Laboratory Evaluation Plan

Bench-Scale Evaluation of a Carbonyl Process for the Decontamination and Purification of Portsmouth In-Process Nickel

Fluor-B&W Portsmouth, LLC (FBP)

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Prepared by

Fluor-B&W Portsmouth, LLC (FBP) Contractor under Contract DE-AC30-10CC40017

1.0 Evaluation Purpose and Objectives

In accordance with U.S. Department of Energy Contract DE-AC30-10CC40017, the DOE has directed Fluor-B&W Portsmouth, LLC (FBP) to perform a bench-scale evaluation of a carbonyl process for purification of Portsmouth radiologically contaminated nickel material. The purpose of the bench-scale evaluation is to obtain sufficient data to provide proof-of-process using actual contaminated nickel from nickel-bearing equipment being deactivated under the Portsmouth D&D Project. In addition, combinations of important processing conditions will be evaluated to determine the optimum combination from the standpoints of both process throughput and nickel product purity. If the viability of the process is supported by a favorable outcome on this evaluation, additional bench studies and larger pilot-scale evaluations are planned to provide detailed data for engineering scale-up to commercial processing. No decisions to go beyond this evaluation have been made by the Department and are subject to further policy review. The goals of this DOE sponsored effort are as follows:

- Demonstrate the ability to perform high-hazard carbonyl processing within the framework of ISMS, the current Safety Basis and applicable QA standards and requirements.

- Produce purified nickel that will meet or exceed DOE standards for future re-use.

- Identify the optimum combination of process conditions that provides favorable processing rates without adversely affecting product purity.

- Demonstrate the repeatability of results and the consistency of the process using a large number (20+) of multiple trials based upon statistical principles.

- Analyze and characterize contaminated residues produced by the process so that a clear disposal pathway(s) can be determined.

This evaluation plan was developed with the DOE to provide a basic proof-of-process for carbonyl processing of nickel to determine viability as a basis for continuing with additional bench and pilot-scale studies in keeping with associated DOE policies for providing funding support for development efforts. This evaluation is intended to be a basic, scoping-type effort for determining if nickel purity targets that will meet or exceed DOE standards for re-use can be achieved. As such, it is not intended to provide detailed process parameters for scale-up to a commercial facility (e.g. accurate processing/production rates and efficiencies and associated economics, environmental impacts, specific NCS controls, etc.)

2.0 Background

The reaction of carbon monoxide with nickel metal to form nickel carbonyl or $Ni(CO)_4$ was discovered by Ludwig Mond about 120 years ago. For this reason, the basic technique is known as the "Mond Process". The process is unique because it is highly selective for the separation of nickel at the conditions to be employed for this evaluation and has been said to "give wings" to this heavy metal[1]. The process has been used extensively in the mining industry for many years for the refining of nickel ore.

The U. S. Department of Energy (DOE) has been interested in recovery of gaseous diffusion complex nickel assets for many years. While electrochemical and smelting processes have been considered and evaluated, the chemical selectivity of a carbonyl process greatly increases the probability of producing nickel of high purity that is essentially devoid of uranium and ^{99}Tc contamination. The FBP principal investigators assigned to this effort have successful prior experience with evaluation of similar carbonyl processes on these same nickel-bearing materials This proven inherent selectivity has prompted the DOE and some commercial interests to consider and/or evaluate variations of the basic method for this application. The nickel made available from the Portsmouth Project has a much higher purity and surface area than ores or commercially available forms of nickel which should allow for a very practical asset recovery process. Bench-scale evaluation of a carbonyl process by FBP, as directed by the DOE, represents the next step in the development and evolution of an approach and methods to harvest the value of U.S. Diffusion Process in-process nickel materials. Additional evaluations will be necessary to determine effective methods for process material feed, extraction of residues, and to provide additional data on parameters that will affect scale-up of the process in a manner that ensures worker protection from the radioactive component of the feed material.

3.0 Design and Fabrication of Evaluation Apparatus

In keeping with Integrated Safety Management System Principles being applied to all FBP activities at Portsmouth, the design, fabrication and acceptance of the evaluation apparatus is being conducted as an Engineering Project in accordance with FBP Engineering Procedure FBP-NSE-PRO-00107, "Modification Design Control". This will provide the engineering framework and rigor to ensure that the appropriate safety, functional and quality requirements are incorporated into the equipment design. FBP Management assigned an integrated Engineering Modification Team that consists of qualified FBP professionals in the areas of Design and Systems Engineering, Nuclear Safety and Nuclear Criticality Safety, Industrial Safety, Radiological Control, Environmental Compliance, Quality Assurance, Fire Protection, Waste Management, Security and Laboratory personnel who will be involved in the performance of the evaluations. The Engineering Modification Team is currently engaged in the preparation of a formal Systems Requirements Document and technical specifications for test apparatus and engineered systems to ensure evaluation safety.

The apparatus will consist of custom-fabricated components combined with commercially available components including a thermal degradation unit, gas circulation and vacuum pumps, flow meter, bottled gas and associated fittings, valves and pressure regulators, an emissions trapping system for thermal degradation system off-gases and associated instrumentation, hardware, valves and tubing for the overall system. With the exception of the emissions trapping system, which will be located in a standard laboratory fume hood, the evaluation apparatus and process will be contained within a vacuum enclosure to enhance safety and portability. The FBP design is specifically engineered to ensure containment of the hazardous and radioactive materials during the processing. A schematic diagram of the evaluation apparatus is shown in Attachment 1.

FBP Engineering is developing the design and specifications for the processing apparatus and controlled atmosphere chambers, and other custom fabricated components. Engineering specifications completed to date are as follows:

Engineering Specification TFR-SE-2013-0599, "Technical and Functional Requirements for Bench Test Equipment for Nickel Recovery Study" contains the requirements for the custom-fabricated components to be employed in the apparatus.

Engineering Specification TFR-SE-2013-0581, "Technical and Functional Requirements for Controlled Atmosphere Chamber" contains the requirements for the apparatus vacuum enclosure.

4.0 Analytical Methods

Rationale for Selection of Sample Material

Approximately 5 pounds (about 2300 grams) of contaminated in-process nickel material removed from Portsmouth equipment will be used as feedstock for the evaluation trials. This amount of nickel feedstock will be sufficient to support the carbonyl reactor charges and associated evaluation trials needed to evaluate all combinations of important processing conditions and to meet statistical requirements for replicate trials to ensure repeatability. Volatile forms of ^{99}Tc that were present in the process migrated up the diffusion cascade and concentrated in X-326. The nickel materials in the X-330 and X-333 are expected to be contaminated with ^{99}Tc. However, use of X-326 nickel material for these evaluations will provide worst-case conditions for ^{235}U and ^{99}Tc holdup.

Nickel Processing

A split sample of the nickel feedstock will be prepared for each evaluation trial by preparing composites of each specimen for both the material charged into the reaction vessel and unprocessed material that is submitted for radiochemical and metals analyses. The unprocessed material will provide a reference/control for the purified material produced during

each evaluation trial. When the carbonyl reaction has reached completion (nickel portion of feedstock consumed), the reaction will be terminated and the apparatus will be carefully purged and evacuated. Evacuated gases will be passed through the emissions trapping system. Although it is extremely unlikely that nickel carbonyl could withstand thermal degradation temperatures to be employed, if the evacuated gases contain low concentrations, the emissions trapping system will ensure that they are degraded to nickel metal that will be retained in the trap and CO and H_2S promoter gas that will be vented to a standard laboratory fume hood.

Various combinations of important processing parameters will be evaluated in multiple trials to determine optimum processing conditions. These parameters and the range to be evaluated, based on prior experience with similar processes, are provided in Table 1 below.

Table 1
Range of Processing Parameters Examined

Process Parameter	Nominal Range Examined
Carbonyl Reactor Pressure	1 – 10 psig
H_2S Promoter Concentration	2 – 6 mole %
Reagent Gas Flow	5 – 25 cc/min
Thermal Degradation Temperature	350 - 400°F

Statistical Experimental Design

All combinations of important process parameters, each over a target range of levels, will be evaluated to determine the optimum combination of process conditions using statistical experimental design techniques. A factorial design will be employed which has the advantage of allowing the study of the interaction between important parameters affecting throughput and product quality. Based on statistical analysis of ongoing test data, it will likely be possible to introduce a fractional factorial design sequence to reduce the required number of combination trials by eliminating non-optimum combinations. This will allow a larger number of replicate trials to be performed at near optimum conditions which will enhance the reliability of evaluation results by providing the opportunity to demonstrate repeatability[2].

Harvesting and Analysis of Purified Nickel and Wastes

After purging and evacuation, the system will be opened to harvest purified nickel, remove residual unreacted materials from the reaction vessel and to prepare the entire system for the next evaluation trial. The purified nickel metal and unreacted materials will be sub-sampled and submitted for radiochemical analyses for uranium, ^{99}Tc and other radionuclides and ICP analyses for metals/purity. Analyses of the unreacted material will provide information on the

amount of nuclides retained on the unreacted material and whether or not RCRA metals exist at hazardous levels.

A Sampling and Analysis Plan (SAP) is currently being prepared that will establish the necessary analytical requirements to ensure accurate, defensible radiochemical and metals data is obtained that can be used for comparison against DOE standards for future re-use of the purified nickel materials. The SAP will also ensure that analysis of unreacted residual materials is suitable for determining appropriate disposal pathways that could later be applied to a commercial processing operation. The content of the SAP is described in more detail in Section 5.0.

5.0 Quality Assurance Requirements (Apparatus Design/Fabrication and Sample Analysis)

General Requirements and References

The Quality Assurance (QA) program is designed in accordance with FBP's *Quality Assurance Program* (FBP-QA-PL-00001)[3], FBP's *Sample Analysis Data Quality Assurance Project Plan (SADQ) at the Portsmouth Gaseous Diffusion Plant* (FBP-ER-PRO-WD-PL-0006)[4] and EPA's *Guidance on Systematic Planning using the Data Quality Objectives Process* (EPA/240/B-06/001)[5] .

Design/Fabrication Requirements

The following QA/QC requirements will be associated with the design, fabrication and procurement of the evaluation apparatus specialty components that will be provided by vendors:

- Both the apparatus components and the vacuum enclosure must be constructed of materials compatible with the reagents (CO and H2S promoter gas) and intermediate product ($Ni(CO)_4$).
- Assembly drawings showing component material types will be provided for FBP Engineering review prior to fabrication.
- FBP QA, Engineering and Laboratory inspection of assembled components prior to testing will be performed.
- As-built drawings with materials listing will be provided to FBP engineering prior to delivery to ensure fabrication meets the requirements of the FBP specifications.
- FBP QA inspects all welds prior to testing.
- FBP QA will observe leak testing of all apparatus components.

- A final on-site inspection to ensure the components will meet project requirements will be performed by FBP QA, Engineering and Laboratory personnel prior to delivery.

Sampling and Analysis Plan (SAP)

A Sampling and Analysis Plan (SAP) is currently being prepared to provide the requirements necessary to demonstrate that DOE standards for future re-use of the purified nickel materials will been met or exceeded. The SAP will contain the analytical suite including radionuclides and metals, analytical methods including required lower limits of detection for comparison to DOE standards, requirements for obtaining composite split samples and other sampling protocols, and associated Quality Assurance and Quality Control requirements including data validation to ensure that definitive and defensible analytical results are obtained. These include general laboratory requirements and specific sample preparation and analysis requirements. For example, radiochemical count times will be specified to ensure lower limits of detection suitable for comparison against DOE standards for future re-use of the purified nickel are employed. While DOE standards for future re-use have not yet been established, the results of a DOE-sponsored study of commercially available domestic nickel and nickel from foreign sources performed under the ORISE (Oak Ridge Institute for Science and Education) Contract by ORAU (Oak Ridge Associated Universities) will be used to establish lower limits of detection for radiochemical analysis performed in conjunction with this study[6]. The following quality assurance records are generated by this analysis effort.

- Chain of custody (COC) records
- Sample data
- Data validation

Chain of Custody

Samples shall remain in the custody of the Principal Investigator until they are transferred to the analytical laboratories in accordance with Procedure FBP-ER-PRO-00186, "Uranium Chain of Custody"[7]. This procedure describes the protocol for documenting possession (i.e., custody, transfer, and movement) of samples from the point of collection to the point of acceptance by the designated laboratory to ensure integrity of the samples. This procedure includes the requirements for the generation, use, and completion of COC forms.

Laboratory Requirements

All laboratories used to analyze characterization samples of process buildings waste will be audited by FBP QA group or by a DOE Consolidated Audit Program (DOECAP). FBP and/or DOECAP performs annual audits and periodic assessments, as necessary, of all participating FBP laboratory facilities in areas, including but not limited to laboratory QA program, information management systems, materials management operations, waste disposal, and analytical method

performance and compliance. The analytical laboratories shall be approved for use by FBP and consistently generate data of defensible quality.

Sample Preparation and Analysis
The laboratory will perform sample preparation, analyses, and QC requirements in accordance with the laboratory SOW, DOE QSAS, approved Security Plans/Requirements and approved standard operating procedures (SOP).

Sample Waste Management
The laboratory will archive leachate for 90 days prior to disposal. The laboratory will dispose of leachate solutions (after the archival period) and sample media at an appropriate, approved disposal facility.

Independent Analytical Verification & Validation of Test Results
A split sample of the nickel product will be prepared and sent to an off-site laboratory for independent analysis. The results of the independent analysis will be reported directly to the DOE for comparison to the FBP results.

6.0 Security Considerations and Requirements

FBP and WEMS Security technical and administrative professionals who will be involved with all aspects of this evaluation possess diffusion process access authorizations that meet (DOE L-Clearances) or exceed (DOE Q-Clearances) the requirements for physical access and access to C-RD/ECI classified data associated with diffusion process nickel materials. In addition, many of the individuals involved have a working knowledge of the design and characteristics of these materials which was developed through many years of experience during the period of operation of the diffusion equipment. This working knowledge has not only influenced the design of the apparatus and the experimental approach to be used for this evaluation but provides a high level of confidence that the classified information associated with these materials will be protected in accordance with DOE Security and Classification requirements throughout this effort.

Nickel feedstock material employed in these evaluations was obtained from components to be deactivated that are classified as Confidential-RD (C-RD). These materials will retain C-RD features after removal and staging/storage for the evaluations. For these reasons, all of the work associated with the evaluation trials described herein will be conducted in the X-710 Process Chemistry Laboratory or other areas reviewed and approved by WEMS Security.

Purified nickel and unreacted residual materials produced during these evaluations will be handled as C-RD until it is determined by the DOE Site Classification Officer that the classified features of the material no longer exist.

7.0 Safety Considerations and Reviews

In accordance with DOE Order DOE G 450.4-1B, "Integrated Safety Management System (ISMS)" and in keeping with FBP contractual requirements contained in DOE Contract No. DE-AC30-10CC40017, ISMS guiding principles and core functions will be employed in conjunction with all aspects of this evaluation including design, fabrication and assembly of apparatus, performance of the evaluation trials, performance of analytical work on the evaluation sample materials and disposal of evaluation and analytical waste materials.

Carbon monoxide and promoter reagent gases used in the carbonyl process are toxic gases that must be handled appropriately during bench-scale evaluations. In addition, gaseous nickel carbonyl that will be produced and handled during the evaluations is extremely toxic requiring special precautions to be taken. Reagent gases will be supplied from vendor obtained high-pressure cylinders. Since high-pressure cylinders and toxic reagents and intermediate products (nickel carbonyl) will be handled during the evaluations, a Safety/IH assessment will be performed to identify hazards and determine safe handling practices during the evaluations. Since nickel tetracarbonyl is a known carcinogen, a Carcinogen Control Plan (CCP) will also be developed and implemented. The reagent gases are also highly flammable. Therefore an assessment of fire hazards will be performed by the FBP Fire Protection Group to identify measures to minimize fire hazards during the evaluations.

The entire bench-scale apparatus will be contained in a vacuum enclosure. While it is unlikely that a leak would develop, any out-leakage from the apparatus would be contained within the enclosure. A vent to a standard laboratory fume hood will be provided to accommodate apparatus purging and evacuation activities needed to harvest purified nickel and load nickel feedstock for subsequent trials. The vent stream will be passed through an emissions trapping system before venting to the fume hood. Trace quantities of nickel carbonyl could be present in the vent gases. The process design and procedures will ensure that these trace quantity gases are be degraded to carbon monoxide and promoter gas and nickel metal that will be collected in the system trap. Vent gases should contain primarily dry purge gases (nitrogen, oxygen) with lesser amounts of carbon monoxide and promoter gas. An Environmental Review will be performed to ensure that this mixture of gases can be safely vented to a standard laboratory fume hood.

The nickel feedstock for these evaluations was removed from X-326 equipment to be deactivated. The samples were obtained in conjunction with previous environmental and waste characterization sampling activities in X-326. As such, the feed material will contain significant levels of uranium and ^{99}Tc contamination. Pure nickel will be selectively removed from the raw feed materials during the carbonyl process, leaving contaminated residues behind in the carbonyl reactor. Periodically, it will be necessary to remove the contaminated residues from the reactor. Movement of raw feedstock into the enclosure and purified nickel and

contaminated residue out of the enclosure will be necessary during performance of the evaluations. Since these activities must be performed in a manner that minimizes the spread of contamination and maintains nuclear criticality safety, both a Formal Radiological Review and an NCS/Nuclear Safety Review will be performed to determine requirements necessary to perform this work safely. Results of the radiological review will be incorporated into the Radiological Work Permits. All work will be performed in accordance with the RWP by trained and qualified Radiological Workers.

A Job Hazard Analysis (JHA) will be performed that addresses all evaluation activities. Hazards and appropriate controls to perform the work safely that were identified by each specific area review will be incorporated into the JHA. These reviews include the following as outlined above:

- Safety/IH Assessment
- Fire Hazards Assessment
- Environmental Review
- Formal Radiological Review
- NCS/Nuclear Safety Review

The hazards and controls identified in the JHA and associated reviews and assessments will be incorporated into a procedure for the bench-scale evaluations as an integral part per current policies.

8.0 Evaluation Coordination, Emergency Planning and Response

Nuclear Safety will perform an Unreviewed Safety Question (USQ) screen to determine whether any conflicts exist with the current Safety Basis and what, if any, documentation and approvals are required to implement the plan.

In addition, performance of the evaluations will be coordinated with the Plant Shift Superintendent and Emergency Response, Industrial Safety/IH and Radiological Control personnel to ensure that the proper notifications are promptly made if an emergency response is necessary. The following entities will be notified immediately prior to initiating evaluation activities (entering the procedure), if unexpected conditions arise and/or if an emergency response is necessary and immediately after terminating evaluation activities (exiting the procedure). In this way, all critical response areas are aware when evaluation activities are ongoing or not.

- Plant Shift Superintendent (PSS)
- Fire Services/Emergency Management
- Industrial Safety/IH
- Radiological Control

Contingency actions to be taken if unexpected conditions arise during the evaluations, including a plan for emergency response, are summarized below:

- If possible, the test apparatus will be placed in a safe, stable condition as soon as possible.
- If judged to be necessary by the Principal Investigator, an emergency response will be initiated.
- Evaluation actions are suspended until the problem/issue is resolved.
- A Problem Report is written and submitted to determine any necessary corrective actions and to document the problem(s) encountered.
- The Principal Investigator conducts a debriefing with all involved personnel.
- Before resumption of evaluation trials, the Principal Investigator ensures that all necessary corrective actions have been implemented and conducts a pre-job briefing.

9.0 Evaluation Prerequisites

1. Nuclear Safety personnel have completed a USQ screening of the evaluation plan/procedure and the use of X-710 Laboratory facilities for the evaluation against the current safety basis and associated documentation to determine if there are any conflicts and to determine what, if any, additional reviews and approvals are required to perform the evaluations.
2. NCS personnel have completed an NCS review of the evaluation plan/procedure and have provided requirements to maintain nuclear criticality safety during the evaluation.

3. Radiological Control personnel have completed a Formal Radiological Review of the evaluation plan/procedure and have provided requirements to minimize the spread of radioactive contamination.

4. Fire Protection and Emergency Management personnel have completed an assessment of fire hazards associated with the evaluation and have provided measures to minimize the risk of fire.

5. Industrial Safety/Industrial Hygiene (IH) personnel have competed a review of the evaluation plan/procedure to assess industrial and chemical hazards and have provided protective measures to ensure the evaluations can be performed safely.

6. IH personnel have prepared a Carcinogen Control Plan (CCP) to cover activities associated with the evaluation.

7. ESH&Q personnel have completed an environmental review of the waste gas venting activities associated with the evaluation and have determined any necessary requirements.

8. A Job Hazard Analysis (JHA) on the evaluation plan/procedure has been completed and approved. The specific reviews described in prerequisites 1 through 7 will be integral to the JHA.

9. An FBP technical procedure has been prepared and is approved for use in performing the evaluations.

10. Laboratory equipment and materials have been obtained and meet the requirements of associated Engineering Specifications including QA inspection and testing requirements.

11. The laboratory fume hood in that will be used for venting of waste gases during the evaluation has a "Radiological" service rating based on the latest face velocity testing (posted on hood).

12. The laboratory apparatus has been assembled per the requirements of the associated approved Engineering Design.

13. Apparatus pressure, flow and temperature measurement instrumentation has been properly configured, tested and calibration checks have been performed.

14. At least two qualified individuals trained on the evaluation procedure and the use of PPE specified below for removing purified nickel from and loading nickel feed material into the apparatus enclosure are present during all such loading/unloading activities.

15. PPE required for removing purified nickel from and loading nickel feed material into the apparatus enclosure and safety monitoring equipment have been obtained and are staged in the laboratory area. This equipment is as follows:

    a. PVC Gloves (rubber not acceptable)
    b. Full-face respiratory protection with supplied air
    c. Hand-held CO Detectors
    d. Nickel Carbonyl Detection Tubes and Hand Air Pump (staged within the apparatus enclosure before each trial)
    e. Other PPE as specified by Industrial Hygiene

16. Purified nickel materials and residual, unreacted materials generated by the processes performed during this evaluation will be handled as Confidential-RD until it is determined by the DOE Site Classification Officer that the classified features of the material no longer exist.

17. A Pre-Job Briefing has been performed with all personnel who will be involved with the evaluation by the Principal Investigator. The briefing will include discussion of the hazards, precautions and contingency actions associated with the Carbonyl Process to be evaluated.

10.0 Evaluation Steps

The evaluation steps are contained in an associated FBP technical procedure that is currently being developed.

11.0 References

[1]"The Winning of Nickel", The International Nickel Company of Canada, Limited, 1967.

[2]Personal communication with Brian Lanning, FBP Statistician, June 27, 2013.

[3]FBP-QA-PDD-00001, "Quality Assurance Program Description", Fluor-B&W Portsmouth, March, 2013.

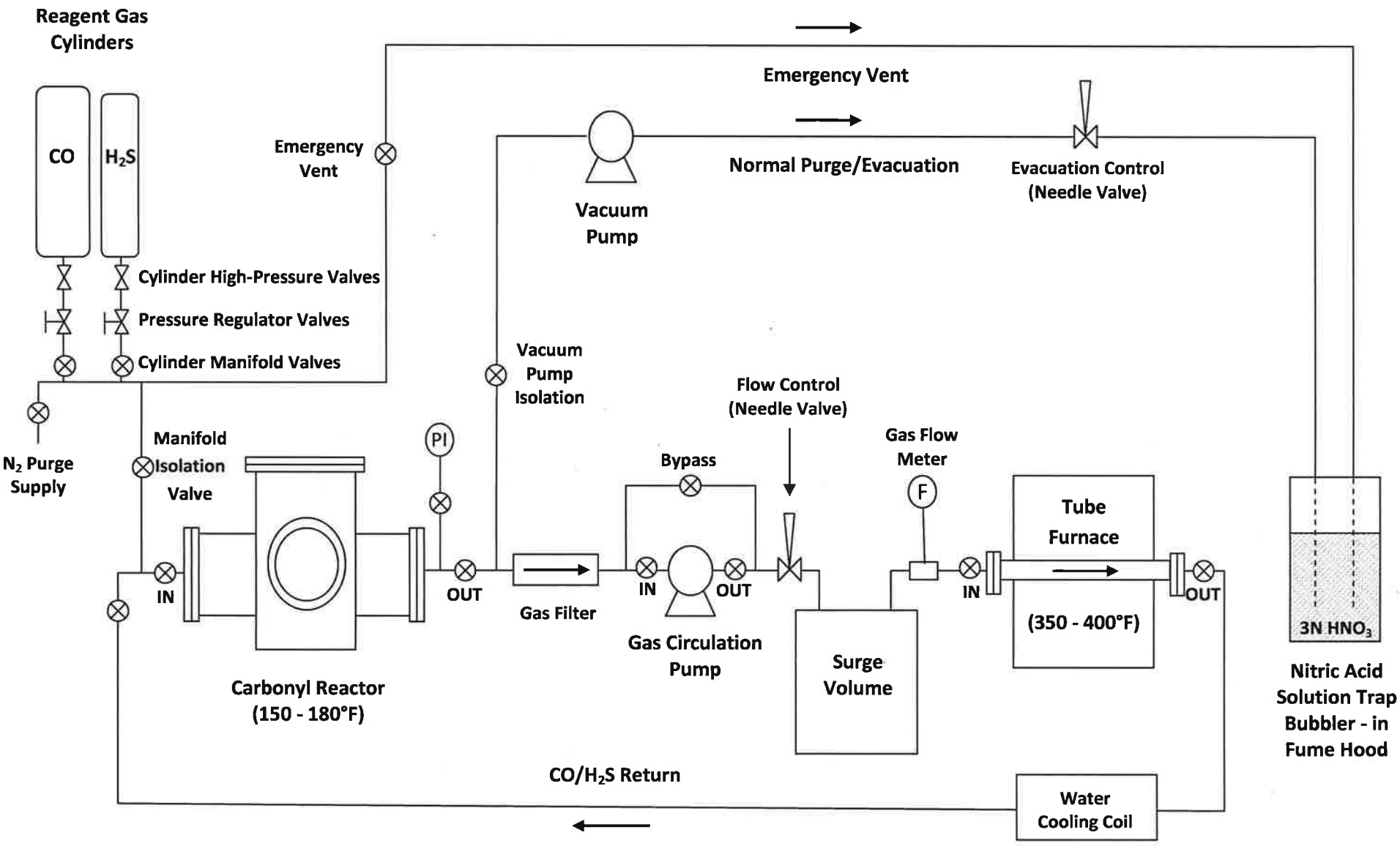
[4]FBP-ER-PRO-WD-PL-0006, "Sample Analysis Data Quality Assurance Project Plan (SADQ) at the Portsmouth Gaseous Diffusion Plant, Fluor-B&W Portsmouth.

[5]EPA/240/B-06/001, "Guidance on Systematic Planning using the Data Quality Objectives Process", U.S. Environmental Protection Agency, February, 2006.

[6]"Report for Commercial Grade Nickel Characterization and Benchmarking", David A. King, Oak Ridge Associated Universities (ORAU) in cooperation with Oak Ridge Institute for Science and Education (ORISE) under DOE contract number DE-AC05-06OR23100, December 2012.

[7]"Uranium Chain of Custody", FBP Procedure FBP-ER-PRO-00186, Fluor-B&W Portsmouth, Portsmouth Gaseous Diffusion Plant, January 30, 2012.

Attachment 1
Apparatus for Bench-Scale Evaluation of Low-Pressure Carbonyl Process for Nickel Purification



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