INL/CON-24-76292-Revision-0

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May 2025

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Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

Development and Demonstration of a Prototype Molten Salt Sampling System

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ABSTRACT

Molten salt reactors (MSRs) offer potential operability and safety advantages compared to commercial light water reactors. However, operating experience with MSRs is sparse in comparison to what exists for LWRs. Further, the chemical and isotopic composition of the fuel and/or coolant salt is dynamic and still being characterized—posing potential safety, operability, and safeguards unknowns that need to be addressed. A molten salt sampling system (MSSS) is regarded as a necessary subsystem within first-generation MSRs used to remove grab samples of salt for chemical and isotopic analysis in support of the need to monitor and control salt composition during operation. The MSSS is being developed using the Safety in Design (SiD) methodology, which incorporates incremental integration of safety analysis into the design process. The MSSS conceptual design emerging from the application of the early stages of the SiD methodology consists of a sample collection system and its housing, a freeze port, and an inert gas control and a delivery system. This article describes the prototypes developed to test the functions of the above MSSS subsystems and provides a status update on the ongoing testing and reliability data collection activities being performed within the relevant testing environments in accordance with the principles of SiD.

Keywords: Molten salt reactor, Molten salt sampling system, Safety in design, reliability

1. INTRODUCTION

Molten salt reactors (MSRs) offer potential safety and operability advantages compared to light water reactors (LWRs) in future energy portfolios due to their ability to achieve higher core inlet/outlet temperatures, larger prompt negative temperature coefficients, and lower operating pressures [1]. However, the limited operational experience with the MSR concept and the dynamic chemical and isotopic composition of the fuel and/or coolant salt pose challenges to these potential safety and operability benefits, as well as reactor safeguards [2]. These challenges have led to the theorization and development of a molten salt sampling system (MSSS)—a subsystem tasked with removing grab samples of salt during operation for chemical and isotopic analysis to support the overall operational and safety goal of monitoring and controlling the composition of the primary salt inventory(ies).

The MSSS has been developed using the Safety in Design (SiD) methodology pioneered by the Electric Power Research Institute and Vanderbilt University [3], which incrementally integrates safety analysis into the design process, commensurate with design maturity. The MSSS concept can be broken into subsystems—the sample collection system (SCS) capsule and its housing, the freeze port, and the inert gas control and delivery system. Prototypes to demonstrate the functions of these subsystems are undergoing testing within relevant environments. This article: (1) gives background on the concepts of SiD and molten salt sampling; (2) describes the MSSS developed using the SiD methodology; (3) conveys the incremental approach being used for prototype testing; and (4) provides preliminary results of prototype testing.

1.1. Early Incorporation of Safety into Advanced Reactor Design: The SiD Methodology

Historically, safety analysis of nuclear reactors has been stylized in nature and performed after a design has largely been completed [4]. Growing interest in advanced reactors has sparked research into alternative avenues available, including the early incorporation of safety analysis into advanced reactor design. The SiD approach leverages hazard evaluation methodologies [3], such as process hazards analysis (PHA) techniques [5], that can be tailored to meet the needs of advanced reactor developers. The use of PHA methods within advanced reactor design is intended to be iterative [4], starting with qualitative hazard evaluations from the earliest stages of design to foster early design decision-making. After a number of iterations, such analyses mature into quantitative assessments of initiating events, their frequencies and consequences, and an understanding of the structures, systems, and components in place to ensure public and worker safety [4]. To-date, SiD has been applied only partially to the advanced reactor design process—it has been used for limited scope case studies on preconceptual commercial reactor designs and test loops [3] and for *post hoc* analysis of the Molten Salt Reactor Experiment (MSRE) [6-7]. However, until the development of the MSSS, the SiD methodology had not yet been demonstrated or applied over the duration of the advanced reactor system design cycle (conceptualization, prototyping, testing, and iteration).

1.2. Molten Salt Sampling: Historical Implementation and Modern Perspectives

MSR operating experience is limited to two previous reactors, only one of which—the MSRE—resembles commercial interpretations of the concept that exist today. The MSRE, which operated from 1965 to 1969, utilized a system called the Sampler-Enricher (S-E) [8] to accomplish the function of molten salt sampling. The S-E was designed to remove 10- to 25-gram grab samples approximately three times per day [8]. During the MSRE's four years of operation, the S-E was used more than 700 times; however, a detailed review of the system's operational history revealed a need for frequent corrective maintenance that makes the original design unsuitable for use within a future MSR, primarily on the basis of its unreliability [9].

The MSRE was the most-recent MSR operated. Correspondingly, research into the development of a more reliable MSSS has only re-emerged upon recent government, academic, and industry interest in MSRs spurred when the concept was selected as promising by the Generation IV International Forum in 2002 [10]. Since then, modern perspectives on the role of molten salt sampling in future MSRs have been documented within the literature. Reports produced by Oak Ridge National Laboratory have discussed the role that an MSSS may play in fuel qualification [1], the measurements to be taken (including redox conditions, structural material corrosion progression, and actinide/lanthanide concentrations) on samples removed with an MSSS [11], and the need to discuss sampling frequency in a license application for a non-power MSR [12]. A recent study of postulated initiating events for the Molten Salt Fast Reactor also includes one event related to MSSS failure ("complete rupture of the pressurized sampling device") [13].

1.3. Implementation of SiD in Support of MSSS Conceptual Design

The MSSS has been developed using many of the elements of the SiD methodology, outlined in blue in Figure 1. One of the first tasks performed in developing the MSSS was to find and collect operating data from the predecessor to the MSSS—the MSRE S-E. This data, found in publicly available reports, consisted of narrative descriptions of 64 operational occurrences and 140 corrective actions that were evaluated for relevant design and phenomena insights for the MSSS using the principles of the Systems Theoretic Accident Model and Process (STAMP) [9]. These insights were used to help produce the first iteration of the MSSS concept, on which preliminary go/no-go testing was performed [14]. A highly detailed PHA, specifically a Failure Modes and Effects Analysis (FMEA), was then performed on this iteration of the MSSS, shown in Figure 2. [15] The concept consists of the following subsystems:

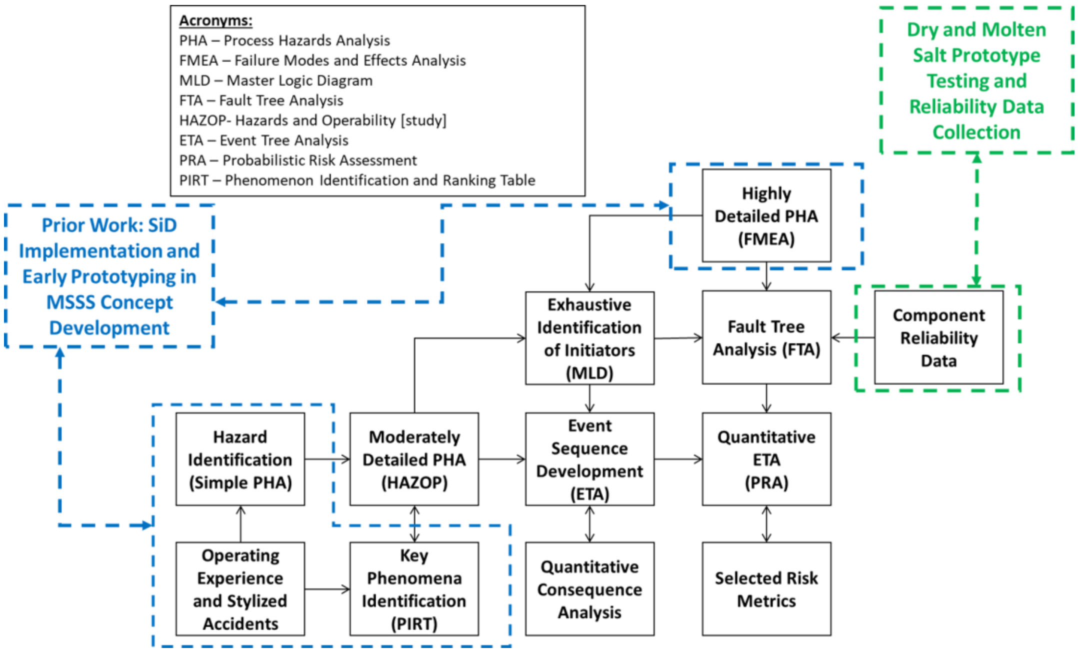

Figure 1. Implementaion of SiD methodology (adapted from Ref. [3]) for MSSS development.

1. SCS capsule (purple): the SCS isolates a small sample of primary salt from the bulk salt volume and confines the sample during transfer. The SCS consists of an oblong-shaped capsule (3” L x 3/8” OD) at lower pressure than the sampled system, penetrated by a 1/16”-OD needle whose external tip is filled with solid salt. Upon insertion to the primary salt, the salt in the tip of the needle melts. The sample flows up through the needle and into the capsule driven by the pressure difference between the capsule and the primary system salt. The sample spills into the bottom of the capsule where it is below the elevation of the needle extending up into the capsule, and is retained by gravity. The top of the capsule is connected to a magnet, which facilitates its retention at the top of the capsule housing following the sampling process;
2. Capsule housing (magenta): the capsule housing provides a pressure boundary to contain the SCS and isolate radiological or chemical contaminants released from the salt during sampling;
3. Inert gas control and delivery systems (blue and green, respectively): the inert gas control and delivery systems supply inert gas to the MSSS for three functions—purging contaminated gas from the capsule housing before and after sampling, pressurizing system internals to control the salt level within the freeze port during sampling, and providing pneumatic motive force to move capsules into and out of the sampling position at the bottom of the capsule housing; and
4. Freeze port (orange): the freeze port directly interfaces with the primary salt system by performing the functions of isolation (when the MSSS is in standby) and providing access to the primary salt below the port (when sampling is ongoing). The freeze port design is similar to the freeze valve design used within the MSRE (and pursued by modern MSR designers) [16], in that a small-diameter tube (6” L x ½” OD) filled with salt is used as an alternative to traditional electromechanical valves. When a sample needs to be collected, external heaters surrounding the tube are used to melt the salt, such that the needle of the SCS can penetrate the freeze port and contact the primary salt volume below. When sampling is complete and the capsule has been retracted, the heaters are turned off, and forced convection air flow at room temperature can be used to re-freeze the port and isolate the MSSS from the primary salt system.

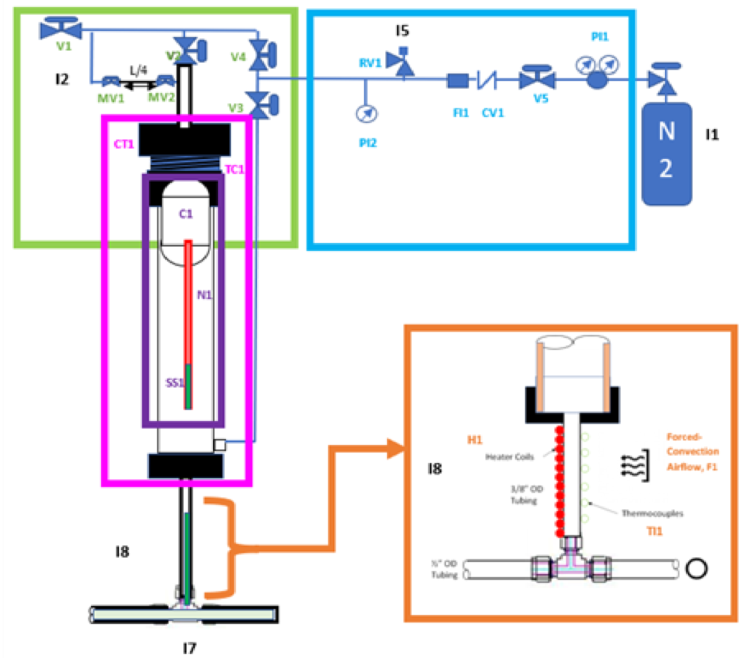

Figure 2. Conceptual design of the MSSS [14].

2. METHODS

The SiD approach to MSSS development, and more specifically the FMEA, indicated that the performance of three of the functions of the system described above needed to be assessed via laboratory testing: (1) the reliability of the freeze port to open (melt) and close (freeze) in lieu of a traditional valve, (2) the reliability of a pneumatic means of moving SCS capsules into and out of the sampling position, and (3) the reliability of the SCS design to obtain a sample of sufficient size to be used for analysis to characterize bulk salt behavior. In accordance with the SiD methodology, and also due to time and resource constraints for the project itself, the individual functions of the physical MSSS prototype are being systematically proven out in increasingly hazardous environments—starting with low-hazard testing of those functions that do not require molten salt testing to demonstrate proof of concept (i.e., dry testing) and subsequently moving to a higher hazard environment for testing of functions that require testing in a molten salt environment for proof of concept demonstration—prior to full system testing in a molten salt environment.

2.1. Dry Testing and Reliability Data Collection Methods

We determined that the reliability of a pneumatic means of sample capsule movement was suitable for preliminary testing in a dry (i.e., not molten salt) environment to demonstrate proof of concept. The activities described below were undertaken to perform dry testing in a manner suitable for reliability data collection to satisfy the need for component reliability data within the SiD methodology (indicated in the box outlined in green in Figure 1). First, we performed pre-planning—we determined the physical boundaries of the conceptual design to be tested and procured off-the-shelf components from Swagelok and McMaster Carr to construct the prototype. Next, we prepared a data collection plan using as a basis existing guidance for the Data Quality Objectives (DQO) Process [17]. We determined that each test would consist of two elements: (1) testing the reliability of the sample collection capsule seating in the sampling position upon insertion and (2) testing the reliability of the pneumatic means to unseat the sample collection capsule. For the purposes of the study, we initially defined the criteria for reliable seating (insertion of the SCS into the sampling position) as: (1) the body of the SCS successfully traversing the length of the capsule housing and residing in the low interface position upon application of high pressure; (2) the needle of the SCS successfully traversing the length of the capsule housing and lower transition reducer and residing within the ½” section of piping upon application of high pressure; and (3) no visible damage caused to the SCS or capsule housing during the movement process. We defined reliable unseating (removal of the SCS from the sampling position) as: (1) the body of the SCS successfully traversing the length of the capsule housing and residing in the high point of the capsule housing upon application of high pressure; (2) the needle of the SCS successfully traversing the length of the capsule housing and lower transition reducer and residing within the high point of the capsule housing upon application of high pressure; and (3) no visible damage caused to the SCS or capsule housing during the movement process.

We also developed a procedural checklist to be used for dry testing, including the development of data collection sheets to record reliability data described above for capsule seating and unseating. The procedural checklist was developed using U.S. Department of Energy (DOE) guidance for procedures development [18]. We then fabricated the prototype and performed shakedown testing. Shakedown testing included the implementation of any design revisions required to support successful prototype testing. We next performed prototype dry testing—collecting reliability data for each test performed. Initially, one week was allotted to perform shakedown and dry testing, with the goal of running as many successful tests as possible during that time. Finally, we analyzed the data collected during testing for reliability insights.

2.2. Molten Salt Testing and Reliability Data Collection Methods

We determined that the remaining two MSSS functions required testing in a molten salt, but nonradioactive, environment to demonstrate proof of concept. Molten salt testing is ongoing, and is being performed following the same approach described in Section 2.1 above, with unique data collection plan(s), procedures, and data collection sheets being developed for the tests. To maximize resources usage, many of the components (e.g., valves, tubing, etc.) from the dry test are being repurposed for molten salt testing within the University of Michigan’s Molten Salt Facility for Instrumentation Tests. This is possible because the components used for dry testing are composed of SS 316—and are thus suitable for short-term use within molten salt. Molten salt testing is being performed in two phases. The first phase is intended to prove out the function of the freeze port to reliably “open” and “close” on demand, and the second is intended to prove out the ability of the sample collection capsule to obtain a sample.

3. RESULTS & DISCUSSION

Prototype testing is ongoing. Accomplishments to-date for dry testing of pneumatic sample collection capsule movement are described in Section 3.1. Progress toward completing molten salt testing of the freeze port and sample collection capsule is described in Section 3.2.

3.1. Prototype Dry Testing and Reliability Data Collection

Shakedown testing occurred between August 25, 2023 and September 8, 2023. Given the large amount of time spent refining the prototype design during shakedown testing, dry testing was limited to one day. Dry testing occurred within Vanderbilt University’s structure’s lab on September 11, 2023. The original dry test schematic and bench-scale prototype are shown in Figure 3 below. The prototype was designed such that the SCS can be inserted into the capsule housing through the screw cap (C1) at the top of Figure 3. C1 included a magnet on the inside to retain the capsule, until inert gas flow through the capsule housing’s top tee (facilitated by opening valves V6, V5, V4, V2, and V7) disconnected the capsule and forced it into the sampling position at the bottom of the capsule housing. A pressurized nitrogen canister was used as a surrogate for inert gas supply. Another cap, C3, could be opened to visually determine whether the needle of the SCS capsule, shown in Figure 4, was seated in the proper position. The capsule, which included a magnet adhered to the top of C2, could then be unseated and reattached to the magnet connected to the bottom of C1 pneumatically by flowing nitrogen through the capsule housing's bottom tee (by opening V6, V5, V3, V2, and V1).

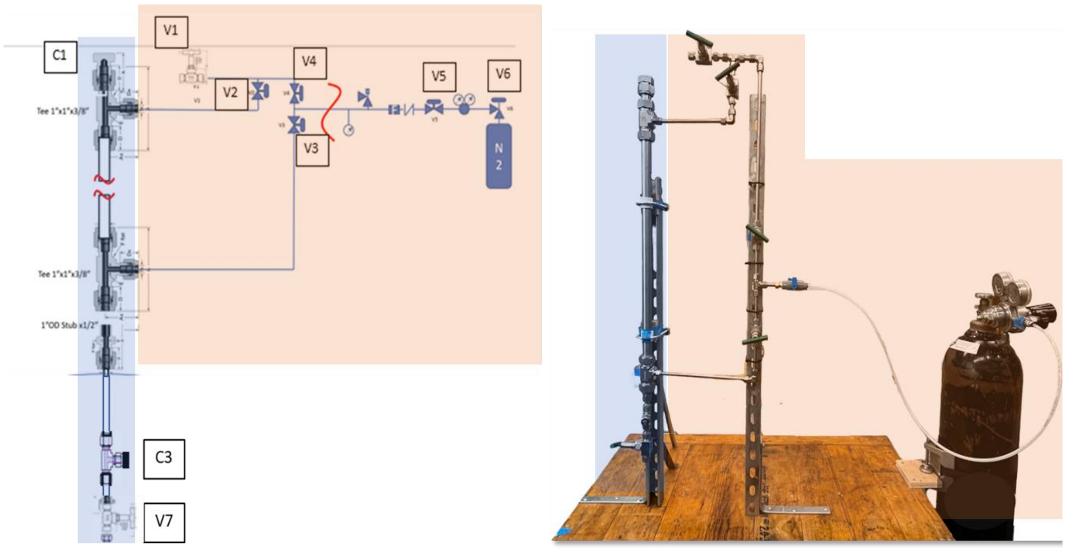

Figure 3. Initial dry testing schematic (left) and prototype (right). The capsule housing is shaded in blue and the inert gas control and delivery systems are shaded in orange.

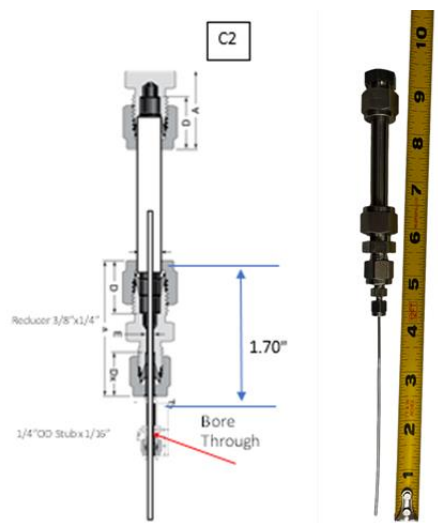

Figure 4. Initial sample collection capsule schematic (left, not to scale) and prototype (right).

3.1.1. Shakedown testing design refinement

As evident in examining the schematics in Figures 3-4 in relation to that shown in Figure 2, a number of differences exist between the fabricated dry test prototype and the conceptual MSSS that would be installed within a future MSR. Specific deviations from the conceptual MSSS in the dry test prototype include: (1) as opposed to running the tubing delivering nitrogen gas to the top of the capsule housing through C1 itself (as shown in Figure 2), the gas was run through a tee to the right of where the capsule sits in the unseated (top) position; (2) the tee supplying nitrogen to the bottom tee of the capsule housing (to remove the capsule from the seated position) sits near the bottom of the capsule, but not below, when it is in the seated (bottom) position; and (3) the capsule itself does not feature the half spherical end caps shown in Figure 2, rather it is fabricated of 3/8” tubing connected to a hexagonal end cap (C2) on one side and a Swagelok bored-through reducer attached to the needle on the other. These differences are attributed to the need to perform dry testing on an expedited schedule using commercially available materials and components; however, these differences required adjustments to the prototype during shakedown testing.

The first adjustment needed during shakedown testing was due to the issue of the SCS being unable to be pneumatically lifted from the bottom of the capsule housing when pressures of 40, 60, 80, and 110 psi were applied. The first adjustment was to increase the length of the needle protruding from the capsule such that the needle rested on top the internal structures of V7, effectively lifting the capsule above the bottom tee of the capsule housing such that gas flow is directed upward during the unseating process before contacting the bottom of the capsule. Further, because of the differences between the prototype sample capsule (with hexagonal end caps) and that pictured in Figure 2, additional clearance existed between the capsule housing and SCS that prevented unseating of the capsule because it allowed too much gas leakage flow around the sides of the capsule. An annular washer with a diameter just less than the inner diameter of the 1” tubing (0.834”) was added between the bored-through reducer and its lower compression fitting (at approximately the 5-½” mark of the prototype in Figure 4) to reduce the clearance between the capsule and capsule housing.

The difference in the insertion point of the nitrogen gas near the top of the capsule housing caused two challenges during shakedown testing. The first challenge had to do with the ability to pneumatically perform the capsule seating test. Because the position of the sample collection capsule in the unseated position (connected to C1) resulted in force being applied to the side of the capsule (as opposed to the top), it was not possible to pneumatically detach the capsule from C1. Given that this was a low priority item for dry testing, the capsule was held in place manually in the unseated position (with C1 detached) and dropped via gravity into the seated position at the bottom of the capsule housing. Second, within an MSSS implemented within a future MSR, it is expected that the off-gas system would be activated by opening V1 in Figure 2, and correspondingly draw a negative pressure on the top of the capsule housing, assisting in maintaining the capsule in the top of the capsule housing. However, the arrangement of the dry test prototype did not include this function. After the adjustments described above were made, it was thus possible to raise the capsule to the top of the capsule housing by applying a nitrogen pressure of 119 psi; however, the magnet connected to C1 would not retain the capsule, thought to be due to a local low pressure on the backside of the capsule caused by the induced high flow velocity zone between the magnets connected to C1 and C2. Given the time and resource limitations that existed for the project, a simple refinement was thus made to demonstrate the ability to magnetically retain the capsule at the top of the capsule housing—a high strength magnet was used to cap the capsule housing (as opposed to C1). However, even with this change, retaining the sample capsule at the top of the housing proved to be challenging, which was believed to be due to the frictional pressure drop caused by the 90˚ bends and valves (V1 and V2) when venting gas from the top of capsule housing. Disconnection of V1 solved this issue.

3.1.2. Preliminary reliability insights

In total, 18 seating and unseating runs were completed with the dry test prototype. As described above, due to differences between the conceptual MSSS and the dry test prototype, the ability to pneumatically seat the SCS was not possible. However, all 18 gravity-driven drops of the capsule otherwise met the remaining seating success criteria defined in Section 2.1 (capsule body residing in the sampling position, needle residing within the freeze port, no visible damage to SCS or capsule housing). Although the seating test did not prove out the concept of pneumatically moving the SCS capsule into position, it did provide evidence to support that the SCS capsule’s needle could reliably pass through the 1” to ½” reducer.

Within a future MSSS, we anticipate that each SCS would be used to take a single sample. However, one capsule was used for all 18 dry tests due to resource constraints. Starting in unseating test 6, we observed that the small magnet attached with adhesive to the top of C2 began to break apart; however, it was still retained by the adhesive in place atop the capsule. In tests 7 through 11, we observed further degradation of the magnet with each test. At test 12, we added more adhesive to the magnet to retain it in place for the remaining tests. We believe this degradation to be due to two factors: (1) the difference in strength between the magnet atop the capsule housing and that attached to C2 and (2) the high pressure needed to unseat the capsule, and the resultingly high speed at which the capsule collides with the magnet atop the capsule housing. Although further characterization of this degradation may be needed in the future, we disregarded criterion 3 (no visible damage to the SCS or capsule housing) in our preliminary reliability estimates, since we envision the SCS to be a single-use item in a future MSSS.

Seventeen out of the 18 pneumatic unseating tests met criteria 1 and 2. The single observed failure was considered to be due to operator error. While performing shakedown testing, it was observed that that the gas supply must be turned off slowly for the capsule to be retained by the magnet above the capsule housing—by slowly closing either V3 or V5 over approximately a 5-10 second period. In the failed unseating test, V5 was shut off too quickly, causing the capsule to dislodge from the magnet and drop back into the seated position. Disregarding criterion (3), and assigning a successful test a value of 1 and an unsuccessful test a value of 0, we can assess an unseating functional failure rate for the prototype, assuming that classical statistics are valid, of 0.056 per demand, according to Equation 1 below, where "F" is the unseating failure rate, "R" is the reliability, "n" is number of tests performed, and "S" represents a successful test. The unseating test provided proof of concept for the following elements of the MSSS design: (1) ability to pneumatically unseat the SCS capsule from the sampling position, and (2) ability to magnetically capture and retain the capsule in the unseated position after it has been lifted out of the sampling position.

$$F = 1 - \mathrm{R} = 1 - \frac{\sum S}{n} = 1 - \frac{17}{18} = 0.056/demand \quad (1)$$

3.1.3. Insights for future design, testing, and analysis

In accordance with the principles of SiD [3], the results of the dry testing can support the design iteration process—providing insights for design modifications, as well as additional testing/characterization and analysis. Examples of each of these categories from the MSSS dry testing are as follows:

- Design Modifications: Although the SCS was retained in the unseated position during the dry test purely using magnets, magnet degradation during repeated testing and differences in the nitrogen gas insertion point in a future MSSS suggest investigation of using an electromagnet atop the capsule housing. Further, the method of securing the magnet was a function of limited time and funds. Because of the intended application in high temperatures, a more robust method to adhere the magnet to C2 is recommended—either a magnetic cap or by welding the magnet to the cap.
- Additional Testing/Characterization: Once the design modifications above are evaluated, additional reliability data specific to these modifications should be collected.
- Additional Analyses: Although the reliability data provided above are preliminary and additional statistical analysis is ongoing, these estimates can be used to in future PHA evolutions. For example, the prototype testing provides details on failure modes (e.g., operator error in timing of V5 closure) to be considered in future PHAs and may also be used to develop "first cut" estimates of failure rates for the system as a whole, while additional prototype iteration and testing is still ongoing.

3.2. Prototype Molten Salt Testing

To date, we have acquired all materials/components for molten salt testing and developed a data collection plan. Prototype fabrication and procedures development are ongoing. Reliability data will be collected during molten salt testing. The conceptual model of the freeze port test, shown in Figure 5a, consists of a crucible of molten salt contained inside a small secondary containment. The crucible of molten salt contains a ½” OD section of tubing fitted with a custom heater containing two thermocouples (.e., the freeze port). The ½” OD tubing penetrates the top of the secondary containment, as well as out of the larger primary stainless steel containment vessel through a custom fabricated flange. The initial purpose of the test bed is to test the ability to pneumatically control the salt level within the port, monitored using thermocouples on the top and bottom of the custom heater. Pressure control of the primary and secondary containment control volumes are provided by a ¼” OD tubing penetration, and leads for instrumentation into the test bed are provided through a third penetration. The second purpose of the test bed is to test the ability to reliably freeze the salt within the ½” section of tubing—using a fourth penetration through the primary and secondary containment. This penetration vertically passes through the secondary containment and features a 90˚ bend such that it can blow room-temperature air on the lateral surface of the tubing to freeze the salt.

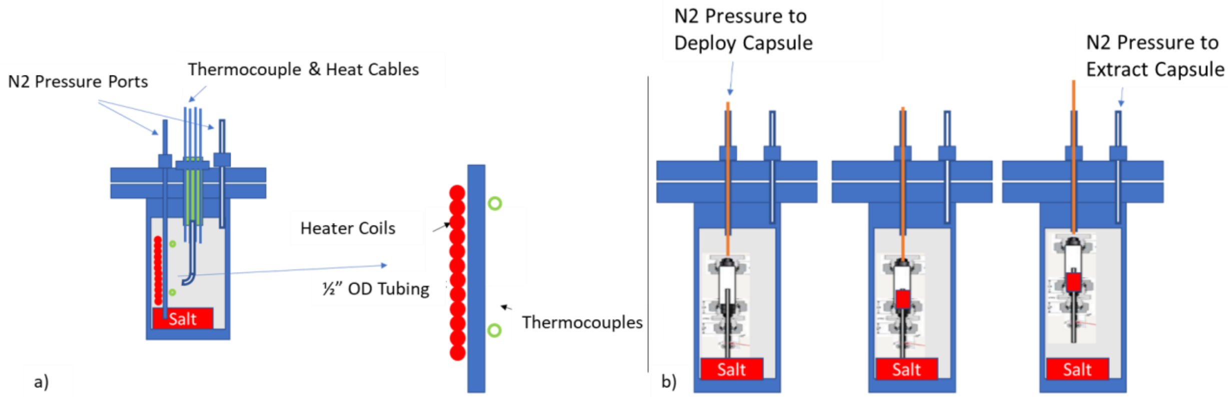

Figure 5. Conceptual model of (a) freeze port test and (b) sample collection test.

After freeze port testing, the test bed will be reconfigured to test the ability of the SCS to obtain a salt sample. The SCS in Figure 5b will be attached to a push rod that can be used to lower the capsule through the ½” OD secondary containment tube such that the needle is lowered into the crucible of salt. This can be done using the additional penetrations to increase the pressure in the secondary containment. This pressure difference simulates the pressure difference between the sample capsule and the primary molten salt system, which induces molten salt flow up into the body of the capsule.

4. CONCLUSIONS AND FUTURE WORK

The development of the MSSS design using the SiD methodology has led to the testing of the system within increasingly relevant environments. This testing is still ongoing; however, initial proof of concept has been demonstrated for a number of the MSSS’s subfunctions to-date. Future work consists of completing molten salt testing and compiling reliability data, developing additional reliability/failure rate estimates, and integrating the results into more detailed safety analyses of the MSSS.

ACKNOWLEDGMENTS

This work was supported by U.S. DOE’s Office of Nuclear Energy, through Integrated University Program Graduate Fellowship DE-NE0000646 and the Nuclear Energy University Program (DE-NE0008905).

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