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Design of Fieldable Head Space Manifold

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OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

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ABBREVIATIONS

1S	cylinder size containing up to 0.45 kg of UF_6
2S	cylinder size containing up to 2.22 kg of UF_6
5B	cylinder size containing up to 16.9 kg of UF_6
8A	cylinder size containing up to 115.7 kg of UF_6
12B	cylinder size containing up to 208.7 kg of UF_6
30B	cylinder size containing up to 2,277 kg of UF_6
48X	cylinder size containing up to 21,030 kg of UF_6
48Y	cylinder size containing up to 27,560 kg of UF_6
ASTM	American Society for Testing and Materials
ORNL	Oak Ridge National Laboratory
VIMS	variable inlet mass spectrometer

EXECUTIVE SUMMARY

A system has been designed and built to meet the expected requirements for sampling large UF_6 cylinders in the field and to provide similar purification and analyses to those currently performed for 1S and 2S samples at Oak Ridge National Laboratory (ORNL). The system was designed to be modular, allowing flexibility to perform under a variety of conditions. At a minimum, a large cylinder can be accessed to collect head space samples. These samples can additionally be processed in the field to generate 10 cc purified samples that should be similar to ones currently generated at ORNL from sampling of smaller cylinders. Variable inlet mass spectrometer operation can be performed at the time of purification or at a different location on a purified sample. This design should allow collection, processing and analysis of one extracted sample per day based on similar operations using the ORNL manifold. Reduced operations can be performed at a slightly faster rate although movement between cylinders does require time to evacuate and dry the connection adequately for accessing the UF_6 head space.

1. PURPOSE

This document introduces the design of a fieldable manifold that could be deployed to connect to a cylinder of uranium hexafluoride (UF_6) and collect head space gas samples, process one or more head space gas samples, and measure the noble gas content of a processed sample.

The design has been divided into modules that encompass the simplest base operations. This approach allows for the highest degree of flexibility to adapt operations to potential operational restrictions (e.g., spatial, power, export control, or political). The first unit collects a subsample of head space from the target cylinder and will be employed for the larger cylinder range. The second unit processes the head space gas to remove reactive species that may interfere with the analysis. Finally, the third unit performs the analysis. The carts can be operated separately or as a single unit. This design allows for measurements to be made at a remote site or head space or purified samples can be shipped to a regional location or back to a US facility.

2. DESIGN

The proposed design consists of three separate systems that can be connected and used in unison or operated independently. This approach provides maximum flexibility to meet the various operational constraints. The sections can be summarized as a sampling manifold, a processing manifold, and an analysis manifold.

2.1 CARTS

During development of the manifold layout, a cart was designed to accommodate the largest system. The same cart design (Figure 1) was employed for all three units. This decision was made to simplify the design, minimizing parts and allowing potential repurposing as needed. The cart dimensions are 62 in. (height) × 23.75 in. (width) × 47.75 in. (length), which should fit through most doors (The standard minimum door is typically 78 in. tall and 24 in. wide.). The construction used 80/20 T-slotted aluminum, which is light, adaptable, and able to carry large weights.

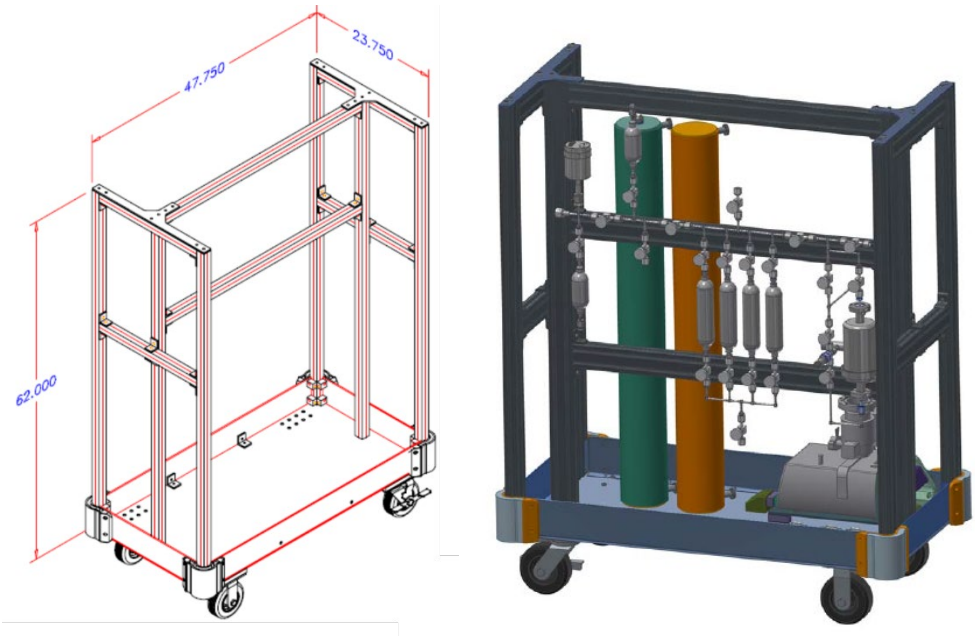

Figure 1. Cart dimensions and 3D rendering.

2.2 SAMPLING MANIFOLD

Knowing the fraction of the total head space gas that has been extracted is critical for sampling, analyzing head space gas, and interpreting the findings. To determine this volume, the design will attempt to extract at least ~5% of the total head space of the cylinder and analyze samples from the beginning and end of that extraction. The selection of 5% is somewhat arbitrary but allows adequate head space extraction to allow the total head space volume to be estimated through serial dilution of an analyte while removing a minimal UF_6 gas mass, reducing solid perturbation and mass of uranium disposal, trap size or handling considerations. As shown in Table 1, this volume[1] will vary depending on the size of the UF_6 cylinder being sampled. An expansion volume is required for any cylinders larger than an 8A. Subsamples would be collected before and after this expansion volume. For 5A/B and 8A, the large sample bottle will not be required because the extracted head space volume is small enough to be collected directly as the sample. Smaller samples, such as those required for 1S and 2S cylinders, will be extracted directly into the processing manifold. A range of sample bottles, chemical traps, and fittings will need to be available to accommodate the range of ASTM-certified and noncertified cylinder types that may be encountered.

Table 1. Sample sizes to extract 5% of the head space for various sized cylinders

Cylinder	V (L)	Head Space (L)[A]	5% of V (L)	Expansion Vessel
1S	0.15	0.06	0.003	—
2S	0.74	0.30	0.015	—
5A/5B	8.04	3.22	0.16	150 cc
8A	37.4	15.0	0.75	1 L
12B	67.4	27.0	1.35	2.25 L
30B	736.0	294.4	14.7	15 L
48X	3,084	1,234	61.7	15 L × 4 (60 L)
48Y	4,041	1,616	80.8	15 L × 5 (75 L)

[A] Calculation of head space volume assumes a 60% full cylinder at typical 40% ullage.

Note, this manifold was designed to accommodate the largest sample sizes. This required some design decisions that would not be optimal for the smallest samples (in which, for example, reduced manifold volume would be desirable).

Further confidence in the effectiveness of a ^3He spike hopefully will eliminate the need for collection of 5% of the head space, although there may be interest in using both methods to improve the confidence in the volume determination. Collection of multiple liters of UF_6 necessitates availability of large chemical traps in a form that can be replaced as needed for possible repeat operations. Additional 15 L chemical traps were fabricated to fill this need. Note, that 15 L is a convenient size because traps can be fabricated with a diameter of less than 5 in. to reduce criticality concerns and still have a reasonable mass to allow movement when loaded with absorbent. Traps this size can also be staged in series to accommodate multiple cylinders of the largest scale. The traps will be loaded with alumina that has been dried at ~300 °C under flowing dry gas until the relative humidity is less than 10%. These conditions were selected based on current work for other customers that suggests that the current as-received alumina may not be adequately dry when processed by vacuum drying.

The sampling manifold will be used to collect an adequate portion of the target cylinder head space and provide subsamples to be processed and analyzed with the basic form shown in Figure 2. This system may be used on its own or possibly joined with the processing manifold. The UF_6 cylinder would attach at the far left. The left portion of the manifold would allow for an initial pressure reading and injection of a

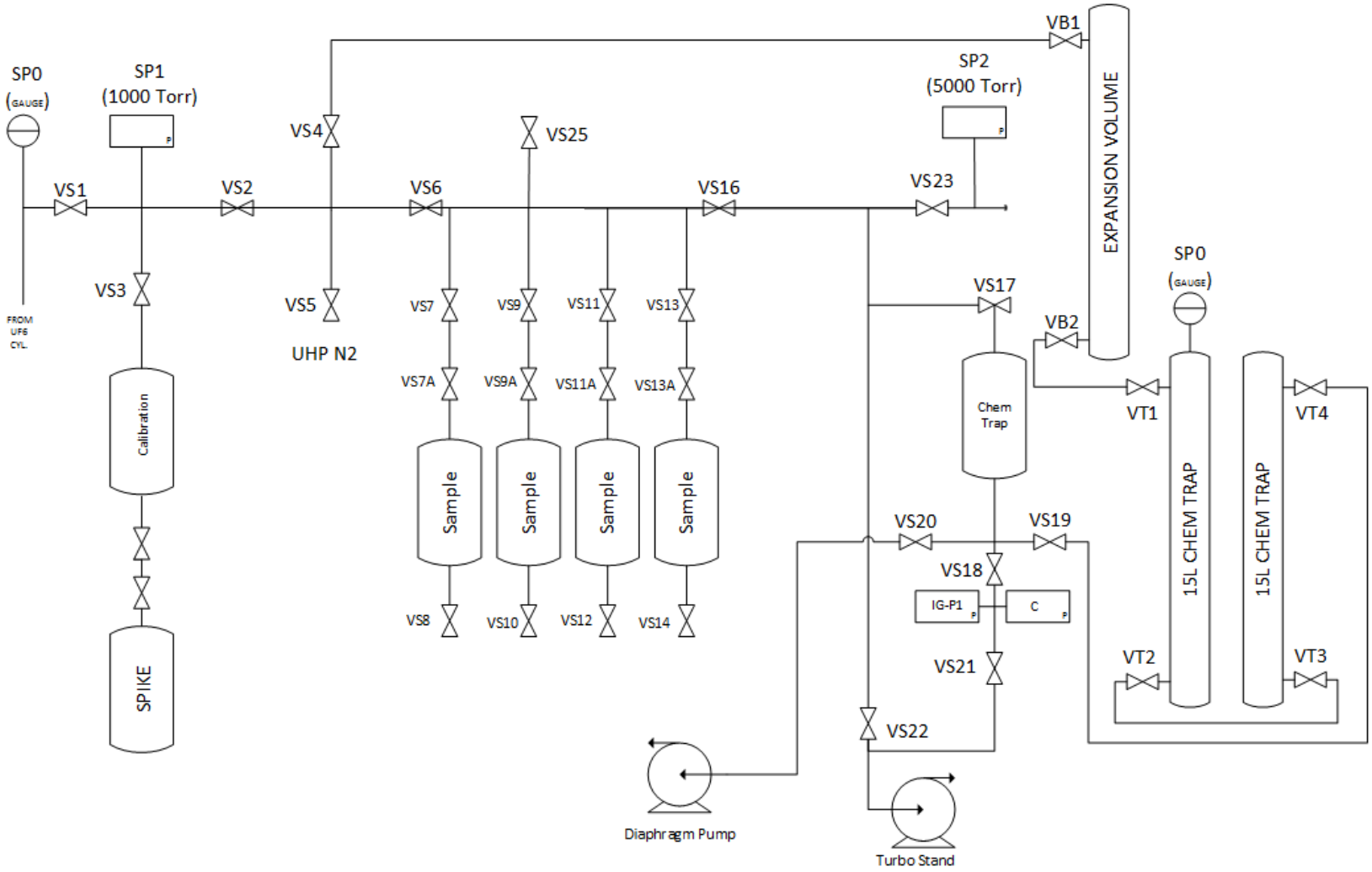

Figure 2. Sampling manifold.

noble gas spike, if used, into the UF_6 cylinder. Large and small volume cylinders/bottles will be used as needed for the sampling and subsampling of the head space.

The completed manifold is shown in Figure 3 with three 300 cc head space sample bottles. Operation requires a single 15 A, 110 V electrical circuit to power the required pumps and gauges and a lecture bottle of inert (water free, but also largely helium free) gas such as ultra–high purity nitrogen.

Figure 3. Sampling cart as built. Note, the system is shown in a combined operation configuration.

The product from this manifold would be two 300 cc samples of cylinder head space that would be transported for additional processing and analysis. If an additional sample is required, two 300 cc samples of head space can be collected per extraction, or the sample bottles can be replaced with larger volume

vessels. Note, this manifold can be modified to collect other samples in the field as needed. For instance, one or more of the sample bottles can be replaced with P10 tubes or other sample bottles to collect UF_6 for laboratory-based isotopic ratio analysis. Collection of these samples could be performed onto alumina (variation on the ABACC-Cristallini Method[2]) using cryogens to freeze the UF_6 or other methods as they become available.

2.3 PROCESSING MANIFOLD

The processing manifold, shown in Figures 4 and 5, accepts a head space sample bottle collected by the sampling cart, a small sample bottle (such as a 1S), or can be connected directly to the sampling cart for sample transfer. The operation of this manifold was designed to mimic the current system at ORNL,[3] including collecting P10 samples of the UF_6 gas that would be returned to a laboratory for determination of isotopic ratios using high-precision mass spectrometry, monitoring infrared active gas concentrations using a Fourier transform infrared spectrometer, and trapping reactive gas species using a sodium fluoride (NaF) chemical trap (primarily for UF_6 and HF) and a heated copper trap (for fluorine and other reactive gases). After removal of the reactive gas species, subsamples can be collected and shipped for off-site analyses by noble gas mass spectrometer. The system can be operated in a stand-alone mode for a sample bottle (i.e., the 300 cc head space sample collected by cart 1) or a small UF_6 cylinder such as a 1S or 2S.

If operations allow, gas can also be transferred directly to the analysis manifold and passed to the variable inlet mass spectrometer (VIMS) for more real-time, on-site analysis. If not on-site, gas bottles can be collected and sent to a regionally deployed VIMS if required.

The processing cart can run on a 15 A, 110 V circuit and needs a small bottle of ultra–high purity gas (no helium or water) such as nitrogen.

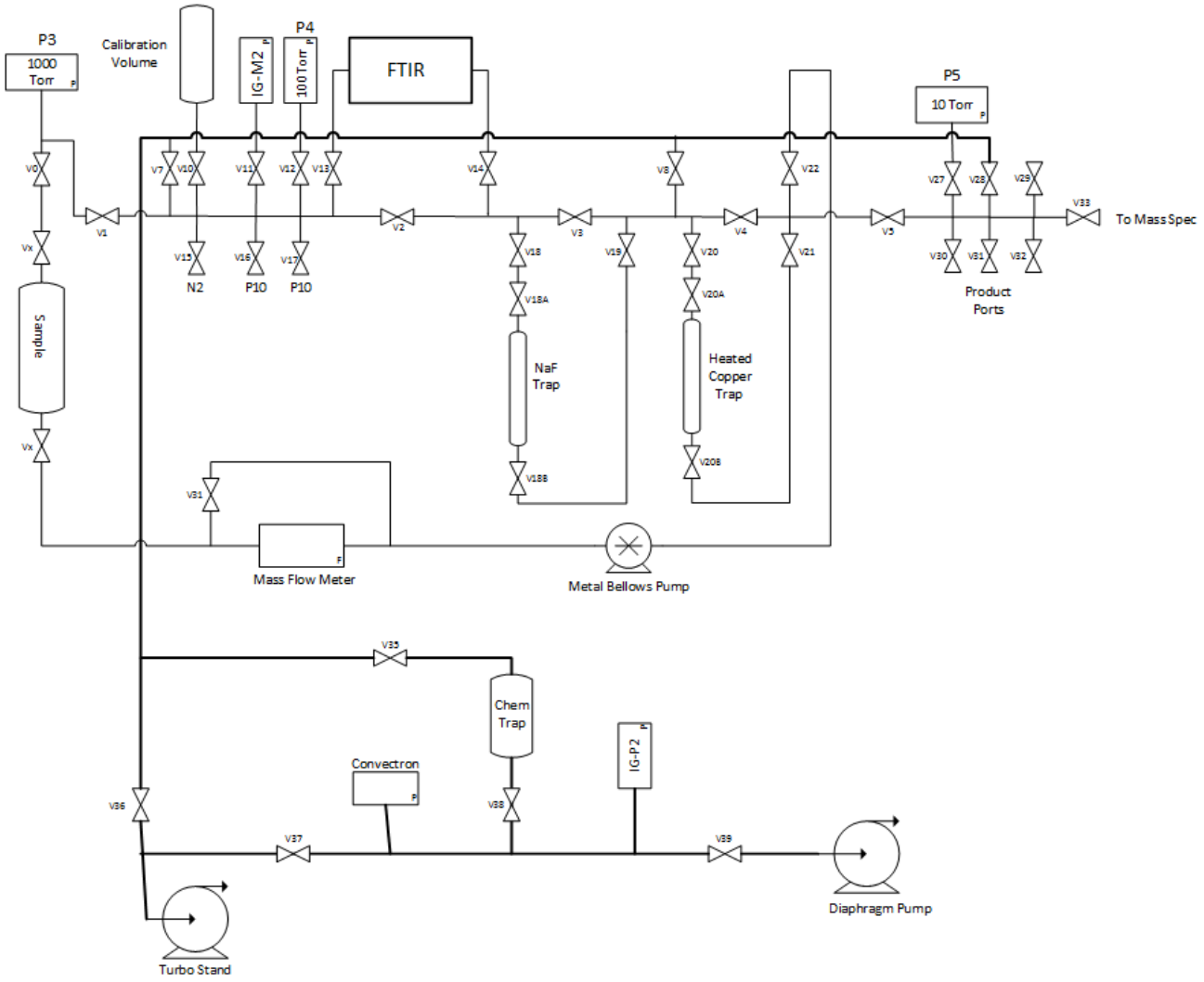

Figure 4. Processing manifold design.

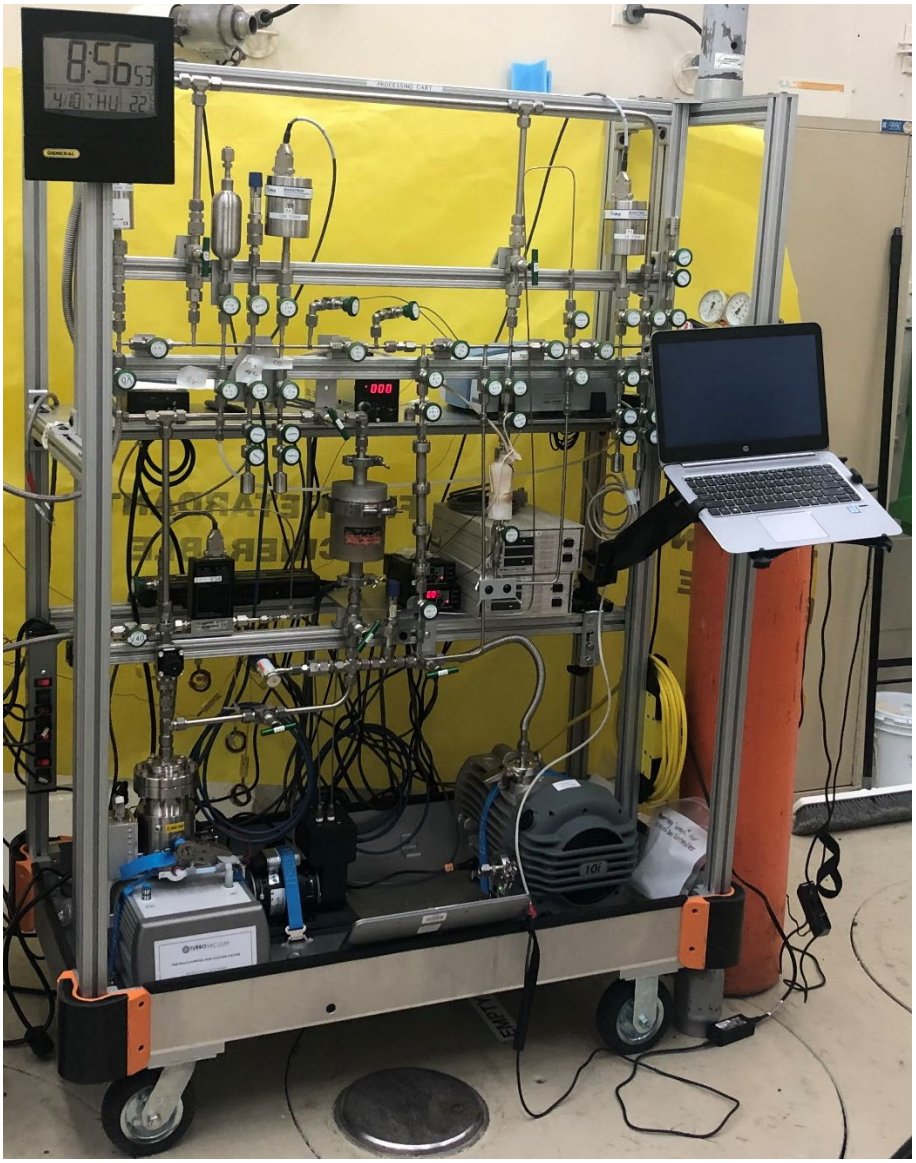

Figure 5. Processing manifold as built (note that system is shown in combined operation configuration).

2.4 ANALYSIS MANIFOLD

The analysis manifold would provide the VIMS with a known volume air sample used for calibration as well as a known volume of purified gas from the processing manifold. A new manifold (Figure 6) was designed by Lawrence Livermore National Laboratory and procured that had required tools (vacuum, pressure gauge and air pipette) while also allowing connection to the processing manifold and addition of a spike and sample bottles. The analysis cart requires a 15 A, 110 V circuit for operations.

The analysis manifold has been used with the existing Oak Ridge National Laboratory (ORNL) sampling/processing manifold during 2018 to perform measurements.

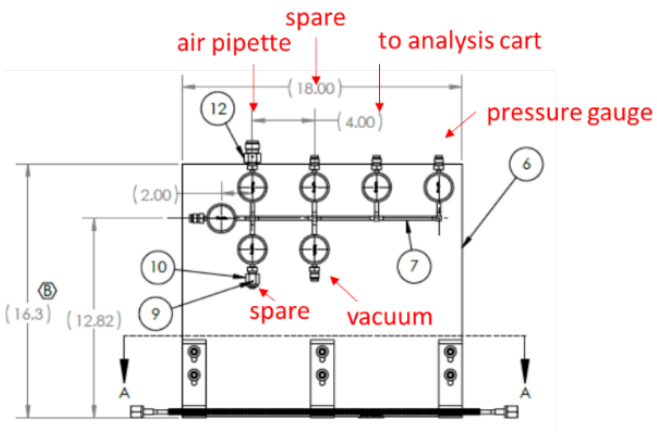

Figure 6. Analysis manifold.

Figure 7. Analysis manifold as built.

2.5 COMBINED OPERATIONS

Combined operation of carts 1 and 2, shown in Figures 8 and 9, will allow sample collection and processing in the field. Operations and capabilities are as stated for the separate sampling and processing sections above. The system as shown can be run off two 15 A, 110 V circuits and requires a cylinder of high purity (no helium or water) gas such as nitrogen. The addition of the analysis cart will increase the requirements to three 15 A, 110 V circuits. The actual power demand may be lower but has not been determined.

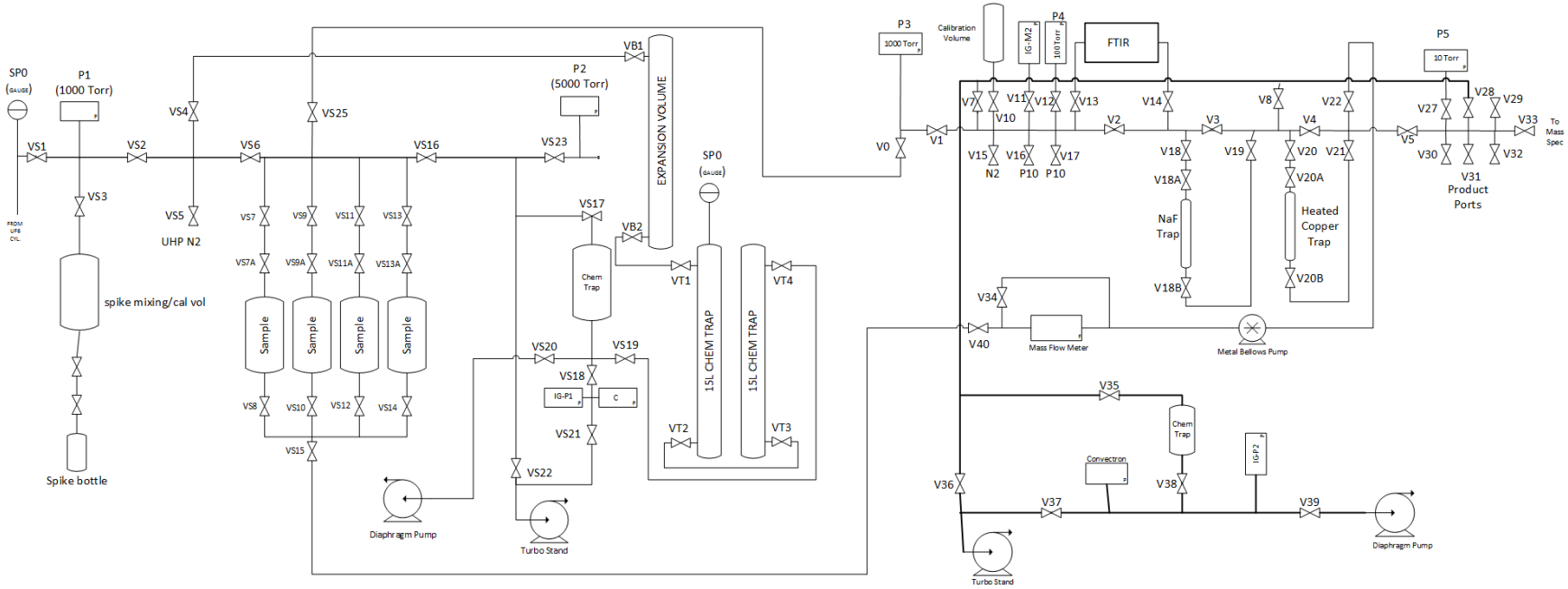

Figure 8. Combined sampling and processing manifold.

Figure 9. Combined sampling and processing manifold as built.

The current plan is to collect two 300 cc head space samples per large UF_6 cylinder. Larger volumes of head space can be collected using either two 300 cc bottles or larger bottles. Then, each 300 cc head space sample will be processed to produce one P10 sample of UF_6 for isotopic assay and four purified 10 cc gas samples. While not shown in the report, the analysis cart can be connected at V33 (far right) to allow for VIMS measurements.

3. CRITICALITY REVIEW LIMITS

A criticality review[4] was performed by staff at ORNL to evaluate the design and potential operations. The primary concern of the review is the accumulation of uranium on the larger external chemical traps. All criticality concerns will be addressed by implementing the following limits:

- Establish a 6 ft perimeter around the sides of the carts
- Only sample a single cylinder at a time
- Locate the cylinder being sampled at least 2 ft from the cart
- Only have two alumina traps located on the cart at any given time (including empty spares)
- Unload spent alumina traps one at a time and place them in appropriate shipping containers

Additionally, the enrichment of UF_6 is limited by the cylinder sizes shown in Table 2. This approach will accommodate standard sized cylinders as well as out-of-specification vessels.

Table 2. Enrichment and maximum dimension limits for UF_6 cylinders.

Cylinder	Max % ^{235}U	Max OD (in.)	Max Length (in.)
8 in.	100%	10	60
12 in.	40%	15	50
30 in.	5%	36	90
48 in.	5%	56	180

4. CONCLUSION

A system has been designed and built to meet the expected requirements for sampling large UF_6 cylinders in the field and to provide similar purification and analyses to those currently performed for 1S and 2S samples at ORNL. The system was designed to be modular, allowing flexibility to perform under a variety of conditions. At a minimum, a large cylinder can be accessed to collect head space samples. These samples can additionally be processed in the field to generate 10 cc purified samples that should be similar to ones currently generated at ORNL from sampling of smaller cylinders. Variable inlet mass spectrometer operation can be performed at the time of purification or at a different location on a purified sample. This design should allow collection, processing and analysis of one extracted sample per day based on similar operations using the ORNL manifold. Reduced operations can be performed at a slightly faster rate although movement between cylinders does require time to evacuate and dry the connection adequately for accessing the UF_6 head space.

5. ACKNOWLEDGMENTS

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6. REFERENCES

1. *Uranium Hexafluoride: A Manual of Good Handling Practices,* United States Enrichment Corporation, 2006.
2. Galdoz, E.; Esteban, A.; Cristallini, O.; Perrotta, J. A. "UF_6 Sampling Method Using Alumina," In *Annual Meeting Proceedings of the Institute of Nuclear Materials Management*, 2008, p. 199.
3. Singleton, M.; William Cassata, W.B.; Peterson, J.; Cox, R.; Trowbridge, L.; Simmons, D.; Fugate, G. Helium Age Dating of UF_6 Sample Cylinders, LLNL-TR-756167, Lawrence Livermore National Laboratory, Livermore, CA, 2018.
4. Nuclear Criticality Safety Evaluation for UF_6 Sampling Cart, *Nuclear Criticality Safety Evaluation 154*, Revision 0, 2019.