Technical report

CALORIMETRY OF THE IBP SYSTEM

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September 2, 2016

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Abstract

The IPB calorimeters at Brillouin Energy Corp (BEC) are power compensation calorimeters designed to measure heat flow from internally heated reactor cores. Exothermic reactions are expected to occur on the surface of the reactor cores when they are exposed to high current density pulses, temperatures of ~200 - 600°C, and pressurized hydrogen gas. This report details the IPB calorimetry methods; it is intended for anyone working with the IPB calorimetry systems or investigating the validity of experimental results obtained with an IPB calorimeter.

This report is separated into five sections: section 1 describes the derivation of equations used to calculate reaction power, section 2 describes the three pulse power measurement methods, section 3 lists the known potential sources of systematic error, section 4 describes the calorimetric control tests, and section 5 lists recommended improvements for minimizing measurement error.

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Nomenclature

$Q_{reaction}$	Reaction power
Q_{flow}	Main heat flow out of the calorimeter
Q_{loss}	Heat flow out of the calorimeter not measured in Q_{flow}
Q_{heater}	Heater power
Q_{pulse}	Pulse power
Q_{out}	Heat flow out of the calorimeter (Q_{flow} + Q_{loss})
T_r	Temperature of reactor core
T_j	Jacket fluid temperature
T_{ext}	Temperature of air outside the calorimeter
k_{flow}	Heat transfer coefficient of the main heat flow term
k_{loss}	Heat transfer coefficient of heat loss term
V	Voltage
I	Current
Z_{term}	Termination resistance
P	Electric power

Acronyms

BEC	Brillouin Energy Corp
IPB	Isoperibolic
COP	Coefficient of Performance

1 IPB calorimetry

Eq.(1) is the steady-state isothermal heat flow balance equation of the IPB calorimeter. The terms in the left bracket represent heat outflows and the terms in the right bracket represent heat inflows. $Q_{reaction}$ is the heat flow from reactions, Q_{flow} is the heat flow captured by the calorimeter's jacket, Q_{loss} is heat flow to the ambient air (not captured by the jacket), Q_{heater} is the heater power, and Q_{pulse} is the power dissipated into the reactor core from electric pulses.

$$Q_{reaction} = \left(Q_{flow} + Q_{loss}\right) - \left(Q_{heater} + Q_{pulse}\right) \quad (1)$$

Eq. (3) and Eq. (4) are not used in calorimetric measurements with the IPB system, but they are presented here to help the reader understand concepts that will be described later. The following is the heat flow balance equation of Q_{flow} where T_r is the core temperature, T_j is the jacket temperature, and k_{flow} is a calibration constant determined by using the core heater as a calibration heat source.

$$Q_{flow} = k_{flow}\left(T_r - T_j\right) \quad (2)$$

In reality, Eq.(2) is a fourth order equation with calibration constants determined by curve fitting, but the measurement concept is the same. Furthermore, Q_{loss} is determined with a different calibration constant k_{loss} and the temperature of the air outside the calorimeter T_{ext}.

$$Q_{loss} = k_{loss}(T_r - T_{ext}) \quad (3)$$

The geometry of heat input from the heater is not equivalent to the geometry of heat input from electric pulses at the surface of a reactor core (the heater is approximately half the length the surface of the core where electric pulses are applied). Therefore, when pulses are applied to a core, the constants k_{flow} and k_{loss} deviate from the values found with the heater as a calibration heat source. The consequence of this deviation on reaction power measurements is discussed later.

Less calibration is required and less sources of error are present if the IPB calorimeter is operated as a power compensation calorimeter. The heat balance equation for power compensation calorimetry is derived from the heat balance equation for heat flow calorimetry at two different conditions (e.g. 1 and 2):

$$\begin{aligned} Q_{reaction2} - Q_{reaction1} \\ &= \left(Q_{flow2} - Q_{flow1}\right) + (Q_{loss2} - \mathrm{Q_{loss1}}) \\ &- (\mathrm{Q_{heater2}} - Q_{heater1}) - \left(Q_{pulse2} - Q_{pulse1}\right) \end{aligned} \quad (4)$$

In order to reduce the number of variables, no pulses are applied during condition 1 and it is then assumed that no reactions are occurring ($Q_{pulse1} = Q_{reaction1} = 0\ W$). Condition 1 can be thought of as a calibration. To further simplify Eq. (4), the term Q_{pulse2} is replaced with Q_{pulse} and

$Q_{reaction2}$ with $Q_{reaction}$, and the terms Q_{flow} and Q_{loss} are grouped into a single term Q_{out} for total heat flow out of the calorimeter.

$$Q_{reaction} = (Q_{heater1} - \mathrm{Q}_{\mathrm{heater2}}) - Q_{pulse} + (\mathrm{Q}_{\mathrm{out2}} - Q_{out1}) \quad (5)$$

In an ideal power compensation calorimeter, the heat inputs from pulses and the heater would not cause variation in the heat flow out of the calorimeter (constant Q_{out}), simplifying Eq.(5) to the following:

$$Q_{reaction} = (Q_{heater1} - \mathrm{Q}_{\mathrm{heater2}}) - Q_{pulse} \quad (6)$$

However, the current configuration of the IPB calorimeter is not ideal because heat flow out of the calorimeter Q_{out} depends on pulse power Q_{pulse}. Therefore, Eq.(5) is used to find reaction power with the IPB calorimeter, and it can be generalized as follows:

$$Q_{reaction} = \Delta Q_{heater} - Q_{pulse} + \Delta Q_{out} \quad (7)$$

The variation in heat flow out of the calorimeter ΔQ_{out} also depends on the temperature of the reactor core T_r. The dependence of Q_{out} on T_r and Q_{pulse} is determined by calibration: pulses are replaced with DC current applied at the surface of the core (Q_{DC}) that is not expected to cause reactions ($Q_{reaction} = 0\ W$), and Eq.(7) is rearranged to find ΔQ_{out}. Referring to conditions 1 and 2 where DC power is applied at condition 2 but not condition 1, the following equation is used to find ΔQ_{out}:

$$\Delta Q_{out} = Q_{DC} - (Q_{heater1} - \mathrm{Q}_{\mathrm{heater2}}) \quad (8)$$

As a side note to this information, it's useful to determine which pulse parameters (e.g. pulse width, voltage, frequency) produce the same pulse power in order to minimize variability of Q_{out} when testing a set of pulse parameters.

At BEC, the results of calorimetric experiments are usually expressed as a coefficient of performance (COP). For research purposes, the COP of reactions is more useful than the COP of an entire heat producing system. When calculating the COP of reactions, the amount of energy required to catalyze reactions is conservatively assumed to be the total amount of pulse power dissipated in reactor cores.

$$COP = \frac{\Delta Q_{heater} + \Delta Q_{out}}{Q_{pulse}} \quad (9)$$

COP values greater than 1 means that there is a heat input to the core/gas part of the calorimeter. It should be noted that the commercial value of reaction power cannot be determined directly from Eq.(9) because it does not take into account the total power consumed by the heat producing system.

2 Pulse power measurement methods

As of this writing, two methods have been used to measure pulse power dissipated in cores, which are described in sections 2.1 and 2.2. The data required by both methods is voltage across the dielectric layer at each end of coaxial cores. The oscilloscope used for data acquisition is a Tektronix MDO3104 with 5 GHz sample rate (temporal resolution of 0.2 nanoseconds/sample). The Nyquist criterion is satisfied if all the frequency components of pulses are less than 2.5 GHz.

With both of these pulse power calculation methods, current is calculated by dividing the voltage drop across the termination resistor (i.e. the voltage across the dielectric at the termination end of the core) by the termination resistance. Figure 1 is a schematic of the coaxial core with voltage measurements at each end and the termination resistor represented by Z_L.

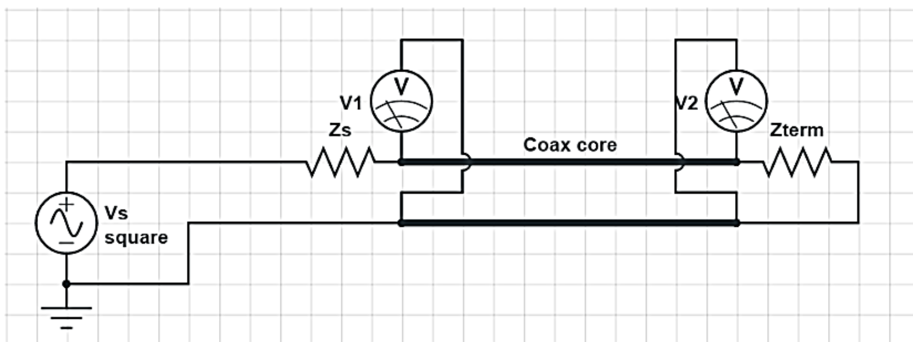

Figure 1 – Schematic of the pulse alignment and RMS measurement configurations

Unless the RMS voltage method is further developed, it can only be used to measure pulses produced by half-H boards, whereas the pulse alignment method can be used to measure pulses produced by both full-H and half-H boards. Also, multiple tests have shown that the pulse power values from both methods agree to within 5%.

Both of these measurement methods give apparent power, which is greater than true power and is therefore a conservative approximation. However, a third method is being developed that will give true power values. This new method is based on the measurement of real-time power at each end of the core by using dedicated current sensors; the details of the method are described in section 2.3.

2.1 Pulse alignment method

Since pulses are extremely short (on the order of 50 to 200 nanoseconds long), the time delay for the electric field to travel through the core is a significant fraction of the pulse length. With this pulse power measurement method, real-time voltage data from each end of the core is aligned and then subtracted by using a data processing script in DIAdem.

Pulses are aligned at a point on the leading edges of the pulses for each voltage channel. The alignment point is determined with a loop that identifies the first data point to surpass a value which is a specified fraction of the pulse's amplitude. Alignment at 1/10 of the pulse amplitude was determined to be optimal by qualitative analysis of several pulse alignments at different alignment fractions. The noise at the baseline voltage limits how small of a fraction can be used to align the voltage curves. Figure 2 illustrates aligned pulses at 1/10 and 1/5 of the pulse's amplitude.

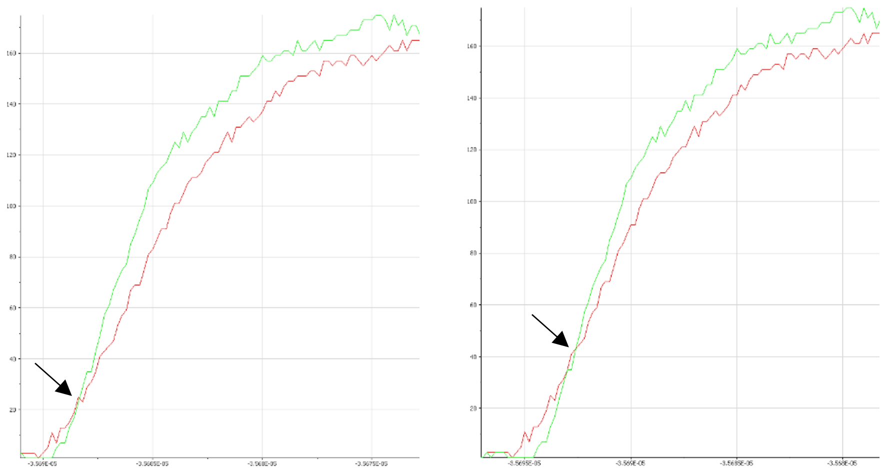

Figure 2 – Examples of aligned voltage curves for an alignment fraction of 1/10 (left) and 1/5 (right)

The next step in the data analysis is to determine the current in order to calculate power. Current I is obtained from Ohm's Law:

$$I = \frac{V_{term}}{Z_{term}} \tag{10}$$

where V_{term} is the voltage across the dielectric at the termination end of the core and Z_{term} is the impedance matched termination resistance. The direction that pulses travel are alternated to avoid defect migration in the conductive inner and outer layers of the core. Therefore, the script has to select the smaller of the two voltage channels as V_{term}. Instantaneous pulse power P is obtained from the following equation:

$$P = \Delta V \cdot I \tag{11}$$

where ΔV is the difference between the aligned voltage channels. Figure 3 is an example of instantaneous voltage difference, current, and power for a single pulse.

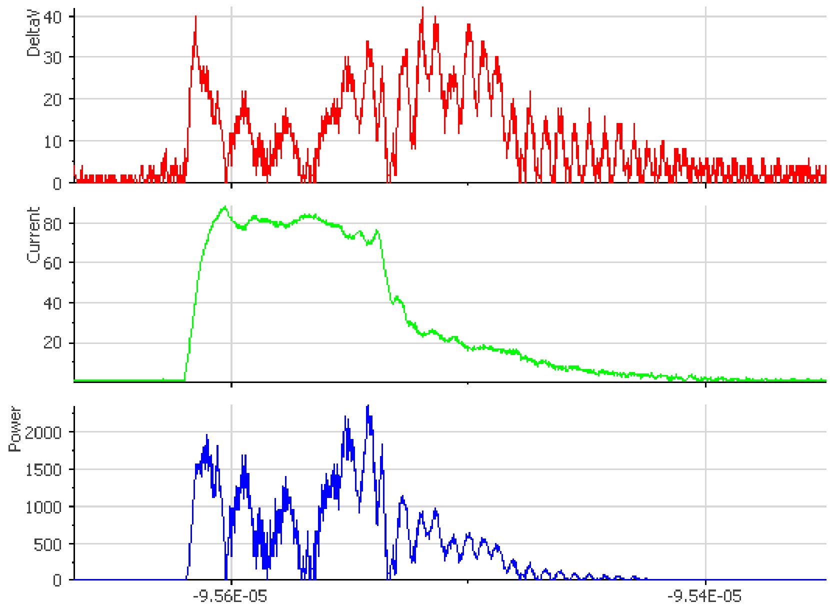

Figure 3 – Voltage drop across a core (top), current (middle), and power (bottom) of a pulse. This image is a screenshot from the DIAdem user interface; the horizontal axis is time (seconds).

Before calculating pulse power, the script removes noise data surrounding the pulses. Instantaneous pulse energy is calculated by time integrating the instantaneous pulse power, and the energy per pulse is calculated by dividing the total pulse energy by the number of pulses in a dataset. Finally, the energy per pulse is multiplied by the pulse frequency to find pulse power (Q_{pulse}).

2.2 RMS voltage method

The RMS voltage method is simple in comparison to the pulse alignment method. It's based on the principle that RMS voltage V_{RMS} is equal to the value of DC voltage that would produce the same power dissipation. For n data points with voltage V, RMS voltage (V_{RMS}) is calculated as follows:

$$V_{RMS} = \sqrt{\frac{\sum_{i=1}^{n} V_i^2}{n}} \qquad (12)$$

Denoting the voltage measurement at each end of a core as V_{RMS1} and V_{RMS2}, where V_{RMS2} is also the voltage across the termination resistor, current is calculated as follows:

$$I_{RMS} = \frac{V_{RMS2}}{Z_{term}} \qquad (13)$$

Then, pulse power is calculated with the following equation:

$$Q_{pulse} = (V_{RMS1} - V_{RMS2})I_{RMS} \quad (14)$$

RMS pulse power can be calculated with a spreadsheet software program such as Excel. However, this method was recently improved so that pulse power is measured continuously. In addition to minimizing the amount of work required to run an experiment, continuous pulse power measurement captures drifts in pulse power that may cause inaccurate COP values.

2.3 Real-time power method

The real-time power measurement method involves real-time current and voltage sensing at both ends of coaxial reactor cores. This method has not yet been implemented, but in theory, it will give true power (instead of apparent power) and it will avoid error from using the termination resistor for current measurement. Figure 4 is the schematic of the current/voltage sensors used to measure real-time current and voltage; R_{sense} is an RF resistor dedicated to current measurement.

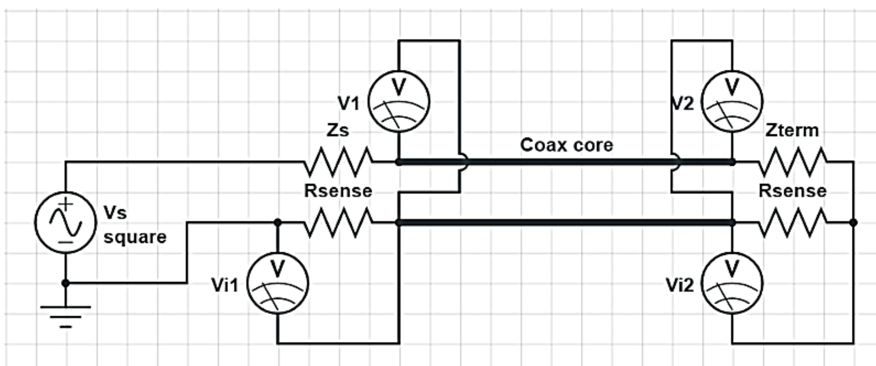

Figure 4 – Schematic of the real-time pulse power measurement configuration

The power measured on the pulse generator side of the core (V_1/V_{i1} in the schematic) is the total power dissipated in the core and the components after the core such as the transmission line and the termination resistor. The power measured on the termination side of the core (V_2/V_{i2} in the schematic) is the same except that it doesn't include power dissipated in the core. The following equations are used to calculate power, where P_1 and P_2 are real-time power at each end of the core.

$$P_1 = V_1 I_1 = V_1 \left(\frac{V_{i1}}{R_{sense}}\right) \quad (15)$$

$$P_2 = V_2 I_2 = V_2 \left(\frac{V_{i2}}{R_{sense}}\right) \quad (16)$$

These signals are RMS averaged and then subtracted to find power dissipated in the core alone. With regards to nomenclature, the letter Q represents thermal power and P represents electric power.

$$Q_{pulse} = P_{1RMS} - P_{2RMS} \tag{17}$$

With this real-time power method, pulse power is continuously measured (similarly to the RMS voltage method).

3 Potential sources of measurement error

As can be demonstrated by uncertainty analysis, random error is negligible for the amount of reaction power that the IPB calorimeter was designed to measure. Therefore, section 3 is limited to investigating possible sources of *systematic* measurement error.

When considering a source of error, it's important to find out if it causes more conservative or less conservative COP values. The objective is to avoid any error that increases COP values, i.e. false negatives are preferable to false positives.

3.1 Heater power Q_{heater}

In the current IPB systems, the heater control loop is susceptible to electromagnetic interference from the pulse system. In the IPB system, most measurements such as temperatures, flowrates, etc. are made during the pulse off-time that occurs during approximately 1 out of every 10 seconds. However, the heater controller operates continuously; it has not been restricted to operating to when pulses are off. A heater controller which produces random swings in heater power/core temperature is easy to identify and the accompanying data is discarded, but a continuous bias or drift in heater power measurement from the true heater power can be difficult to identify. The consequences (conservative or not) of such an error are unknown.

3.2 Pulse power Q_{pulse}

One issue with pulse power measurements is that inductance in the oscilloscope probe leads will cause 'ringing' in the signal. This effect can be observed by reducing the length of the probe leads.

Another issue is mismatch of the termination resistance or termination lines with the core, which will cause 'reflected' pulses to travel in the reverse direction from the initial pulse. The voltage and current of these reflections will be measured, but the resulting power will negatively contribute to pulse power (error that reduces pulse power is non-conservative and vice versa).

The transmission lines between the core and the termination resistor are about 3-4 feet long and they conduct current at about 2 feet/second. Therefore, pulses take 12-16 nanoseconds to travel to the termination resistor and back, which results in reflections that overlap with the initial pulse. When there is poor impedance matching, reflections are observed as distortions or noise in the flat part and the decay of pulses. The current method of impedance matching the termination resistor and transmission lines to the core is by adjusting their impedance until the pulse looks the way it does when it comes out of the pulse generator.

Since reflections overlap with the initial pulse, it's not possible to know the magnitude of the reflections' power dissipated in cores even when it seems as though the impedances are well matched. A solution to this problem is to make the transmission lines to and from the termination resistor long enough so that reflections don't overlap with the initial pulse, allowing for the power dissipated from reflections to be isolated. If the magnitude of power dissipated from reflected pulses is significant, it should be added to overall pulse power or there should be better impedance matching.

Moreover, there is more certainty in pulse power measurements if different measurement methods produce the same results. Tests have shown that the pulse alignment method and the RMS voltage method agree to within 5%. Therefore, any sources of error likely apply to both of these methods. For example, current is measured with the termination resistor in both cases – a potential source of error.

The accuracy of pulse power measurements can be confirmed by applying a known sinusoidal input power to reactor cores and comparing with power measured using the methods described in section 2. Due to the unknown frequency response of the components in the pulse system, it's important to determine the accuracy of pulse power measurements for the range of frequencies found in pulses. At BEC, this type of calibration has not yet been done.

3.3 Correction term Q_{out}

There is a source of systematic measurement error in the ΔQ_{out} term which results in reaction power values that are smaller than the actual reaction power. This error originates from not knowing the heat input profile of reactions and assuming that the heat input profile from reactions is the same as that of the heater (i.e. assuming Q_{out} does not depend on $Q_{reaction}$). This error will not cause false claims of excess heat, and the mathematical reasoning behind it is detailed in the appendix of this report.

There is less potential error from inaccuracies in the ΔQ_{out} term if its magnitude is reduced, which can be done by designing reactor cores with the same length heater and outer conductive layer where pulse heat is dissipated. The heater cannot exceed a certain length for a given T_r operating point because of temperature limitations of materials at the ends of the cores. However, pulse heat dissipation can be reduced at the ends where there is no heater by decreasing the electrical resistance of those sections. For example, the palladium/nickel could be replaced with a highly conductive material such as silver.

Furthermore, the magnitude of ΔQ_{out} and any associated error is reduced by operating the IPB calorimeter in isoperibolic mode, which is when the metal heat spreader is kept at constant temperature (instead of isothermal mode, which is when the core is kept at constant temperature). This is because there is less variation in heat flow out of the calorimeter (ΔQ_{out}) if the heat spreader is at constant temperature than if the center of the core is at constant temperature. However, in isoperibolic mode, an issue remains with variability of the heat leaving the calorimeter past the ends of the heat spreader and through the ends of the core, so there would still be a (reduced) ΔQ_{out} correction term. The drawback of the isoperibolic operating mode is that it increases the IPB calorimeter's time constant. Therefore, the amount of experimental parameters that can be tested is maximized when the IPB calorimeter is operated in isothermal mode, and the accuracy of measurement is maximized when the IPB calorimeter is operated in isoperibolic mode.

4 Control tests

Control tests are an important way of confirming the accuracy of calorimetry. If a negative control tests is producing COP results greater than one, there's systematic error in the calorimetry. Another approach to the use of control tests is to subtract the reaction heat measurement from the control from that of the actual experiment; if systematic error is common to both, it will cancel out.

4.1 Helium

According to the reaction mechanism hypothesis that is guiding this research work, hydrogen is a reactant for nuclear reactions in reactor cores. Replacing the hydrogen with an inert gas such as helium stops any reactions from occurring, allowing for the exact same experiment parameters and core that produce excess heat to be used as a negative control test.

If a reactor core has been in a pressurized hydrogen environment at high temperatures for an extended period of time, it's likely that hydrogen has absorbed deeply into the metal lattice (more so for palladium than nickel). If such a core is used as null core and exposed to helium, it's difficult to know if and when hydrogen is fully displaced. Therefore, the best time to do a helium null test is when a core has not yet been exposed to hydrogen (i.e. when it's untested for excess heat).

4.2 Null core

A null core is a reactor core that is known to not produce excess heat regardless of the gas to which it's exposed. The issue with this method is the uncertainty in assuming that a core will not produce excess heat since we have very limited knowledge of what conditions could cause reactions. If there is possibility of systematic error that occurs specifically when hydrogen is used (e.g. caused by a difference in thermal conductivity), a null core exposed to hydrogen is the appropriate control test. Otherwise, a null core exposed to helium is ideal.

5 Recommended work

The IPB calorimeter is a significant improvement over previous calorimetry methods at BEC. However, there is still potential for improving the accuracy of measurements. At the moment, there are three main improvements to be made:

1. Implementing the new pulse power measurement method that involves finding true power from instantaneous current and instantaneous voltage measurements at each end of reactor cores.
2. Limiting core heater control to when pulses are off.
3. Matching the heat input profile of core heaters to that of pulses by reducing the resistance of the core surface where there is no heater and/or by increasing the length of the heater (minimizing ΔQ_{out}).

Appendix – Analysis of systematic ΔQ_{out} error

The heat flow out of IPB calorimeters depends on core temperature, pulse power, and reaction power. For a given temperature, we can assume that ΔQ_{out} has the following linear relationship with pulse power and reaction power:

$$\Delta Q_{out} = m\left(Q_{pulse} + aQ_{reaction}\right) + b \tag{18}$$

m and b are calibration constants determined with Eq.(8), and a is the calibration constant which represents the unknown contribution of $Q_{reaction}$ to ΔQ_{out}. In order to understand this source of error, there are three scenarios to consider:

1. If the heat input profile from reactions is the same as the heater ($Q_{reaction}(z) = Q_{heater}(z)$, where z is the axial dimension of the core), $a = 0$.
2. If the heat input profile from reactions is the same as the pulses ($Q_{reaction}(z) = Q_{pulse}(z)$), $a = 1$.
3. If the heat input profile from reactions is not the same as the heater or the pulses ($Q_{reaction}(z) \neq Q_{pulse}(z) \neq Q_{heater}(z)$), $0 < a < 1$ or $a > 1$.

Unfortunately, a is unknown and difficult to determine. However, we can find that assuming $a = 0$ will result in conservative reaction heat measurements. Plugging Eq.(18) into the general reaction power equation (Eq.(7)) gives the following equation (simplified by assuming that there are no pulses initially):

$$Q_{reaction} = \Delta Q_{heater} - Q_{pulse} + m\left(Q_{pulse} + aQ_{reaction}\right) + b \tag{19}$$

Since a is unknown, it's not possible to solve for reaction power; rearranging Eq.(19) to find the dependence of reaction power on the calibration constant a:

$$Q_{reaction} = \frac{\Delta Q_{heater} - Q_{pulse} + mQ_{pulse} + b}{1 - ma} \tag{20}$$

In any scenario, Eq.(20) is defined for $a \geq 0$ and $Q_{reaction} \geq 0$. Therefore, the minimum value of reaction power is obtained for $a = 0$, and its value will be the numerator in Eq.(20). Hence, assuming $a = 0$ results in conservative values of reaction power.

It's possible to determine a by calibration, but this would require changes in hardware and complex methods. First, the temperature profile from reaction power would be determined with a row of thermocouples inside the core (instead of the single thermocouple that is currently used). Then, the heat input profile could be determined from this temperature profile. In order to find the calibration constant a, the heat input from reactions would be simulated with a Joule heater that can produce any heat input profile.

Also, assuming $a = 0$, ΔQ_{out} is eliminated if the outer conductive layer of the core is used as the resistive compensation heater since the heat input profile from pulses and compensation heating would be the same. In order to avoid overloading the outer conductive layer, the core heater could be used as a steady-state source for the majority of the heat that maintains high core temperatures. A potential issue with this approach is that the DC compensation power overlapped on the pulses may affect the reactions in the outer layer.