



Standardization in Cryogenic Insulation Testing and Performance Data

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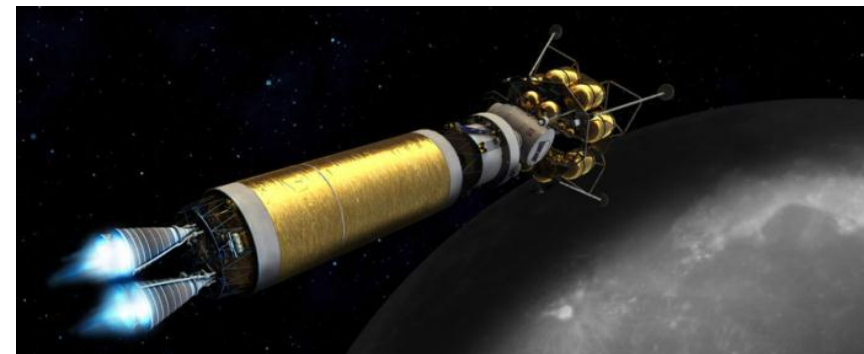
**Cryogenics Test Laboratory
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Background



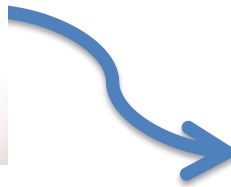
- Thermal insulation provides:
 - energy savings over time,
 - system control,
 - and/or process safety.
- Adverse effects of sudden loss of vacuum, flow induced vibrations, pressure transients, transient structural loads, or inability to achieve certain conditions.
- What thermal insulation system is the best for a given application depends on the operational environment, mechanical design, and insulation materials.
- Economic objectives underscore the technical approach to be taken: *thermal performance must justify the cost.*
- Standard sets of thermal data for are needed to make proper design trade-offs and come up with the best solution.
- But we first need *standard ways of testing and reporting* those data.



Electrical Performance



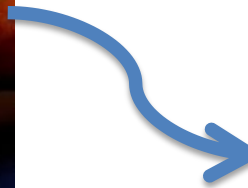
- Circuit boards below are for the brains of the Tesla automobile.
- Measure **electrical resistance** of a wire and then determine the electrical performance of the boards? **No!**



Thermal Performance



- The tank below is for a truck's LNG fuel supply.
- Measure **thermal resistance** of a piece of material and then determine the thermal performance of the tank? **No!**



Motivation for Standardization



- Broader use of cryogenic systems in commercial enterprises worldwide.
- One area in particular is the emerging **cryofuels** usage for transportation.
- From the future passenger car, commercial space launch vehicle, to the floating liquefied natural gas platform, the successful proliferation of these efforts will be partly enabled by the *adequate isolation of liquid hydrogen or liquefied natural gas from the ambient environment*.
- The end-user applications involving transportation necessarily include many **transient operations**.
- Transient, or on and off, cold and warm, operations are a challenge for the design of **robust, safe systems** that meet their overall cost objectives.

Key Terminology

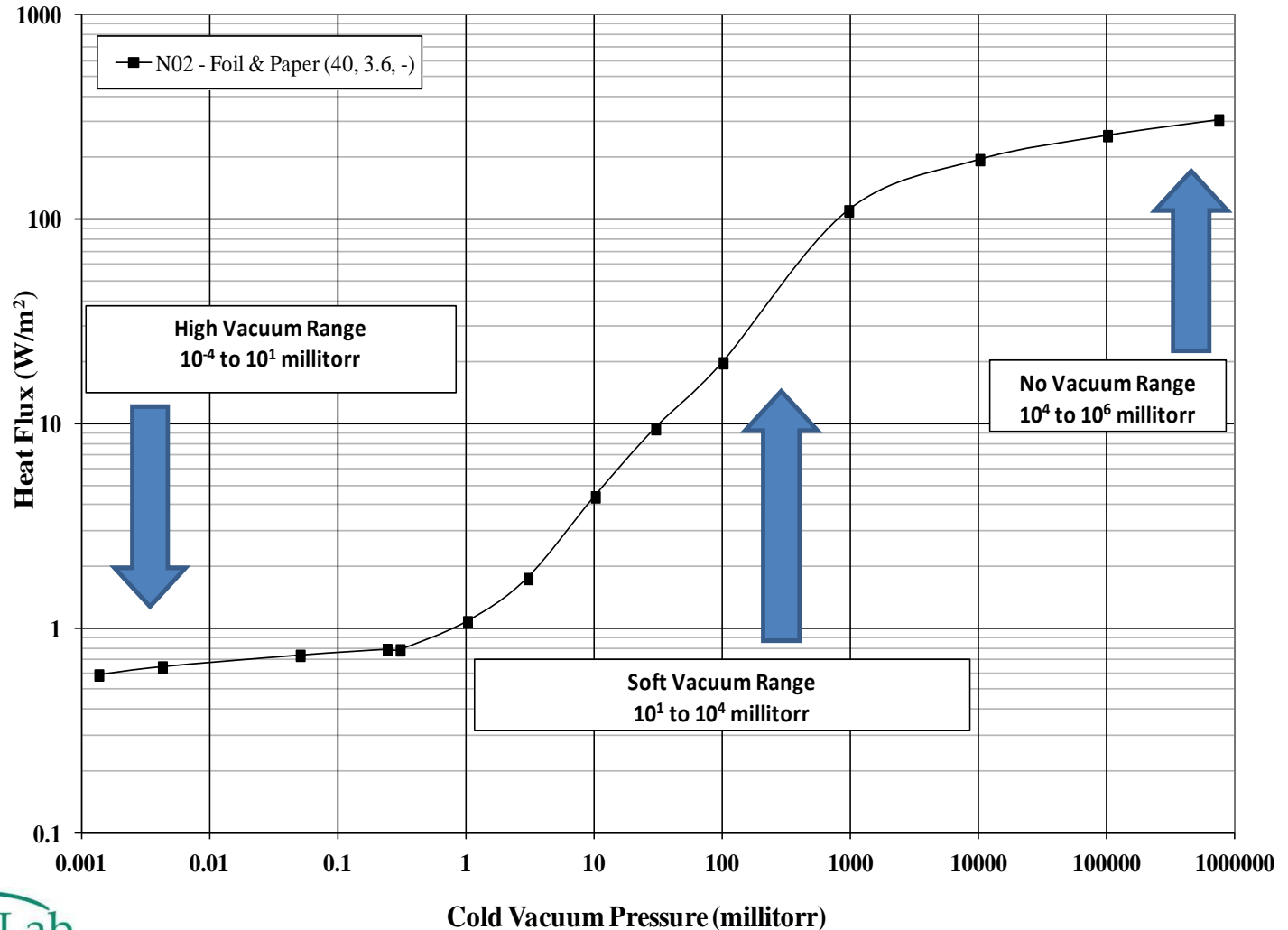


- **Cold boundary temperature (CBT)**—the cold temperature imposed on cold-side surface of the insulation system by the cold mass (K).
 - *May be cooled by a cryogen or a cryocooler.*
- **Warm boundary temperature (WBT)**—the warm temperature imposed on the warm-side surface of the insulation system by the warm mass (K).
 - *May be heated by an electrical heater, liquid bath heat exchanger, or ambient environment.*
- **Cold vacuum pressure (CVP)**—the steady-state vacuum pressure level within the insulation system achieved after cooldown (Pa or millitorr; 1 millitorr = 0.133 Pa).
 - *Can be any pressure from high vacuum to no vacuum, with or without a residual gas.*
- **Warm vacuum pressure (WVP)**—the vacuum pressure level within the insulation system before cooldown.
- **Heat flow rate (Q)**—quantity of heat energy transferred to or from the insulation system in a unit of time (W).
- **Heat flux (q)**—heat flow rate, under steady-state conditions, in a direction perpendicular to the plane of the insulation system (W/m^2).
 - *Based on the effective heat transfer area (A_e)*
- **Effective thermal conductivity (k_e)**—the thermal conductivity through the total thickness of the insulation system between the reported boundary temperatures and in a specified environment ($\text{mW}/\text{m}\cdot\text{K}$).
 - *The insulation system 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 system plus any ancillary elements such as packaging, supports, getter packages, enclosure, outer jacket, etc. ($\text{mW}/\text{m}\cdot\text{K}$).

Cold Vacuum Pressure



Three categories of CVP using the example of heat flux through an MLI system (boundary conditions are 77 K and 293 K with residual gas of nitrogen). The MLI system is aluminum foil and micro-fiberglass paper (40 layers at a density of 3.6 layers per millimeter)





- Through ASTM International, Committee C16 on Thermal Insulation, two new standards were recently published:
 - *ASTM C1774 - Standard Guide for Thermal Performance Testing of Cryogenic Insulation Systems (2013)*
 - *ASTM C740 - Standard Guide for Evacuated Reflective Cryogenic Insulation (2014)*
- Both standards are **comprehensive guides** that provide the necessary terminology, analytical approaches, and reporting requirements for the technology area of cryogenic insulation systems.
- Advances in test apparatus, methods, and materials have provided a foundation for these new standards.

Guide to Cryogenic Thermal Performance Testing



- Covers six different testing approaches/apparatus including:
 - Boiloff calorimetry and electrical power methods.
 - Absolute and comparative methods.
- Applicable to a wide variety of specimens, from opaque solids to porous or transparent materials to composite systems:
 - A “system” may be composed of one or more materials that may be homogeneous or non-homogeneous.
 - Flat, cylindrical, or spherical geometries.
 - Highly anisotropic materials and layered systems such as multilayer insulation (MLI).
- Includes a wide range of environmental conditions:
 - Including various gases and over a range of pressures.
 - Boundary conditions from 4 to 400 K.
 - Environments from high vacuum to an ambient pressure of air or residual gas.
- Laboratory measurement and calculations of the steady-state thermal transmission properties and heat flux.
- A key aspect of this guide is the notion of an insulation system, not an insulation material. *Under the practical use environment of most cryogenic applications, even a single-material system can be a complex insulation system.*



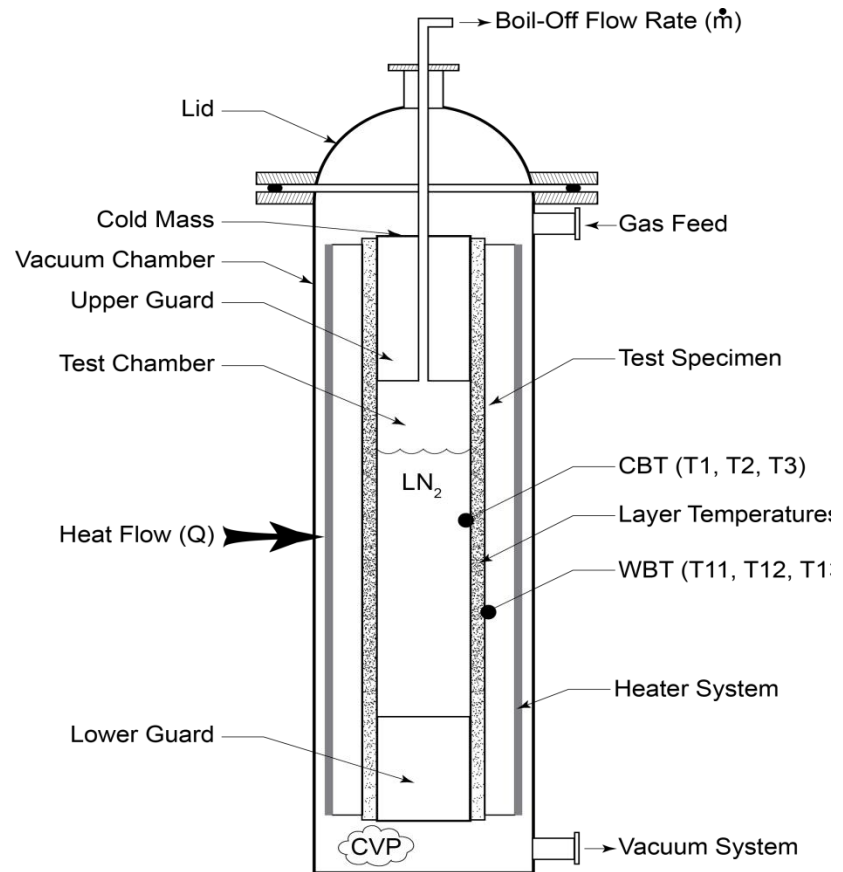
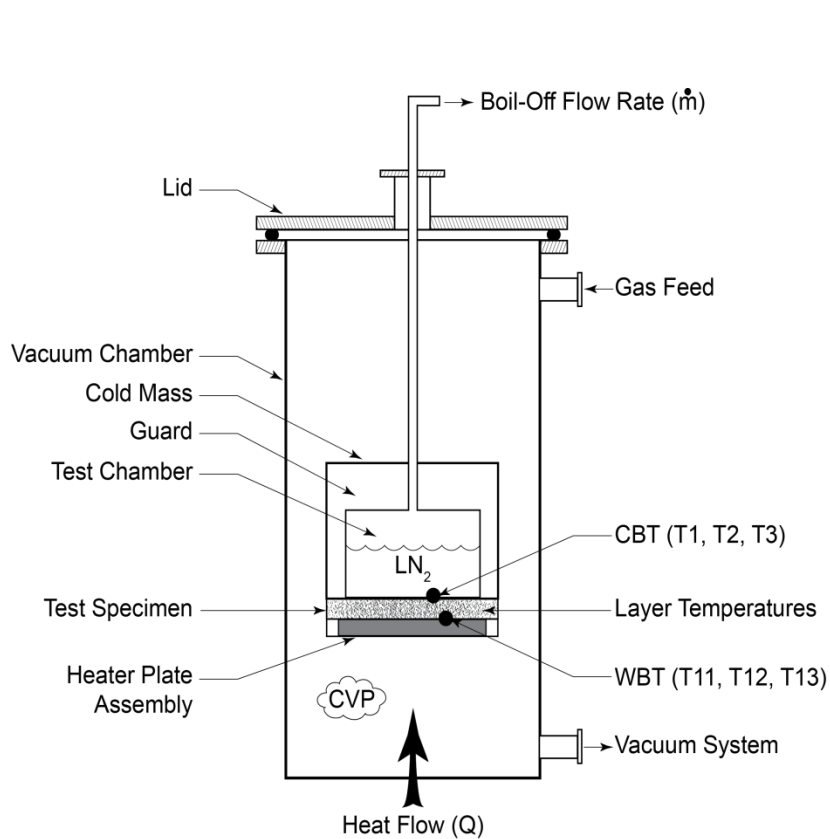
Typical characteristics of boiloff calorimeter instruments (cryostats):

Type	Name	Heat Flux (q) W/m ²	k_e mW/m-K	Specimen Size
Cylindrical Absolute	Cryostat-100	0.1 to 500	0.01 to 60	1-m length; up to 50-mm thickness
Cylindrical Comparative	Cryostat-200	1 to 500	0.1 to 60	0.5-m length; up to 30-mm thickness
Flat Plate Absolute	Cryostat-500	1 to 1,000	0.05 to 100	200-mm diameter; up to 30-mm thickness
Flat Plate Comparative	Cryostat-400	10 to 1,000	0.5 to 100	200-mm diameter; up to 30-mm thickness

Guide to Cryogenic Thermal Performance Testing



General arrangement of a flat plate boiloff apparatus (left) and a cylindrical boiloff apparatus (right):



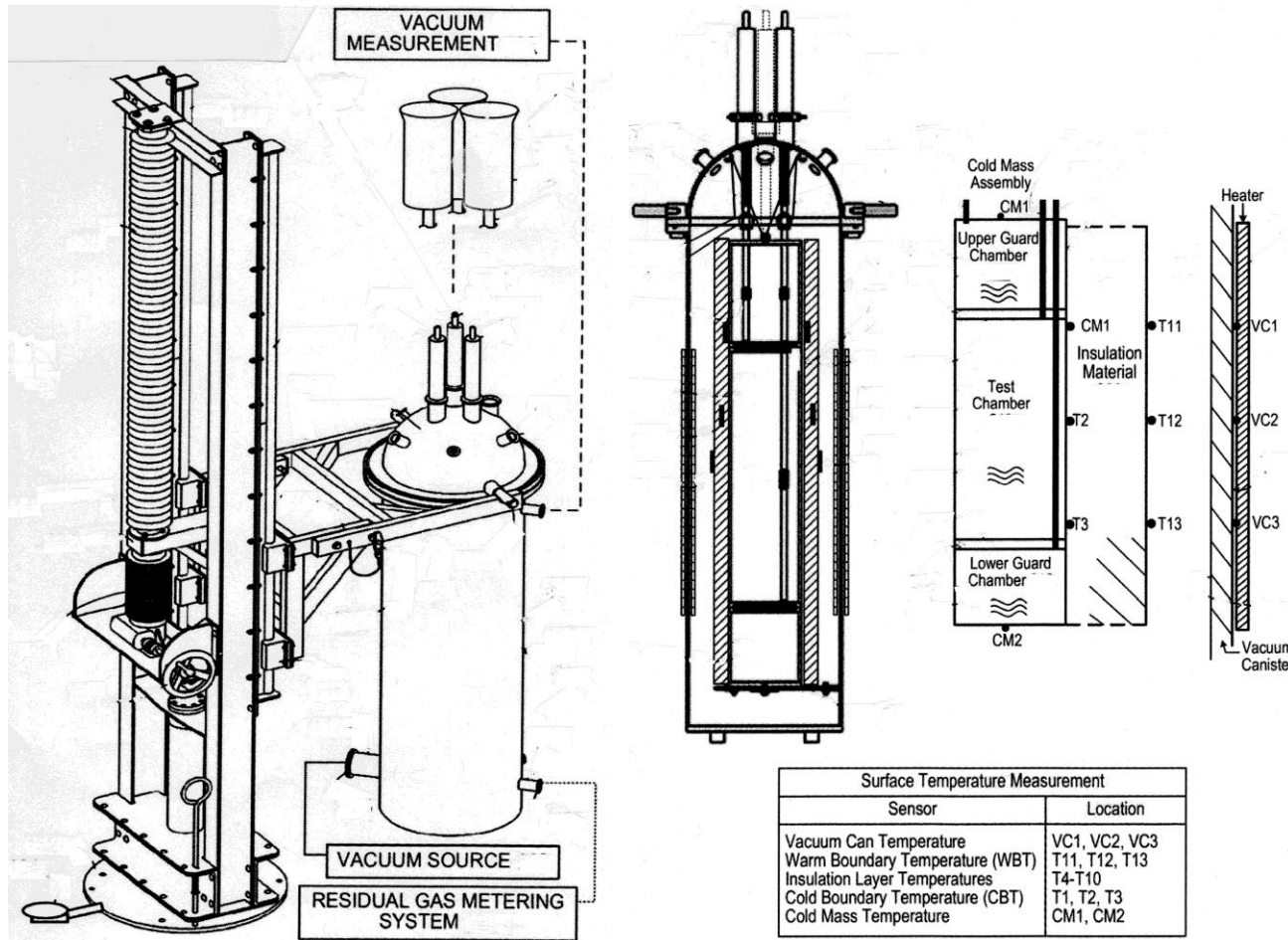


- Covers the heat flux or thermal conductivity data, performance considerations, typical applications, manufacturing methods, material specification, and safety considerations:
 - Warm boundary temperatures of **300 K or higher** and cold boundary temperatures from **4 K to 111 K**, but any temperature below ambient is applicable.
 - Typically used in a **high vacuum** environment (evacuated), but **soft vacuum or no vacuum environments** are also applicable.
- Heat flux values well below 1 W/m^2 are achievable in high vacuum MLI systems. Other evacuated insulations (bulk-fill materials) can provide heat flux values in the range of 5 to 20 W/m^2 .
- For comparison among different systems, or for space & weight considerations, the effective thermal conductivity of the system can be calculated for a specific total thickness:
 - Effective thermal conductivities of less than 1 mW/m-K [R-value 143] are typical.
 - Values on the order of 0.01 mW/m-K have been achieved [R-value 14,300].
- Applications categories: storage, transfer, thermal protection, and low-temperature processes. Very low temperature (4 K and below) refrigeration is a major technical capability for basic physics research world-wide.

Cylindrical Absolute Boiloff Test Apparatus



- Cylindrical absolute boiloff test apparatus ([Cryostat-100](#)): Lift mechanism, vacuum-pressure regulation ports, and funnel filling assembly (left); schematic, guard chambers, and temperature sensor locations (right).



Surface Temperature Measurement	
Sensor	Location
Vacuum Can Temperature	VC1, VC2, VC3
Warm Boundary Temperature (WBT)	T11, T12, T13
Insulation Layer Temperatures	T4-T10
Cold Boundary Temperature (CBT)	T1, T2, T3
Cold Mass Temperature	CM1, CM2

Cylindrical Absolute Boiloff Test Apparatus



- The steady-state heat flow rate (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). This heat flow rate through the insulation test specimen and into the cold-mass tank is directly proportional to the liquid nitrogen boiloff rate.
- The heat flux (q) is calculated by dividing the total heat transfer rate by the effective area for heat transfer:

$$q = \frac{Q}{A_e}$$

- Calculations of k_e are highly sensitive to the thickness of the test specimen:

$$k_e = \frac{Qx}{A_eDT}$$

- For Cylindrical geometry:

$$k_e = \frac{Q \ln\left(\frac{d_o}{d_i}\right)}{2\rho L_e DT}$$

- The steady-state condition is reached when the boiloff flow rates from all three chambers are stabilized, the temperature profile through the thickness is stabilized, and the liquid level in the test chamber is at least 90% full.
- The total test duration may be several hours to several days depending on the range of heat flow involved.

Thermal Performance – Data Summary



Benchmark data summary from Cryostat-100 testing with LN₂:

- Boiloff flow rate (m-dot)
- Heat (Q)
- Heat Flux (q)
- Effective thermal conductivity (k_e)

Materials include:

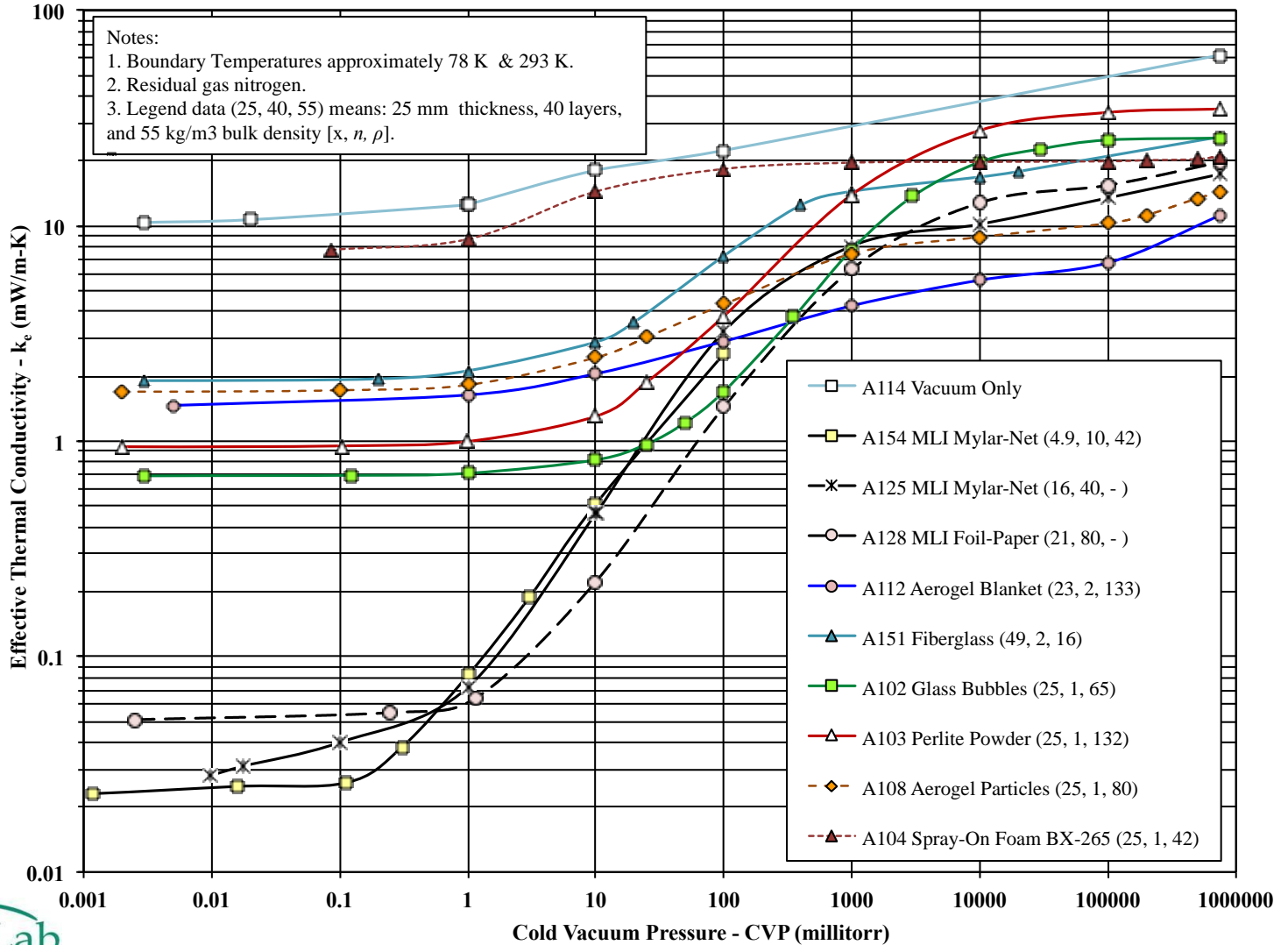
- Vacuum Only (black sleeve)
- Three different MLI systems
- Aerogel blanket
- Fiberglass blanket
- Glass bubbles
- Perlite powder
- Aerogel particles
- Spray-on foam

Test Specimen	CVP millitorr	WBT K	Flow sccm	Q W	q W/m ²	k _e mW/m-K	Specifications
A114 Vacuum Only Black sleeve.	0.003	293.1	7447	30.8	88.4	10.44	x n A _e ρ ^β
	0.02	292.9	7620	31.5	90.5	10.69	mm - m ² kg/m ³
	1	292.8	8917	36.9	105.9	12.52	- - 0.304 -
	1	291.9	8912	36.9	105.8	12.57	
	10	292.4	12907	53.4	153.3	18.16	
	100	292.5	15961	66.0	189.5	22.44	
	760000	242.9	34094	141	404.8	62.37	
A154 MLI Mylar-Net (4.9, 10, 42) Double-aluminized Mylar and polyester net spacer. Layer by layer installation.	0.001	293.0	75	0.312	0.997	0.023	x n A _e ρ ^β
	0.02	290.7	83	0.341	1.09	0.025	mm - m ² kg/m ³
	0.1	293.4	88	0.363	1.16	0.026	4.9 10 0.313 42
	0.3	293.3	126	0.519	1.66	0.038	
	1	291.5	271	1.12	3.58	0.082	
	3	293.0	623	2.58	8.24	0.187	
	10	290.0	1688	6.98	22.3	0.512	
100	285.3	8220	34.00	109	2.55		
A125 MLI Mylar-Net (16, 40, -) Double-aluminized Mylar and polyester net spacer. Layer by layer installation.	0.01	293.8	32	0.132	0.398	0.028	x n A _e ρ ^β
	0.02	293.1	35	0.145	0.431	0.031	mm - m ² kg/m ³
	0.1	293.1	44	0.182	0.549	0.040	15.5 40 0.332 -
	1	292.8	81	0.335	1.00	0.072	
	10	293.3	518	2.14	6.46	0.464	
	10	292.9	521	2.16	6.51	0.468	
	100	292.5	3604	14.9	45.0	3.24	
1000	292.8	8983	37.2	112	8.06		
10000	292.4	11341	46.9	142	10.2		
100000	293.0	15058	62.3	188	13.5		
760000	292.2	19376	80.2	242	17.4		
A128 MLI Foil-Paper (21, 80, -) Aluminum foilContinuous rolled and microfiberglass paper spacer. Continuous-rolled installation.	0.003	292.0	42	0.176	0.516	0.051	x n A _e ρ ^β
	0.3	293.6	46	0.192	0.563	0.055	mm - m ² kg/m ³
	1.1	293.1	54	0.221	0.648	0.064	21.1 80 0.341 -
	10	294.0	188	0.780	2.29	0.223	
	100	293.0	1214	5.03	14.7	1.44	
	1000	292.7	5293	21.9	64.2	6.30	
	10000	293.4	10943	45.3	133	12.8	
100000	293.2	13013	53.9	158	15.5		
760000	291.7	16548	68.5	201	19.3		
A112 Aerogel Blanket (23, 2, 133) Cryogel aerogel composite.	0.01	293.0	1160	4.800	12.4	1.47	x n A _e ρ ^β
	1	293.2	1299	5.377	13.9	1.64	mm - m ² kg/m ³
	10	292.7	1626	6.729	17.4	2.06	23 2 0.344 133
	100	292.7	2299	9.514	24.6	2.91	
	1000	293.0	3367	13.934	36.0	4.26	
	10000	293.0	4427	18.318	47.4	5.60	
	100000	292.6	5328	22.047	57.0	6.75	
760000	293.4	8894	36.803	95.2	11.24		
A151 Fiberglass (49, 2, 16) Micro-fiberglass batt.	0.003	293.0	802	3.32	8.59	1.90	x n A _e ρ ^β
	0.2	294.1	809	3.35	8.59	1.95	mm - m ² kg/m ³
	1	294.0	903	3.74	9.68	2.13	48.6 2 0.386 16
	10	293.0	1219	5.05	13.1	2.89	
	20	294.2	1487	6.16	15.9	3.59	
	100	293.0	3099	12.8	33.2	7.26	
	400	295.8	5227	21.6	56.0	12.51	
1000	296.6	6080	25.2	65.2	14.49		
10000	297.4	7129	29.5	76.4	16.90		
20000	293.6	7943	30.7	79.5	17.92		
760000	293.0	10724	44.4	115	25.99		
A102 Glass Bubbles (25, 1, 65) Type K1 hollow microspheres. Black sleeve.	0.003	292.6	494	2.043	5.9	0.69	x n A _e ρ ^β
	0.1	293.0	495	2.049	5.9	0.70	mm - m ² kg/m ³
	1	292.9	506	2.096	6.0	0.71	25.4 1 0.349 65
	10	293.1	585	2.419	6.9	0.82	
	25	293.3	691	2.861	8.2	0.97	
	50	293.6	875	3.620	10.4	1.22	
	100	293.8	1220	5.048	14.5	1.70	
350	293.5	2696	11.158	32.0	3.77		
1000	293.0	5547	22.953	65.9	7.78		
3000	292.6	9795	40.535	116.3	13.76		
10000	293.3	14161	58.602	168.2	19.84		
30000	293.5	16294	67.427	193.5	22.80		
100000	292.7	17861	73.913	212.1	25.09		
760000	293.6	18308	75.763	217.4	25.91		
A103 Perlite Powder (25, 1, 132) High density. Black sleeve	0.002	292.6	666	2.756	8.1	0.94	x n A _e ρ ^β
	0.1	292.7	679	2.808	8.1	0.95	mm - m ² kg/m ³
	1	292.9	712	2.945	8.5	1.00	25.4 1 0.349 132
	10	293.5	935	3.867	11.1	1.31	
	25	293.0	1342	5.555	15.9	1.88	
	100	293.2	2721	11.261	32.3	3.81	
	1000	292.7	9961	41.220	118.3	13.99	
10000	292.6	19792	81.903	235.0	27.82		
100000	292.7	23978	99.227	284.7	33.68		
760000	293.3	24954	103.265	296.3	34.95		
A108 Aerogel Particles (25, 1, 80)	0.002	293.0	1204	4.981	12.6	1.69	x n A _e ρ ^β
	0.1	293.2	1232	5.100	12.9	1.73	mm - m ² kg/m ³
	1	293.0	1303	5.392	13.6	1.83	

Thermal Performance Data - k_e



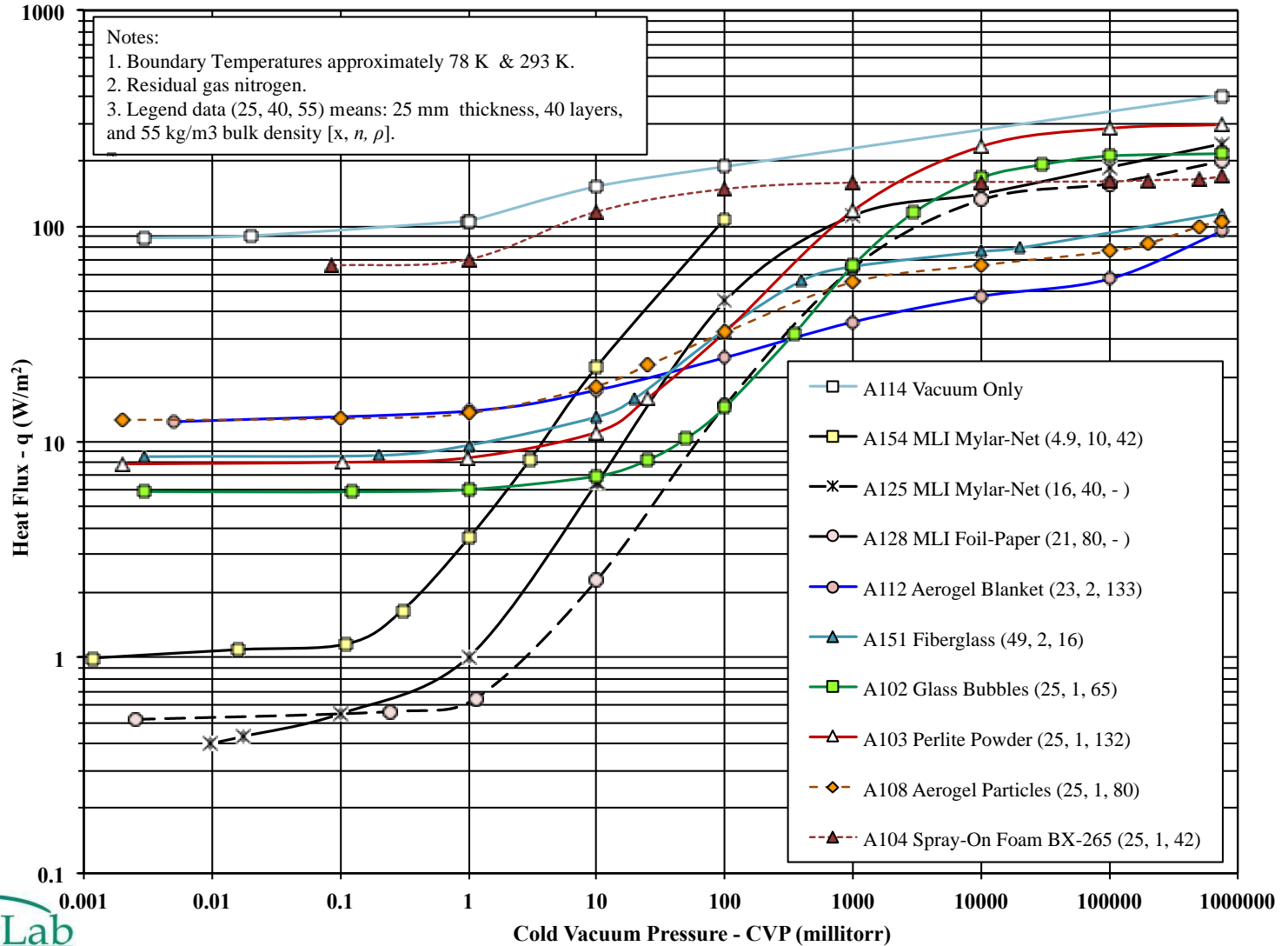
Variation of effective thermal conductivity (k_e) with CVP for different cryogenic insulation systems and materials. Boundary temperatures: 78 K and 293 K. Residual gas is nitrogen.



Thermal Performance Data - q



Variation of heat flux (q) with CVP for different cryogenic insulation systems and materials. Boundary temperatures: 78 K and 293 K. Residual gas is nitrogen.





Current Standards

Cryogenic Insulation Standards:

- ISO 21014 Cryogenic Vessels: Cryogenic Insulation Performance
- ASTM C1774 Standard Guide for Thermal Performance Testing of Cryogenic Insulation Systems
- ASTM C740 Standard Guide for Evacuated Reflective Insulation in Cryogenic Service

Insulation Materials Standards with Mention of Cryogenic Temperatures:

- ASTM C1728 Standard Specification for Flexible Aerogel Insulation
- ASTM C534 Standard Specification for Preformed Flexible Elastomeric Cellular Thermal Insulation in Sheet and Tubular Form
- ASTM C549 Standard Specification for Perlite Loose Fill Insulation
- ASTM C552 Standard Specification for Cellular Glass Thermal Insulation
- ASTM C578 Standard Specification for Rigid, Cellular Polystyrene Thermal Insulation
- ASTM C591 Standard Specification for Unfaced Preformed Rigid Cellular Polyisocyanurate Thermal Insulation
- ASTM C1029 Standard Specification for Spray-Applied Rigid Cellular Polyurethane Thermal Insulation
- ASTM C1482 Standard Specification for Polyimide Flexible Cellular Thermal & Sound Absorbing Insulation
- ASTM C1594 Standard Specification for Polyimide Rigid Cellular Thermal Insulation

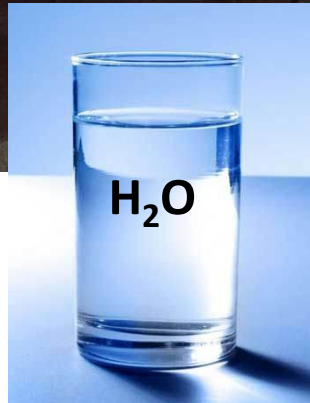


- Future technical consensus standards are envisioned for both **test methods and materials practices**.
- Specific test methods would be formulated for **cylindrical and flat plate geometries** covering **absolute and comparative approaches**, as required by mutual industry needs. Also:
 - Tanks and Pipelines
 - Water Vapor Sorption and Evacuation/Purging/Heating
- Materials of interest include, for example:
 - Non-Vacuum Multilayer Insulation Systems in Cryogenic Service
 - Glass Microspheres Loose-Fill Insulation Materials in Cryogenic Service
 - Aerogel Loose-Fill Insulation Materials in Cryogenic Service
 - Aerogel Blanket Insulation Materials in Cryogenic Service
 - Low-Density Cellular Foam Insulation in Cryogenic Service
- Standard data sets for specific materials would then be produced through a **round robin of cryogenic testing** among laboratories.

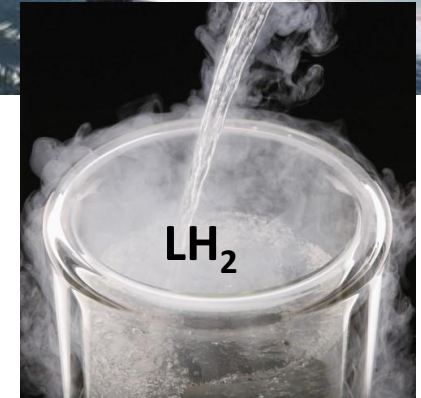
Preservation of the Cold



$\Delta T = 500\text{ }^\circ\text{F}$



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- **Energy efficiency, system control, and operational safety** are inter-related aspects of deciding the best thermal insulation system for a particular application.
- Emerging cryofuels enterprises including LNG and LH2 are a particular challenge due to the **transient operational processes** to be addressed and the competitive economic targets to be met.
- New ASTM standards (C1774 and C740) are one step toward providing thermal performance data and **materials specifications** for the cryogenic industry.
- Cryostat-100 insulation test apparatus has provided **thermal performance data** for a wide range of different materials under relevant conditions.
- Benchmark thermal performance data can be used to calibrate comparative instruments and support detailed studies of **insulation system designs** for specific applications.
- **Future technical consensus standards** are envisioned for both test methods and materials practices to support the cryogenic industry and further the proliferation of new industrial opportunities in the areas of transportation and energy.

Standardization in Cryogenic Insulation Testing and Performance Data





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