Below Ambient and Cryogenic Thermal Testing

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Introduction / Measurement of Heat Flow

Thermal insulation systems operating in below-ambient temperature conditions are inherently susceptible to moisture intrusion and vapor drive toward the cold side. The subsequent effects may include condensation, icing, cracking, corrosion, and other problems. To test for thermal performance of a cold insulated pipeline under relevant conditions we must be able to measure the heat (energy) flow. But what is energy? No one really knows, but whatever it is, energy is the same at temperatures above ambient and below ambient. And we do know one thing: it always flows from the hotter side to the colder side. No one has ever seen a joule (unit of energy) or a bucket of joules, but we do know from experience that hot water put into a cold bucket will lower the energy (heat) of the bucket. We can get an indication of the change in energy by measuring the bucket's temperature, but we cannot measure the energy.

For below-ambient cases, the heat flow rate can be measured directly by the technique of boiloff calorimetry. Although it is a misnomer (there is no such thing as a "calorie-meter"), the boiloff fluid such as liquid nitrogen (LN_2) provides a direct measurement of the rate of heat flowing through an insulation test specimen. The *energy-going* is the heat flow rate (Q), or power, in units of joules per second (W). The boiloff fluid vaporizes at a rate according to the rate of heat flowing through the test specimen. The boiloff flow rate is therefore directly proportional to the heat flow rate by the enthalpy of vaporization (h_{fg}) as shown below. This simple equation is the essence of cryogenic boiloff calorimetry.

 $Q = \dot{m} \times h_{fg}$

Q = heat flow rate (W) \hat{m} = mass flow rate (g/s) h_{fg} = enthalpy of vaporization (J/g) = 199 J/g for LN₂

Boiloff Calorimetry for Below-Ambient Thermal Testing

Cryogenic boiloff calorimetry is a steady-state method using a static, fixed volume of boiloff fluid. The boiloff fluid can be thought of as the "energy meter" for direct measurement of heat flow. The most typical boiloff fluid is liquid nitrogen (LN2), but other fluids such a Freon or even water have been used. The LN2 is saturated at ambient pressure for stability. The steady-state thermal equilibrium provides a heat flow rate that is the same through all layers of a test specimen. This fundamental principle is a key advantage over electrical-based thermal test methods as complex, non-isotropic, non-homogeneous, or

layered insulation systems can be tested as directly as uniform slabs of isotropic, homogeneous materials. How? Boiloff calorimetry measures the heat flow rate, not just temperature or voltage. The temperature range for current standard capabilities is from about 100 °C (373 K) down to -196 °C (77 K). Typical is a large temperature difference (Δ T) that be more representative of the real-world conditions. With a large Δ T to work with, thermal conductivities at different mean temperatures (T_m) can also be calculated. In this way, multiple test points can be obtained from a single test.

Configurations of boiloff calorimeter systems can be flat plate or cylindrical. A horizontal cylindrical design is used for pipe insulation. The thermal measurement method can be absolute or comparative depending on the test objectives. The new standard published by ASTM International, C1774 – *Standard Guide to Thermal Performance Testing of Cryogenic Insulation Systems*, includes three different approaches (boiloff or electrical power) and six different apparatuses (four boiloff). Section X1.2 states that the approaches, techniques, and methodologies can be adapted for use in the cryogenic thermal performance testing of pipelines: cryogen boiloff (static) or flow-through (dynamic). Another new and related standard of ASTM International is ASTM C740 – *Standard Guide for Evacuated Reflective Cryogenic Insulation*. This standard includes thermal performance data for multilayer insulation (MLI) and other cryogenic insulation systems, foams, aerogels, and bulk-fill materials operating between ambient and cryogenic temperatures. Key definitions from ASTM C1774 and ASTM C740 are listed as follows:

- Effective thermal conductivity (k_e) the thermal conductivity through the total thickness of the insulation test specimen between the reported boundary temperatures and in a specified environment (mW/m-K). The insulation test specimen may be one material, homogeneous non-homogeneous, or a combination of materials.
- System thermal conductivity (k_s) the thermal conductivity through the total thickness of the insulation test specimen and all ancillary elements such as packaging, supports, getter packages, enclosures, etc. (mW/m-K).
- Heat flow rate (Q) quantity of heat energy transferred to a system in a unit of time (W).
- Heat flux (q) heat flow rate, under steady-state conditions, through a unit area, in a direction perpendicular to the plane of the thermal insulation system (W/m²).

Below-Ambient Testing

By placing a first insulation layer on the cold side, the cryogenic boiloff method can be used for any belowambient temperature application.



Figure 1. Below-ambient temperature range.

Establish a steady warm boundary temperature (WBT) on an outer surface. Establish a steady cold boundary temperature (CBT) on an inner surface. By placing a first insulation layer on the inner cold boundary, the cryogenic boiloff method is used for a wide range of below-ambient temperature applications. Three main phases: 1) Cooldown/Fill, 2) Cold Soak, and 3) Test (steady-state boiloff). After thermalization, the heat flow rate (Q) through the insulation is constant and the same through all layers of the insulation system. Heat flow rate: Q = boiloff flow x heat of vaporization.

Flat plate boiloff instruments are listed in Table 1. The Cryostat-500 insulation test instrument, per ASTM C1774, Annex A3, provides the following capability: testing 204-mm diameter, 25-mm thick specimens under representative-use conditions; direct energy rate measurement by LN2 boiloff calorimetry; and reliable thermal conductivity data for non- homogenous, non-isotropic thermal insulation systems. A Cryostat-500 instrument is shown in operation in Figure 2.

			ASTM Test		Heat Flux
Instrument	Туре	Test Specimen Size	Standard	Environment	(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

Table 1. Flat plate boiloff instruments for insulation testing.



Figure 2. Cryostat-500 during test specimen installation (left) and cooldown operation (right).

Cylindrical boiloff instruments are listed in Table 2. The Cryostat-100 insulation test instrument, per ASTM C1774, Annex A1, provides the following capability: testing 1-meter long, 218-mm diameter specimens under representative-use conditions; direct energy rate measurement by LN2 boiloff calorimetry; and reliable thermal conductivity data for non- homogenous, non-isotropic thermal insulation systems. A Cryostat-100 instrument is shown in operation in Figure 3.

Instrument	Туре	Test Specimen Size	ASTM Test Standard	Environment	Heat Flux (W/m ²)
Cryostat-100 (1 unit)	Absolute	1 m long, 167 mm diameter, up to 50 mm thick	C1774 Annex A1	Full range vacuum 77 K–353 K	0.2–200
Cryostat-200 (2 units)	Comparative	0.5 m long, 132 mm diameter, up to 50 mm thick	C1774 Annex A2	Full range vacuum 77 K–353 K	1–200
Cryostat-P100 (1 unit)	Absolute	12.2 m long, 25 - 88 mm diameter up to 200 mm OD	C335	No vacuum or vacuum-jacket 77 K–353 K	4–400
Cryostat-P200 (future)	Comparative	1.8 m long, 33 mm diameter, up to 110 mm OD	C335	No vacuum 77 K–353 K	100–500

Table 2. Cylindrical boiloff instruments for insulation testing.



Figure 3. Cryostat-100 during test specimen installation (left) and cooldown operation (right).

Below-Ambient Insulated Pipe Testing

Of particular interest is the application of boiloff calorimetry to the testing of pipe insulation for cold applications. The potential revision of ASTM C335 to include below-ambient method based on cryogenic boiloff is under review ASTM International's Committee C16 on Thermal Insulation. An apparatus and method for thermal performance testing of cryogenic piping systems has been established by the

Cryogenics Test Laboratory at NASA Kennedy Space Center. This apparatus, called Cryostat-P100, provides heat leak data for pipelines under "real world" conditions and a standardized thermal test for low-temperature piping systems. A comparative type, bench-top cold pipe tester, Cryostat-P200, is currently under development. Current research work of the Cryogenics Test Laboratory includes testing of below-ambient thermal insulation materials/systems for energy-efficient transfer lines and piping systems.

The cold pipe tester, Cryostat-P100, is a liquid nitrogen boil-off test apparatus, thermally guarded for absolute heat leak rate measurements. This apparatus has provision for two insulated test pipes of the following dimensions: 12-m long (40-feet) and up to 3-inch diameter pipe size (NPS). The insulated test pipes are tested in parallel. The piping can be continuous or segmented and rigid or flexible. Components such as valves, expansion joints, ports, and so forth can also be incorporated, if desired, as part of the test pipe configuration.

The system provides a 3° slope to provide a high point tap for the boiloff flow rate. An optional externallyapplied heater wrap provides control of warm boundary temperature control. The anchors of the system are the upstream and downstream cold boxes as depicted in Figure 4. The cold boxes are filled with liquid nitrogen (LN2) supplied from a normal low-pressure supply tank. The downstream cold box is filled from the upstream cold box through a vacuum-jacketed pipeline located in the middle between the two cold boxes. The test pipes are supplied with ambient pressure saturated LN2 from a heat exchanger coil routed through upstream cold box. Temperature sensors (Type E thermocouples) are usually installed in the following locations: length-wise (top, side, and bottom), through thickness of insulation, and terminations.



Figure 4. Basic schematic of the cold pipe tester Cryostat-P100.

The thermal end guards and test pipe terminations are shown in Figure 5. These terminations are adaptable to a variety of different end connections. The terminations are thermally guarded by the liquid nitrogen bath of the cold boxes to which they are connected. The connections provide built-in compliance for thermal contraction and ease of installation of the test pipes. The middle line is typically used for downstream cold box supply but can be configured for a third test pipe.

The basic phases of operation are listed as follows: cooldown and fill, cold soak, and boiloff test. Overall views of the cold pipe tester Cryostat-P100 in operation are shown in Figure 6. The test pipes are then refilled and allowed to thermally stabilize for a short period of time after which another test run is

performed. This process is repeated as many times as desired for a complete test series. The system is then drained, purged, and allowed to warm to ambient temperature.



Figure 5. The thermal end guards and test pipe terminations of the cold pipe tester are shown without insulation (left) and with insulation during a test (right).



Figure 6. Overall views of the cold pipe tester Cryostat-P100 in operation.

As an example of the test results, Table 3 presents a summary from a test of a 1.5-inch thick clamshell type insulation system on two 3-inch Nominal Pipe Size pipelines. The two pipes were insulated and installed in the same way and tested in parallel. The boundary temperatures were approximately 293 K (warm side) and 78 K (cold side). The cold soak phase was a minimum of 8 hours followed by multiple test runs within a few hours. The average data for three runs is given in the table.

Parameter	East Pipeline	West Pipeline	
Heat leakage rate (Q)	30.0 W	32.0 W	
Heat leak per unit length (Q/L)	2.45 W/m	2.62 W/m	
Effective thermal conductivity (k _e)	0.95 mW/m-K	1.1 mW/m-K	
Boiloff flow rate	7.25 slpm	7.73 slpm	

Table 3. Example summary of test results from cold pipe tester Cryostat-P100.

Note: slpm = standard liter per minute

Future Plans

Thermal insulation systems operating in below-ambient temperature conditions are inherently susceptible to moisture intrusion and vapor drive toward the cold side. The subsequent effects may include condensation, icing, cracking, corrosion, and other problems. Methods and apparatus for real-world thermal performance testing of below-ambient systems have been developed based on cryogenic boiloff calorimetry. Continuing to develop partnerships among industry, academia, and laboratories are essential for success in producing the needed technical consensus standards for below-ambient testing of pipe insulation.

The newly published standard guide to cryogenic testing, ASTM C1774, provides a foundation for the development of new standard method that is specific to cold pipe testing. Another option is to revise the well-established pipe insulation test method, ASTM C335, to include two parts, one for the current above-ambient test and one for the below-ambient test. A key aspect of the work reported here as well as the ASTM C1774 is that provision is made for both insulation materials and insulation systems. The system aspect for insulated cold pipelines operating in the ambient environment cannot be overstated. The cold pipe tester Cryostat-P100 provides a way of measuring the effective thermal conductivity of insulation materials under relevant conditions as well as determining the real-world thermal performance of insulation systems including installation and environmental factors.

Consistent techniques for establishing different prescribed cold boundary temperatures from about -100 °C up to 0 °C must be verified through testing across the full range of temperatures of interest to industry applications. Advance planning for future round-robin testing of select insulation materials is recommended. Also under development is a comparative type, bench-top cold pipe tester, Cryostat-P200, for 1.5-meter long and 25-mm diameter (nominal) pipe insulation systems.

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