

Flat-plate boiloff calorimeters for testing of thermal insulation systems

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Abstract. Cryostats have been developed and standardized for laboratory testing of thermal insulation systems in a flat-plate configuration. Boiloff calorimetry is the measurement principle for determining the effective thermal conductivity (k_e) and heat flux (q) of test specimens under a wide range of actual conditions. Cryostat-500 is thermally guarded to measure absolute thermal performance when calibrated with a known reference via an adjustable-edge guard ring. With liquid nitrogen as the energy meter, the cold boundary temperature can be adjusted to any temperature between 77 K and approximately 300 K by the interposition of a thermal resistance layer between the cold mass and the specimen. A low thermal conductivity suspension system has compliance rods that adjust for specimen thickness and compression force. Material type, thickness, density, flatness, compliance, outgassing, and temperature sensor placement are important test considerations, and edge effects and calibration techniques for the apparatus are crucial. Over the full vacuum pressure range, the thermal performance capability is nearly four orders of magnitude. The horizontal configuration provides key advantages over the vertical cylindrical cryostats for testing at ambient pressure conditions. Cryostat-500's design and test methods, other flat-plate boiloff calorimeters, and results for select thermal insulation materials (composites, foams, aerogels) are discussed.

1. Introduction

Several cryostat instruments have been developed and standardized for laboratory testing of thermal insulation systems in a flat-plate configuration. Boiloff calorimetry is the measurement principle for determining the effective thermal conductivity (k_e) and heat flux (q) of a test specimen under a wide range of real-world conditions. Cryostat-500 is thermally guarded by a separate cryogen chamber to provide absolute thermal performance data. Absolute (primary) instruments produce the data by which comparative (secondary) instruments can be calibrated. The larger Cryostat-600, with its structural element option, is also a guarded design. Both are similar in design and operation and will be jointly referred to as Cryostat-500.

Cryostat-400 takes the same-size test specimen as Cryostat-500 but is a comparative type of instrument, without a separate cryogen guard chamber. The Macroflash Cup Cryostat is a comparative, benchtop-size instrument for thermal conductivity testing of materials, from aerogel insulation to carbon composites. The Macroflash is designed to test in ambient pressure environments with different purge gases and under compression loads from zero up to approximately 100 kPa. The basic characteristics of four different flat-plate boiloff calorimeters are given in table 1.



Table 1. Insulation test cryostat instruments: flat-plate configuration.

Instrument	Type	Test Specimen Size	ASTM Test Standard	Environment	Heat Flux (W/m ²)
Cryostat-500 (3 units)	Absolute	203 mm diameter, up to 40 mm thick	C1774 Annex A3	Full range vacuum 77 K–353 K	0.4–400
Cryostat-600 (1 unit)	Absolute w/structural element option	305 mm diameter, up to any thickness	C1774 Annex A3	Full range vacuum 77 K–353 K	0.4–400
Cryostat-400 (2 units)	Comparative	203 mm diameter, up to 40 mm thick	C1774 Annex A4	Full range vacuum 77 K–353 K	4–400
Macroflash Cup Cryostat (3 units)	Comparative	76 mm diameter, up to 7 mm thick	C1774 Annex A4	No vacuum 77 K–353 K	80–1000

Both cylindrical and flat-plate cryostats have been standardized for laboratory operation [1]. Although cylindrical configurations are better at minimizing unwanted lateral heat transfer, flat-plate configurations offer a number of potential advantages regarding the test specimens, including (1) the ability to handle small test specimens (when only a small piece can be obtained), (2) compression loading capability, (3) specialized ambient pressure testing with different purge gases, and (4) greater relevance to end-use application. Powder-type insulation testing is more difficult on flat-plate calorimeters, but has been done successfully. The flat-plate cryostats are also easier to adjust for different cold boundary temperatures (CBTs) by the placement of an intermediary material on the cold-side surface.

2. Test apparatus design and setup

The primary instrument for flat-plate boiloff calorimeter testing is Cryostat-500 [2]. The technology is built on the prior technology of flat-plate calorimeters developed by the Cryogenics Test Laboratory [3–5]. This apparatus, which accepts round, 203-mm diameter specimens, is guarded on the top and around its perimeter and can be easily adjusted to measure absolute thermal performance. The basic schematic and a photograph of the overall arrangement, including the workstand, are shown in figure 1. An adjustable-edge guard ring enables calibration with a known material. With the use of liquid nitrogen (LN₂) vaporization as the energy meter, the CBT can be adjusted to any temperature between 77 K and approximately 300 K by the interposition of a known thermal resistance layer between the cold mass and the test specimen or by the use of intermediate temperature sensors.

The cold mass assembly, comprising the heat measurement vessel and thermal guard vessel, is suspended from the vacuum chamber lid as shown in figure 2. The cold mass assembly uses a double wall as an interface between the side wall of the test chamber and the guard chamber, which precludes direct solid-conduction heat transfer between the two liquid volumes. Such isolation is critical for thermal stability and the fine equilibrium necessary for an accurate boiloff measurement.

The right-hand view in figure 1 shows the vacuum pumping system, vacuum instrumentation, mass flow meters, and other ancillary equipment in the laboratory setting. A low thermal conductivity suspension system includes compliance rod assemblies that can be adjusted for the thickness of a given test specimen. Test specimens with thicknesses between approximately 3 mm and 40 mm can be tested. For rigid-type materials, particularly in vacuum, flatness is critical for ensuring good thermal contact on the cold side of the specimen. The suspension system can be readily configured for rigid or fully compressible materials. Compression loading up to approximately 100 kPa (15 psi) can also be applied to the test specimen through the suspension system as required.

Cryostat-500 includes an external heating system for bakeout and a heating plate system for control of the warm boundary temperature (WBT). Two custom-designed funnel filling tubes (7.93-mm outside diameter) interface with the two LN₂ feedthroughs (12.7-mm outside diameter) provide the means for cooldown, filling, and replenishment by pouring from a small nonpressurized dewar. The filling tubes are removed when not being used. Connected to the top ports of the LN₂ feedthroughs are plastic tubing assemblies that route the boiloff flow rates from both the test chamber and the guard chamber to their

respective mass flow meters. Vacuum instrumentation includes a pair of MKS Baratron® capacitance manometers, an MKS Granville-Phillips® Micro-Ion® gage, and a Pfeiffer Compact Full-Range™ gage for backup. The vacuum pumping system includes a directly connected turbopump and a separately plumbed mechanical pump. A gaseous nitrogen (GN_2) supply system provides purging and residual gas pressure control to vacuum levels as low as 5×10^{-5} torr. All instruments are connected to a customized National Instruments LabVIEW data acquisition system for recording and monitoring.

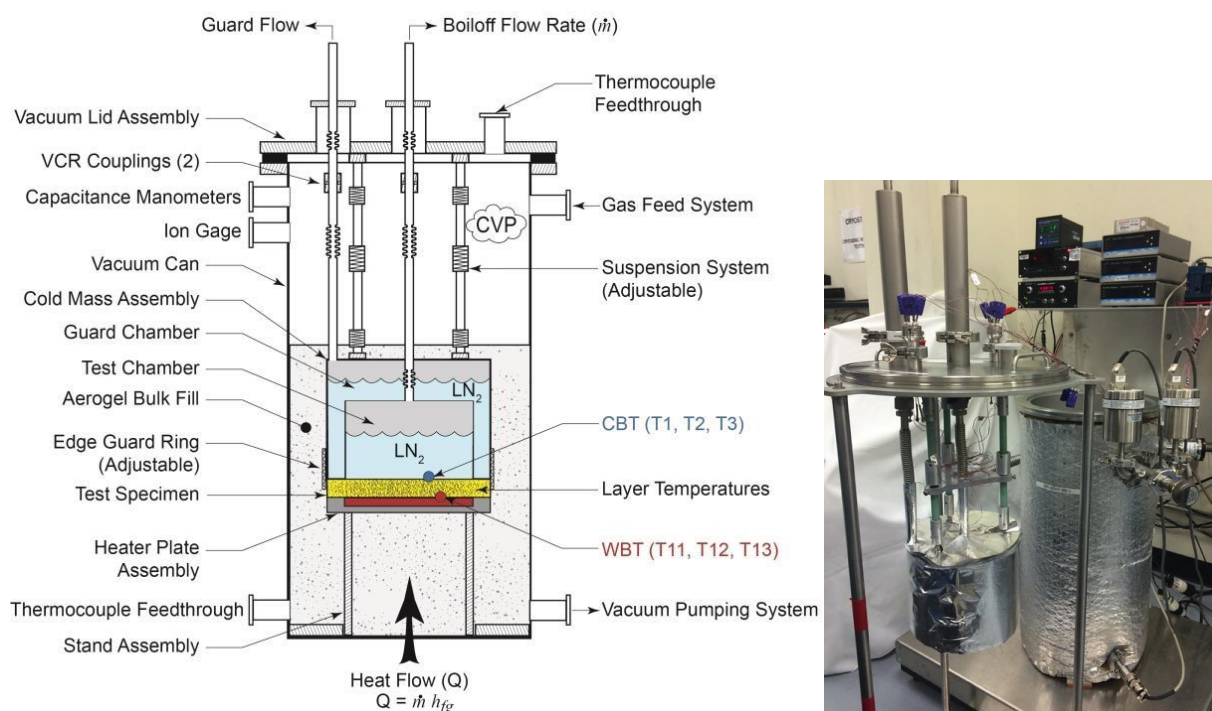


Figure 1. Cryostat-500: basic schematic (left) and overall arrangement with workstand (right).

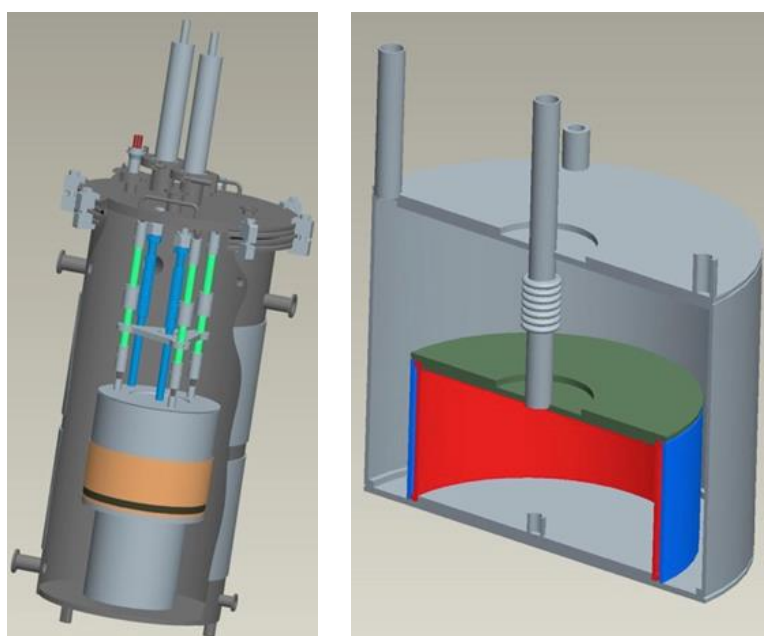


Figure 2. Overall system (left) and cold mass assembly (right).

3. Testing methodology

The principle of heat rate measurement for Cryostat-500 is based on LN₂ boiloff calorimetry, following the guidelines of ASTM C1774, Annex A3 [6]. This standard covers the terminology, rationale, and approaches to thermal performance testing of cryogenic insulation systems while outlining test methods in both boiloff calorimetry and electrical power based categories. The steady-state heat flow rate (\dot{Q}) is the basis for calculating the thermal properties, including effective thermal conductivity (k_e) (or system thermal conductivity, k_s) and heat flux (q). The test specimen may consist of a single material, one type of material in several discrete elements, or a number of different materials working in a specialized design configuration. In reality, a test specimen is always a system, either a single material (with or without inclusion of a gas) or a combination of materials in different forms. Any thermal performance test result is considered along with other results. Therefore, the parameters of the test, the manner in which the test is set up and performed, and the key terminology for the reporting of data are crucial [7].

Calculations of k_e are highly sensitive to the thickness of the test specimen. The thickness, as tested, is carefully measured or calculated, and any assumptions are explained. Materials can be monolithic, layered slabs, blankets, or layered blankets. Bulk-fill materials can also be tested with the Macroflash Cup Cryostat. The adjustable cold mass suspension system allows for the proper set up and testing of rigid or soft materials. From the measured thickness of a given specimen, the suspension system is precisely configured for the desired test thickness. Springs are used for rigid materials to provide a built-in compliance while spacers are used for soft materials to establish a fixed gap (thickness).

The steady-state condition is reached when the boiloff flow rates from both chambers are stabilized, the temperature profile through the thickness is stabilized, and the liquid level in the guard chamber is at least 50% full (that is, covering the top surface of the test chamber). A stable state of the system is indicated by slight oscillation of the temperature sensors with no overall trend in their average value [6]. The liquid level inside the test chamber can be any level between 0% and 100%, which greatly simplifies the overall test operation. The total test duration may be hours to days, depending on the level of heat flow involved. All test data are ordered into standardized files, by test series, for processing and archival purposes.

The variation in boiloff flow rate is primarily determined by the states of the liquid masses in the two chambers. In general, the liquid can be stratified, mixed, or in transition. For steady-state measurement to be achieved, all liquid masses must be stratified and stable, a condition reached only by inherent design of the cold mass assembly. Other important factors in boiloff flow rate stability are the regional variations and twice-daily fluctuations in atmospheric pressure. Without systematic controls to counteract this effect, at very low heat flux rates, these fluctuations can periodically and substantially influence the results. The system therefore includes a backpressure control system to provide a more even ambient pressure.

An advantage to be noted for boiloff calorimetry is the inherently wide range of temperatures involved. A given test condition can be arranged to achieve any set of cold and warm boundary temperatures within the maximum range. The large temperature difference can be subdivided to obtain additional information that can subsequently be used for thermal modeling and analysis or for research and development of products. Furthermore, recent work has shown that multiple data points can be obtained from a single test by the intermediate placement of temperature sensors through the thickness of the test specimen [8]. Standard boundary temperatures are 293 K (warm) and 78 K (cold), but any two temperatures between 78 K and approximately 353 K can be set up. In any case, the heat transfer principle remains the same: heat flows as a function of the temperature difference, not as a function of temperature.

Different materials and varied test objectives require an appropriate combination of apparatus and method. The type, thickness, density, flatness, compliance, and outgassing of the material and the placement of temperature sensors are important considerations. Edge effects and calibration techniques for the flat-plate apparatus are crucial in design and test operation. Over the full vacuum pressure range, the thermal performance capability is shown to be nearly four orders of magnitude. The horizontal configuration provides key advantages over the vertical cylindrical cryostats for testing at ambient pressure conditions as the convection effects in the range of about 100 torr to 760 torr are favorably stratified in uniform horizontal layers in flat plate systems. Conversely, the convection currents in vertical cylindrical systems can be magnified and lead to problems with controlling the warm boundary heat inputs. For testing very low

density materials (below about 32 kg/m³) under ambient pressure conditions, use of the flat plate cryostat is highly recommended.

4. Uncertainty analysis

The rate of heat transfer through the insulation system and into the bottom of the test chamber of the cold mass assembly (Q) is directly proportional to the LN₂ boiloff flow rate (V), as given by (1),

$$Q = V_{STP} \rho_{STP} h_{fg} \left(\frac{\rho_f}{\rho_{fg}} \right) \quad (1)$$

where STP = Standard Temperature and Pressure (0 °C and 760 torr) and the right hand term is the density correction, if any, between the liquid and the saturated liquid conditions. The value of k_e is determined from Fourier's law of heat conduction through a flat plate, as given by (2).

$$k_e = \frac{Qx}{A_e \Delta T} = \frac{4Qx}{\pi d_e^2 \Delta T} \quad (2)$$

For flat-plate geometries, the effective heat transfer area (A_e) is the area defined by the median line of the test chamber side wall. The heat flux (q) is calculated by dividing the total heat transfer rate by the effective area for heat transfer (A_e). The symbols and sources of error attributable to uncertainty for calculating thermal properties from boiloff testing with Cryostat-500 are given in table 2.

Table 2. Symbols and sources of error for the flat plate calorimeter, Cryostat-500.

Symbol	Description	Unit	% Error
V	Volumetric flow rate (boiloff) at STP	m ³ /s	0.500
ρ	Density of GN ₂ (boiloff) [0.0012502 g/cm ³]	kg/m ³	n/a
h_{fg}	Heat of vaporization	J/g	2.00
x	Thickness of insulation specimen	m	3.94
d_e	Diameter, effective heat transfer	m	1.60
A_e	Area, effective heat transfer area	m ²	n/a
ΔT	Temperature difference (WBT – CBT)	K	0.981

The error introduced by each parameter is taken into account for the calculation of the total error, in accordance with the “Error Analysis of Experiments” equation listed in Perry's Chemical Engineers' Handbook [9]. The total uncertainty in k_e is calculated to be 4.8% for the Cryostat-500. Likewise, the total uncertainty in heat flux q is 4.7%. Measurement of the boiloff flow rate is made using a mass flow meter that automatically compensates for gas densities in the range of 273 K to 323 K and makes the density error not applicable. The mass flow meter outputs the data in terms of a volumetric flow rate at STP.

The overall error of k_e is estimated for the worst-case situation. Thickness is the largest source of uncertainty and must be handled carefully, particularly for specimens thinner than 10 mm. In addition, fit-up is crucial for good thermal contact to be maintained through the usual thermal cycles and shrinkage associated with testing. Heat of vaporization is the next largest source of uncertainty, typically 2% for LN₂ [10]. The nominal value for the heat of vaporization is 199.1 J/g based on a saturation pressure of approximately 765 torr (0.1 psig). Because the test specimen is located at the bottom of the test chamber, and that chamber is surrounded by cryogen, ullage vapor heating is not a consideration [11]. In most testing situations, for a given series of tests, the overall repeatability is demonstrated to be within 2%.

5. Example test results and discussion

The results of tests performed by the Cryogenics Test Laboratory with the flat-plate boiloff calorimeters are summarized in table 3. For flat-plate calorimeters, 520 material specimens have undergone more than 2,100 individual tests, representing over 6 years of continuous boiloff run time.

Table 3. Summary of flat-plate cryostat testing by number of material specimens and tests.

Apparatus	Number of specimens	Number of tests	Hours of run time ^a
Cryostat-500	81	~729	~21,870
Cryostat-600	44	~88	~7,040
Cryostat-400	142	~1,056	~25,344
Macroflash	253	~263	~1,052
Total	520	~2,136	~55,306

^aTime does not include that required for evacuation and heating, purging, cooldown, or warmup.

Results for select thermal insulation materials, including composites, foams, and aerogels, are presented in Figures 3 through 5. Figure 3 (left) presents the results of an individual test of a spray-on foam insulation (SOFI) material (BX-265), and Figure 3 (right) presents the results of the complete test series for the same test specimen. Details of these and many other materials are given in the literature [6-7, 12]. The boiloff data for the five-run test of a carbon fiber composite panel at 760 torr CVP are shown in Figure 4. This test specimen was a six-layer stack of panels, with temperature sensors between all layers to enable analysis of the variation of thermal conductivity with mean temperature [7]. Figure 5 presents an example test of a multilayer insulation (MLI) system (11 layers of Mylar/net) showing a stable boiloff flow rate for more than 30 hours.

The large database of thermal performance data can provide a foundation for the development of future standards in insulation material specifications and installation practices. For example, a set of 20 cellular glass specimens (each 203 mm in diameter and 25 mm thick) is available for industry round-robin testing and future precision and bias studies. A program of 45 tests of 14 cellular glass disks using the Cryostat-400 showed variability of approximately 3.0% [13].

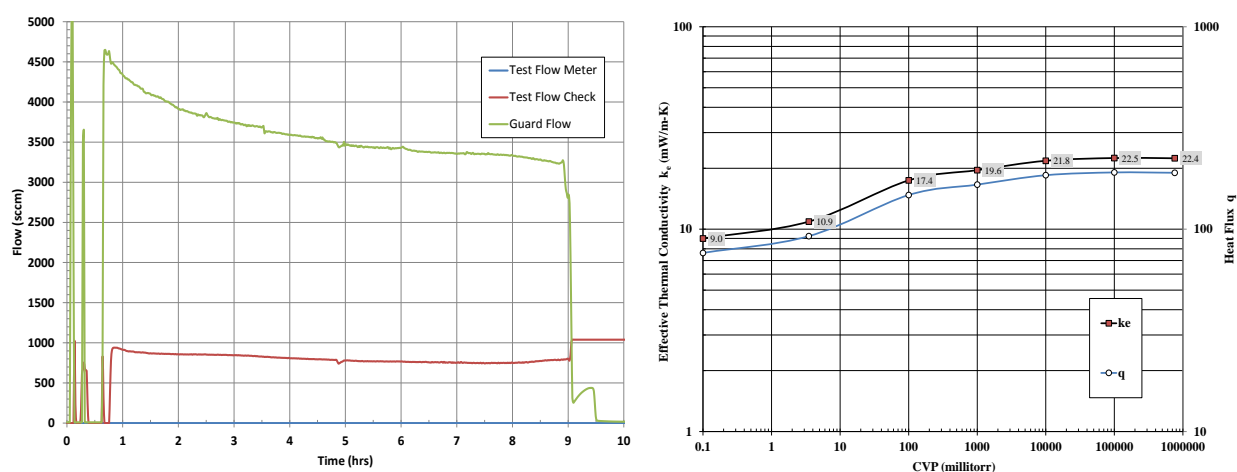


Figure 3. Example Cryostat-500 test results for foam material test specimen G157: 25 mm-thick SOFI BX-265 aged 10 years; boundary temperatures of 293 K/78 K; the residual gas is nitrogen. Boiloff flow rate for a test at 760 torr CVP (left); test data summary, variation of k_e and q with CVP (right).

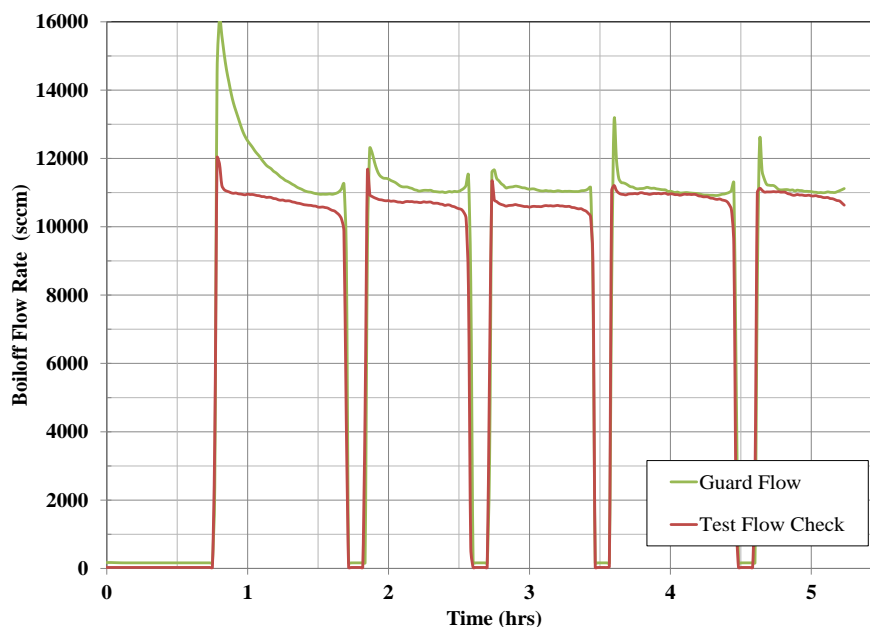


Figure 4. Example test result from carbon fiber composite panel test at 760 torr CVP using Cryostat-500: boiloff flow rates. Test specimen: six-stack of carbon panels; 13 mm thickness; boundary temperatures of 293 K/78 K; the residual gas is nitrogen.

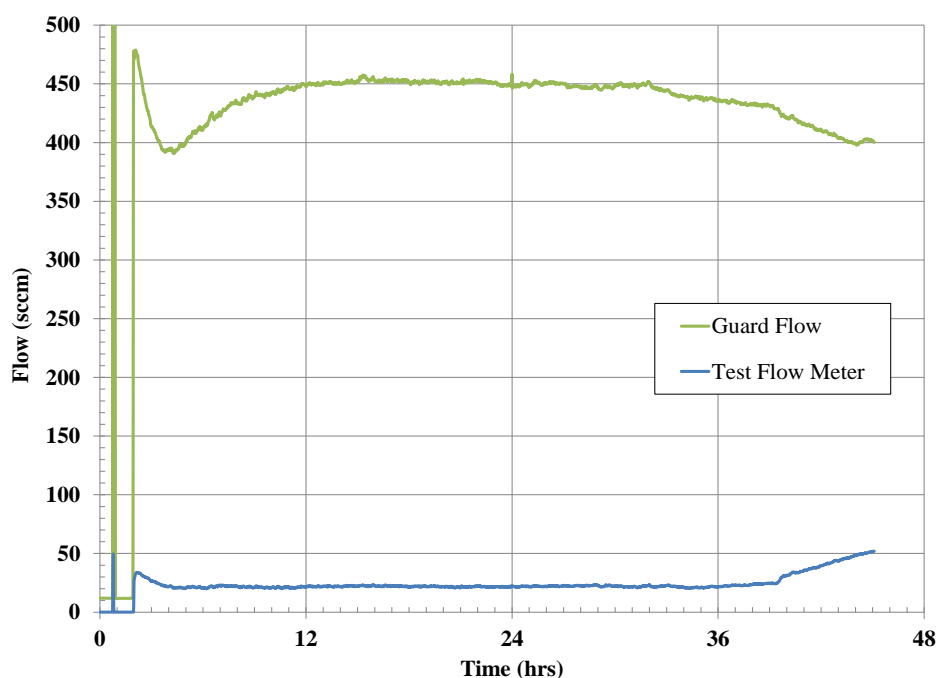


Figure 5. Example test result from MLI test at 1×10^{-5} torr CVP using Cryostat-500: boiloff flow rates. Test specimen: 11 layers (MLI: Mylar and polyester net); boundary temperatures of 293 K/78 K; the residual gas is nitrogen.

With Cryostat-500, a total heat flow on the order of a few milliwatts is achievable because of the thermal stability of the stratified-liquid approach of the cold mass designs in combination with the unique ambient backpressure control system. For example, the test results of a thick MLI blanket are 10 mW with a corresponding flow rate of 4 sccm. In another test series for a structural composite panel, heat loads of up to 100 W were successfully measured to demonstrate a measureable range of over four orders of magnitude in one instrument.

Another advantage of boiloff calorimetry using Cryostat-500 and the other flat-plate calorimeters is the capability for testing novel materials and combinations of materials. These novel composites, multifunctional materials, and thermal management systems are sometimes composed of both high thermal resistance and high thermal conducting materials. Comparing test results of MLI systems between flat plate and cylindrical calorimeters is also of interest. Boiloff calorimetry provides the means to reliably test such systems under the prescribed representative conditions for the targeted engineering application.

6. Conclusion

Based on boiloff calorimetry, new flat-plate cryostats and methods for testing thermal insulation systems have been successfully developed by the Cryogenics Test Laboratory at NASA Kennedy Space Center over the last 15 years. These boiloff instruments (or cryostats) are applicable to a wide range of materials and test conditions. Test measurements are generally made at large temperature differences (boundary temperatures of 293 K and 78 K are typical) and include the full vacuum pressure range. Results are generally reported in effective thermal conductivity (k_e) and mean heat flux (q) through the insulation system. The Cryostat-500 instrument, with its thermal break cold mass design, stratified liquid approach, and single port filling/venting method, provides an effective and reliable way to characterize the thermal performance of materials under subambient conditions. An adjustable suspension system provides for the testing of rigid or soft materials, with or without compressive loading. Proven through thousands of tests of hundreds of different material specimens, these flat-plate cryostats have supported a wide range of aerospace, industry, and research projects.

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