A boiloff calorimetry test configuration for the characterization of thermal insulation systems in flammable background gasses

A M Swanger[1], R W Hughes[2], and M A Guerrero Nacif[3]

[1]NASA Kennedy Space Center, Cryogenics Test Laboratory, KSC, FL 32899 USA
[2]NASA Kennedy Space Center, Safety & Mission Assurance, KSC, FL 32899 USA
[3]GenH2, 5200 S Washington Ave, Titusville, FL 32780

Email: adam.m.swanger@nasa.gov

Abstract. In support of efforts related to the design of future mega-scale liquid hydrogen storage tanks required to facilitate the global hydrogen economy, the Cryogenics Test Laboratory at NASA Kennedy Space Center has recently begun thermal performance characterization of various insulation systems in a gaseous hydrogen background using the Cryostat-100 (CS-100) liquid nitrogen boiloff calorimeter. The CS-100 is a vertical-cylindrical geometry, capable of measuring heat load and vacuum pressure ranges from 100 mW to 100 W, and 10^{-8} torr to ambient pressure respectively, via the ASTM C1774, Annex A.1 standard methodology. Flammable gas testing required numerous augmentations to the standard CS-100 hardware configuration and controls software, and modifications to the Lab facility to ensure a safe test campaign. These included double-containment of the CS-100 vacuum chamber and most supporting hardware, with continuous inert gas purging using nitrogen; remote control of valves and vacuum pumps; and hydrogen and oxygen detection systems. The careful design and implementation of these unique modifications led to safe and successful CS-100 hydrogen testing, and will be presented and discussed in-detail.

1. Introduction

To support a global hydrogen economy, large-scale production, storage, and transport of liquid hydrogen (LH2) are essential. Currently, the world's largest LH_2 storage tank, with a capacity of 4,700 m^3, is located at Kennedy Space Center's launch Pad B [1]. However, projected needs for storage tanks exceeding 50,000 m^3 present significant challenges for traditional vacuum-insulated designs. Beyond a certain threshold, constructing such vessels may become technically or economically unfeasible. If larger LH_2 tanks are required, non-vacuum-insulated designs will likely be necessary, a domain where examples for LH_2 storage are limited outside of space launch vehicle applications.

While non-vacuum designs exist for cryogens like liquid oxygen (LOX) and liquefied natural gas (LNG), the lower boiling point of hydrogen complicates insulation choices for the annular space. Heavy gases such as nitrogen or argon offer superior insulation but risk liquefying or freezing upon contact with surfaces at LH_2 temperatures. Helium, another option, provides less effective insulation and is constrained by scarcity and non-renewability. Hydrogen itself, despite being a possible purge gas, also offers subpar insulation performance.

If non-vacuum LH_2 tanks using hydrogen as a purge gas are to be viable, understanding insulation system performance becomes crucial. Currently, no standardized data exists for insulation performance in a hydrogen background gas. Recently, the Cryogenics Test Laboratory at NASA Kennedy Space Center undertook an experimental campaign using the CS-100 liquid nitrogen boiloff calorimeter to evaluate various insulation materials under hydrogen conditions [4]. The CS-100, renowned for its cryogenic insulation testing capabilities, required significant modifications to safely handle the higher heat loads and ensure personnel safety during testing with a flammable background gas.

2. Boiloff Calorimetry & Cryostat-100

Boiloff calorimetry is a method to experimentally determine heat flow through a thermal insulation system (TIS) using a cryogenic liquid as a constant cold boundary temperature (CBT) heat sink. A resistive heater is used to maintain the warm boundary temperature (WBT), and is typically set to ambient. The steady-state boiloff flow rate is measured and used to determine the heat transmission (i.e. heat watts) via the heat of vaporization of the cryogen. With the heat transmission in-hand, along with the ΔT, and geometric factors of the TIS, the total heat transfer coefficient, k_e, also referred to as the effective thermal conductivity, can be calculated using the Fourier heat conduction equation. This methodology, along with various apparatuses, are formalized in the ASTM C1774 standard [5].

The CS-100 apparatus was designed and built by the KSC Cryogenics Test Laboratory in the early 2000's and has been used to characterize the thermal performance of a wide range of thermal insulation systems using liquid nitrogen (LN_2) [4-7]. It is capable of measuring heat loads from 100 mW to 100 W, over vacuum pressure ranges from 10^{-8} torr to ambient. A thorough overview of the CS-100 can be found in reference 4.

Figure 1 presents a high-level cut-away of CS-100 and the system during LN_2 filling operations. What is not fully captured in the figure is the greater balance-of-plant hardware required to perform testing, such as various instrumentation, vacuum pump(s), data acquisition, and other electrical components. Historically, as all testing has been conducted using nitrogen or other inert background gasses, the system was relatively benign from a safety perspective. However, with the move into testing with hydrogen background gas, numerous improvements/augmentations to the system were necessary to comply with codes and internal safety requirements.

3. Conceptual Design of the Hydrogen Background Test Setup

Two factors were the primary drivers for augmentations to the base CS-100 setup to accommodate testing with hydrogen background gas (GH_2), or indeed any other flammable gas: 1. The increased heat load associated with testing with a lighter background gas at higher pressure ranges; and 2. Safety related to flammability issues and venting. The first driver required various changes internal to the CS-100, while the second required extensive updates external to the instrument, as well as facility modifications.

3.1 Addressing the Increased Heat Load

Historically, the insulation thickness for CS-100 was 25 mm with nitrogen gas, which was sufficient to achieve stable, steady-state conditions. Transitioning to more thermally conductive GH2 would increase heat loads, resulting in unstable data. To mitigate this, the insulation thickness was increased to 80.8 mm, extending the warm boundary to the aluminum heater sleeve. This configuration used the vacuum chamber more effectively, with the heater sleeve becoming the WBT. The update aimed to ensure stable boiloff tests with GH2, incorporating bulk-fill powder insulation thanks to a lower floor design interfaced to the lower guard on the cold mass and a temperature rake assembly with seven thermocouples separated into roughly 10.1 mm layer increments for detailed thermal monitoring.

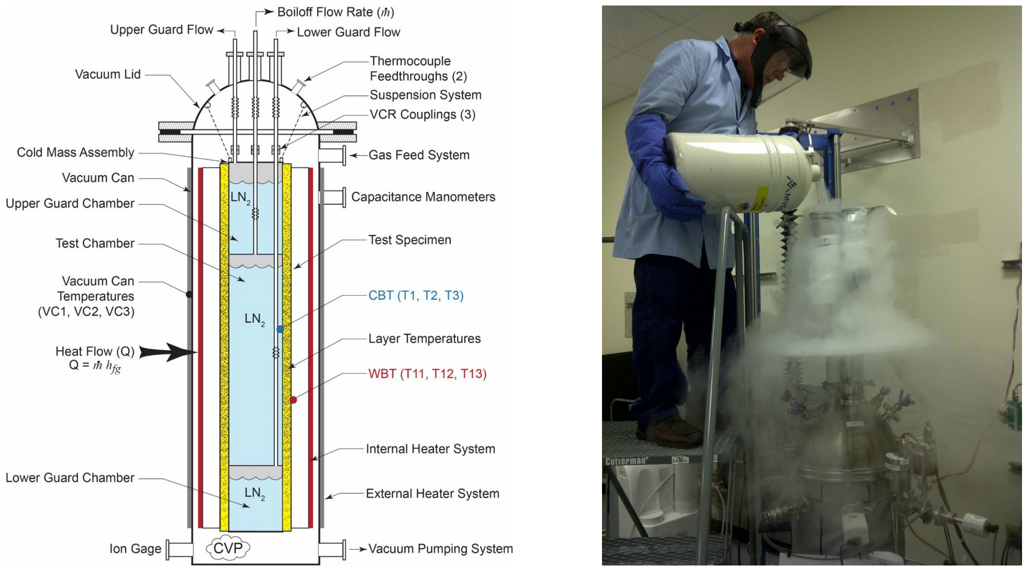

Figure 1. Cut-Away of CS-100 Assembly Showing Major Components/Details (left); CS-100 During LN_2 Fill Procedure (right)

3.2 Addressing the Safety-Related Issues

Since flammable gases had never previously been tested at the Cryogenics Test Laboratory, the facility had no dedicated area approved for such work. Therefore, the challenge for the project team was twofold: 1. Devise, design, and execute augmentations to the existing hardware and facility that met the technical goals for testing; and 2. Gain approval from the Authority Having Jurisdiction (AHJ) and required NASA-KSC safety boards.

Devising a practical solution to conflicting requirements stemming from the National Fire Protection Association (NFPA) 497 code [8] for electrical equipment used in hazardous (flammable) locations, and the non-compliant hardware necessary for testing was the most vexing issue. Per NFPA 497, the area within 4.57 m of any potential GH_2 leak point is considered "classified" (Class 1, Division 2 specifically)—which effectively encompassed the entire CS-100 lab room—and required compliant electrical hardware. Unfortunately, components such as vacuum pumps, valves, various instrumentation, data acquisition hardware, etc. required to achieve the test objectives, and which were standard to all previous CS-100 testing, either were not available with NFPA 497 code compliance, and/or were not technically compatible for the system, and/or were cost prohibitive.

Two facts did, however, play to the team's advantage: 1. The facility had access to a continuous supply of inert GN_2 from a cross-county pipeline at KSC; and 2. The CS-100 room had already been outfitted with a port through the outer wall, immediately adjacent to the vacuum chamber. Exploiting these attributes, the team developed a solution wherein the CS-100 cryostat and most electrical hardware would be contained within an enclosure, and supplied with a continuous GN_2 purge, with only the LN_2 feed ports accessible from the outside. The purge gas, and potentially any flammable gas leaked inside the enclosure, would be captured and vented through the wall, outdoors to a vent stack attached to the side of the building. The hydrogen gas bottle would be located outside the building as well, with gas routed through the wall port and purged enclosure to the test setup via a seamless flex hose to eliminate any potential leak points inside the lab room.

Such configuration complies with NFPA 496 Standard for Purged and Pressurized Enclosures for Electrical Equipment [9], specifically Paragraph 4.4.1, 4.9.1, and 8.3.1. Which mandate a minimum positive pressure of 25 Pa, alarms and indicators to positive pressure failure and the usage of inert gases to lower oxygen contents within the enclosure.

Once the CS-100 enhancements were planned and the operational concept approved, focus shifted to detailed technical design, equipment procurement, and obtaining necessary safety approvals from Kennedy Space Center (KSC). A total of five approvals were required before initiating the test campaign. Initial approvals involved walkthroughs and procedure reviews by the AHJ (Authority Having Jurisdiction) and explosives safety officer. Subsequent approvals included assessments by KSC's Safety Health Review Board (SHRB) and Ground Risk Review Panel (GRRP), requiring comprehensive hazard and risk assessments and involving multiple organizational members. Lastly, compliance with NASA's pressure systems standard necessitated developing a technical package approved by the KSC Pressure Systems Manager.

4. Final Design & Checkout of the Hydrogen Background Test Setup

Enclosing the CS-100 and its support hardware in a continuously purged environment to meet safety requirements posed significant practical challenges. Ultimately, a design was selected featuring oval-shaped upper and lower sheet metal assemblies for the enclosure's floor and ceiling, spacious enough to accommodate all necessary equipment. LN2 fill tubes penetrated the upper assembly, supported by stainless steel struts on the opposite end. Each assembly included 10 cm tall inner and outer sheet metal strips forming a racetrack configuration, providing structural rigidity and mounting surfaces for impermeable plastic curtains that served as enclosure walls. The lower assembly walls facilitated various electrical, pneumatic, and instrumentation feed-throughs using sealed tube sections. Polyvinylchloride (PVC) curtains were procured to specified dimensions, with hook-and-loop strips applied to secure them to the sheet metal assemblies. It was understood that the enclosures could not be perfectly or hermetically sealed. Their only purpose was to maintain both volumes at least 25 Pa above atmospheric pressure to prevent air intrusion, and the redundancy would effectively eliminate the possibility of flammable gas escaping into the lab room. Figure 2 presents the piping and instrumentation diagram (P&ID). Nitrogen purge gas was inputted at one end of the bottom assembly, filling both volumes and exiting through independent vent paths located at the far end of the top assembly. Pressure gauges were linked to both volumes to monitor the pressures, which also satisfied NFPA 496 paragraph 4.9.1 requirements. Determining the proper nitrogen purge gas flow rates to the enclosures was done through trail-and-error on the final configuration, using different sized fixed orifices. As the curtains were flexible, the entire enclosure inflated like a balloon, and too much flow would result in pressures high enough for the curtains to detach, or the leak rate to be too high. Final orifice sizes were 0.762 mm and 1.016 mm on the outer and inner volumes respectively, with a supply pressure of 5.5 bar. This maintained the inner enclosure at roughly 125 Pa, and the outer at 62 Pa during operation, at calculated flow rates of 116 L/min and 65 L/min respectively. Estimated volumes of the inner and outer enclosures were 1.61 m^3 and 0.5 m^3 respectively, resulting in a volume change-over of roughly 14 minutes for the inner volume and 8 minutes for the outer. The maximum probable hydrogen leak rate, determined assuming the primary solenoid needle valve that controlled flow into the CS-100 vacuum chamber was to fail wide open, was estimated to be 11.6 L/min; resulting in a 10:1 ratio of nitrogen-to-hydrogen flow into the inner purged volume in the case of a leak.

Containing the vacuum solenoid valves and pumping station inside the enclosure required remote operation, which was achieved via on/off control switches for the valves, and an interface to the LabVIEW data acquisition software (DAQ) for the pump. Also, as the testing campaign required characterization of insulations over the full vacuum pressure range (760 tor to 10^{-7} torr), with some powder-type specimens, three independent pumping paths were used, with one employing a small, fixed orifice to slow the pump-down rate from ambient pressure. These are represented in figure 2, with the upper vacuum chamber pump-down port splitting into three paths. Also shown in figure 2 is a nitrogen supply to the exit of the vacuum pumping station, through a fixed orifice. This leg was included for two reasons: 1. To aid in purging all air out of the system during pretest procedures; and 2. To dilute the exit gas during flammable gas testing with active pumping. An oxygen sensor was installed on the primary vent stack to verify all air had been purged from the system. Figure 3 shows the completed sheet metal assemblies and installed curtains, and the final assembly of the CS-100 configuration for flammable background testing.

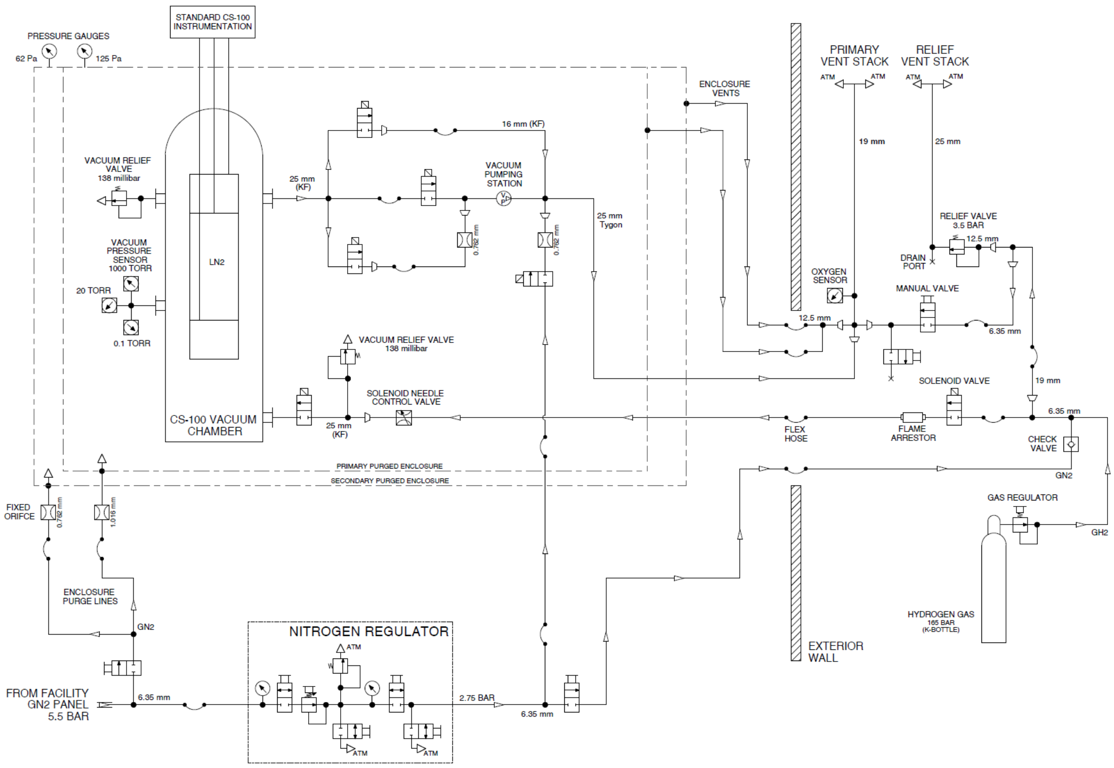

Figure 2. Piping and Instrumentation Diagram of the CS-100 Test Configuration Using Hydrogen Background Gas (all tubing and hoses are stainless steel except where noted; line sizes noted)

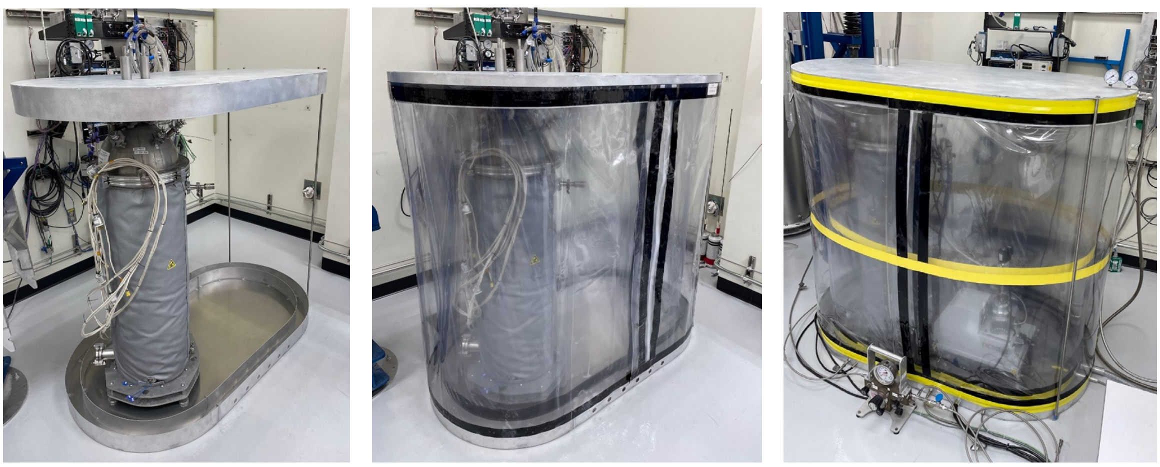

Figure 3. Upper and Lower Sheet Metal Assemblies Installed on CS-100 (left); Enclosure with PVC Curtains Installed (middle); Completed Setup with Purged Enclosure Inflated (right; black strips are hook-and-loop, yellow are vinyl tape)

5. Lessons Learned

The testing program has shown excellent performance overall, with no hydrogen leaks or unexpected issues detected. However, two minor challenges surfaced. First, installing PVC curtains and sealing them with vinyl tape was cumbersome due to narrow inner and outer sheet metal flanges. Future designs should consider widening inner flanges and expanding lower flanges to facilitate easier installation and better sealing. Second, maintaining optimal temperature control in the inner enclosure proved challenging due to heat from the vacuum pumping station. While safe for equipment, the warmer environment affected the boiloff test's Warm Boundary Temperature (WBT), requiring testing at a slightly higher WBT of 293 K. Improving purge gas flow rates, reducing its temperature, or enhancing environmental control in the inner enclosure would be beneficial for future iterations.

6. Conclusion

A new configuration for boiloff calorimetry per ASTM C1774, Annex A.1 using flammable background gas was presented. The new setup was designed, and implemented at the NASA Kennedy Space Center Cryogenics Test Laboratory using the CS-100 instrument, and uses a double-containment-style enclosure around the cryostat and most test equipment. The enclosure is supplied with a continuous inert nitrogen gas purge to eliminate the presence of oxygen in the case of flammable gas leak, which is captured and vented outside the building through a dedicated vent stack.

To-date, the new CS-100 setup has been used to characterize two different insulation samples safely and successfully over the full vacuum pressure range with hydrogen background gas. In total, twenty individual tests have been conducted, with a combined test time of roughly 300 hours.

7. Acknowledgments

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