Rapport BIPM-09/01

Construction of an Absorbed-Dose Graphite Calorimeter

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ABSTRACT

The development of a calorimetric primary standard for absorbed dose to water at the BIPM has three major aspects: determination of the specific heat capacity of graphite, the construction of a graphite calorimeter, and development of a conversion procedure to determine absorbed dose to water. This report describes the second stage, the construction of an absorbed-dose graphite calorimeter.

1. INTRODUCTION

The development of a calorimetric primary standard for absorbed dose to water at the BIPM comprises three major parts: determination of the specific heat capacity of graphite, the construction of a graphite calorimeter and development of a conversion procedure to determine absorbed dose to water. The first part, in which the specific heat capacity of ultra-pure graphite was determined using two different methods, underpinned by a study of a reference material, has been reported previously [1-3]. The second part, consisting of the construction of the calorimeter, gives rise to three different but complementary experimental configurations.

The BIPM calorimeter, described below, differs in several ways from those made by other groups, e.g. [4-12]. One reason for this is the intention to have a dismountable calorimeter that is inherently quasi-adiabatic. The procedure applied to determine the absorbed dose to water consists of three different measurements: the calorimetric measurements are combined with ionometric measurements made in a similar geometric configuration and, subsequently, in a water phantom. When combined with the results of Monte Carlo calculations using the same geometrical models, the absorbed dose to water can be determined.

With the objective to reinforce traceable absolute measurements of absorbed dose of ionizing radiation at the BIPM, a graphite calorimeter has been constructed. This report describes the design of the calorimeter itself.

2. DESIGN AND REALIZATION OF THE CALORIMETER

2.1. Strategy

The absorbed dose calorimeter is made of graphite and consists principally of a graphite core enclosed by a graphite jacket. Several requirements were identified when designing the calorimeter, in particular:

- to make the calorimeter modular and dismountable, so as to identify and evaluate systematic uncertainties more easily;
- to avoid a temperature gradient between the calorimeter core and its jacket and therefore operate in a quasi-adiabatic mode;
- to apply a mass thickness in front of the centre corresponding to 5 g cm^{-2} in water for ^{60}Co beams, and 10 g cm^{-2} for high energy photon accelerator beams $\geq$ 6 MV;
- to employ only low Z materials;
- to make the calorimeter transportable.

Further, as one of the ionometric measurements consists of replacing the calorimetric core by a parallel-plate transfer ionization chamber, it is necessary that

- the dimensions of the hollow space inside the jacket are precisely those of the parallel-plate ionization chamber type used for many years as a primary standard at the BIPM;
- the thickness of the graphite core equals the total graphite thickness of the ionization chamber.

2.2. Calorimeter Core

The graphite core was machined in the form of a solid cylinder: 6.7 mm long and with a diameter of 45 mm. The core was first put into a temperature-controlled oven at 110 °C, according to the recommendations given in [13-15]. Once cooled down to ambient temperature in a recipient filled with a silica gel desiccant, it was placed on a balance using soft-tipped forceps. The mass measurements were repeated, controlling the zero reference between measurements. The balance is located in an air-conditioned laboratory where ambient temperature, atmospheric pressure and humidity are stabilized and measured. These parameters are required for the correction of the air buoyancy effect according to [16, 17].

Each time a hole was drilled or material was added to the core, the mass was re-measured to allow appropriate corrections to be made for the perturbation of the heat capacity, taking into account the radiation interaction properties of any non-graphite material.

Two holes separated by 2 mm, 2 mm deep with a diameter of 0.6 mm, are drilled into the curved face of the cylinder at half height. A glass-beaded thermistor, 1 mm long and 0.4 mm wide, is glued into each hole using an epoxy of high thermal conductivity. The two thermistors form a pair, mounted in opposite arms of a Wheatstone bridge in which the two other resistances are of fixed values and with a low temperature coefficient (5 parts in 10^6/K). In total, three such thermistor pairs are positioned on the curved face, separated by an angle of 120°, *cf.* fig.1.

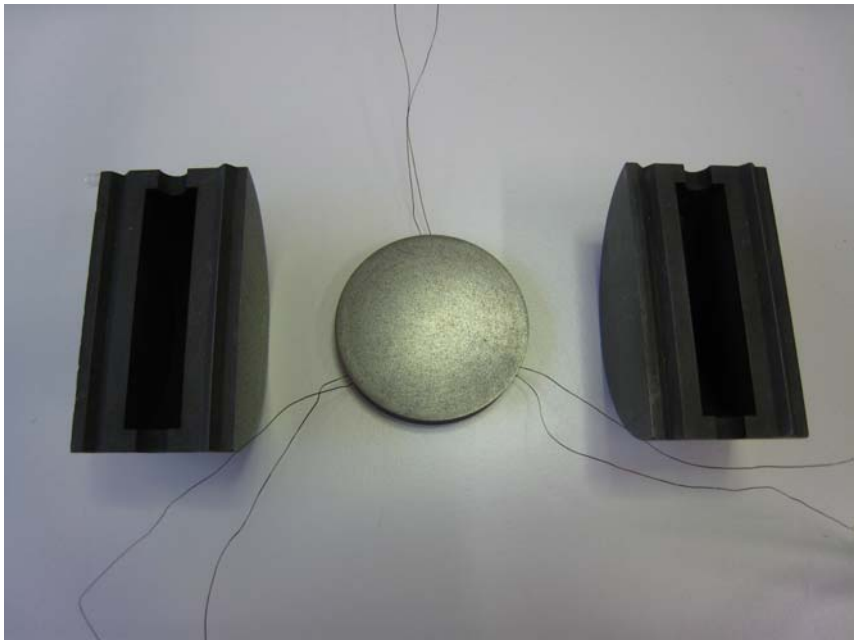

Figure 1. The graphite core, equipped with three thermistor pairs, is shown in the centre, with the two halves of the graphite jacket positioned to the left and right. Four holes are drilled in the curved surfaces of the core and jacket. These are used to accept the four pins that support the core inside the jacket.

Each of the three Wheatstone bridges is powered by a low noise d.c. power supply of low drift (2 parts in 10^4 in 2 years) at 1.14 V. To achieve a temperature calibration, the core is placed in thermal contact with a water-tight copper container into which a platinum resistance thermometer (PRT) is also placed. The container assembly is immersed in a water tank with a temperature stability of about 1 mK. The voltage signal from each Wheatstone bridge and the d.c. power supply, as well as the PRT signal, are recorded simultaneously while stabilizing the water at temperatures from 19 °C to 28 °C, in steps of 0.25 °C. The relative standard uncertainty of a measured temperature difference due to the temperature calibration is typically less than 2 parts in 10^4.

Four additional holes with a diameter and depth of 1 mm are drilled into the curved surface of the core to accept the pins that mount the core in its jacket.

Two cores have been machined, identified as **A1** and **A3**. The impurities added to each core have been traced as shown in table 1.

Table 1. Materials and corresponding masses of the components of the calorimeter cores.

	A1		**A3**	
material	m /g	$u(m)$/g	m /g	$u(m)$/g
C	19.0400	5×10^{-4}	18.9388	5×10^{-4}
6 beads	3.0×10^{-3}	0.3×10^{-3}	3.0×10^{-3}	0.3×10^{-3}
epoxy resin [18]	13×10^{-3}	0.1×10^{-3}	8.2×10^{-3}	0.1×10^{-3}

2.3. Jacket

The assembled jacket forms a hollow cylinder, to house either the graphite core or the parallel-plate transfer ionization chamber. The simpler option to construct the jacket from front and back components was abandoned in favour of top and bottom half-cylinders. Although more complicated to produce, the latter configuration should result in a more homogenous temperature distribution, with no barrier to heat conduction from front to back, while in the simpler design this conduction would be hampered by relatively poor conduction between the two components. The jacket has an outer diameter of 60 mm and is 32 mm high. The hollowed interior forms a cylinder of diameter 51 mm and 11.2 mm high. The contact surfaces of each half-cylinder are wedged to prevent a direct line of sight to the core by incident photons. The wedges also assure a stable and reproducible positioning.

The density of the graphite piece used for the jacket was determined before machining. After machining, the jacket was placed in a pumped oven to remove any impurities added to the

material during machining. Several holes were drilled in the two half-cylinders. Firstly, two diametrically opposite holes of 6 mm diameter were made though the curved cylinder wall at the junction between the two halves, to accommodate the ionization chamber stem, *cf.* fig 2. Secondly, seven holes, each of diameter 4 mm, were made at half height on this same circular wall. Three of these holes are rotationally aligned with the three pairs of holes of the core to allow the thermistor leads to pass. The four remaining holes each accommodate a nylon plug through which a thin metal pin is passed to support the core.

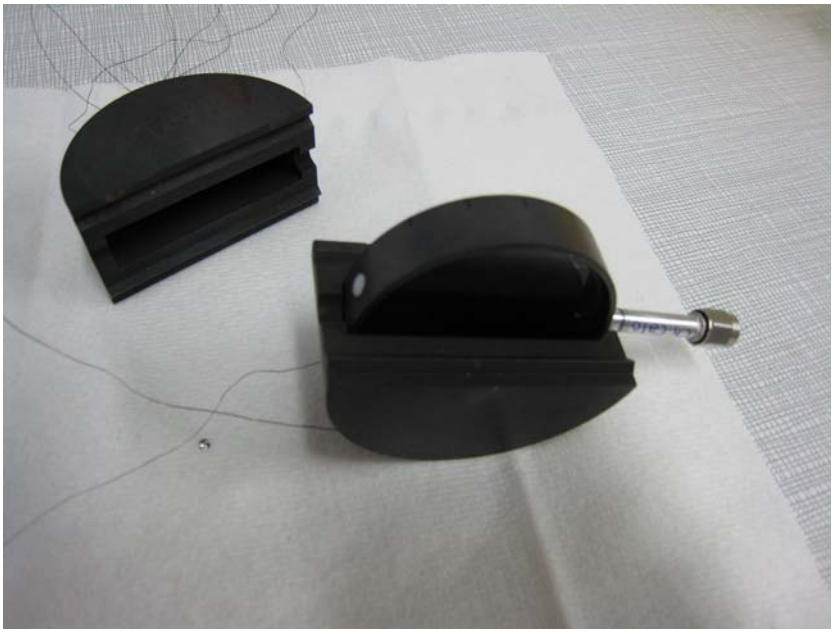

Figure 2. The graphite jacket shown now with the parallel-plate transfer ionization chamber.

Finally, two further holes, 2 mm deep with a diameter of 0.6 mm, were drilled on the curved face of each half-cylinder. One glass-beaded thermistor, 1 mm long and 0.4 mm wide, was glued into each hole using an epoxy of high thermal conductivity. These thermistors measuring the temperature of each half of the jacket were calibrated in a similar way as for the core, but rather than employ a Wheatstone bridge the thermistor resistance is recorded directly as a function of temperature.

Two nominally identical jackets have been constructed, named **J1** and **J2**. The similarity of the two jackets, and in particular the effective thickness of the front faces, has been evaluated by measuring the ionization current from the parallel-plate transfer ionization chamber when placed inside each jacket in turn and irradiated in the BIPM ^{60}Co reference beam. A relative difference of 3 parts in 10^4 was measured, which indicates that, provided a small correction is applied for this effect, the jackets can be considered interchangeable. This simplifies the use of the calorimeter in practice; in particular, one jacket can be used for calorimetric measurements and the other for ionometric measurements.

2.4. Vacuum Container

A maximum temperature rise of a few mK is generated when irradiating the calorimeter in the BIPM reference ^{60}Co beam or in a high energy accelerator beam. To minimize heat losses, a vacuum container made of PMMA[a] was constructed to house the calorimeter. The external dimensions of the container were chosen to be the same as those of the reference water phantom used at the BIPM for the ionometric measurements in water, as it is considered that similar dimensions will reduce certain systematic effects by cancellation, *cf.* fig. 3.

Figure 3. Calorimeter vacuum container made in PMMA, without reflectors inside.

To make the container mechanically resistant under vacuum, the walls are 30 mm thick. However, for calorimeter measurements at a depth of 5 g cm^{-2}, the entrance window cannot be so thick. Nor can it be as thin as the 4 mm PMMA window of the BIPM reference water phantom, which would not support the vacuum. Ideally, for the graphite absorbed dose measurement the build-up material should be entirely graphite, although this is not feasible for the entrance window because of the porosity of graphite. As a compromise, a 10 mm thick circular graphite window with a trapezoidal profile was constructed. This is placed into a 150 mm diameter hole machined on the front face of the container, and covered by a 4 mm thick PMMA window, *cf.* fig. 4. Small grooves were machined on the outer edge of the graphite window to improve the evacuation of air situated between the PMMA window and the graphite. The graphite window serves not only as a build-up material but also as a mechanical support for the PMMA window (whose thickness is chosen to match that of the window of the BIPM reference water phantom).

[a] polymethylmethacrylate

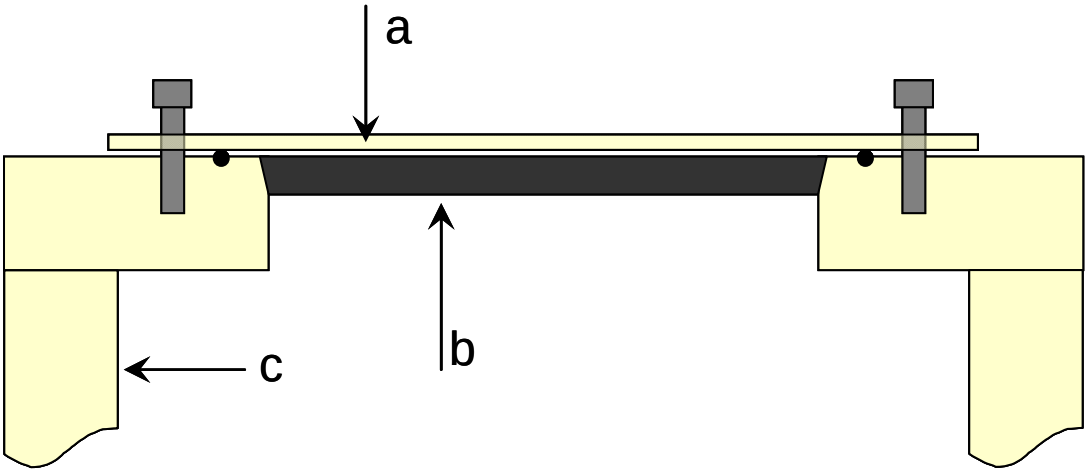

Figure 4. Schematic of the PMMA and graphite window combination. a – 4 mm thick PMMA window; b – 10 mm thick circular graphite window; c – container wall 30 mm thick.

The calorimeter is mounted in a circular PMMA frame that can be fixed on a PMMA support, itself attached to a small translation table of micrometric resolution. When using a ^{60}Co beam, this arrangement is placed behind the entrance window of the container, *cf.* fig. 5. For accelerator beams, the mount is displaced 50 mm towards the back face of the container, and a 160 mm square graphite block 27 mm thick is placed between the entrance window and the calorimeter jacket to fulfil the requirement for a total mass thickness of 10 g cm^{-2}.

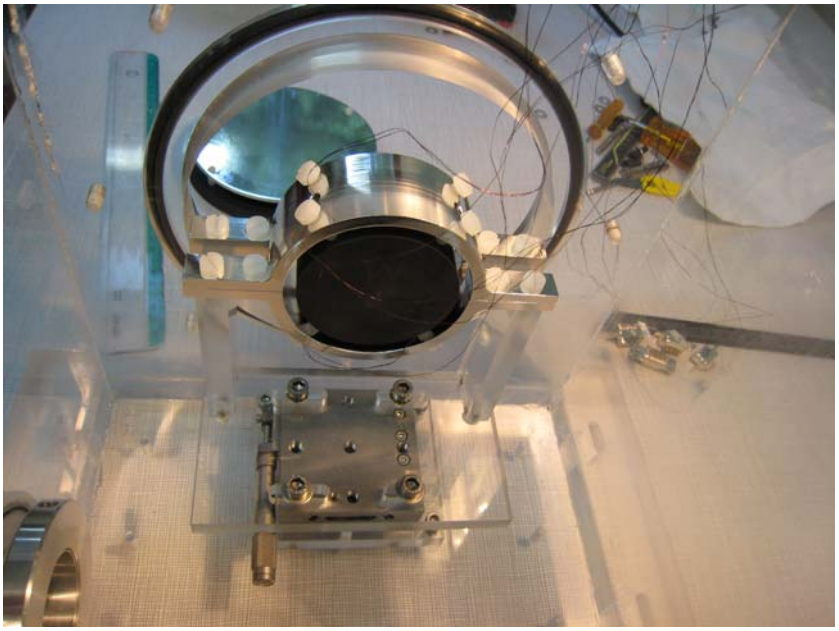

Figure 5. The calorimeter in its aluminized PMMA support mounted on the translation table, viewed from the rear. Behind the calorimeter is the graphite window, although it is obscured by the aluminized PMMA reflector positioned between the window and the calorimeter.

Three reflecting surfaces were introduced to reduce radiative heat transfer. Firstly, a 2.5 mm thick aluminized PMMA plate was mounted directly behind the graphite entrance window, to arrange back reflection of heat radiation originating from the front face of the jacket, *cf.* fig. 6-a. This component serves also to complete the build-up material, which totals 5 g cm^{-2}. Secondly, the circular PMMA frame was aluminized to maximize back reflection of heat radiating in the radial direction. Thirdly, an aluminized concave spherical lens of PMMA was placed at the back of the container in such a position that heat radiating from the rear of the jacket is reflected back, *cf.* fig 6-b. The radius of curvature of the lens is 190 mm for ^{60}Co beams and 140 mm for accelerator beams, *cf.* Section 3.

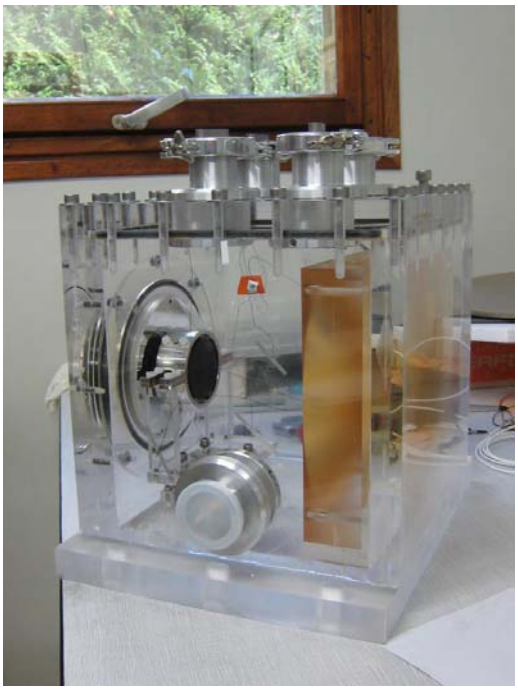

Fig. 6-a. The mounted calorimeter assembly, from the side. A flat aluminized PMMA plate acts as build up material as well as a reflector for heat radiation.

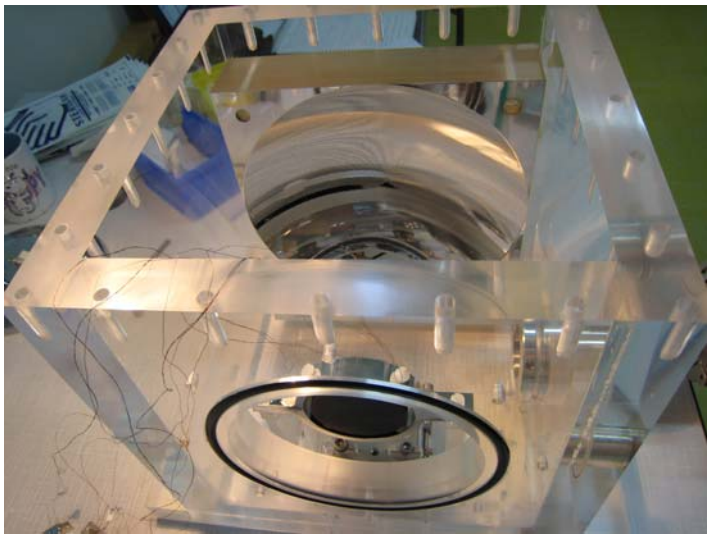

Figure 6-b. From above. A concave mirror is placed behind the calorimeter for back-reflection of heat radiation generated by the jacket.

The top of the PMMA container is a 30 mm thick PMMA lid, fixed by screws and a vacuum seal onto the container. The lid is equipped with four vacuum flanges, allowing various electrical signals including the thermistor leads to pass to the measurement system, *cf.* fig 6-c.

The connection to a turbo molecular pumping system is made via a flange placed on the lower part of a side wall. A pressure of less than 10 mPa[b] was typically achieved under continuous pumping.

[b] Corresponding to 10^{-4} mbar.

Fig. 6-c. The graphite window is seen to the right, covered by the PMMA window. Four vacuum flanges on the lid of the vacuum container allow the passage of electrical signals.

2.5. Thermal Insulation and Electrical Shielding

To ensure a stable temperature environment, a double thermal barrier has been constructed of a plastic honeycomb structure, combined with expanded polystyrene[c]. Each of the two components is covered by aluminized PET[d], mainly to provide shielding against electromagnetic interference that might arise in an accelerator area, but also contributing to the thermal insulation.

2.6. Mechanical Support

As for the BIPM reference water phantom, the vacuum container is placed on cylindrical feet 100 mm high. The feet are made of PTFE[e] to ensure temperature insulation and are fixed on a 30 mm thick PMMA slab. This slab is placed on a mechanical support that is adjustable in the horizontal directions using translation stages, and in the vertical direction using four screws.

[c] Also known as Styrofoam or Styron.
[d] Polyethelene terephthalate, also known as Mylar or Melinex.
[e] Polytetrafluoroethylene, also known as Teflon.

3. DETERMINATION OF MASS THICKNESS

For the calorimetric measurements, a source-to-detector distance of 1 m is used for all beams. The measurement depth for reference measurements in ^{60}Co radiation is normally 5 g cm^{-2} [19-23], which corresponds to a depth of around 5 cm in water. Consequently, the source-to-surface distance (SSD) is chosen to be 0.95 m for ^{60}Co beams. For accelerator photon beams, the reference depth is 10 g cm^{-2} and consequently SSD = 0.90 m is used. To calculate the precise mass thickness, all materials are scaled by their mass density. Therefore, the density and thickness were determined for each component placed in the beam in front of the measuring plane. The dimensional measurements were made using a calibrated coordinate measurement machine with 1 µm resolution. Tables 2-a and 2-b summarize the values obtained for the BIPM graphite calorimeter.

Table 2-a. Measured contributions to the mass thickness of build-up material when using a ^{60}Co beam.

material	component	thickness / cm	bulk density /(g cm^{-3})	mass thickness /(g cm^{-2})
PMMA	window	0.40	1.187	0.47
C	window	0.995	1.774	1.76
PMMA	substrate	0.25	1.190	0.30
Al+varnish[f]	coating	-	-	0.01
C	jacket front	1.04	1.781	1.85
C	half core thickness	0.335	1.783	0.60
	total			4.99

Table 2-b. Measured contributions to the mass thickness of build-up material when using accelerator beams.

material	component	thickness / cm	bulk density /(g cm^{-3})	mass thickness /(g cm^{-2})
PMMA	window	0.40	1.187	0.47
C	window	0.995	1.774	1.76
PMMA	substrate	0.25	1.190	0.30
Al+varnish	coating	-	-	0.01
C	jacket front	1.04	1.781	1.85
C	half core thickness	0.335	1.783	0.60
C	compensation block	2.73	1.830	5.00
	total			9.99

[f] Aluminizing plus varnishing added a mass of 1.43 g to the substrate of diameter 14.2 cm.

4. MEASUREMENT APPARATUS

The data collection is computer-assisted. Each of the three resistance bridges are connected to a nanovoltmeter. The resistance of the two thermistors of the graphite jacket and the voltage of the d.c. power supply are measured using three multimeters. The six instruments are triggered externally via a signal generator. All measurements are made by firstly collecting the data in the local buffers. When the measurement is complete, the data are transferred to the computer for automatic data treatment and storage. The irradiation source can be piloted via a TTL signal generated either by one of the commercial meters, or by a digital acquisition card. An electrometer with an external feedback capacitor is used to measure the ionization current from the transfer chamber. A thermistor is used to measure the chamber temperature, and air pressure and humidity are also recorded. For accelerator beams, a monitor chamber is used for normalization. In this case, a second electrometer and thermistor are used.

5. DISCUSSION AND CONCLUSION

The reduction of all sources of heat transfer by conduction is important, in particular isolation of the calorimeter from the varying ambient temperature. For this reason, a vacuum is applied, insulating materials are used and contact surfaces are minimized, for example by using only pins to support the core inside the jacket. However, the suppression of radiative heat transfer is arguably even more important, as it is non-linearly dependent on temperature. The addition of the reflecting surfaces reduced the heat loss experienced after heating the core by a factor of three. Initial measurements using the calorimeter indicate a potential relative statistical uncertainty of less than 3 parts in 10^3 using three thermistor bridges over two working days.

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