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# Conceptual Design of a Salt Dechlorination and Vitrification Apparatus

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Changing the World's Energy Future

David D Tolman, Brian J. Riley

Idaho National Laboratory

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**http://www.inl.gov**

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**U.S. Department of Energy**
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# Conceptual Design of a Salt Dechlorination and Vitrification Apparatus (DeVA)

## Nuclear Fuel Cycle and Supply Chain

***Prepared for***
***U.S. Department of Energy***
***Material Recovery and Waste Form Development Campaign***


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# SUMMARY

The dechlorination and vitrification apparatus (DeVA) discussed in this report is a prototype conceptualization of a device that could be used to remove halogens from salt-based waste streams so that the remaining salt cations can be converted to oxides and immobilized in a glass-based waste form. The process described is designed around allowing for the utilization of a single system to perform both the dechlorination and the vitrification steps required to treat salt wastes. The chlorine is removed either as HCl, $NH_4Cl$, or a mixture thereof and these byproducts can either be captured directly or reacted with uranium metal to create $UCl_x$ within a separate chamber within the device. Several additional ideas are captured within this report that include a variety of features that can be included in the system design to accommodate different needs such as a way to charge dry or wet chemicals into the crucible, stir the melt, and actively cool the melt.

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# ACRONYMS AND ABBREVIATIONS

| | |
|---|---|
| An | actinide |
| BMI | Battelle Memorial Institute |
| BN | boron nitride |
| DeVA | Dechlorination and Vitrification Apparatus |
| DOE | Department of Energy |
| EBR-II | Experimental Breeder Reactor-II |
| EMM | Electro-Mechanical Manipulator |
| FCF | Fuel Conditional Facility |
| FOR | functional and operational requirements |
| HALEU | high-assay low-enriched uranium |
| HFEF | Hot Fuel Examination Facility |
| INL | Idaho National Laboratory |
| LEU | low-enriched uranium |
| MFC | Materials Fuel Complex (at INL) |
| MSR | molten salt reactor |
| natU | natural uranium |
| NE | (Office of) Nuclear Energy |
| PLC | programmable logic controller |
| PNNL | Pacific Northwest National Laboratory |
| PSIG | pounds per square inch in gauge |
| SDD | system design description |
| TC | thermocouple |
| TRISO | tristructural isotropic (fuel) |
| TSR | Technical Safety Requirement |
| USHYZ | ultrastable H-Y zeolite |

# 1. INTRODUCTION

Salt-based nuclear wastes are generated during different processes including electrochemical reprocessing of used nuclear fuel and from operation of molten salt reactors (MSRs). These generally include chloride-based and fluoride-based salt compositions. For electrochemical reprocessing, the LiCl-KCl eutectic salt is used, and common MSR salt compositions are fluoride mixtures (e.g., $LiF-BeF_2$) or chloride mixtures ($NaCl-MgCl_2$) with burnable actinide (An) fuels (see Table 1 for examples). While these are examples of electrolyte types, many other options exist. Also, MSRs can either be salt cooled with solid fuel (e.g., tristructural isotropic or TRISO fueled) or salt cooled with salt-based fuel – examples of these different types of MSRs are shown in Figure 1-1.

**Table 1. Summary of molten salt reactor concepts being explored including the reactor type, neutron spectrum, salt application, and salt system. The information in this table is courtesy of Ted Besmann. Note that "An" denotes actinide.**

| Reactor Type | Spectrum | Application | Salt System |
|---|---|---|---|
| Molten Salt Breeder Reactor | Thermal | Fuel | $^{7}LiF-BeF_2-AnF_4$ |
| | Fast | Secondary coolant | $NaF-NaBF_4$ |
| | | Fuel | $^{7}LiF-AnF_4$ |
| | | | $NaCl-MgCl_2-UCl_3-PuCl_3$ |
| | | | $LiF-NaF-BeF_2-AnF_3$ |
| Advanced High Temperature Reactor | Thermal | Primary coolant | $^{7}LiF-BeF_2$ |
| Very High Temperature Reactor | Thermal | Heat transfer coolant | LiF-NaF-KF |
| Liquid Salt Cooled Fast Reactor | Fast | Primary coolant | $LiCl-NaCl-MgCl_2$ |
| | | Intermediate coolant | $NaNO_3-KNO_3$ |

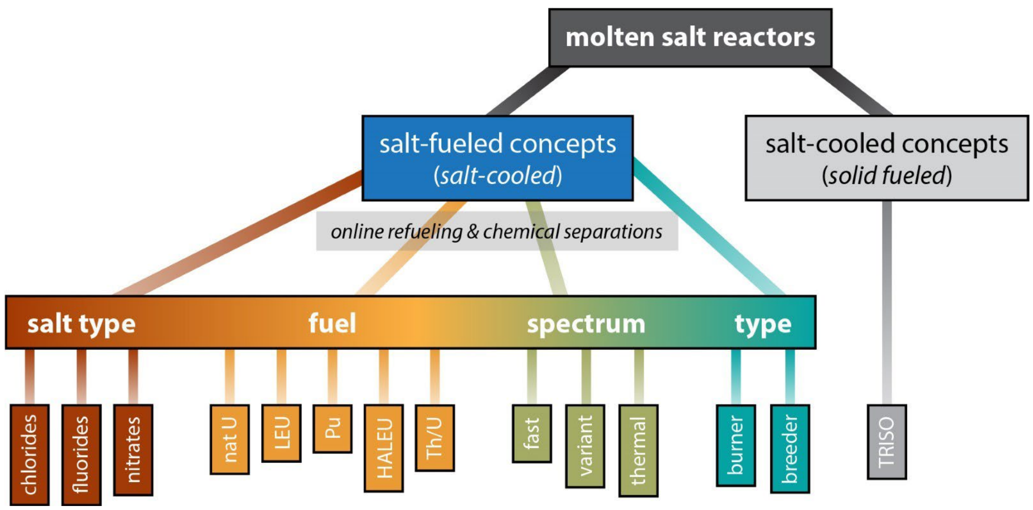


**Figure 1-1. Different types of MSRs. Definitions of abbreviations are provided in the text. This drawing was modified from the original from Patricia Paviet (PNNL) by B. Riley. Note that natU, LEU, HALEU, and TRISO denote natural U, low-enriched U, high-assay low-enriched U, and tristructural isotropic (fuel), respectively.**

All of these streams contain a wide range of fission products. In order to immobilize these wastes in a stable form for long-term (geological) time scales, the salts will likely require some type of stabilization process(es) unless direct disposal is an option. The options for stabilization include removal of actinides to prevent generation of volatile halide compounds (e.g., $UF_6$). Also, radiolysis of solidified halide salts containing fission products can lead to the production of gaseous byproducts (e.g., $F_2$) (Davis et al. 2023). Thus, one of the most promising options for salt stabilization is a process called dehalogenation where the halide fraction of the salt stream is removed. Dehalogenation can be conducted using a variety of processes including the following:

- Reacting salt with phosphates [e.g., $H_3PO_4$, $NH_4H_2PO_4$, $(NH_4)_2HPO_4$] (Donze et al. 2000; Siemer 2012; Lee et al. 2019; Riley and Chong 2020; Riley et al. 2020; Riley et al. 2021). Reactions with these precursors result in the removal of Cl as HCl or $NH_4Cl$ that can be captured and recycled (if enriched in $^{37}Cl$) or discarded. An example reaction is: 2 NaCl + 2 $NH_4H_2PO_4 \rightarrow Na_2O$-$P_2O_5$ + 2 $NH_4Cl$+ 2 $H_2O$. Once converted to an oxide, these compounds can be immobilized in a glass-based waste form.

- Reacting salt with organic acids like oxalic acid ($C_2H_2O_4$) (Dong et al. 2022). Reacting halide salts with organic acids results in the production of HCl as a byproduct followed by the conversion of the salt cation to an organic salt. An example reaction is: 2 NaCl + $C_2H_2O_4 \rightarrow Na_2C_2O_4$ + 2 HCl. Once converted to an organic salt, higher heat treatment temperatures result in conversion of the organic salt to a carbonate and then to an oxide that can be immobilized in a glass-based waste form.

- Reacting salt with ultrastable H-Y zeolite (USHYZ) (Gardner et al. 2020; Gardner et al. 2021). Reactions between USHYZ and halide salts release HCl as a byproduct and the salt cations are incorporated into the zeolite. Once loaded, these can be incorporated into a glass-bonded ceramic waste form.

In order to perform dechlorination processes, the reactions require a careful balance of temperature, heat treatment schedules (temperature versus time), byproduct capture and management, and chemical additions at different stages. The current study was aimed to provide conceptual designs of an apparatus to support both dehalogenation (temperatures up to 650°C) and vitrification (temperatures greater than 1100°C) efforts for treating and immobilizing the cations of salt-based nuclear wastes to be deployed at the Fuel Conditioning Facility (FCF) within the Materials and Fuels Complex (MFC) at the Idaho National Laboratory (INL). It is possible that a single system could be designed to implement a variety of different dehalogenation options for treating both chloride-salts and fluoride-based salts to support closing the backend of the advanced reactor nuclear fuel cycle. This work is building off previous experiments performed at INL as proof-of-concept for converting uranium dendrites into $UCl_3$ in a single process through dechlorination of chloride salt (Frank et al. 2018; Riley et al. 2020).

# 2. SYSTEM DESCRIPTION

## 2.1 System Identification and Location of Deployment

A series of designs for a general-purpose salt Dechlorination and Vitrification Apparatus (DeVA) are provided in the sections below. In the initial conceptual design, the system is designed for processing chloride-salts from nuclear fuel pyroprocessing applications and could potentially be used for MSR used fuel salt processing. This engineering design will focus on salt waste from the EBR-II used fuel pyroprocessing in the Fuel Conditioning Facility (MFC-765) and Hot Fuel Examination Facility (HFEF) Integrated Recycle Test Electrorefiner. Potential locations for the DeVA include the FCF argon cell or the HFEF main cell.

## 2.2 Limitations of the System Design Description

This system design description (SDD) is currently at the conceptual design stage and is used to document the initial functional and operational requirements (FOR) of the apparatus that will be used for verification of the design and final validation of the fabricated apparatus. This SDD scope is limited to the DeVA and does not include facility confinement, shielding, criticality, mass tracking, etc. requirements that are required to handle the highly radioactive salt material that will be used in the DeVA. This SDD focuses on the research, process, and facility interface requirements.

## 2.3 Definitions/Glossary

*Condenser*: The condenser refers to the portion of the DeVA that is located above the hot zone (furnace) and captures the condensable off-gas from the dechlorination process.

*Dechlorination*: For the purposes of this system, dechlorination is the process step that takes place at approximately 500-650°C where the salt is converted into an intermediate glass. This step in the process produces off-gas constituents such as HCl and $NH_4Cl$.

*Hotcell*: a confined space that is heavily shielded for radiation that is used to handle and process highly radioactive materials. Typically, operation of equipment in a hotcell is done using remote means (manipulators, cranes, etc.) and cannot be operated by hand.

*Salt*: In this document, salt is referring to a compound containing chlorine such as LiCl, KCl, NaCl, CsCl, and other chloride compounds that may be present in the electrorefiner salt.

*Thermocouple*: a temperature sensing element consisting of two different metal types. A voltage is created that can be correlated to temperature. For the purposes of this system, thermocouple (TC) is referring to Type K thermocouples.

*Vitrification*: For the purposes of this system, vitrification is the process where the intermediate glass is heated to approximately 1000–1100°C. Additives (such as $Fe_2O_3$) may be added to form a durable final waste form.

# 3. GENERAL SYSTEM OVERVIEW

## 3.1 System Functions

The DeVA will be used for dechlorination and vitrification experiments to establish a process to create a final durable waste form from salts used in spent fuel electrorefiner reprocessing and molten salt reactors.

## 3.2 System Classification

The DeVA does not have any nuclear safety related (safety significant or safety class) functions. It will be housed within a shielded hotcell that provides the safety functions for confinement and shielding of the nuclear and radiological materials.

## 3.3 Basic Operational Overview

The DeVA is a batch process system in which the waste salt and dechlorination reactants are loaded into a crucible and placed in a well (retort) type furnace. A condenser unit is placed on the top of the furnace well. The condenser may or may not (depending on the experiment being performed) contain dendritic uranium metal to capture the $NH_4Cl$ off-gas, converting the uranium metal to uranium chloride. If uranium is not used, the condenser provides relatively cold surfaces for the off-gas constituents to condense. As the well furnace is heated to dechlorination temperatures (typically 500°C to 650°C) the condenser throat temperature can be independently controlled by a condenser heater. Following the dechlorination step, reactants for vitrification (such as iron oxide) may be added via a funnel into the crucible. The well furnace is heated to vitrification temperatures (typically 1000°C to 1100°C). In some experiments it may be desired to rapidly cool the final vitrified product. Typically, in lab or industrial settings, this might be done by pouring the material onto a cold surface or mold. This is not practical for highly radioactive material in a remote hotcell environment. Alternatively, the material may be cooled by inserting a relatively large copper chill rod into the furnace. There may need to be some additional lab work to verify that the chill rod concept provides the desired results. The following figures provide general apparatus configurations.

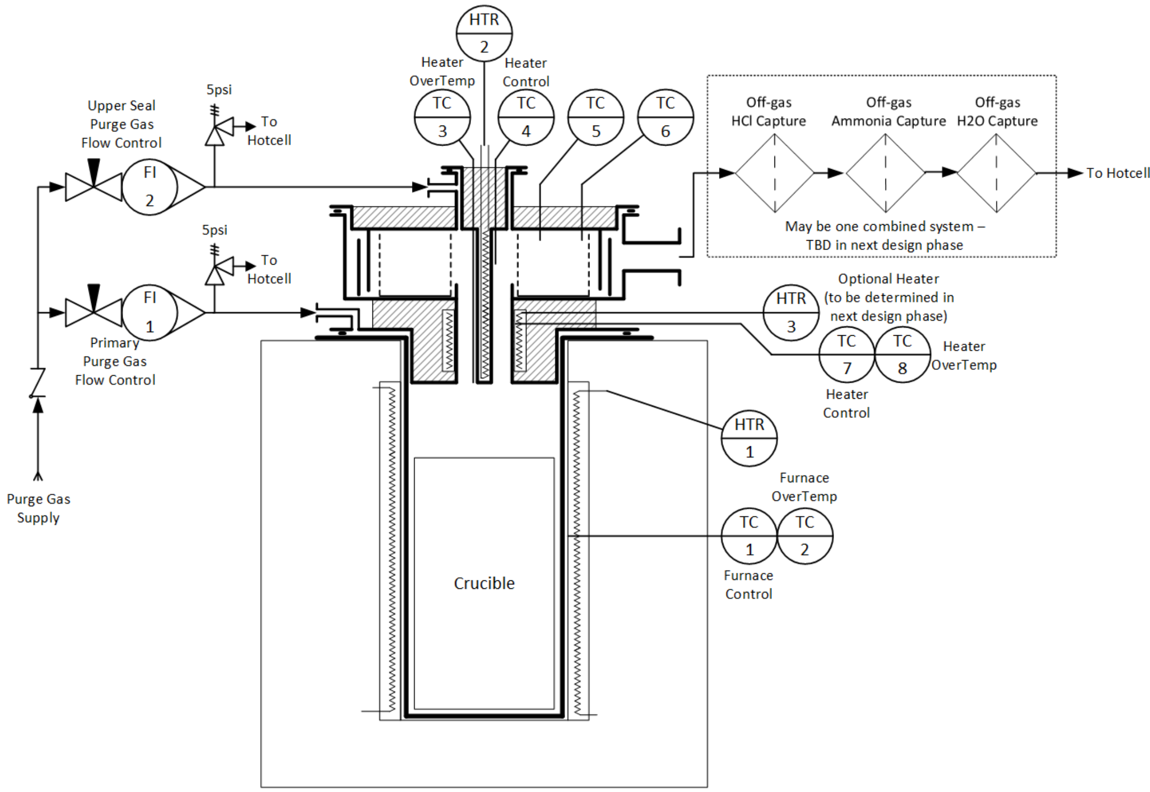


**Figure 3-1. Conceptual Process and Instrumentation Diagram.**

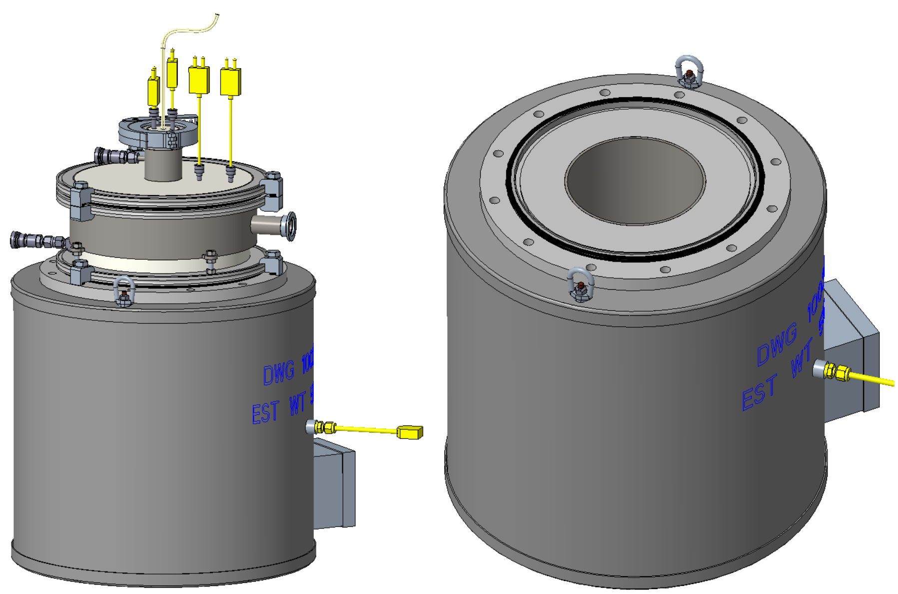

**Figure 3-2. Conceptual model shown (left) with the condenser and (right) without condenser.**

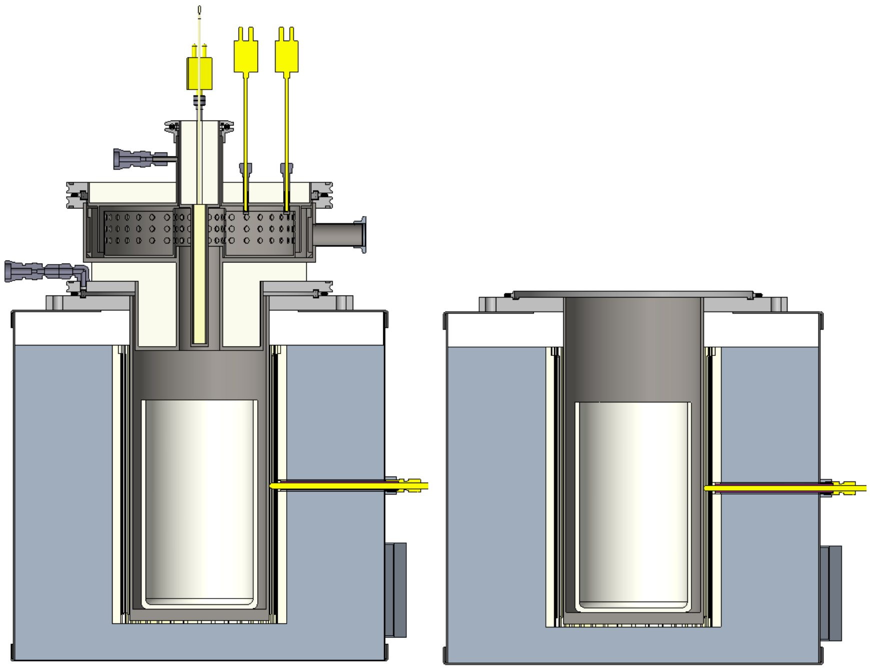

**Figure 3-3. Conceptual model section view showing the system (left) with the condenser and (right) without condenser.**

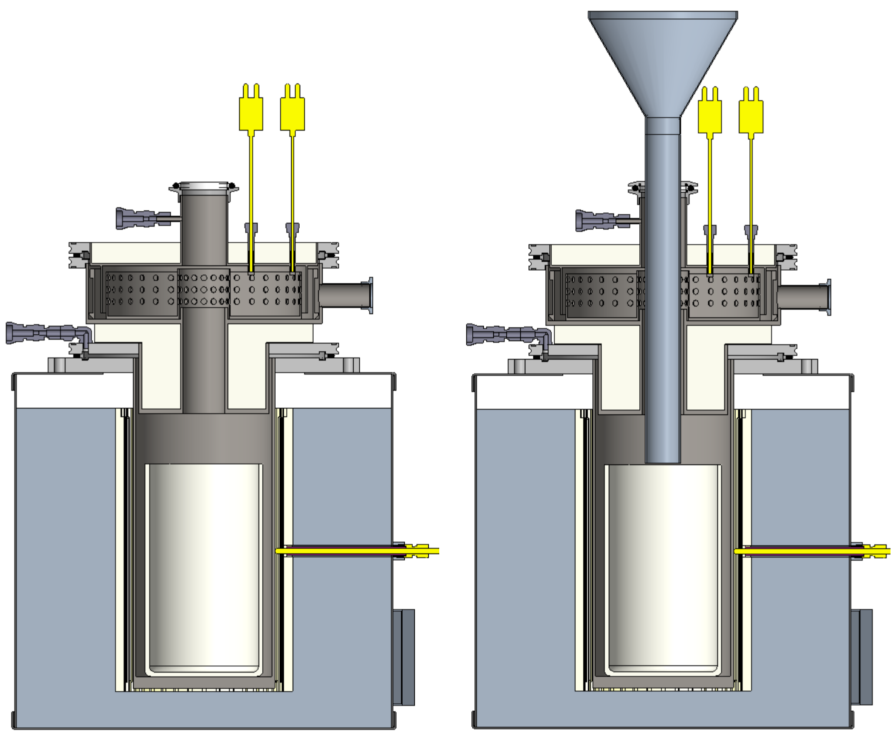

**Figure 3-4. Conceptual section view – (left) without condenser heater and (right) with funnel.**

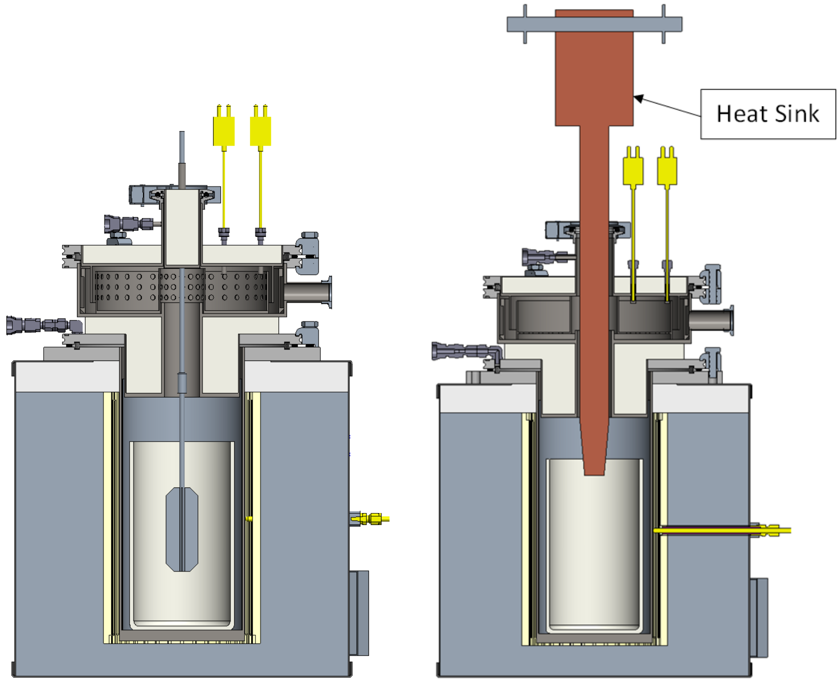


**Figure 3-5. Conceptual model section view shown (left) with a stirrer and (right) with chill (heat sink) rod.**

# 4. REQUIREMENTS AND BASES

## 4.1 Research/Process-Related Requirements

The list of research and process-related requirements are described below in more detail but, in general, include the following:

1. The apparatus shall accommodate a feed salt batch size of at least 500g.
2. The apparatus shall be able to be used for the following processes: iron-phosphate (i.e., $Fe_2O_3$-$P_2O_5$ glass-forming system) process (Donze et al. 2000; Siemer 2012; Riley et al. 2020; Riley et al. 2021), silica-alumino-phosphate (SAP) process (Park et al. 2007a; Park et al. 2007b; Park et al. 2008; Park et al. 2011; Lee et al. 2019), and the oxalic acid process (Dong et al. 2022).

The list of general process requirements for the general apparatus includes the following items:

- The apparatus shall be capable of providing a maximum process temperature of 1200°C. *Basis: process requirement.*
- The apparatus shall have a condenser to capture ammonium chloride ($NH_4Cl$) by either reacting it with uranium dendrite or condensing it on a cold surface (cold relative to the furnace hotzone) *Basis: process requirement.*
- The off-gas flow through the condensing region should be horizontal to prevent $NH_4Cl$ from falling back into the hot zone. *Basis: recommended improvement #2 to the Generation 2 Dechlorination apparatus at PNNL* (Riley et al. 2021).
- The apparatus shall utilize a purge gas to drive condensates out of the furnace efficiently. *Basis: recommended improvement #2 to the Generation 2 Dechlorination apparatus at PNNL* (Riley et al. 2021).
- The apparatus should have the ability to add (funnel in) chemical reactants (such as iron oxide particles) while the device is hot such that the vitrification step may be performed following dechlorination without cooling down. *Basis: recommended improvement #3 to the Generation 2 Dechlorination apparatus at PNNL* (Riley et al. 2021).
- Consideration for quenching the final product should be considered in the design; however, due to the challenges with doing this in a scaled up and remotely operated system, this is not a firm requirement. *Basis: rapid cooling is desired to form an amorphous final product for increased durability.*
- Consideration for a stirring device should be considered; however, due to the challenges with doing this in a remotely operated system, this is not a firm requirement. *Basis: mechanical mixing capability is desirable in this process.*
- The apparatus shall have the capability of handling the following off-gas constituents:
    - Hydrogen chloride gas (HCl)
    - Ammonia gas ($NH_3$)
    - Hydrogen gas ($H_2$)
    - Water vapor ($H_2O$)
    - Ammonium chloride ($NH_4Cl$)

  *Basis: process requirement*
- The apparatus shall be capable of providing oxygen-containing purge gas for salt processes that may require oxygen. *Basis: process requirement*

The list of general process requirements for the condenser portion of the system includes the following items:

- The uranium dendrite volume (for capturing $NH_4Cl$) shall be of similar volume as the salt crucible. *Basis: It is anticipated that the required dendrite volume will be similar to the process volume. This is based on rough order calculations based on estimated dendrite density.*
- The condenser shall have temperature monitoring thermocouples within the condensing region. *Basis: process requirement*
- The condenser shall have heater/temperature control such that the off-gas constituents remain gas until the desired condensing zone. *Basis: process requirement*
- The condenser outlet should have the ability to be connected to a vacuum system. *Basis: while none of the current salt processes require vacuum; it may be desirable for future experiments. Additionally, providing a negative pressure (vacuum) on the offgas may be advantageous in keeping the off-gas constituents contained in the apparatus and help to drive the off-gas flow in the desired flow pathway.*

The list of general process requirements for the material containment (i.e., the crucible and/or liner for the crucible) includes the following items:

- The apparatus shall be designed with secondary containment such that if primary crucible failure occurs the material will be contained and will not adversely affect the apparatus. *Basis: process requirement*
- The apparatus shall be designed to accommodate a variety of crucible materials to support a variety of salt dechlorination and vitrification experiments. Some common materials that may be considered are:
  - Alumina ($Al_2O_3$)
  - Carbon (graphite / glassy carbon)
  - Nickel or nickel alloys (e.g., Inconel, Hastelloy)
  - Boron nitride (BN)
  - Silica (fused quartz or high silica)

  *Basis: process requirement*

## 4.2 Facility-Related Requirements

A list of facility-related requirements is provided below:

- The device shall meet the requirements of F3000-0001-AJ, "Fuel Conditioning Facility Argon Cell and Air Cell Equipment" section 2.2 "Design Requirements"
- The device shall utilize existing hot-cell general purpose, spare, or unused feedthroughs connections.
- All in-cell electrical connections shall be manipulator friendly and shall be made using Amphenol connectors where practical.
- An equipment stop button shall be provided for manual shut down of the furnace.
- Training Requirements will be determined later in the design process.

## 4.3 Engineering Discipline Requirements

The mechanical and material requirements will be determined as the design progresses.

The electrical power requirements include the following:

- It is anticipated that the well furnace will require 230V, 20A, 1PH, 60Hz (4600 W). *Basis: HFEF Bakeout Furnace Requirements, Dwg 1003684 and 755147*
- The condenser heater may require 120V or 230V as determined in the design. A preliminary estimate of 735 Watts. *Basis: Watlow Cartridge Heater Model Number CSH-306735/* was used in the conceptual design*
- Additional incidental 120V power will be required for control cabinet, etc.

The instrumentation and control requirements include the following:

- Thermocouple should be of Type-K in order to utilize existing facility feedthroughs.
- Electronic components may be highly susceptible to damage and should be tested before installation, shielded (or shown to withstand the radiation fields), or placed outside the hotcell.

Computer hardware and softwarae requirements will be determined in the detailed design process.

Fire protection requirements include the following:

- The furnace shall conform to the requirements of NFPA 86, "Standard for Ovens and Furnaces".
- Independent over temperature cutoff shall be implemented in the furnace design and heaters to prevent overtemperature if the primary furnace controller fails.

Radiological protection requirements include the following:

- The equipment should be designed to minimize radioactive contamination traps such as notches, cracks, crevices, and rough surfaces. *Basis: standard radiological contamination control practice*

## 4.4 Testing and Maintenance Requirements

Regarding testability, the device shall undergo validation testing in a phased approach prior to implementation in the hotcell. *Phase I*: test the equipment functionality in a hands-on setting. *Phase II*: test the equipment operability in a remote mockup setting. *Phase III*: Installation and final location functionality and operability testing in the remote hotcell environment.

- Technical Safety Requirement (TSR)-Required Surveillances: none.
- Non-TSR Inspections and Testing: to be determined as the design progresses.
- Maintenance: Since this device is deployed in a remote operations hotcell environment, it is anticipated that no hands-on maintenance can occur. The design should include features such as to allow individual components to be replaced remotely using remote manipulators and electro-mechanical manipulators.

## 4.5 Other Requirements

- Security and Special Nuclear Material (SNM) Protection: none. The facility where this device is housed will provide the necessary security and SNM protection.
- Special Installation Requirements: This device is deployed in a remote operations hotcell environment. It must be designed to be transferred through the hotcell airlocks using existing facility handling equipment (cranes, EMMs). All connections (pneumatic, electrical, I&C) must be made using remote manipulators.
- Reliability, Availability, and Preferred Failure Modes: it is anticipated that the equipment will not provide any safety functionality.

- Quality Assurance: Specific quality assurance requirements will be determined later in the design process. At a conceptual level, it is anticipated that this equipment will not provide any nuclear safety function.
- Assumptions: These assumptions should be validated in lab scale tests prior to equipment implementation.
    - The chill rod method is adequate for salt quenching (as opposed to dumping the product onto a chill plate)
    - Chemical addition at temperature (post dechlorination / pre vitrification) can be done via funneling into the crucible.
    - Shock-sensitive trihalide compounds (i.e. $NCl_3$, $NBr_3$, $NI_3$, and $NH_4I_3$) will not form in significant quantities to pose a risk to the system or facility (Riley and Chong 2022).

## 4.6 Supporting Information

Supporting information for various categories of interest include the following:

- *Need for Configuration Management*. The permanent furnace components and facility interfaces will be configuration managed via the FCF master equipment list. Items internal to the furnace (crucibles, condensers, etc.) that are reconfigured from experiment-to-experiment will not be configuration managed in the FCF master equipment list.
- *Sensitive Information*. Documentation shall be reviewed for Controlled Unclassified Information (CUI) per LWP-11200, "Classified Matter Protection and Control."
- *Export Control*. All information exported to sources outside the Idaho National Laboratory shall be submitted to the Scientific and Technical Information Management System (STIMS) for export compliance review per LWP-1450, "Export Compliance".
- *Need for Engineering Change Control*. The INL engineering change process will be followed for design control and equipment configuration management per KB0015843, "Engineering Initiation".
- *Level of Verification Needed*. The functional and operational requirements shall be verified via checking (at a minimum) and documented in a verification matrix as part of the INL engineering change control process. Remote operation requirements will be validated via mockup qualification testing using remote handling equipment.
- *Technical Integrator*. The technical integrator for this equipment will be designated by the MFC engineering review board.

# 5. CONCEPTUAL DESIGN - SYSTEM DESCRIPTION

## 5.1 Description of System, Subsystems, and Major Components

Regarding the general system overview, the DeVA consists of a well furnace, retort well, condenser, flange seals, purge gas system, off-gas system (post condenser), chemical addition funnel, crucible(s), chill rod, remote handling tooling, and instrumentation and control (see Figure 5-1). This conceptual design is based on using well furnace with a 6" diameter and 12" tall, heated zone. If it is determined in the detailed design process that a larger batch size is desired, there should not be anything that would prohibit slight scaleup while using the same design concept (such as scale up to 8" diameter or 10" or potentially even 12").

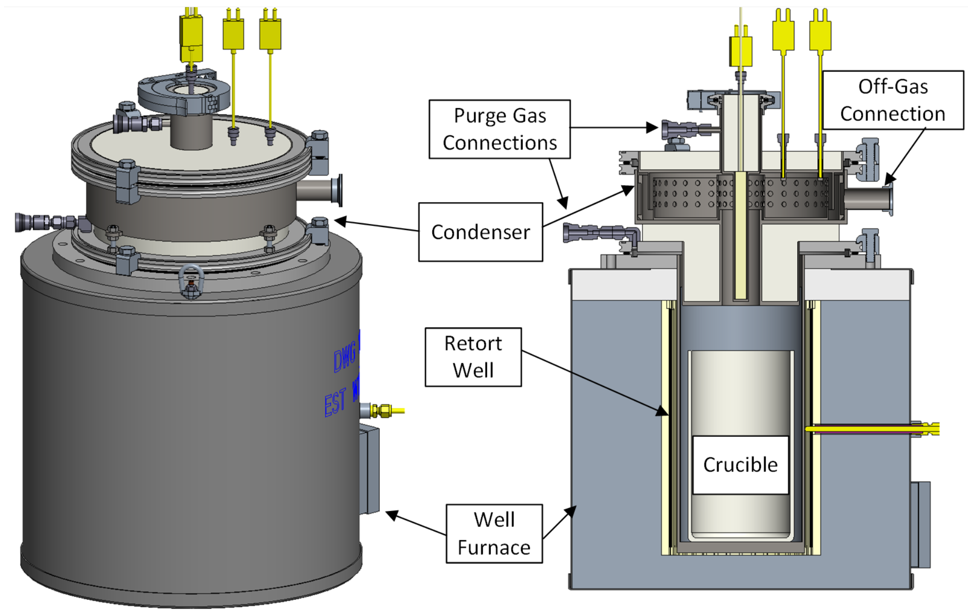


**Figure 5-1. Concept of DeVA general layout including (left) the assembled apparatus and (right) the section view.**

If the DeVA is to be deployed in HFEF the existing bakeout furnace may be used. See drawing 1003684. If the DeVA is to be deployed in FCF a furnace like the HFEF bakeout furnace would be deployed (see Figure 5-2). A retort well provides a sealed boundary between the furnace heating elements and insulation and the process gasses. The retort well is constructed of an Inconel cylindrical flat bottomed well welded to a 300 series stainless steel ISO Large-Flange NW250 Vacuum Chamber Flange. The flange portion will not be subjected to as high of temperature and therefore will not need to be Inconel. The well is nominally 6-inch diameter and 14-inches tall. The well wall thickness is 0.120-inch. This provides enough volume for the anticipated crucible size as well as secondary containment – this is shown in Figure 5-3.

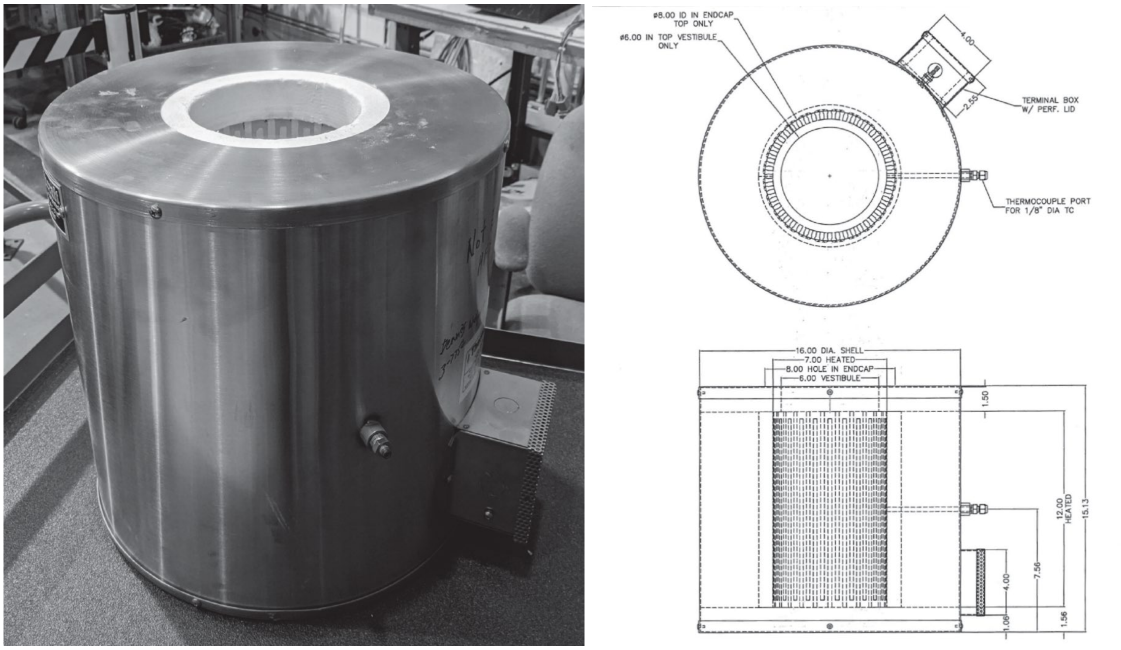


**Figure 5-2. HFEF bakeout furnace – excerpt from drawing 1003684.**

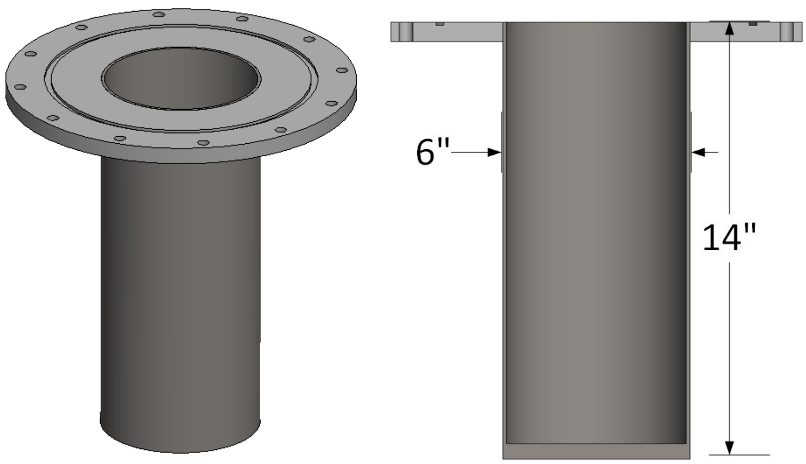


**Figure 5-3. Retort well – isometric view (left) and section view (right).**

The DeVA condenser (see Figure 5-4) provides a means to capture the condensable off-gas and also provides a location for reaction of chlorinated off-gas (such as $NH_4Cl$) to react with high-surface-area uranium to form $UCl_3$ for electrorefiner use (Herrmann 2017; Riley et al. 2020). The condenser is designed such that the thermal gradient is primarily horizontal, such that any condensate should stay in the condenser pan instead of falling back into the hot zone. Toward the outside of the condenser, vertical baffle surfaces provide a large surface area for vapors to condense while not plugging up the off-gas flow path. The condenser also serves as a lid for the furnace well.

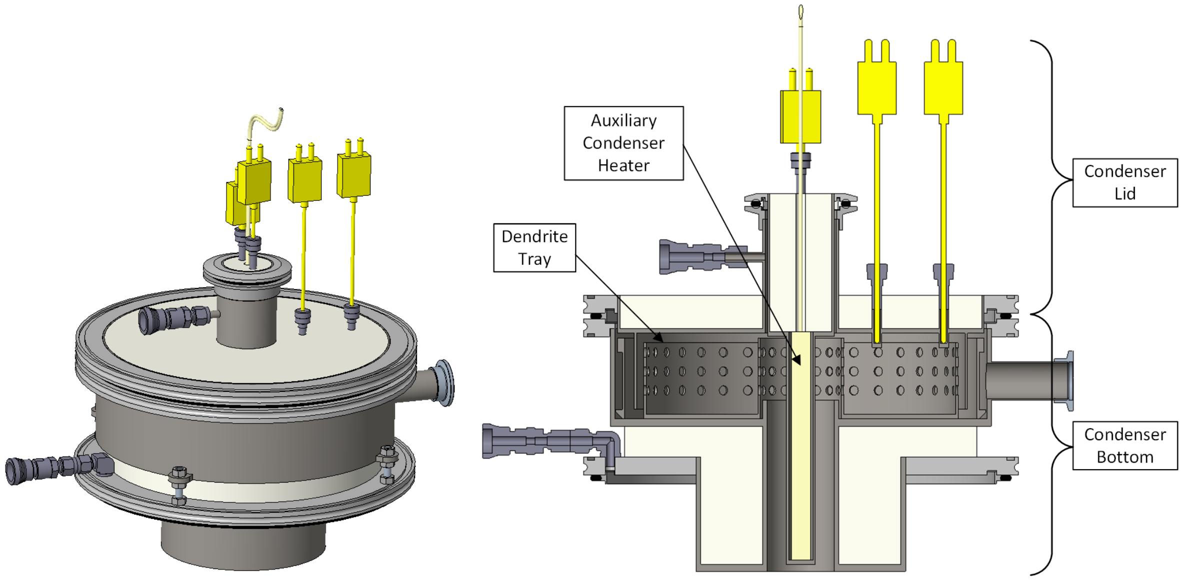


**Figure 5-4. Condenser Layout – Isometric view (left) and section view (right)**

The condenser is comprised of several subcomponents including the bottom, the lid, the dendrite tray, the heater, the flange/sealing components, the purge-gas system, and the off-gas system (post condenser). Several items can be inserted into the melting chamber through the top of the condenser lid including a chemical addition funnel, chill rod device, and stirring device. Additional information is given for the crucible material, but this could be exchanged for other crucible sizes and materials of construction.

*Condenser bottom* (Figure 5-5). The condenser bottom is the primary component of the condenser. It serves as a lid for the furnace well providing insulation to the top of the hot zone. A center opening/port "condenser throat" provides a path for the off-gas to migrate from the furnace well to the condensing region. The condenser bottom surfaces exposed to the hot off-gas stream is constructed from Inconel. The flanges will not be exposed to hot off-gas and are 300 series stainless steel. An option to embed a heater around the throat is shown. Determination if this is needed or desired will be made as the design progresses.

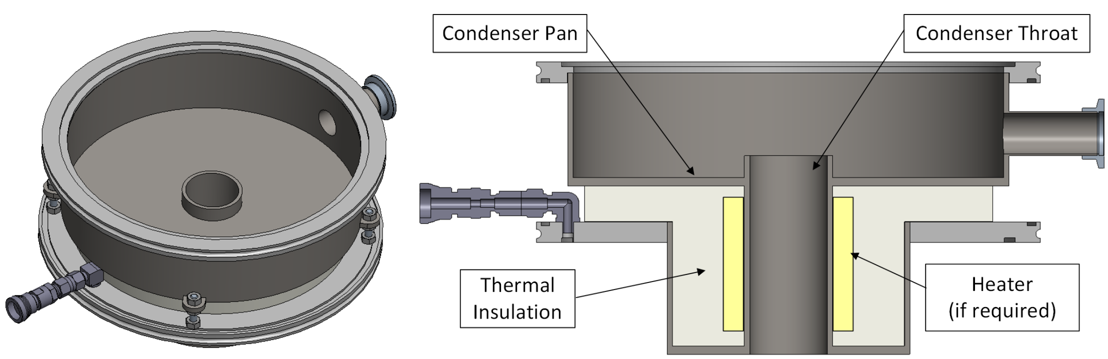


**Figure 5-5. Condenser Bottom – (left) isometric view and (right) section view.**

*Condenser lid* (Figure 5-6). The condenser lid includes a top port for the condenser heater, chemical addition, and chill rod insertion. Thermocouple wells are provided to monitor the temperature in the condensing region. Thermal insulation helps to maintain the desired condenser temperature and thermal gradient from the hot center to the cooler outer. If it is determined in the detail design, or through testing, it may be possible to add heater(s) to the top surface of the lid to provide another method to control condenser temperature.

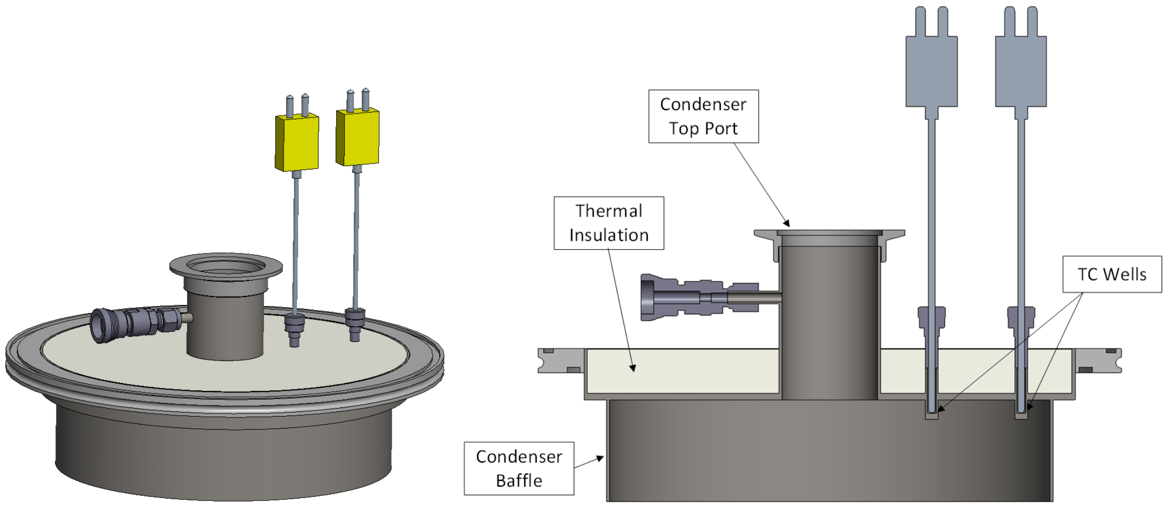


**Figure 5-6. Condenser lid including (left) isometric view and (right) section view.**

*Dendrite tray* (Figure 5-7). The dendrite tray is a removable tray that sits in the condenser pan when in use. It provides a means to contain uranium dendrite and any uranium chloride produced. Two options are presented, but it is anticipated that other dendrite tray options may be constructed for individual experiment needs. The tray shown below on the left provides off-gas cross flow while the tray on the right will provide off-gas flow from the bottom up.

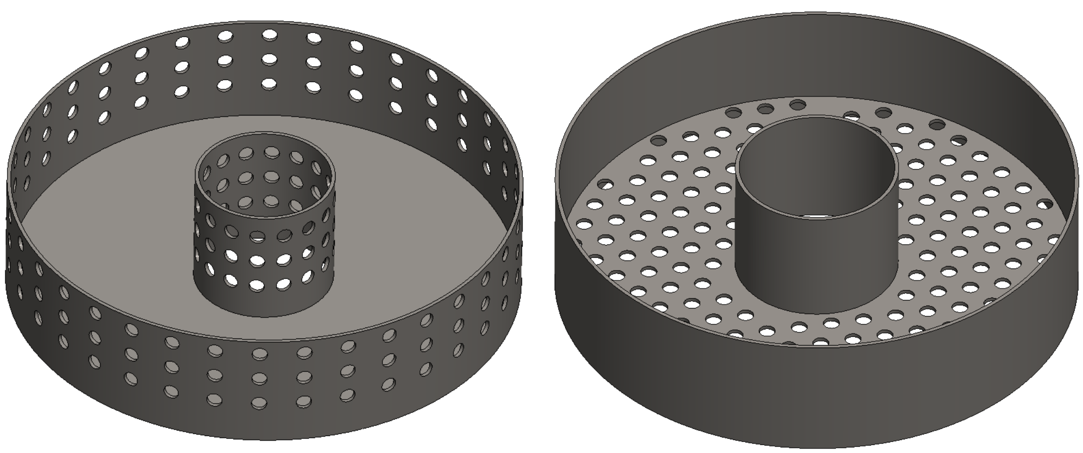

**Figure 5-7. Dendrite tray options including (left) horizontal flow and (right) vertical flow.**

*Condenser heater* (Figure 5-8). The condenser heater is inserted into the condenser top port. It contains a cartridge heater. The primary purpose is to keep the throat of the condenser at a high enough temperature such that condensable off-gas (such as ammonium chloride) makes it to the condenser pan and dendrite tray as a gas. Two type K thermocouples are used as process monitoring and heater temperature control feedback. The top portion of the condenser heater is a plug for the condenser port and provides thermal insulation.

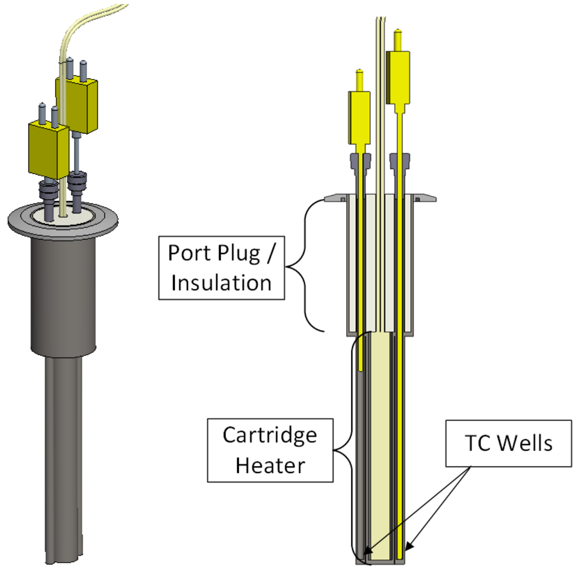


**Figure 5-8. Condenser heater showing (left) isometric view and (right) section view.**

*Flange/sealing components*. Although there is not a hard requirement for a hermetically sealed system, it is desirable to maintain control of the reaction off-gas. Standard vacuum flanges are used for this purpose. The large flanges (between the retort well, condenser, and condenser lid) are ISO Large-Flange size NW250. Viton o-ring seals are used due to successful history of use of this material in the hotcell environment and the relatively high temperature capability. It may be determined as part of the detailed design that the seals may need to be further removed from the hot zone (make the flanges bigger) in order to use an elastomeric o-ring. Alternatively, if it is determined that the seal needs to be at higher temperatures than what an elastomer material can handle, graphite or metal gaskets may be an option to be determined in the detailed design process. While the current design does not require (or is not even desired) to be operated under vacuum conditions, the mechanical design of the system is such that it could be used under rough vacuum conditions if desired.

*Purge-gas system*. The purge gas system serves two purposes: 1) to provide a carrier gas to sweep/carry the off-gas away from the hot zone, through the condenser, and off-gas system and 2) to keep condensable gases out of the sealing areas. The purge gas flow is controlled via rotameters and needle valves. The gas flow rate will be determined in the detailed design and through equipment testing to determine the optimal flow rate for operation. Overpressure relief valves will be provided to ensure that the system is not over pressurized. It is anticipated that the system will be maintained much less than 15 PSIG (pounds per square inch in gauge). In the conceptual design, two purge locations have been identified:

1) Near the seal between the condenser and retort well. This purge flow keeps condensable gasses away from this annular seal area. This will also be the primary carrier gas for the off-gas since it will sweep across the top of the hot zone and up into the condenser throat.

2) The second purge gas location is at the top port annulus. The primary purpose is to keep condensable gasses away from this seal area.

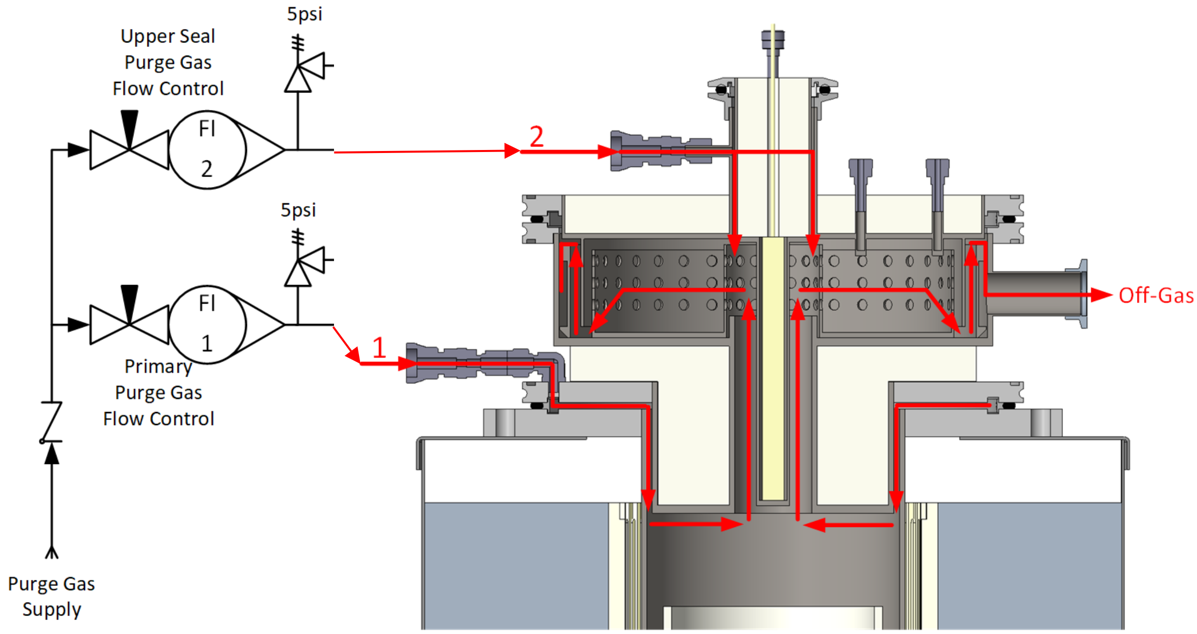


**Figure 5-9. Purge gas flow path.**

*Off-gas system (post condenser)*. A post condenser off-gas filter/absorber will remove any non-condensable gases/vapors (such as ammonia and HCl). Steel wool and activated carbon filters may be used to capture these off-gas constituents. This is not shown in the conceptual model but is anticipated to be canister style filter(s) with replaceable filter/absorption media. Depending on the size, the filter may be rigidly attached to the condenser outlet, or a separate component attached by a flexible stainless-steel hose. Consideration for moisture in the off-gas will also be made as the design progresses. It is anticipated that approximately 175 mL of water will be generated from a 500 g salt batch. The final design will need to consider collecting the water, absorbing it, or potentially venting it into the hotcell atmosphere.

*Chemical addition funnel*. In some salt waste processes, addition of chemicals following dechlorination and prior to vitrification is desired. The condenser top port allows access with the use of a funnel to add chemicals while the crucible is hot. This requires removal of the condenser heater from the condenser top port. The funnel is made of a 1.5”-diameter stainless steel tube approximately 12” long with a 6” diameter funnel welded to the top.

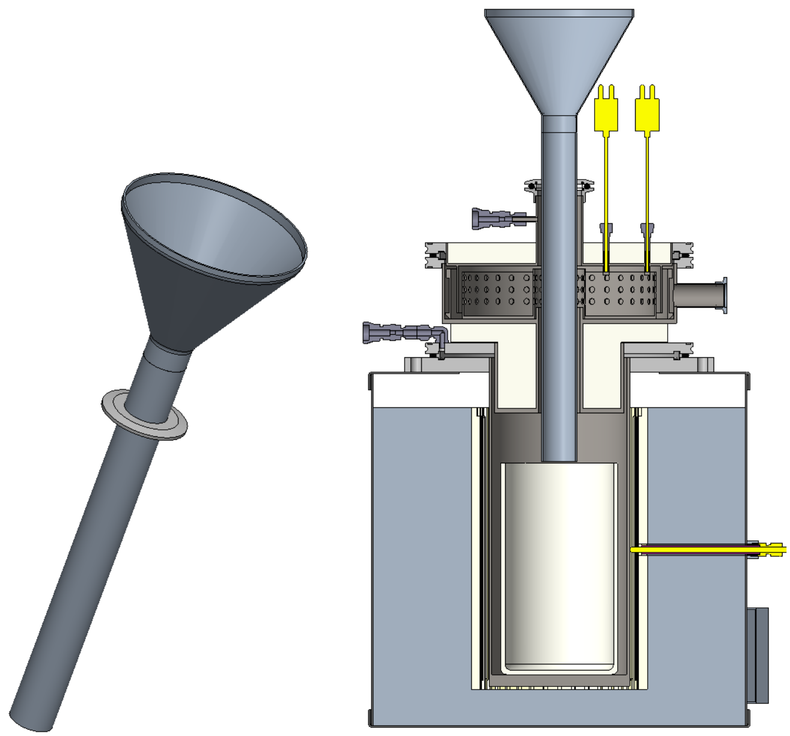

**Figure 5-10. (left) Chemical addition funnel and (right) section view installed in condenser.**

*Chill Rod Device* (Figure 5-11). The chill rod device can be used if the salt waste vitrification process requires rapid cooling. The rod is constructed of copper which has a high thermal conductivity and high thermal mass. The final rod design will be sized such that the entire thermal capacity of the waste product can be absorbed by the copper without raising the cooper temperature above 200°C. For the conceptual design, the chill rod is shown as a 1-1/2" diameter copper rod that is 18" long with a 4" diameter x 6" tall heat sink at the top. It has a has a mass of approximately 15kg. If a larger thermal mass is required, the heat sink size can be easily increased.

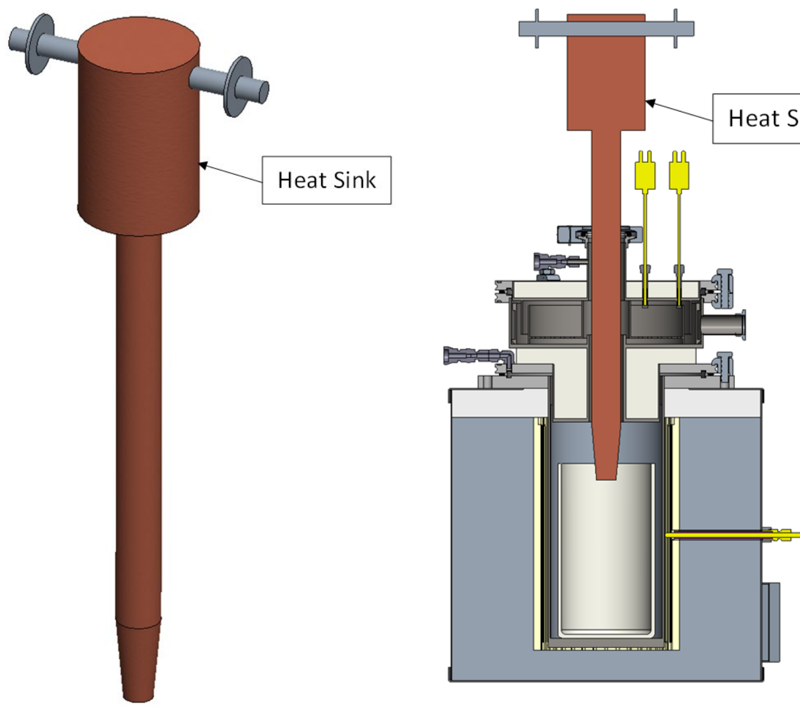


**Figure 5-11. Chill Rod – Isometric view (left) and section view installed in condenser (right)**

*Stirring Device* (Figure 5-12). Conceptually, a stirring device may be inserted through the condenser top port following funneling in vitrification additives (such as iron oxide). The salt/glass wetted stirring materials may be dependent on the specific experiment being performed. The design incorporates a high temperature coupling so that the wetted stirrer can be replaceable. The stirrer is driven by a motor that will withstand the hotcell environment.

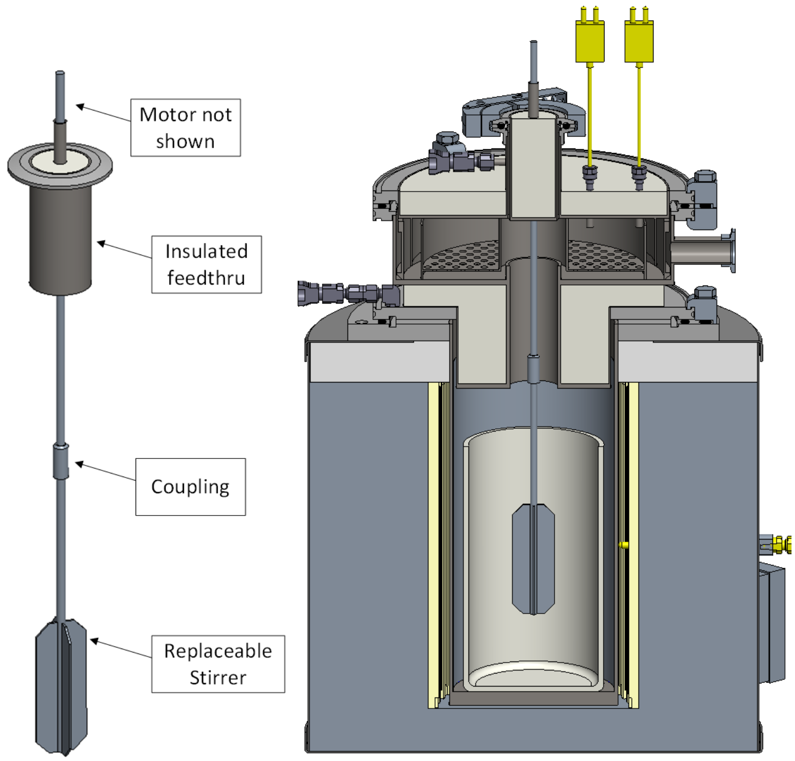


**Figure 5-12. Stirring device that could be added into the crucible through the top port to assist with stirring the melt during dechlorination or vitrification.**

*Crucible(s)* (Figure 5-13). The design intent for the DeVA is such that a variety of crucible materials and crucible sizes can but used. Shown in most of the figures in this concept is a 127-mm (5") diameter × 228-mm (9") high alumina crucible with 2.4-L capacity (CoorsTek Part #65547). While not shown in the conceptual design, a method to retrieve crucibles (crucible holder with grapple attachment point) will be provided in the final design. Regarding crucible volume considerations, the design input is to have a 500 g salt capacity per batch. Assuming a salt loading of 25%, the total waste form would be on the order of 2 kg. The salt and reactants for the dechlorination process would typically have densities of 2 g/cm$^3$ yielding a total volume of 1 L. Since the constituents will have a packing factor and a desire for head room, a crucible with minimum capacity of approximately 2.5 L should be used. This volume value may be revised through more detailed calculations or testing, but for a general concept, development a crucible of 2.5 L volume is conservatively large enough. It is anticipated that as this process is developed the system may be capable of larger amounts of salts than 500 g.

**Figure 5-13. Picture of CoorsTek cylindrical alumina crucible.**

## 5.2 Remote Handling

At this conceptual design stage, the remote handling features have not been fully considered or modeled. However, it is anticipated that each DeVA component will be well below the 750 lb lifting capacity of the hotcell electro-mechanical manipulator (EMM) and will have lifting features compatible with the hotcell EMM. Potentially, a specialized cradle or grapple for the condenser might be designed that would have the function to be able to tilt the condenser toward the hotcell window in order for the operator to access the pan of the condenser with greater ease than if the condenser is sitting vertically on a hotcell worktable. It is also recognized that the crucible placement and retrieval from the retort well may be a remote operational challenge. A specialized grapple might be used. Another option might be a crucible cradle or platen that hangs from the condenser bottom such that the crucible is lowered into the well when the condenser is put in place.

## 5.3 Instrumentation and Control

The primary process parameters are well furnace temperature, condenser throat temperature, and purge gas flow rates. It is anticipated that these process parameters will be controlled independently from individual controllers instead of integrated into one control console or programmable logic controller (PLC). If an existing well furnace is used, the existing controller for said furnace will be used. If a new furnace is used, then the furnace controller and the condenser heater may be collocated in the same panel. It is anticipated that the purge gas flow will stay constant throughout the entire dechlorination process. Therefore, simple manual rotameters with integral needle valves will be used. Type-K thermocouples or other types with higher temperature ratings (i.e., Type-B, Type-R, Type-S), if possible, will be used in this process.

## 5.4 Boundaries and Interfaces

*Electrical and I&C feedthroughs*. If deployed in HFEF, the DeVA will use existing general purpose electrical and I&C feedthroughs and breakout boxes. If deployed in FCF, the DeVA will use existing general purpose electrical and I&C feedthroughs and breakout boxes.

*Purge Gas Source*. If deployed in HFEF, the DeVA would use argon as a purge gas from one of the standard argon supply manifolds in the HFEF main cell. If deployed in FCF, the DeVA would use argon as a purge gas from the spare argon supply connection on the Multi-Function Furnace Vacuum and Argon Skid (INL drawing 818792).

## 5.5 Potential Physical Layout and Location

The potential physical layout and location are FCF Argon Cell Workstation P10 or HFEF Main Cell Workstation to be determined.

## 5.6 System Reliability Features

Independent overtemperature shutoff is used to protect the equipment in the event of a primary controller failure. Redundant thermocouples are used such that in a single thermocouple failure, processing can continue.

## 5.7 System Control Features

*System monitoring*. The primary form of system monitoring is temperature via type K thermocouples. When the device is disassembled and assembled the o-ring seals and other components will be inspected for damage or degradation.

*Control capability and locations*. To be determined.

*Automatic and manual actions*. The furnace and condenser heater are controlled via programmable controllers (e.g., Watlow) and will automatically control furnace temperature.

*Setpoints and ranges*: To be determined.

*Interlocks, bypasses, and permissive*. To be determined.

## 5.8 Operations

*Initial configuration (pre-startup)*. To be determined.

*System startup*. To be determined.

*Normal operations*. To be determined.

*Off-normal operations*. To be determined.

*System shutdown*. Typical system shutdown conditions include the furnace control system powered down and purge gas feed shut-off.

## 5.9 Testing and Maintenance

*Temporary configurations*. None.

*TSR-required surveillances*. None.

*Non-TSR inspections and testing*. To be determined as the design progresses.

*Post-maintenance testing*. To be determined as the design progresses.

*Post-modification testing*. To be determined as the design progresses.

# 6. SUMMARY AND CONCLUSIONS

This document outlines the details for a conceptual design for a dechlorination and vitrification apparatus (DeVA) for removing halogens from halide-salt wastes and converting them to oxide-based products. The primary components of the system include: the base unit, the condenser, and the crucible. The condenser is comprised of several subcomponents including the bottom, the lid, the dendrite tray, the heater, the flange/sealing components, the purge-gas system, and the off-gas system (post condenser). Several items can be inserted into the melting chamber through the top of the condenser lid including a chemical addition funnel, chill rod device, and stirring device. Additional information is given for the crucible material, but this could be exchanged for other crucible sizes and materials of construction. The system design is aimed to be flexible for sizes, required temperatures, and materials processing chemistry to be adaptable for different waste treatment and processing paradigms.

# 7. ACKNOWLEDGEMENTS

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# 9. APPENDIXES

Appendix A, Source Documents

Appendix B, System Drawings and Lists

Appendix C, System Procedures

Appendix D, Alternative Concepts Evaluation

# Appendix A

## Source Documents

1. CREO 3D Models, located in the Windchill server location under sandbox/Tolman, D/Salt-Waste-Furnace

2. Dechlorination Apparatus for Treating Chloride Salt Waste: System Evaluation and Scalte-up. Riley, B. J.; Chong, S; Lonergan, C. E. ACS Omega 2021, 6, 32239-32252

3. F3000-0001-AJ, Rev. 03, "System Design Description – Fuel Conditioning Facility Argon Cell and Air Cell Equipment"

4. KB0015843, "Engineering Initiation".

5. LWP-11200, "Classified Matter Protection and Control."

6. LWP-1450, "Export Compliance"

7. NFPA-86, "Standard for Ovens and Furnaces"

# Appendix B

## System Drawings and Lists

Appendix reserved for future use.

# Appendix C

## System Procedures

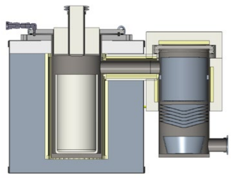
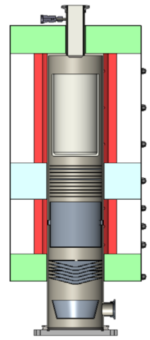
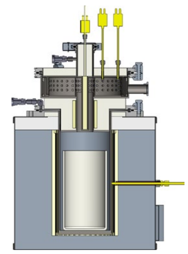
Appendix reserved for future use.

# Appendix D

# Alternative Concepts Evaluation

This appendix is to document alternative concepts that were developed and evaluated as part of the conceptual design process. Three concepts were evaluated and are summarized in table D1. Concept #3 was selected primarily due to the ability to leverage existing equipment at INL and the ability to deploy the apparatus in the hotcell environment.

**Table D-1. Summary of alternative concept evaluation.**

| Category | Concept #1 – Side Condenser | Concept #2 – Bottom Condenser | Concept #3 – Top Condenser |
|---|---|---|---|
| Drawing |  |  |  |
| Leverage of Existing Equipment | **Poor** –would require all new equipment | **Poor** –would require all new equipment | **Good** – would utilize existing well furnaces (e.g., HFEF bakeout furnace) |
| Remote hotcell handling ability | **Poor** – remote assembly and operation of this setup would be difficult due to high temperature seals | **Fair** – there are a couple of distillation apparatus that are in similar design as this that have been deployed remotely, but with some difficulty. The overall height of the assembly may make it difficult to access both the top and the bottom for operations. | **Good** – this arrangement allows for most operations take place at tabletop level. Vertical handling of larger components leverages the hotcell overhead handling electromechanical manipulator. |
| Maintainability | **Poor** – access to the horizontal tube between the hotzone and condenser would be difficult to access for inspection or cleaning. | **Good** – This design provides for generally good access to items for maintenance. | **Fair** – This design provides for generally good access to items for maintenance however since the condenser bottom is fairly complected piece, if there is a heater embedded in it, heater failure may require complete condenser bottom replacement. |
| Hotcell electrical conductor feedthrough required | **Poor** – This would require several heat zones (furnace, horizontal tube, condenser side, condenser lid) | **Fair** - This would require two heat zones (furnace, condenser) | **Fair** – This would require as few as two heat zones (furnace, condenser throat), with potential for a second in the condenser if required. |
| Meets process needs | **Fair** – The meets the needs, but one process concern is worry about buildup of materials in the horizontal tube from the hot zone to the condenser. It will be difficult to inspect or clean this region. | **Fair**– This meets the needs, but there is some concern about adequate containment if a crucible fails; there is potential to get final waste product in the condensing region in the event of crucible failure. | **Fair** – The initial concern is that $NH_4Cl$ may prematurely condense in the vertical tube leading to the condenser and fall back into the hot zone. This is mitigated via heating of the vertical tube. |

Alternative Concept #1

The first concept for the DeVA consists of an arrangement where the primary furnace is loaded from the top and the off-gas vents through a horizontal side port connection (see Figure D1 below).

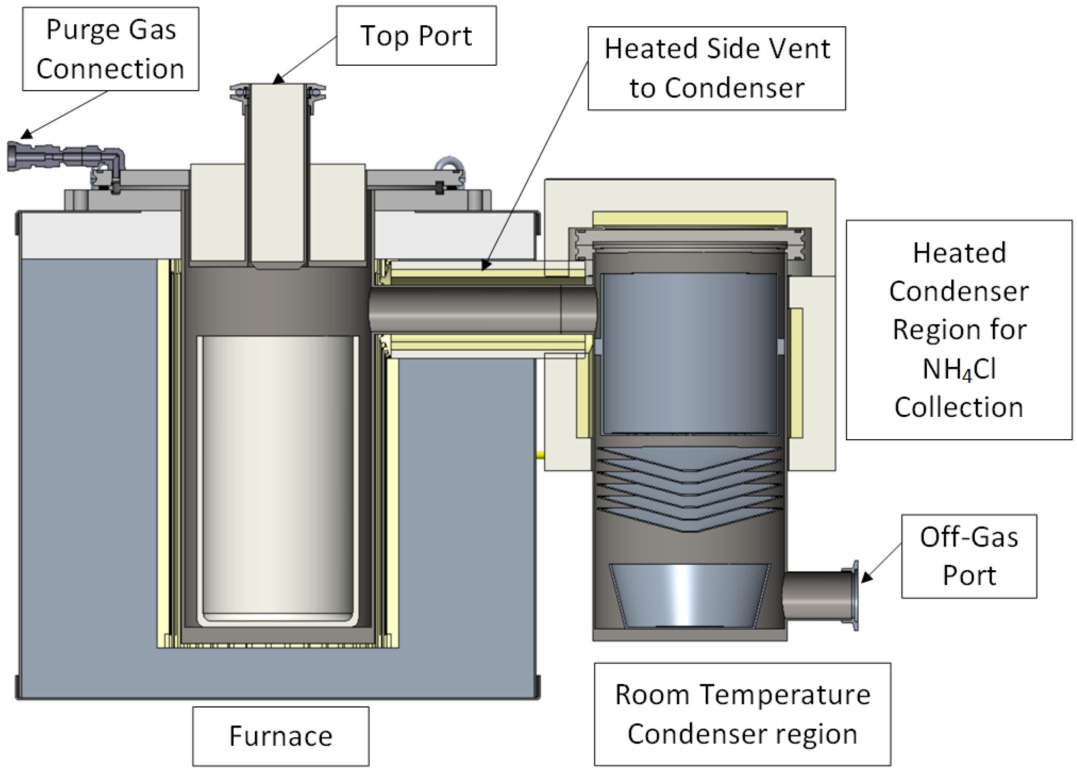



**Figure D-1. Alternative concept #1 with side vent arrangement.**

This concept was an attempt for a system that allowed a top port for vitrification chemical addition (such as iron oxide) while the system is still hot from the dechlorination process step. It was also desired to not have the condensing region as a vertical tube directly above the hot zone in order to prevent the condensed ammonium chloride from falling back into the hot-zone like in previous lab systems.

Alternative Concept #2

In this concept, the hot zone where the salt waste is processed resides at the top and the condenser region is below (see Figure D2). This concept improves on some of the advantages and eliminates some of the challenges of concept #1.

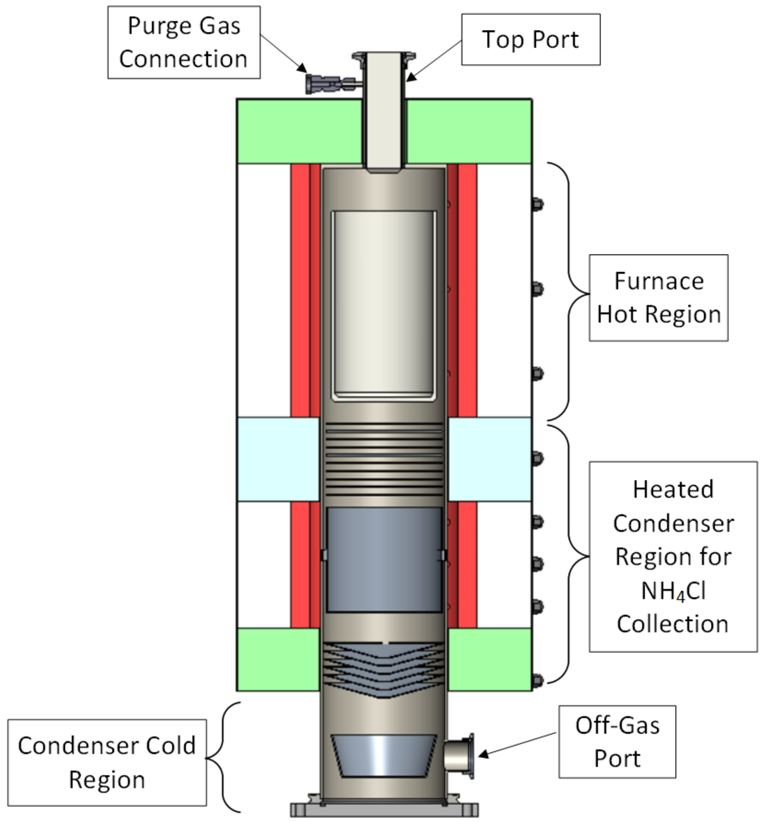



**Figure D-2. Alternative concept #2 with vertical arrangement and condensing region below.**

<u>Alternative Concept #3</u>

This concept was selected as the preferred concept and is discussed in detail in section 4 of this document. Generally, this concept consists of a well furnace with condenser unit on the top with the condensing region arranged such that flow is radially from the inside out (see Figure D3). This concept is selected mostly due to the ability to leverage existing equipment (bakeout furnace) and generable ability to deploy the apparatus in the remote hotcell environment.

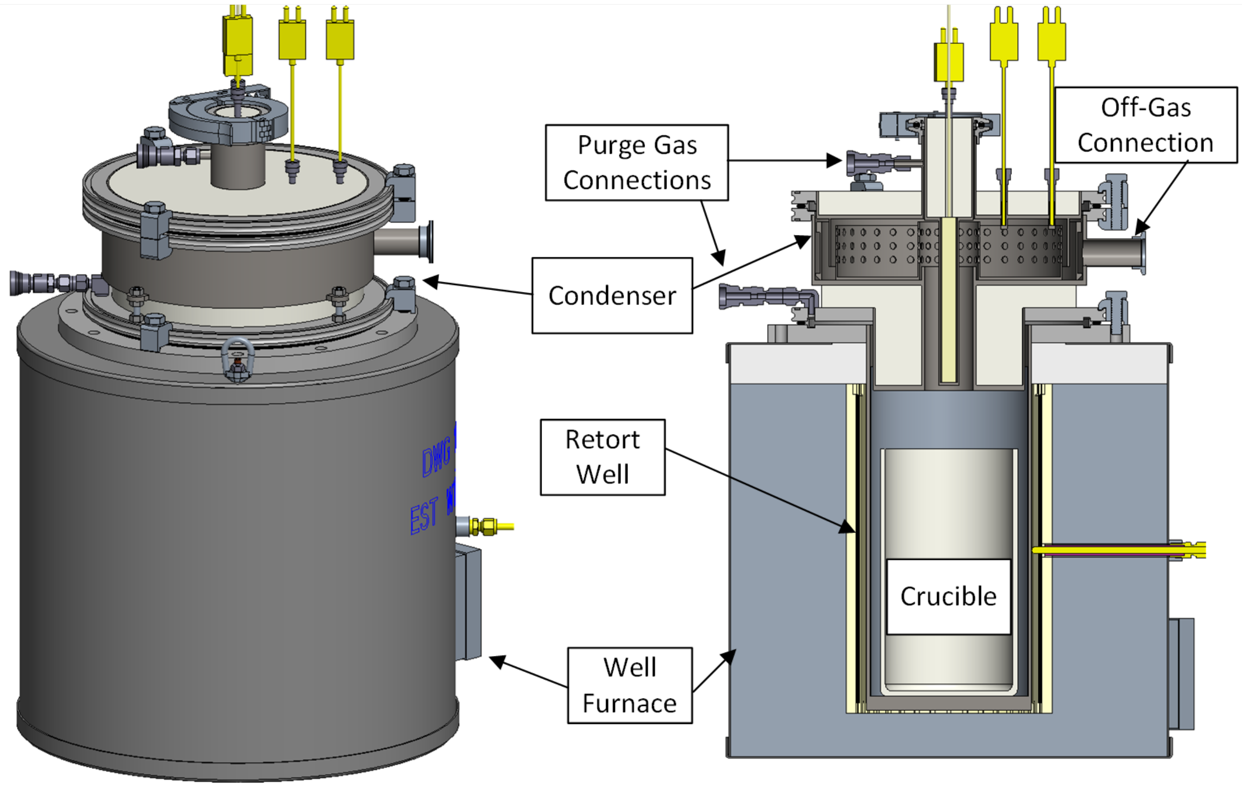



**Figure D-3. Alternative Concept #3 – Arrangement with condensing region above hot region.**