Warm-up calorimetry of Dewar-Detector Assemblies

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Abstract. Boil-off isothermal calorimetry of Dewar-Detector Assemblies (DDA) is a routine part of their Acceptance Testing Procedure. In this approach, the cryogenic liquid coolant (typically LN2) is allowed to naturally boil-off from the Dewar well to the atmosphere through a mass flow meter; the parasitic heat load is then evaluated as the product of the latent heat of vaporization and the "last drop" boil-off rate.

An inherent major limitation of this technique is that it may be performed only at the fixed boiling temperature of the chosen liquid coolant. A further drawback is related to the explosive nature of "last drop" boiling, manifesting itself as an uneven flow rate. This especially holds true for advanced High Operational Temperature Dewar-Detector Assemblies, typically featuring short cold fingers and working at 150 K and above.

In this work, we adapt the well-known technique of dual-slope calorimetry and show how accurate heat load evaluation may be performed by comparing the slopes of the warm-up thermal transients under different trial added heat loads. Because of the simplicity, accuracy and ability to perform calorimetry literally at any temperature of interest, this technique shows good potential for replacing traditional boil-off calorimetry.

1. Introduction

Manufacturing and maintaining Dewar-Detector Assemblies (DDA) requires accurate calorimetry. An elevated self-heat load may indicate a loss of vacuum due to natural outgassing, release of trapped contaminations or insufficient mechanical integrity. This is why the evaluation of the parasitic heat load has become a regular part of Acceptance Test Procedures.

Accurate calorimetry is also needed for calibrating so-called Simulation Dewars. These are a cost-effective substitute to actual DDAs, where the heat flow produced by the detector Read-out Integrated Circuitry is mimicked by electrically heating an auxiliary shunt mounted directly upon a mock-up substrate.

The best known and widely accepted practice of boil-off calorimetry is based on an evaluation of the boil-off rate produced by the cryogenic liquid boiling inside the Dewar's cold finger as it evaporates to the atmosphere through a mass flow meter. The total heat flow (comprised of conductive, radiative and convective heat inflows) is then calculated as the product of the latent heat of vaporization and the said boil-off rate [1-3]. Liquid Nitrogen (LN2) which boils at approximately 77K is the most popular cryogenic liquid ideally suited to traditional DDAs, the detectors for which are intended to operate at or near such a temperature.
In order to minimize inaccuracies related to the presence of uncontrollable residuals of liquid coolant inside the cold finger, the said boil-off rate value is usually taken as the last drop boils off. At this instance the cold finger is considered to be practically empty. Unfortunately, the said “last drop” usually boils off in a rather explosive manner; this results in abnormal bubbling, explosions and geysering effects. The presence of gaseous residuals of the evaporated liquid along with boiling micro-drops at atmospheric pressure affect the temperature distribution along the cold finger and produce additional parasitic – convective and conductive – heat flows, evidently negating the measurement accuracy and its repeatability.

These phenomena result in typically irregular time dependence of the flow rate, making the entire measurement quite challenging and, to some extent, inaccurate. Additional inaccuracy results from unavoidable heating and, therefore, expansion of the vapor in a tube connecting the Dewar and flow-meter [3]. Another source of inaccuracy is the substantial error introduced by the mass flow meter.

With the aim of improving the measurement repeatability, this technique has been enhanced by using a trial heat load calibration performed on a pilot Simulation Dewar. In this approach, the “last drop” boil-off rate is recorded at different measurable and controllable (trial) amounts of added heat load applied to the cold finger tip by using an auxiliary resistor of a Simulation Dewar. The obtained, typically linear, dependence of the flow rate on the value of the trial added heat load is then extrapolated backwards. The ordinate of the "zero crossing" returns the negative value of the self heat load. This point corresponds to the condition of virtual thermal equilibrium characterized by a zero total heat flow at which the cryogenic liquid is virtually not boiling at all and gas flow is virtually absent. Such a method appears to be less sensitive to the systematic errors mentioned above and knowledge of the latent heat of vaporization is not required at all. For more accurate results, a linear curve-fitting technique is applicable.

The obtained slope is then used as a factor for evaluating the parasitic heat load of operational DDA by running the natural boil-off test with no added heat load. From experience, this technique, unfortunately, is not capable of delivering better than 10% repeatability.

Recent advances in High Operational Temperature (HOT) infrared (IR) detectors have resulted in an increase of the operating temperature to 150K or even above, along with using very compact, typically Stirling, mechanical cryo-coolers featuring very short (20mm and below) cold fingers. There are three major reasons which should prevent attempts to perform LN2 calorimetry on such DDAs. First, from experience, LN2 calorimetry of Dewars featuring shortened cold fingers is not repeatable at all, see above explanations and comments below. Second, since the conductive, radiative and convective heat inflows cannot be separated, there is no way to estimate the heat load at the higher temperatures based on a 77K evaluation. Third, since the effect of cryogenic pumping is temperature dependent, the convective heat inflow resulting from vacuum imperfections and a release of trapped gaseous contaminants from the cold surfaces inside the evacuated envelope may be quite different at 77K as compared to the higher temperatures.

The above considerations indicate the importance of performing calorimetry at or at least near the detector working temperature. This may require using of exotic cryogenic coolants boiling at higher than LN2 temperatures, which results in further complications. For example, possible cryogen choices for a DDA working at 150K, are Xenon (165K) and Carbon Tetra-Fluoride CF$_4$ (145K). Transportation and on-the-spot liquefaction of such cryogens present serious logistical difficulties, not to mention major health and safety hazards.

Unfortunately, other methods of Dewar calorimetry have not been developed yet [4]. There is a need, therefore, to advise on a novel technique for the evaluation of the DDA self-heat load over a wide range of working, preferably at any arbitrary, temperature, while avoiding the use of "exotic" cryogenic liquids.

The method advised below may be thought of as a modification of the "dual-slope" calorimetry as is used for the evaluation of heat capacities by making a direct comparison of the heating and cooling rates of a sample, as explained in [5-9]. In the modified technique, we will compare the heating rates during warm up of an initially precooled cold finger in the pilot Simulation Dewar under different trial
added heat loads. In doing so, we will evaluate the self heat load along with aggregate heat capacity at all temperatures. Assuming the consistency of the heat capacity, the self heat load of operational DDA will be then evaluated using single natural warm-up test (no added heat load).

2. Typical LN2 boil-off calorimetry of Simulation Dewar
Figure 1.a shows typical SCD Simulation Dewar (a) of a HOT IR detector featuring short (20 mm cold finger) and Figure 1.b shows the boil-off calorimetry setup. Figure 2 portrays four typical superimposed LN2 time histories of the boil-off rate recorded using this simulation Dewar with a 50mW added heat load.

![](image1.png)

**Figure 1.** Simulation Dewar (a) and boil-off calorimetry station

![](image2.png)

**Figure 2.** Typical LN2 boil-off curves
The drawbacks of boil-off calorimetry mentioned above manifest themselves in the form of obvious discrepancies in Figure 2.a between the “should be” similar boil-off curves, and may be only partially explained by the different amounts of LN2 poured initially into the cold finger well. It is worth noting that only the final portions of the boil-off curves represent “last drop boiling” (encircled in Figure 2.a) and are used for the “last drop” boil-off rate monitoring and heat load evaluation. Figure 2.b shows the typical patterns in more detail, where the boil-off curves are synchronized at 53 sccm. In Figure 2.b the ”last drop boiling” flow rate is not repeatable at all. Using the flow rate corresponding to the typical “plateau” preceding the typical peak produces more repeatable data, however, results in erroneous heat load evaluation since the cold finger is not empty and partially filled by LN2 at this moment of time.

3. **Warm-up rate as a measure of the heat inflow**

Reducing the distributed heat capacity along with conductive, convective, radiative and added "trial" heat flows to the tip of a cold finger, allows using the well-known differential Fourier heat equation for the warm-up process of a cold finger plug starting from the particular initial temperature which is assumed to be well below the temperature of interest. This is

\[ C(T)T'(T) = H_0(T) + H, \]  

where \( C(T) \) and \( H_0(T) \) are the aggregate heat capacity and heat inflow due to heat conductivity, convection and radiation at instantaneous temperature \( T \) at time \( t \). In (1), \( T'(T) = dT/dt \) is the instantaneous warm-up rate at time \( t \) and \( H \) is the trial heat flow added locally to the cold finger tip; it is assumed to be constant at all times. Since during all the warm-ups, \( C(T) \) and \( H_0(T) \) remain invariable, from (1), the warm-up rate at arbitrary temperature \( T \) should be an explicit and linear function of the amount of added trial heat flow \( H \).

Let us assume now that the cold finger tip was initially precooled to a temperature well below the temperature of interest by using, say, LN2 and then consequently allowed to naturally warm-up firstly due to a parasitic heat flow \( H_0(T) \) and secondly with added trial heat load \( H \), respectively.

Using (1), for the two warm-up transients we have two linear equations in \( C(T) \) and \( H_0(T) \)

\[ C(T)T'_0(T) = H_0(T), \quad C(T)T'(T) = H_0(T) + H, \]  

where \( T'_0(T) \) and \( T'(T) \) are the instantaneous warm-up rates at temperature \( T \).

From (2),

\[ C(T) = \frac{H}{T'(T) - T'_0(T)}, \quad H_0(T) = \frac{H}{T'(T)/T'_0(T) - 1} \]  

From (3), comparing the warm-up slopes of the two separate warm-up tests with and with no additional trial heat load it is possible to evaluate the heat capacity and parasitic heat load at any temperature covered by the above thermal transient.

Increasing evaluation accuracy is possible by running multiple warm-up tests at different trial heat loads, measuring the instantaneous warm-up rates at the temperature of interest and evaluating the "warm-up rate vs added heat load" linear curve. The ordinate of the zero crossing returns a negative value for the Dewar self-heat load. Similar to above explanations, this point corresponds to the condition of virtual thermal equilibrium, characterized by a virtual zero total heat inflow and virtual steady state temperature, resulting in a zero warm-up rate. For more accurate results, the linear curve-fitting technique is applicable.

The obvious and major advantage of this approach is that the heat load may be evaluated at any temperature over a wide temperature range without making use of exotic cryogenic coolants, as used in traditional isothermal boil-off calorimetry. Further, there is no gas pipe and flow meter involved in
the measurement. Instead, we use a thermometer (temperature diode) having much better accuracy as compared with a flow meter. Finally, since the warm-up rate is a very smooth and monotonous function of time (see below), the accuracy and repeatability should be further improved.

4. Multi-slope warm-up calorimetry at 150K

Figures 3. a,b show a diagrammatic view of the experimental setup and the simulation Dewar under test. In Figure 3.a, the Simulation Dewar is installed inside a vacuum jar. A temperature diode is connected to a Data Logger via a current source, as shown in Figure 3.b, so that the temperature of the cold end may be monitored during warm-up transient (resolution 0.05 K; sampling rate 50 Hz).

The above mentioned auxiliary resistor is thermally interfaced with the substrate mock-up and connected to a regulated power supply maintaining a constant power output independent of the resistor temperature. A control thermocouple is mounted on the cold finger base, the temperature of which is also monitored by the Data Logger. A small amount of LN2 was poured inside the cold finger well until the equilibrium temperature was reached, as monitored by the temperature diode. Upon opening of the vacuum port and evacuation of the LN2 residuals, the cold tip warm-up was recorded between 145 K and 155 K, thus allowing for an accurate estimation of the warm-up rate at 150K. This warm-up protocol has been repeated 4 times for each trial heat load, at 0, 50, 100 and 150mW, respectively.

Using a linear approximation, the warm up rates were evaluated for each run thus producing the dependence of the warm up rate on the magnitude of the added heat load. Figure 4 shows superimposed nearly linear and coinciding warm-up curves for (a) DDA #1 and (b) DDA #2 at a trial heat flow of 100 mW.

![Figure 3. Warm-up calorimetry setup](image)

From Figure 4, the warm-up test produces quite repeatable data. The temperature values over the limited temperature range involved may be accurately recorded and approached as linear trends. R-squared values of 0.9998 indicate that the linear fit is of sufficient quality (see also the superimposed linear trends in (a) and (b)).
Figures 5. a,b show superimposed dependencies of the warm up rates on the added heat load for the above two DDAs as collected during 3 independent tests. From Figure 5, this behavior may be approximated quite accurately by linear trends, which are also superimposed for reference. Also, from Figure 5, the repeatability of this method can be seen to be high.

Table 1 summarizes the results of calculations of the self heat load for the above two DDAs, and the associated testing repeatability. In particular, \( a \) and \( b \) are the curve-fitting coefficients of a linear warm up rate approximation \( T' = aH + b \). The negative value of the self heat load may be evaluated as the solution to equation \( T' = aH_0 + b = 0 \), namely \( -H_0 = -b/a \).
Table 1. Summary - self heat loads and accuracies at 150 K

| Run # | a       | b       | \( H_0 \), mW | a       | b       | \( H_0 \), mW |
|-------|---------|---------|---------------|---------|---------|---------------|
| 1     | 0.000833| 0.121375548| 145.68       | 0.000841| 0.121311  | 144.22        |
| 2     | 0.000833| 0.121217626| 145.55       | 0.000838| 0.121301  | 144.75        |
| 3     | 0.000826| 0.120200975| 145.51       | 0.000835| 0.120522  | 144.34        |
| Average| 145.58  |          |               | 144.44  |          |               |
| Normalized STD | 0.06% |          |               | 0.2%    |          |               |

Table 1 also shows the averaged values (which are quite close) and estimates of the pretty high testing repeatability. Further improvements in terms of experimental accuracy (if needed) may be obtained by averaging the warm-up curves in time domain.

Based on the above evaluations, the aggregate heat capacity of a DDA may be calculated easily. From Figure 5, for example, the averaged warm-up rates at 150K are 0.1202 K/s and 0.1198 K/s for the first and the second DDAs, respectively. From (1) and Table 1, the heat capacities are approximately \( C(150) = 1.2 J/K \) for both DDAs.

5. Evaluation of heat load of operational DDAs

The procedure described above, of evaluating the heat load of a simulation DDA at any arbitrary temperature, is time consuming and is not really feasible in mass production environment for operational DDAs, which are usually not equipped with an auxiliary shunt. It is important to note that for a given DDA design, the aggregate heat capacity is more or less consistent. The variations of the heat load may be due to a vacuum loss (outgassing, release of trapped contamination or insufficient mechanical integrity) and/or deviation of the cold finger wall thickness from its nominal value. It goes without saying, all these factors do not affect much the heat capacity, which may be evaluated using a pilot simulation DDA or calculated using an exact knowledge of the individual heat capacities of the DDA parts involved. A prior knowledge of the aggregate heat capacity \( C(T) \) and an evaluation of the slope of a natural warm up rate, \( T'_0 \), with no added heat load, allows for a simple calculation of the heat load using equation (1).

Figure 6. Heat load evaluation using thermal short
In case the pilot Simulation Dewar does not exist and prior information on the above mentioned heat capacity is not available, we can advise on slightly different procedure, where trial heat load is applied not by activating the auxiliary shunt, but by artificially shortening the cold finger at its warm end.

Figure 6.a shows the setup schematic. The heat conductive ring made of Copper (encircled) has been placed and thermally interfaced with the warm side of the said cold finger. This resulted in a thermal shortening of the cold finger without adding heat capacity. Assuming good thermal contact between cold finger and the said trial ring, from the cold finger geometry (nominal OD and wall thickness) along with material heat conductance at room temperature, we may evaluate additional heat load.

Figure 6.b shows typical warm-ups of DDA with (Short) and with no (Reference) heat conductive ring.

From Figure 6.b, effective shortening the cold finger by 3.8 mm resulted in an additional heat flow \( H = 12.5 \text{mW} \) through the cold finger, the distal (cold) and proximal (warm) ends of which are at 150\( K \) and 300\( K \), respectively. This manifested itself in 8.6% increase of the warm-up rate from \( T'_0 = 0.1236 \text{K/s} \) to \( T' = 0.1343 \text{K/s} \). From (3), total self-heat load is, therefore, 145.8 \( \text{mW} \), which is quite close to the above evaluated value.

As explained above, better evaluation accuracy may be obtained using a number of trial rings of different lengths providing for different, but controllable cold finger shortening and, therefore, for different trial added heat load. Based on this data, the DDA heat capacity may be further evaluated.

**Conclusions**

The proposed techniques of warm-up multi-slope calorimetry are free of the drawbacks typical of traditional isothermal boil-off calorimetry and allow an accurate evaluation of the DDA heat load at any temperature without using "exotic" cryogenic coolants and mass flow meters.

Along with these lines, the suggested technique allows for a simple assessment of the heat load in a mass production environment.

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