'excess heat' (either positive or negative) with a known chemical reaction, simply by changing the position of the heater or temperature sensors. Because the type of calorimeter illustrated in Fig. 1A has many potential applications other than 'cold fusion', this study defines the conditions under which accurate results can be obtained.

2. The calorimeter

The isoperibol, heat-conduction calorimeter (Fig. 2) was built from a brass tube, 38 mm (1.5 inch) i.d. and 178 mm long, with brass plates soldered on both ends. The top plate has 16 tubes of 6.35 mm (0.25 inch) i.d. soldered into holes around the perimeter, and one large hole in the center for the reaction vessel. Seven of the holes in the top are inlets from the constant temperature bath and discharge at the bottom of the jacket while seven others are outlet tubes, alternately placed between the inlets, to carry water off the top of the jacket. (Because of the appearance of the 14 curved tubes coming out of the top in a symmetrical pattern, the instrument has been nicknamed the *Octopus*.) The remaining two openings have tubes sealed and soldered at the bottom to serve as wells for thermocouples and thermistors which measure the jacket temperature. The wells are filled with water to improve thermal conductivity. Water is circulated through the jacket at > 5 l min^{-1} from the constant temperature bath (Hart Scientific Model 5024) that maintains temperature within ±0.2 mK. The bath was set at 25.2°C. The bath could more easily serve as the outer jacket, but this particular calorimeter was designed to be operated in a location remote from the bath.

The reaction vessel is a 135 mm × 20 mm Pyrex test tube glued to a threaded brass head that screws into the brass jacket (Fig. 3). A Teflon bearing is fitted in the

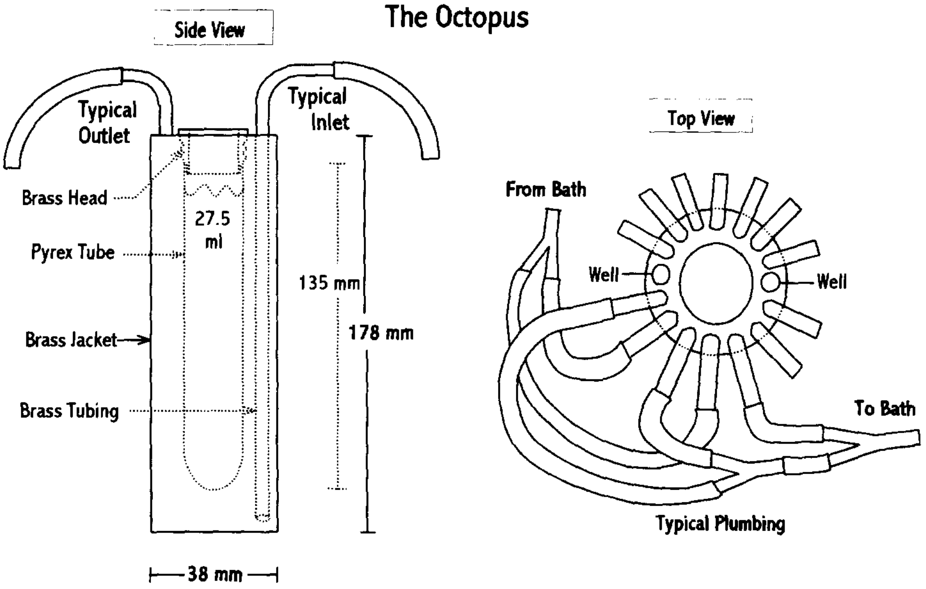


Fig. 2. Octopus calorimeter – design and dimensions.

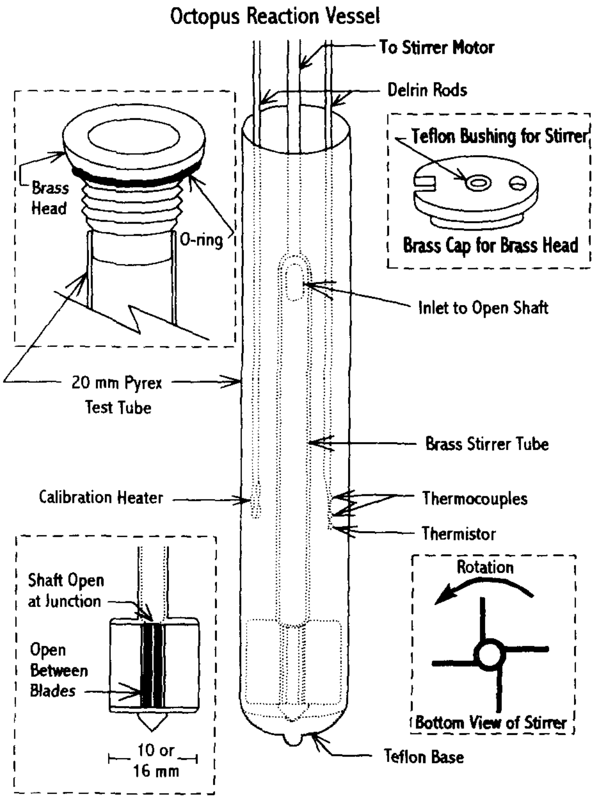


Fig. 3. Octopus reaction vessel details.

bottom of the test tube to stabilize the stirrer. The vessel holds 27.5 ml of solution. The stirrer (epoxy coated brass) is driven by a 1000 rpm synchronous motor. The brass cap contains a Teflon bushing in the center through which the stirring rod extends, two stationary Delrin rods ($\approx$2 mm o.d.) that extend down into the cell, and two holes to allow for escaping gas and for adding or removing liquids. The calibration heater (37.2 Ω total) is constructed of three wire-wound resistors in series to increase surface area and heat transfer to the solution. The heater leads are 32 gage Cu ca. 50 cm long. Four leads were used, two for current conduction and two to measure the voltage at the resistors. The heater and a 1 mm o.d. Teflon tube for liquid addition are attached to one of the Delrin rods. The end of the Teflon tube is in the middle of the calibration heater. The voltage for the calibration heater is supplied by a control panel from a Tronac Model 450 calorimeter. The wire-wound resistor (2.051 Ω) used to determine heater current was calibrated against 1, 10 and 100 Ω standard resistors.

The other Delrin rod supports two thermocouples and a thermistor. The temperature sensors were first coated with epoxy, then placed in Teflon heat-shrink tubing and the ends sealed with silicone rubber. The sensors were attached to the rod to prevent lateral or vertical movement. The thermocouples (copper–Constantan) provide redundant confirmation of the thermistor response and, though not as sensitive to ΔT as thermistors, thermocouples are not susceptible to errors caused by self-heating. The thermistors are glass encapsulated 10 kΩ with a 10 V power supply across the bridge. Inside the vessel, wiring for the heater, thermistor and thermocouples was encapsulated in heat-shrink Teflon tubing. To avoid unwanted Seebeck effects in the temperature sensor circuits, connections were made on a terminal block fastened to an aluminum block ($112 \times 112 \times 13$ mm^3) attached at the top of the jacket.

To eliminate the possibility of systematic errors in the ΔT measurement, the calorimeter vessel has three temperature sensors, one thermistor and two thermocouples. The differential thermistor bridge ($\approx$0.1 V K^{-1}) measures the ΔT between the vessel and a well in the jacket (Figs. 2 and 3). One thermocouple pair ($\approx$40 μV K^{-1}) measures ΔT between the vessel and the other well of the jacket in a similar way. The second thermocouple pair measures ΔT between the cell and an exterior ice bath. If the calorimeter is working properly all three sensors should record equivalent responses to a heat effect.

Data collection is done through an IEEE board attached to a Keithley Model 195 digital multimeter and a Hewlett–Packard 3488A multiplexer (Fig. 4). Data collected during an experiment typically include elapsed time interval, heater voltage, heater current (voltage across a calibrated resistor), thermocouple voltage 1 (vessel and jacket), thermocouple voltage 2 (vessel and ice bath) and thermistor–bridge–unbalance voltage (vessel and jacket). Data are typically collected every 22 s.

The shape of the reaction vessel (a tall, narrow test tube) made mixing in the vertical direction difficult, so the stirrer was designed to pump solution downward through the tubular shaft. Four blades at the base of the open shaft create a reduced pressure at the junction of the shaft with the blades (Fig. 3). Mixing was tested

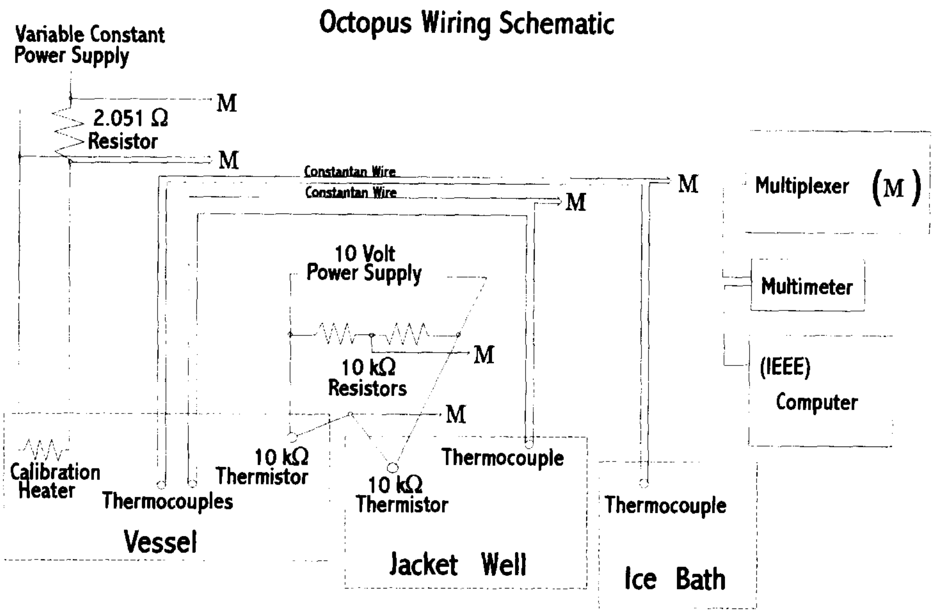


Fig. 4. Octopus wiring schematic.

by observing the mixing of dye injections. Visually, stirring appeared to be very rapid, at 1000 rpm (< 1 s).

The heat of dilution of 1-propanol was used as one standard test reaction to verify the electrical calibration. The amount of heat generated (176 ± 3 J), when 1.02 ml (4 min injection at 255 ± 1 μl min^{-1} from a motorized buret) of neat 1-propanol was added to 27 ml of a 0.030 M solution, was first determined on a Tronac model 450 titration calorimeter. Total heat at intermediate points was also determined, and the heat generated was linear with quantity added, the intercept being equal to zero within the limits of error. A second standard test reaction, addition of perchloric acid solution (4.42 M) to Tris (tris[hydroxymethyl]aminomethane) base solution (0.25 M prepared by weight) was used to confirm the 1-propanol dilution results. The enthalpy change of the reaction determined with a Tronac Model 450 titration calorimeter (47.24 kJ mol^{-1}) was within the limits of error of the literature [21] value (47.36 ± 0.25 kJ mol^{-1}).

3. Results and discussion

Table 2A gives the baseline noise and temperature detection limits for the temperature sensors with the first stirrer constructed. By switching the heater on or off, the calorimeter time constant (τ), defined by the equation $V = ae^{-(t/\tau)}$ (where V is the thermistor bridge unbalance voltage, a a constant and t the time), was determined to be ≈0.5 min by fitting the voltage–time curve. After the heat transfer rate stabilized with the heater on, the temperature rise was constant with less than 5% peak-to-peak noise.

Table 2
Baseline noise and detection limit

	Standard deviation of the baseline [a]/ μV	Detection limit [b], ΔT/ K
A. Original stirrer		
thermistor	94	0.0028
thermocouple vs. bath	0.71	0.053
thermocouple vs. ice	1.0	0.075
B. Improved stirrer		
thermistor	51	0.0015
thermocouple vs. bath	0.78	0.059
thermocouple vs. ice	0.66	0.049

[a] The noise level over a 6 h run.

[b] The detection limit is three times the standard deviation of the noise divided by the sensitivity.

The calibration constant was calculated in two different ways. First, G was calculated as the slope of the line in a plot of the change in voltage of the thermistor bridge against the heater power. The value of G' was calculated as the slope of the line in a plot of the area under the temperature–time curve against the total heat from the heater. Both plots were linear from 0.02 to 4 W (5 to 1200 J) with zero intercepts. Area was determined with the trapezoidal rule: $\int f(t)\mathrm{d}t \approx \sum 1/2[V_2 + V_1][t_2 - t_1]$. (To distinguish G from G', G values are given with units of W mV^{-1} and G' values are displayed with units of J s^{-1} mV^{-1}; note that J s^{-1} = W.) The value of G^{-1} was found to be 25.3 mV W^{-1}, and G'^{-1} - 25.1 mV s J^{-1}. Because the volume changes during the experiment, the calorimeter was calibrated at various liquid volumes. The response of the thermistor bridge (V) was plotted against the input wattage while the volume of water was changed from 25.5 ml to 28 ml. The calibration constant varied only slightly. The change in G is < 1.7 W mV^{-1} or $\approx 2\%$ per ml change in volume (at 0.8 W of power).

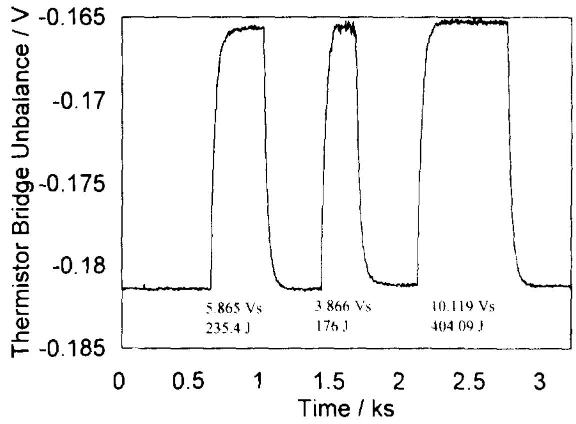


Fig. 5. Typical calibration data from the thermistor bridge (see D1, Table 3).

Several tests were performed in which the calibration heater was on for a period, after which a four minute injection of 1-propanol was made, followed by turning on the calibration heater again (Fig. 5). The response of the thermistor bridge to the calibration heater was 8% greater than the response produced by the injection of 1-propanol (Table 3). It was apparent that large systematic errors were present. These results were further tested with the perchloric acid-Tris reaction. The acid-base reaction in the Octopus gave the same 8% difference, indicating that the problem was not with the chemical reactions, but with the calorimeter.

Moving the calibration heater gave a different calibration constant at each new position. The results with position were random, i.e. the lowest position (one-third of the way up the vessel) gave a constant ca. 18% high, while the highest position (two-thirds of the way up the vessel) gave a constant in agreement with the chemical reaction heats, and an intermediate position (middle of the vessel) gave a constant ca. 18% low. We concluded that the point temperature sensors were not measuring the average solution temperature. This source of systematic error probably accounts for many of the reports of 'excess heat' and much of the

Table 3
Calorimetric constant determined with electrical heating compared to injection of 1-propanol (original stirrer)[a]. Note the 8% difference between results of electrical and chemical calibrations

Run	Heater energy/ J	Response/ V s	$[G']^{-1}$/ mV s J^{-1}	Injection energy/ J	Response/ V s[b]	$[G']^{-1}$ in V sJ^{-1}	Heater energy/ J	Response/ V s	$[G']^{-1}$/ mV s J^{-1}
A1	234.1	5.878	25.1	176	4.061	23.7	331.1	8.277	25.0
B1	607.4	15.21	25.0	176	3.929	22.9	952.1	24.06	25.3
C1	247.2	6.251	25.3	176	3.925	22.9	275.7	6.969	25.3
D1	235.4	5.865	24.9	176	3.866	22.6	404.1	10.12	25.0
E1	275.9	6.925	25.1	176	3.968	23.1	262.6	6.599	25.1
F1	193.7	4.893	25.3	176	4.096	23.9	249.9	6.320	25.3
G1	485.1	12.18	25.1	176	4.041	23.6	606.4	15.38	25.4
H1	1562	38.48	24.6	176	3.877	22.6	438.8	10.78	24.6
Average			25.1			23.2			25.1

[a] $G^{-1} = 25.1 \pm 0.3$ mV s J^{-1} when determined with electrical heating and 23.2 ± 0.5 mV s J^{-1} with chemical injection.[b] Values adjusted for heat change due to the introduction of a liquid cooler than the calorimeter.

Table 4
Calorimetric constant determined with electrical heating compared to chemical reaction [a] (improved stirrer)

Run	Injection energy/ J [b]	Response/ V s	$[G]^{-1}$/ mV s J^{-1}	Heater energy/ J	Response/ V s	$[G']^{-1}$/ mV s J^{-1}
A2	176	4.37	25.6	189.6	4.75	25.1
B2	176	4.32	25.3	151.7	3.82	25.2
C2	176	4.37	25.6	151.7	3.82	25.2
D2	176	4.29	25.1	151.7	3.83	25.2
J2	176	4.31	25.2	151.7	3.82	25.2
K2	176	4.32	25.3	151.7	3.80	25.0
A3	215	5.24	25.0	151.6	3.78	24.9
B3	215	5.28	25.2	151.6	3.81	25.1
C3	215	5.17	24.7	151.6	3.75	24.7
D3	215	5.20	24.8	151.6	3.80	25.1
Average			25.2 ± 0.3			25.1 ± 0.2

[a] Runs A2-D2 and J2-K2 are 1-propanol dilution. Runs A3-D3 are perchloric acid-tris reactions.
[b] Values adjusted for heat change due to introduction of liquid cooler than the calorimeter.

irreproducibility of 'cold fusion' calorimetry. The percent 'excess heat', as also the production of any 'excess heat', is seldom a reproducible result in the 'cold fusion' articles reviewed [9–14]. Previous explanations for the irreproducibility usually focused on defects, 'contamination', or improper manufacture of the cathode, but deficiencies in calorimeter construction and calibration are a more probable explanation.

The influence of the stirrer was investigated by changing the stirrer motor from 1000 to 600 and 200 rpm. 'Excess heat' increased as the rpm was reduced, from ca. 8% at 1000 rpm to ca. 11% at 600 rpm to ca. 40% at 200 rpm. Despite visual indications, using dye injection, that stirring was adequate [11], these results show that it was not.

The stirrer was improved by reducing the shaft from 6.35 to 4.76 mm o.d. and increasing the impeller diameter from 10 to 16 mm. The new stirrer was operated only at 1000 rpm. Both chemical reactions were again used to test the calorimeter. The results are given in Table 2B and Table 4. A slight decrease in baseline noise was noted with the improved stirrer. Difference between response to the calibration heater and a chemical reaction was $< 1\%$ (Table 4). The calorimeter was retested at various liquid volumes. The response of the thermistors (V) was plotted against the input heat rate while the volume of liquid was changed from 26.5 to 29 ml. The change in G was again $<2\%$ per ml volume change (at 0.8 W of power).

4. Conclusion

Stability and repeatability are not sufficient verification of the accuracy of a calorimeter. A chemical reaction with a known heat effect should always be used to verify electrical calibration. The accuracy of heat-conduction calorimeters with point temperature sensors can be significantly affected by inadequate mixing. The time constant of mixing must be much smaller than the time constant of heat transfer to the surroundings, otherwise the rate of heat transfer measured by point sensors will depend on the location of the heat source. Even when mixing appears to be rapid, electrical heating can produce calibration constants with large errors. Up to a 40% different response (i.e. 'excess heat') between the heater and a chemical reaction was observed in this study. If the calorimeter is designed properly and mixing speed is adequate, location of point temperature sensors in the vessel will not affect the results. The lack of reproducibility in 'cold fusion' experiments is the expected and observed result from systematic errors arising from use of single-point temperature sensing in a heat-conduction type of calorimeter.

Problems in 'cold fusion' calorimeters include inadequate stirring, unstable heat paths, and inadequate calibration to validate use of point temperature sensors. Claims of 'excess heat' based on measurements with the type of calorimeter illustrated in Fig. 1A should not be accepted until all these problems have been resolved. Observations of 'excess heat' are more likely due to calorimetric errors than to violations of the laws of thermodynamics, or to known or unknown nuclear or chemical reactions.

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