# Report on Mizuno's adiabatic calorimetry

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#### **Retraction**

Some calibrations performed after this paper was written cast doubt upon the results. I now believe that most if not all of the apparent excess heat was caused by changes in ambient temperature. This is described in Appendix A.

### Introduction

T. Mizuno published a paper in 2013 describing anomalous excess heat from nanoparticles produced on palladium or nickel wires. [1] The wires are carefully cleaned, then installed in the reactor. The reactor is filled with deuterium or hydrogen gas. The wire is then heated, and the gas is purged and refilled. This heat-and-purge cycle is repeated several times to clean the wire. Then the wire is eroded with glow discharge for several days, leaving nanoparticles on the wire surface. Because they are created *in situ*, the nanoparticles are never exposed to air. The reactor is then filled with hydrogen or deuterium gas, heated again, and the wire absorbs the gas and produces anomalous heat. Once the nanoparticles are formed, glow discharge is no longer needed. The wire is heated with a resistance heater, and it produces anomalous heat. <sup>1</sup> The heat turns on within minutes. In late 2013 the experiment produced heat in 16 out of 22 tests. [1] In recent months it has worked in every test.

The method of calorimetry described in the 2013 paper was crude, with a large error margin. The stainless steel reactor was left open to ambient air, and the air temperature and airflow were poorly controlled. The method was mainly a form of isoperibolic calorimetry based on the temperature inside the reactor, and also on the temperature measured at two locations on the reactor surface. The surface temperature of the reactor varied widely from one location to the other, being warmest at locations close to where the reaction occurred. The resistance heater was run continuously to keep the reactor internal temperature high. This made the reactor outer surface too hot to touch. Mizuno developed complex calorimetric formulas to measure the heat based on various factors, such as the heat captured by the reactor metal. The main heat loss path was from the reactor surface to the surroundings, which was measured based on the surface and

<sup>&</sup>lt;sup>1</sup> The 2013 paper did not clearly explain that the effect is triggered by heating the wire with a resistance heater. The glow discharge phase may also produce anomalous heat, but this is difficult to measure because the input power is complex.

ambient temperatures recorded by thermocouples. This is not a good technique because a small error in the temperature measurement causes a large error in the estimated heat.

The 2013 paper reports large excess heat, but in my opinion the method of calorimetry was so problematic these numbers are open to question.

Over several months starting in December 2013, Mizuno took steps to improve the calorimetry. He insulated the reactor somewhat, and then wrapped it in a plastic tube cooling loop.

Starting in August 2014, I began working with Mizuno. I reviewed the calorimetric data from 2013 and 2014, as well as schematics and photographs of the equipment layout. I discovered some errors and shortcomings in the data, including a possible instrument error in January 2014.

In August and September 2014 Mizuno made important changes to the experiment. He added a great deal of additional urethane foam insulation above the plastic tube, so that the cooling water captures a larger fraction of the heat. He reduced input electric heating power and overall energy. He simplified the calorimetric methods and analysis. He now uses adiabatic calorimetry, described below.

In October 2014, I visited the laboratory and spent ten days learning more about the equipment, watching the experiment in operation, and taking data with my own instruments. This report describes the calorimetry of the system as it exists today, and it describes recent results. I will not discuss previous configurations or data, because that would confuse the issue. Based on the recent data, I believe there probably was anomalous heat in previous experiments, but I cannot be sure of that. I have more confidence in the recent results taken with improved calorimetry.

In the tests I observed, the reactants were palladium wire in deuterium gas. The wire is 0.2 mm thick and 3 m long (Nilaco, PD-341265). It weighs about 1 g.

## **Description of apparatus**

The equipment consists of a large reactor interfaced to high voltage power for glow discharge, a Variac to heat a resistor to heat the palladium wire, two neutron detectors, a gamma detector, and a quadrupole mass analyzer. The data is collected with a logger and output in the form of comma separated text (.CSV) which is easily converted into spreadsheets for analysis.



#### Figure 1. The experimental system.

Figure 2 is a photograph from Ref. [1] showing the electrodes and thermocouple that are placed inside the reactor. From left to right: the counter-electrode, the palladium wire wrapped around a white ceramic block, and an RTD to the right in front of the ceramic block. The ceramic block has a resistance heater and another thermocouple inside it. To trigger the reaction, the ceramic block is heated by a Variac.



Figure 2. The electrodes and thermocouple placed in the reactor.

The calorimetric system consists of the reactor which is wrapped in polyethylene plastic tubing with cooling water flowing through it. The cooling water is pumped from a Dewar reservoir. The reactor and plastic tube are covered with insulation. The tubing is wrapped around the entire reactor body including the arms, so that it covers most of the outer surface of the reactor. A pump circulates the cooling water rapidly (8 L/min) through the loop and through the Dewar vessel.



Figure 3. Calorimetric system.



Figure 4. Photo of equipment. The reactor wrapped in white urethane foam pad insulation is on the left, the neutron and gamma detectors are in front, and the Dewar is on the right, sitting in the white tray. The polyethylene plastic tube from the Dewar to the reactor is covered with black insulation.



Figure 5. Reactor without insulation.

#### **Adiabatic calorimetry**

Adiabatic calorimetry is used to measure the heat produced by the reactor. This method is described by Hemminger and Hohne as follows: "under ideal circumstances, no heat exchange whatever occurs between the measuring system and the surroundings. The measuring system is separated from the surroundings by an 'infinitely large' thermal resistance, i.e. thermally insulated in the best possible way." (Ref. [2], p. 84) In practice, this means you insulate with an envelope around the reactor to capture as much of the heat as you can inside the envelope. You measure the heat based on the temperature change of the material in the envelope multiplied by the specific heat of that material. You can also calibrate by inputting a measured amount of heat with a resistance heater.

In this experiment, the material in the envelope consists of 4 kg of water (specific heat 4.18 J/g °C) and the 50.5 kg stainless steel reactor body itself (SUS 316, specific heat 0.49 ~ 0.53 J/g °C).

Adiabatic calorimetry has only two parameters: the input electric power and the temperature change. In this experiment the electric power is AC to a single resistance heater. It is measured by two stand-alone meters, which agree. The temperature change is measured with four thermocouples, which agree to within 0.1°C.

As shown in Fig. 3 and Fig. 4 this system is actually made of two envelopes which function as one, because they are connected with cooling water flowing through insulated tube.

We did not try to rigorously account for heat losses through the insulation. Some heat must escape because there is no such thing as perfect insulation, <sup>2</sup> and the ambient temperature is lower than the cooling water temperature. There are various other unaccounted for losses from the system, which if we were to measure carefully would increase the excess heat. We chose not to include these losses in this preliminary study because based on previous calibrations they are probably marginal and they would obscure the issue. The anomalous heat produces a large change in the water temperature that can be measured with confidence. At this stage, we see no need to search for other factors to establish a more precise heat balance.

As far as we know, there are no unaccounted-for gains in the system. All assumptions are conservative.

Mizuno has performed extensive calibrations. For example, the resistance heater was run without palladium or nickel wires in the reactor. These calibrations were performed with the previous configurations of the reactor and will be repeated with the latest, heavily insulated version.

#### Method

As noted above, once the nanoparticles are formed by glow discharge, the anomalous heat can be triggered any number of times by heating the wire. Glow discharge is not needed again unless the wire is contaminated and must be cleaned. Each time the wire is heated the anomalous heat turns on within minutes. I observed the second phase of the test only, and not the glow discharge.

Until September 2014, Mizuno generally performed this experiment by turning on the resistance heater to heat up the wire, and then leaving the heater on at 20 to 40 W during the entire experimental run. This probably produced a great deal of excess heat. However, it made the reactor hot to the touch which caused large heat losses to the environment even with the heavy insulation. Also, the input electric power complicated the calorimetry. To avoid these problems Mizuno recently began using another technique. He inputs a pulse of heat at ~20 W lasting 500 s (~10,000 J in 8 minutes, 20 seconds). This raises the reactor internal temperature above 300°C and triggers the anomalous heat reaction. After the pulse, when electric power is turned off, the cooling water temperature rises as the heat transfers from the inside of the reactor through the walls into the cooling water. The temperature continues to rise at a modest pace for 2

<sup>&</sup>lt;sup>2</sup> Nearly perfect pseudo-insulation can be made with a thermostatically controlled heated barrier that stays at the furnace outer wall temperature. This "quasiadiabatic" method is complicated. [2] In Fig. 12 we achieved this for a few hours inadvertently.

hours. One or two additional 10,000 J pulses are added, and the temperature rises throughout the day. At the end of the day, the total temperature change of the cooling water is measured.



Figure 5 shows an example of this method. In the first 4.4 hours of this test, three pulses of ~10,000 J each were input. Exact input energy computed from the spreadsheet was 30,794 J.

Figure 6. A test on Oct. 21. Left axis: water and reactor wall temperatures. Right axis: input power.

The temperature of the cooling water rose from 23.12°C to a peak of 25.67°C 6.2 hours later. That is an increase of 2.54°C. Heat output captured by the cooling water is therefore:

 $2.54^{\circ}C \times 4.18$  specific heat of water  $\times 4,000$  g = 42,469 J

This far exceeds the input energy of 30,794 J.

Not all of the heat was captured by the water. That is not possible. Some of the heat radiated from the reactor body (see the discussion at Fig. 14), while a great deal more was captured by the reactor vessel itself. We know this because the second law of thermodynamics ensures that the reactor vessel surface had to be warmer than the water while the temperature was rising, or there would be no heat transfer to the water. This is confirmed by the fact that the two thermocouples attached to the reactor surface showed overall temperature increases slightly higher than the cooling water. At the 6.2 hour peak the right reactor wall Delta T is 2.63°C, the left reactor wall 2.57°C, and the water is 2.50°C.

The reactor weighs 50.5 kg. The specific heat of metal is roughly 10 times less than water, but the mass of this reactor is more than 10 times greater than the water. If we assume the entire reactor vessel temperature rose by  $\sim 2.54^{\circ}$ C, this indicates an additional 62,852 J:

 $2.54^{\circ}C \times 0.49$  specific heat of SUS  $316 \times 50,500$  g = 62,852 J

In this case total output would be 105,321 J.

The total heat captured by the reactor metal may not have been quite this high because it is unclear whether the temperature of entire mass of the reactor vessel rose as much as the cooling water did. An exposed portion of the metal was no warmer than ambient temperature. The steel vessel temperature distribution may have been uneven, and some parts may have been warmer than others. On the other hand, the cooling water tube was wrapped around a large fraction of the surface, so most of the surface was warmer than the water.

The computer data from the thermocouples attached to the reactor surface show persistent 0.3°C lower temperatures than the cooling water in equilibrium. This must be an instrument bias, because the three thermocouples track one another closely, and overall they rise in temperature a little more than the water. Figure 7 shows the change in water and the two wall temperatures. That is to say, it shows all three thermocouple values normalized to their own starting temperatures.

With this method of adiabatic calorimetry, all output heat is computed based on a single temperature measurement, taken at the peak, shown here with the purple arrow. In this case, the temperature change is 2.5°C. This is large and easy to measure with any thermometer. With other methods of calorimetry, numerous individual temperature measurements must be taken and they are usually much smaller than this. For example, if this were flow calorimetry at 60 mL per minute, this test would produce an average temperature difference of 0.12°C between the inlet and outlet cooling water.



Figure 7. Oct 21 test showing change in temperatures of the water and reactor wall right and left side thermocouples. Output heat is based on one measurement only: the total 2.5°C change in temperature at the peak, shown with the purple arrow.

The bias in Fig. 6 shows that the thermocouples are only accurate to within 0.3°C. The fact that they all show the same temperature rise (when compared to themselves) shows that they are precise to within 0.1°C. Precision is more important than accuracy with adiabatic calorimetry because you only need to compute a delta change in temperature. The thermocouples rose the same amount in all five tests, whenever they were in equilibrium. In four of these tests a handheld electronic thermometer also showed the same temperature rise, to within 0.1°C.

Let us consider what would have happened if there had been no anomalous heat in the Oct 21 test. Assume that all 30,794 J of input electrical heat transferred directly into the 4 L of cooling water. The temperature would have risen by 1.8°C, instead of 2.5°C. With all four of these instruments it is certain we would have detected this difference. In fact, this scenario is manifestly impossible. First, because heat cannot leap from the resistance heater to the water without heating up the reactor vessel. As noted above, the second law ensures that large fraction of the heat has to be captured by the reactor metal itself, otherwise the reactor could not have heated up the cooling water. So the temperature rise would be much lower than 1.8°C. Second, the reactor is continuously losing heat to the surroundings. Figure 13 shows that during 6 hours overnight it cools ~0.9°C. Probably, without anomalous heat, in this test there would be no measureable temperature rise at all. Upcoming calibration tests with a resistance heater should confirm this.

The right and left reactor wall sensors are K-type thermocouples installed on opposite sides of the vessel, at locations 1/3<sup>rd</sup> and 2/3<sup>rd</sup> down from the lid. The right one, 1/3<sup>rd</sup> down, is closer to

the section of the reactor where the wire and resistance heater are mounted on the inside, so it heats up more during the pulse. This shows that the temperature of the reactor wall is not uniform during the pulse, and that it takes a while for the wall temperature to return to equilibrium.

The temperature inside the reactor rises above 300°C during the pulse, so it is not surprising the wall temperature is not uniform for a while. Figure 8 shows the reactor internal temperature (on the right axis), and the two wall temperatures and the water temperature from the first pulse to hour 1.4 when the wall and water temperatures returned to equilibrium. There is a lag between the internal temperature and the wall temperature because the vessel wall is 1 cm thick, and there is another lag in the transfer to the cooling water because polyethylene tube does not conduct heat well.



Figure 8. Oct. 21 test, first pulse. Left axis: the reactor wall right and left, and the cooling water temperatures. Right axis: reactor internal temperature. There is apparently a ~0.3°C instrument bias between the reactor wall and the water temperatures in equilibrium.

The water and wall temperatures continue to drift up long after they have reached equilibrium from the pulse. This indicates there is a source of heat in the reactor. There is no input electricity and no chemical fuel in the reactor, so this is anomalous heat. Figure 9 shows a close-up view. This figure shows the temperature change; not the absolute temperature. The temperatures are rising even though the ambient temperature is falling, and it is lower than the water temperature. This figure also explains the perturbations in the water and wall temperature seen in Fig. 7 from hour 1.4 to hour 2.0. These are caused by changes in the ambient temperature, from the room heater cycling on and off. Heat losses from the reactor increase slightly when the ambient temperature falls.



Figure 9. Left axis: change in water and wall temperatures. Right axis: ambient temperature. (Note that the ambient temperature is actually lower than the water and wall temperatures.)

The temperature changes in the reactor wall and in the center of the reactor shown in Figs. 7 and 8 are interesting observations, but please note that they have no effect on the adiabatic method of calorimetry, because we only use the temperature at the end of the test when all three thermocouples (plus the handheld electronic thermometer) come into equilibrium, as shown by the purple arrow in Fig. 7.

To conclude, there is no question that a large fraction of the reactor vessel itself heated up to a temperature greater than the cooling water. The reactor vessel temperature may not be uniform, but the cooling water temperature is. We know this because it is stirred at 8 L per minute, and because two separate, stand-alone thermocouples in the Dewar registered the same temperatures (as described below). So, we can be sure the water captured 42,469 J.

Table 1 summarizes five recent tests. In the SUS316 J column, we assume that the entire reactor came to the same temperature as the cooling water. The temperature change is measured with high confidence using these instruments. This is not a marginal effect. This form of calorimetry may be somewhat crude and imprecise, but assuming we have made no mistakes in the method, the result is irrefutable.

These results are in reasonable agreement with previous results derived with an uninsulated reactor and complicated calorimetry.

Date	Note	Pulses	Input J	Hours to peak	Peak Temp Delta T	Water J	SUS316 J	Total J	Water out/in	Comb- ined out/in
Oct. 16	А	3	31,205	7.5	3.72	62,198	92,051	154,250	1.99	4.94
Oct. 20		1	10,208	2.8	1.40	23,408	34,643	58,051	2.29	5.69
Oct. 21		3	30,794	6.2	2.54	42,469	62,852	105,321	1.38	3.42
Oct. 22		2	20,819	6.7	1.60	26,752	39,592	66,344	1.28	3.19
Oct. 24	В	5	52,110	7.0	2.21	36,868	54,686	91,554	0.71	1.76

Table 1. Summary of results from five tests

Notes:

A. The temperature was still trending up when data collection halted.

B. Calibration in vacuum.

I observed the tests from October 20 to October 24, but not the October 16 test. I have examined data from many other tests.

This analysis assumes anomalous heat ended abruptly at the peak. It may have continued, in which case the peak only indicates when heat losses overcame anomalous heat, and the temperature started to drop. You can compute total output up to any point in the experiment in which the three temperature sensors are in equilibrium.

Note that the ratio of output divided by input is useful for a comparison only. It is no indication of the potential technological usefulness of the reaction, or a future ability to generate electricity (which calls for a high ratio). This number has no technological significance because it can be adjusted up or down to any ratio simply by changing the insulation around the reactor. It takes a pulse of ~10,000 J to heat the wire enough to trigger the heat. With better insulation and a smaller reactor chamber, the wire could be heated with less energy, perhaps 500 J, or 100 J. After the reaction is triggered, input power is zero. In cold fusion jargon, this is "heat after death" and the actual ratio of output to input (the coefficient of production or "COP") is infinity.

The challenge to developing this into a practical source of energy is to control the reaction and to reliably scale it up. Improving the output to input ratio is a trivial problem. In my opinion this has been the case with all forms of cold fusion reported until now.

Table 2 shows the anomalous energy converted to average power. The duration is the number of seconds it took to reach the peak temperature. For October 21, anomalous joules for water is output energy measured by water minus the input energy 42,469 J - 30,794 J = 11,675 J. Anomalous joules combined assumes the water and steel together measured 105,321 J.

Based on the previous results and calibrations, and the overall performance of the calorimeter system, it seems likely to me there was at least 3.34 W of heat, and probably more when losses

are accounted for. There is roughly 1 g of palladium in the reactor, so this is excellent power density. It is in the same range as the power density of a uranium fuel rod, which is  $\sim 26$  W/g.

		Duration	Anomal	ous Joules	Watts		
Date	Pulses	in seconds	Water	Combined	Water	Combined	
Oct. 16	3	27,000	30,993	123,045	1.15	4.56	
Oct. 20	1	10,080	13,200	47,843	1.31	4.75	
Oct. 21	3	22,320	11,675	74,527	0.52	3.34	
Oct. 22	2	24,120	5,933	45,525	0.25	1.89	
Oct. 24	5	25,200	-15,242	39,444	-0.60	1.57	

Table 2. Anomalous energy converted to power

(The estimate of uranium fuel power density is based on this statement from Argonne National Laboratory: "A 1100 MWe pressurized water reactor may contain 193 fuel assemblies composed of over 50,000 fuel rods and some 18 million fuel pellets." [3] A 1100 MWe reactor produces ~3300 MW of heat. Dividing that by 18 million pellets gives 183 W/pellet. A pellet weighs 7 g, so that is ~26 W/g.)

## A calibration in vacuum

We decided to do a control test during my visit. The best method would be to run the reactor with a resistance heater only, and no palladium or deuterium gas. Unfortunately we did not have time for this. In lieu of this, we left the wire in place and tested the reactor in a vacuum.

This is not a satisfactory control test, because previous tests in vacuum have shown considerable anomalous heat, presumably because there is gas left in the palladium after evacuating the reactor.

In previous control tests in vacuum, the reactor was only evacuated for a few hours with the rough pump. In this case on the evening of October 23, the rough pump (ULVAC, G-100D) was left running overnight. On the morning of October 24, the turbo molecular pump (ULVAC, YTP-50M) was run for a few hours, creating a hard vacuum. The turbo molecular pump was left running during the test.

Five 10,000 J pulses were added, as shown in Fig. 10. Input is 52,110 J (from the spreadsheet). By the end of the day, the water temperature increased 2.21°C, indicating 36,951 J output captured by the water.





When you include heat from the metal, the total rises to 91,554 J, considerably above input. This may indicate there is some anomalous heat, or it may indicate that the reactor vessel metal temperature is not uniform, per the previous discussion. I think it is more likely that there is anomalous heat, as there was in previous tests in vacuum.

Whatever the absolute level of power may be, a comparison shows that for 10,000 J of input, the temperature rose 0.82°C on October 21 but only 0.42°C in this test. Furthermore, the five pulses were spread out over the day and the last one was closer to the final hour then on October 21, so if anything the temperature should have been higher at the end.

To better establish the heat balance and recovery rate for this calorimeter, more calibrations will be performed with a resistance heater only and no palladium metal or deuterium gas. This will be done the next time the palladium wire is changed out. Tests with the palladium wire in place will also be performed before glow discharge nanoparticle production. Mizuno has performed such tests with the previous configurations of the reactor.

#### Ambient temperature fluctuations

The worst problem with the calorimetry at present is probably the ambient temperature, which is poorly controlled and changes as much is 2.5°C per day. The reactor is well insulated, and the calibrations show that these changes in ambient temperature take a long time to affect the reactor and the cooling water temperatures. The ambient temperature seldom exceeds the reactor or water temperature, except for brief periods early in the morning when the experiment starts. So ambient temperature could not cause the cooling water temperature to rise, but it does prevent

it from falling, reducing losses. Since we do not attempt to account for these losses and we are treating this as if it were a perfectly insulated adiabatic calorimeter, the rise in ambient temperature cannot cause spurious excess heat as long as it stays below the cooling water temperature. Still, ambient temperature fluctuations remain a concern. New heating and air conditioning equipment will soon be installed in the lab which should reduce this problem.

In November 2014, Mizuno placed an aluminum foil tent over the reactor and Dewar, and shrink-wrap plastic insulation to the laboratory windows. This reduced ambient temperature fluctuations, allowing an estimate of the heat added to the system by the circulation pump. This estimate is described below.



Figure 11. Aluminum foil tent placed over the reactor to reduce ambient fluctuations.

Here is another look at the October 24 data showing ambient temperature was close to the water temperature on October 24. The two are roughly equal after the third pulse, although actually ambient may be a little cooler than shown here, as explained below in the discussion of Figs. 17 and 18:



Figure 12. Left axis: water and ambient temperature. Right axis: input power.

The ambient temperature was reasonably stable during the October 21 test, and it stayed well below the water temperature most of the time, so ambient temperature fluctuations probably had little or no effect on the results:



Figure 13. Water temperature and ambient temperature fluctuations on October 21.

As shown in Fig. 14, the ambient temperature was much closer to the water temperature on October 22. It fell drastically overnight, because this is in Sapporo, Japan, which is cold at this time of year, and like most office space in Japan, the laboratory is unheated at night. You can see that the space heater was turned on again the next morning, 18 hours into the test. The spreadsheet data for this figure shows that from hour 8 to hour 14, the water temperature fell 0.9°C. Presumably, any anomalous heat has faded out by hour 8, so this is how much the reactor and Dewar cool with no input power. This gives some indication of the heat loss from the reactor during the six hour tests on October 16, October 21 and October 22. The thermal mass of the reactor plus cooling water is equivalent to 9.9 kg of water, so the system lost ~37,300 J in 6 hours, at a rate of approximately 1.7 W.

During the 6-hour test in daytime, the reactor may not have cooled quite as much as this. More heat is lost overnight because the ambient temperature is lower and heat loss is a function of the difference between the warm body and the surroundings (Newton's law of cooling).



Figure 14. Ambient temperature was close to the water temperature on October 22, and it fell overnight.

Here is an interesting look at heat losses from the reactor and Dewar. At around 7.5 hours into the October 21 test, at night, the circulation pump failed. The reactor is not as well insulated as the Dewar, so its temperature fell faster.



Figure 15. The effect of the circulation pump failing; the reactor cools faster than the Dewar.

### This is adiabatic, not flow or isoperibolic calorimetry

Cold fusion experiments often employ either flow or isoperibolic calorimetry. This adiabatic setup resembles both of these other calorimeter types, which may confuse people familiar with other experiments.

This is not flow calorimetry, even though water flows through the cooling tubes and Dewar reservoir. If this were flow calorimetry we would measure the inlet and outlet temperatures of the cooling water, and we would maintain the reservoir at a constant temperature. We would also use a flow rate much lower than 8 L/min. With this system the cooling water flow rate does not need to be recorded and it makes no difference except insofar as the cooling water has to be circulated rapidly through the system and across the entire face of the steel reactor vessel to ensure a uniform surface temperature in the reactor, to capture a large fraction of the heat, and to keep the cooling water well stirred.

Flow calorimeters run at the power levels observed in this experiment will produce a small temperature difference between the inlet and outlet cooling water. A slight change in the temperature measurement or flow rate, or a problem mixing the cooling water in the pipe, will cause a major error. With the adiabatic method even at these relatively low power levels a large, easily measured temperature difference is produced.

Isoperibolic calorimetry does not call for a complete envelope of insulation:

For accurate measurements it is not absolutely necessary to keep heat losses as low as possible. What is more important is that these heat losses be repeatably dependent on the temperature difference between the measuring system and surroundings, in this case they can be determined exactly by (electric) calibration. Of course, large heat losses reduce the sensitivity of the calorimeter to a considerable extent. (Ref [2], p. 83)

With isoperibolic calorimetry you design the envelope to make heat losses predictable, repeatable and consistent across a wide range of power. The cell reaches a fixed temperature in response to a given power level. With adiabatic calorimetry you try to reduce heat losses as much as possible, and keep as much of the heat in the envelope for as long as possible. The temperature continues to rise for as long as the power continues, until the calorimeter overheats and fails.

The adiabatic method works well for this experiment, but it would not have worked with previous cold fusion experiments. This method can only collect a limited amount of heat. It works well in this experiment because both input power and excess heat can be controlled and limited pre-planned amounts, and because the experiment can be turned on and off every day. The heat in this experiment turns on quickly. Previous cold fusion experiments with other materials had to be run continuously for weeks with input electrolysis power to maintain high loading. The heat appeared sporadically and could not be turned on or off on demand. An adiabatic calorimeter run with previous cold fusion experiments would quickly "fill up" with heat. The cooling water might get so hot it boils. Because insulation is never perfect, the reactor would soon lose heat and thus lose track of output energy. It would end up being a poorly-designed isoperibolic calorimeter.

Hemminger and Hohne write that the adiabatic method works best when "the sample reaction takes place so rapidly that no appreciable quantity of heat can leave or enter during the measuring interval." [2] This experiment lasts for hours, so it does not meet that qualification as well as a reaction such as combustion in a bomb calorimeter would.

This method also works well because we know approximately how much heat the reaction will produce, so we can select an appropriate mass of water and metal to ensure that the temperature rises only moderately, 3 to 5°C. This temperature change is high enough to be measured with confidence using conventional instruments, but not so high that it causes large heat losses through the insulation, or evaporation. The insulation leaks enough heat that overnight the reactor cools back down to room temperature and the system is ready for another test the next day.

This adiabatic calorimeter is well-suited for measuring the overall energy release over several hours, but it does not work well for measuring instantaneous power from second to second, or from minute to minute.

While the reactor cannot be run continuously overnight for months with this technique, the palladium and deuterium gas are left in the cell, sealed and intact. The palladium turns on every morning and produces heat every working day. Running a closed cell every day is functionally equivalent to running the reactor continuously. The total hours of heat production per gram of material, and the amount of reaction products captured in the reactor, can both be increased to an extent not yet known. Mizuno has not encountered the limits of production yet.

The pump and cooling water loop are needed because the cooling water equalizes the temperature and captures the heat reliably in one well-mixed body of fluid, whereas the reactor body by itself when not cooled by water tends to have hotspots and uneven heating at the outside surface.

Some of the people who have observed this experiment expressed concerns about the temperature rise in the center of the reactor, and the lag in the temperature of the reactor walls and the cooling water. They worried about the fact that different types of gas and gas pressure in the reactor will cause the reactor temperature to rise to different temperatures with the same input energy. These cause problem with isoperibolic calorimetry, but not with adiabatic calorimetry.

#### Notes on the instruments

The instruments interfaced to the data logger are all expensive and professional grade:

The thermocouples are pre-calibrated and cost \$200 to \$300 each. (Only the high temperature RTD needs adjusting.)

Power is monitored with a Yokogawa PZ4000 Power Analyzer which cost \$16,000. Since this is simple input AC power from a Variac to a resistance heater, it is not difficult to measure and it does not really call for this kind of high precision instrument. [4]

The circulation pump is an Iwaki Co., Magnet Pump MD-6K-N. The motor runs cool and it is well separated from the pump mechanism, so it adds only 0.2 W to the water. This raises the baseline temperature before the test begins, so it has no effect on the adiabatic calorimetry, which is based on the change in temperature, not on a comparison to ambient temperature. This is discussed below. The flow rate is 8 L per minute but this is not important because this is not flow calorimetry. Multiple thermocouples have been inserted into the Dewar reservoir to confirm that the water is well stirred.

The Dewar cooling water reservoir is from Ichikawa Vacuum, 3000 mL capacity. There is a total of 4 L of water in the system, including 1 L in the polyethylene plastic tube. The tube has an inner diameter of 10 mm and as purchased it was 16 m in length, volume 1.26 L. Mizuno ensured that he used one-liter of tube by first wrapping the tube around the reactor, and then holding up the ends up and pouring in 1 L of water. He marked where the water level was. Then

he drained the tube and cut the ends at the marks (leaving enough tube to fit into the Dewar). This method ensures that the tube holds 1 L even if it is somewhat compressed or distorted by being wrapped around the reactor. 100 g of NaHCO<sub>3</sub> is added to the cooling water as fungicide.

An MKD pressure gauge measures the reactor gas pressure.

A ULVAC quadrupole mass spectrometer (QMS), model YTP-50M is on line. It is used to ensure the gas and the metal samples are clean before the run. It is sometimes used to take samples of the gas during the run, but not every day. The QMS accepts such minute samples of gas that continuous sampling can be done all day long without measurably reducing the reactor gas pressure.

The Aloka 161 gamma ray detector and the Fuji and Aloka 451 neutron detectors are high-grade survey meter class instruments rather than the kind used in physics research such as time-of-flight detectors.

## Redundant, standalone instruments for a "reality check"

All parameters are measured with two or more instruments. The critical parameters for adiabatic calorimetry – input power and the cooling water temperature – are measured with two independent, stand-alone instruments. Mizuno installed these at my request.

As noted, input power is simple AC, and it is measured with a \$16,000 PZ4000 capable of measuring far more complex waveforms. This precision instrument is confirmed by plugging the Variac into a \$50 watt meter, Sanwa Supply WattChecker. On the face of it, it may seem absurd to cross-check the performance of a \$16,000 instrument with an off-the-shelf digital meter, but this kind of "reality check" gives me confidence in the measurements.

Here are some results from the WattChecker. When the Variac is turned on but it is not powering anything, the WattChecker shows that it consumes 12.2 W. The Variac is warm to the touch. I confirmed that none of this power reaches the system by disconnecting the Variac from the reactor heater. During a nominal 20 W pulse the WattChecker shows that output increases to 33.6 W, an increase of 21.2 W. This is "nominal" because the power fluctuates during the 500-second pulse and to read the WattChecker you have to stop watching the PZ4000 and scramble behind the equipment cabinet. I conclude that the PZ4000 is correct, and that a Variac wastes a lot of power.



Figure 16. A Sanwa WattChecker watt meter behind the equipment cabinet. The Variac is plugged into it. This \$50 instrument confirms the performance of a \$16,000 instrument.

The temperature of the cooling water and ambient air are monitored with a handheld Omega HH12B Digital Thermometer. The Omega measures to the nearest 0.1°C. It has two K-type thermocouples. One was placed in ambient air close to the Dewar vessel, and the other was placed in the Dewar vessel to measure the cooling water temperature. It tracked Mizuno's computer cooling water thermocouple closely. It was initially 0.1°C higher than Mizuno's thermocouple. The setscrews for both thermocouples were adjusted slightly down by 0.1°C. Here are sample readings from October 24:

Omega Thermometer	Computer Thermocouple
22.9	22.98
23.1	23.09
23.1	23.16
23.2	23.20
24.2	24.17
24.7	24.67
25.2	25.25

Table 3.	Water temperature shown by Omega	Thermometer	and the Computer
	Thermocouples		

The thermometer has a T1-T2 compare mode which showed the difference between the cooling water and ambient temperatures. Based on this, I believe the temperature recorded by Mizuno's ambient thermocouple may be a little higher than the rest of the room, perhaps because it is taped to the MKD pressure gauge sensor, which was in a confined space behind the test rig. (The pressure gauge is temperature sensitive so it must be monitored.) The Omega thermometer

was usually 0.1 to 0.3°C lower than the computer ambient thermocouple. I believe the Omega was more accurate for the entire room.



Figure 17. Computer ambient thermocouple taped to pressure gauge.





With adiabatic calorimetry there are only two parameters: input power and the cooling water temperature. Both have been confirmed with two or more instruments, which show temperature differences far above the minimum threshold of the instruments. The simplest instrument used, the Omega HH12B, is capable of reliably measuring a persistent temperature change of ~0.2°C. In this study, it measured up to a 2.5°C difference, lasting for hours. The other thermocouples,

when measured against themselves can reliably record a persistent change of ~ $0.1^{\circ}$ C. They recorded differences up to  $3.7^{\circ}$ C (on October 16).

With other methods of calorimetry there are additional critical parameters. For example with flow calorimetry you must monitor both the inlet and outlet temperatures and the flow rate; and with isoperibolic calorimetry the temperature outside the envelope will have a larger effect. Much smaller, more sensitive temperature measurements are needed for these other methods.

#### Heat from the circulation pump is in the baseline

After the initial publication of this report, some readers raised concerns that the circulation pump may be adding heat to the water which might be mistaken for anomalous heat. This is not possible, because the heat from the pump is included in the baseline temperature, and anomalous heat is measured as the increase from the baseline to the temperature at the end of the test.

The pump is:

Iwaki Co., Magnet Pump MD-6K-N Maximum capacity: 8/9 L/min Maximum head: 1.0/1.4 100V 12W/60Hz, 12W/50Hz

As noted above, the pump runs cool. Mizuno used the WattChecker watt meter to measure the electric power consumed by the pump, which is 10.8 W. He measured the heat delivered from the pump to the water by running the system for 18.2 hours, starting with pump turned off and the reactor and Dewar cooling water at room temperature. He then turned on the pump only, for the remainder of the 18-hour test. The temperature rose for 1.5 hours until it stabilized  $0.6^{\circ}$ C above room temperature (Fig. 19.) It stabilized because heat losses equal the power from the pump. In other words, with low input power after 1.5 hours, this system acts as an isoperibolic calorimeter. Based on the cooling curve after the ambient temperature fell during the night, this 0. 6°C difference indicates that the pump delivers only ~0.4 W of heat to the circulating water.

More to the point, with this method of adiabatic calorimetry the  $0.6^{\circ}$ C temperature increase over ambient is not included in the calculation of excess heat, because the pump is left on all the time, and it always does the same amount of work, so the temperature is always  $0.6^{\circ}$ C above ambient. To be specific, with this method, the starting water temperature is subtracted from the ending water temperature, and the starting temperature is already  $0.6^{\circ}$ C warmer than ambient.

With other methods of calorimetry, heat is measured by comparing the reactor temperature to ambient. With these methods, heat from the pump has to be subtracted from the total, or it will be mistaken for excess heat.



Figure 19. A test with only the pump running. After 1.5 hours the baseline temperature is stable at 0.6°C above ambient.

# **Pending improvements**

Mizuno plans to make improvements, do continued testing and to submit a paper to the ICCF19 conference. Pending improvements include:

- Increased mass of palladium reactant, the next time the palladium is removed to be analyzed.
- Improved heating and air conditioning equipment, to reduce the noise from ambient temperature fluctuations.
- More calibrations with the present configuration. Several methods of calibration were performed with the past configuration (with less insulation). These calibrations will be repeated.

# The data spreadsheets for this report are available for download

The data logger saves data in the personal computer in comma separated text (.CSV). I converted this to Excel spreadsheets for the analysis and graphs in this report. I have uploaded the temperature and power portions of these spreadsheets to LENR-CANR.org. I invite the reader to download them and have a closer look. The spreadsheets are here:

```
http://LENR-CANR.org/Mizuno/Mizuno2014-10-16.xlsx
http://LENR-CANR.org/Mizuno/Mizuno2014-10-20.xlsx
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http://LENR-CANR.org/Mizuno/Mizuno2014-10-21.xlsx http://LENR-CANR.org/Mizuno/Mizuno2014-10-22.xlsx http://LENR-CANR.org/Mizuno/Mizuno2014-10-24.xlsx http://LENR-CANR.org/Mizuno/Mizuno2014-11-20.xlsx

Here are some notes on the spreadsheets.

Input power, in Watts

<u>Column</u> A	<u>Content</u> Seconds; the number of seconds since the start of data collection. Data is collected every 24.47 s. This fits the data collected from the UVLAC QMS, which apparently only works with that sampling time.
	Columns B through G are for the thermocouples
В	T1 Ambient (at the location of the MKS pressure gauge)
С	T2 Reactor RTD, shown in Fig. 2
D	T3 Reactor thermocouple embedded in the white ceramic block shown in Fig. 2. This is around 30°C too low. It needs to be recalibrated. This is used for high temperatures ranging from 100 to 1000°C, so the 30°C error is not critical.
E	T4 Cooling water
F	T5 Reactor wall right side (right side in the photo), mounted about 1/3 <sup>rd</sup> down from the lid
G	T6 Reactor wall left side, mounted about 2/3 <sup>rd</sup> down from the lid
Н	Reactor gas pressure, in Pascals

I added the columns for Hours and Joules, and various other columns to some of the spreadsheets.

Several columns of the original data were not used in this study, and are not included in these spreadsheets. They include data from the glow discharge power supplies and the particle detectors.

# Conclusion

Ι

NOTE: As described below, these I now believe these conclusions were mistaken.

I conclude that the previously published results are probably correct, but the instruments were too crude, and the methods too complicated to be sure of that.

The present calorimetry is imprecise but highly accurate and reliable. The anomalous heat can be measured using ordinary instruments with high confidence. Input power is limited to 1 to 3 pulses per day, each 20 W lasting 500 s, which is low power and a short time, so it does not interfere with the calorimetry much.

The anomalous heat produces a large temperature increase in the cooling water. The results are large enough that they can be confirmed with confidence even using a handheld \$50 power meter and a \$95 digital thermometer, and with still greater confidence using professional grade instruments backed by the handheld instruments.

There is no likelihood of an instrument error. Only an error in the methodology can disprove these results. Absent the discovery of such an error, I am confident that this system is producing massive amounts of anomalous heat for very long periods, such as 8 hours a day for months from a 1-gram palladium sample in a single charge of deuterium gas, in a closed system. The net energy far exceeds the limits of chemistry.

### **Appendix A**

As discussed in the November 28, 2014 version of this paper, two additional steps were needed to confirm these results. Fluctuations in ambient temperature had to be reduced, and blank calibrations were needed. After November 2014 the ambient temperature fluctuations were reduced but not eliminated. A series of calibrations were performed. Results from these calibrations call into question the original conclusions of this paper.

## **Calibrations**

Three types of calibrations were performed:

- 1. With only the circulation pump running.
- 2. With a resistance heater installed in the cell which was pulsed similar to the way it was pulsed during the October tests.
- 3. With the resistance heater set at one power level for one or two days.

In the first set of calibrations, the reactor was left with only the circulation pump running for one or two days at a time. Although ambient temperature continue to fluctuate in these tests there were several times (usually in the dead of night) when ambient temperature was stable. During these times the temperature of the cooling water, the reactor wall and the reactor core all settled at one temperature within a fraction of a degree. This temperature was typically ~1.2°C above ambient, because of the heat from the pump.

In some cases the temperature settled at considerably more than  $1.2^{\circ}C$  above ambient, up to  $2^{\circ}C$ . It is not clear why, but this may be because the ambient temperature at different places in the room varies as currents of warm and cool air circulate. In other words the air surrounding the reactor vessel and water may have been at one temperature where the ambient temperature sensor was at another temperature. Tests with the handheld Omega thermocouple showed that ambient temperatures in the room varied from one location to the other by ~0.3^{\circ}C. That is not

enough to explain the variation in temperatures, but perhaps the ambient temperature variations were larger at night because there were no people or fans stirring the air in the laboratory.



Figure 20. Temperatures late at night when ambient was stable.

Figure 20 shows the temperatures of the cooling water, the reactor wall and reactor core during an exceptionally stable hour, late at night. The water temperature is 1.09°C warmer than ambient, standard deviation 0.10. This figure shows that one of key assumptions made in the November draft of this paper was incorrect. Mizuno and I assumed that the three thermocouples were not synchronized, so that they had to be normalized to zero at the beginning of the test. This result shows that in stable conditions, the three of them the same temperature to within a fraction of a degree, so they do not need to be normalized. That being the case, figure 8 above does not need to be adjusted and it shows the true temperatures. (That figure is copied here as figure 21 for convenience.)





This shows that in response to the pulse the reactor internal thermocouple rises in temperature and then falls back to the same temperature. The water and reactor wall temperatures rise, fall back and then continue to rise. However, if we assume these temperature differences are real, and the four thermocouples do not need to be normalized to zero, then it is apparent that after 0.8 hours the water temperature is warmer than the two wall temperatures. Therefore the heat must be going from the water to the wall, according to the second law of thermodynamics. The water must be the warmest body in the system, and the heat originates in the water.

The only source of heat in the water is the circulation pump, so it must be causing the temperature to rise. This seems to contradict the conclusion reached in the November version of this paper, namely, that the heat from the pump does not need to be accounted for because it is part of the baseline temperature of the system. That was partly correct. The heat from the pump is in the baseline, but the baseline itself has risen. That is to say, the ambient temperature sometimes rose enough that it kept the reactor and Dewar so warm that from the pump could not escape from the system and built up inside it, causing the temperature to rise. This was shown in much more detail in calibrations performed in the weeks following this initial conclusion.

The palladium reactant was later removed from the reactor and two additional sets of calibrations were performed.

In the first set, heat pulses were added to the reactor, similar to the ones added during the October tests. These tests appeared to support the October results. They recovered far less energy overall from the reactor than the October tests did: 37% and 42% in the water, compared to 195% to 237%.



Figure 22. A pulse calibration test that recovered less heat from the water than the October tests.

In the second set of tests, the resistance heater was set at a fixed power level and left for one or two days. After several hours the temperature stabilized at a given level. In other words, the reactor was tested in isoperibolic mode.

Five of these calibrations were performed at power levels of 1.89, 2.06 4.38, 4.43 and 10.42 W. Figure 23 shows the result of the 1.89 W test. Ambient rose and then fell during the first 9 hours, making the results difficult to interpret. However, as shown in Fig. 24, from hour 21.5 through hour 23.0 the ambient temperature was stable, at an average 16.73°C standard deviation 0.07°C. During this time, the water remained 3.12°C above ambient and the wall 3.80°C above ambient.



Figure 23. Input of 1.89 W from hour 7.1 to hour 23.8.



#### Figure 24. Input of 1.89 W late at night when ambient was stable.

Two aspects of this result indicate that the October results were caused by ambient fluctuations rather than anomalous heat within the reactor:

First, the reactor core and wall temperatures are consistently well above the water temperature, by  $\sim 0.7^{\circ}$ C. This indicates the heat originates inside the reactor, rather than in the water. In this case the heater in the reactor was set to 2 W, which is approximately the level of apparent anomalous heat in the October tests. If there had been anomalous heat in October, the reactor wall should have been warmer than the water by about this same amount.

Second, the results from these five calibration steps were graphed in Fig. 25, and the intercept was measured at positive  $\sim 2^{\circ}C$  above ambient. This positive intercept is caused by heat from the pump. The precise amount of power it represents is unclear, but what it does show is that when ambient temperature approaches the temperature of the water and reactor, heat from the pump cannot escape from the system, and the entire system temperature rises approximately  $2^{\circ}C$  above ambient. Therefore, when ambient temperature is uncontrolled and it rises approaching reactor temperature, the reactor temperature then climbs above ambient.





Looking closely at results from October 21 (Fig. 13) we see that what appears to be a gradual increase in the reactor temperature from anomalous heat is probably caused by changes in ambient temperature (Fig. 26). Even though ambient temperature does not reach the water temperature, it still "pushes up" on the temperature from below (as it were) probably because the heat from the pump cannot escape from the water.



Figure 26. Close up look at October 21 data (from Fig. 13). At hour 5.8 ambient temperature rises, "pushing up" the water temperature probably because heat from the pump cannot escape from the water.

# Possible remaining indications of excess heat.

Two things indicate that there may be excess heat in some cases:

- In one case (Figure 27) the ambient temperature rose to within 0.2°C of the water temperature and it remained within 0.6°C of it for 1.5 hours, yet the water temperature did not appear to rise in response. As noted above, ambient temperature at different places in the room varies as currents of warm and cool air circulate, and that effect may also cause the results shown in this figure.
- 2. During the pulse calibration tests much less heat was recovered by the water than in the October tests. This can probably be explained by the reduced ambient temperature fluctuations, which kept the ambient temperature from coming close to the reactor temperature.

These problems probably invalidate the October results, but not other results obtained by other methods of calorimetry.



Figure 27. After a calibration pulse, ambient rises to between 0.6°C and 0.2°C of the water temperature yet the water temperature does not appear to rise in response.

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