

Tritiated Gas Mixing for Z-GTS Fills and Gas Analysis Round Robin

Jessica A. Bierner, Robert Garcia, Lynelle K. Takahashi, and Russell L. Jarek



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Talk Outline

- Background
- Syringe Pump Gas Mixing Manifold design and operation
- Characterization experiment data
- Applications
 - Complex-wide Gas Analysis Round Robin
 - Z Machine Gas Fills
 - Potential for creating our own tritium gas standards if needed
- Future work and questions to answer

Why do we need a custom Gas Mixing System?

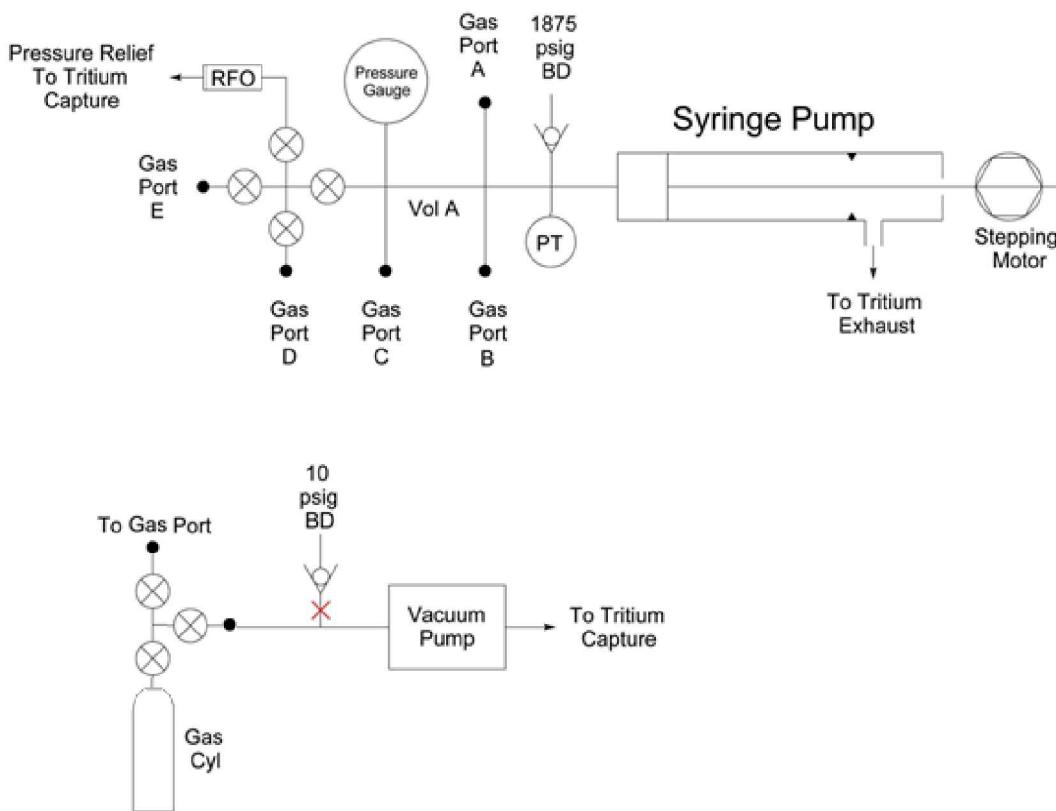
- Previous gas blends came from one of two places:
 - Matheson Tri-Gas for standard gases
 - Savannah River Site for Tritium Gas blends
- In 2015, Matheson lost its vendor certification for Sandia due to failure to supply gases within specification requirements
 - The Gas Lab analyzes all incoming gas cylinders
- Savannah River currently provides mixes in pre-prescribed blends and cylinders.
- Needed a capability to mix custom gas blends for customers-
 - Z Machine Target Fills use a custom apparatus and a unique gas blend for each experiment
- A need arose for a capability in house!



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Syringe Pump Gas Mixing Manifold initially designed for trace tritium capability



- Several manifold configurations are utilized based on the pressure of the activity being performed
- High pressure (up to 1700 psig) and low pressure (up to 13 psig) configurations currently

Syringe Pump System:

- Physical mixing for the gases introduced
- Able to pressurize which is not possible with diffusion mixing apparatus
- Allows for lower pressure gas sources required for ${}^3\text{He}$ and T_2 due to receipt conditions from vendor
- Direct sampling capability for verification of gas fill concentrations
- Useful for both tritiated and non-tritiated gas mixes

GMM System Photos

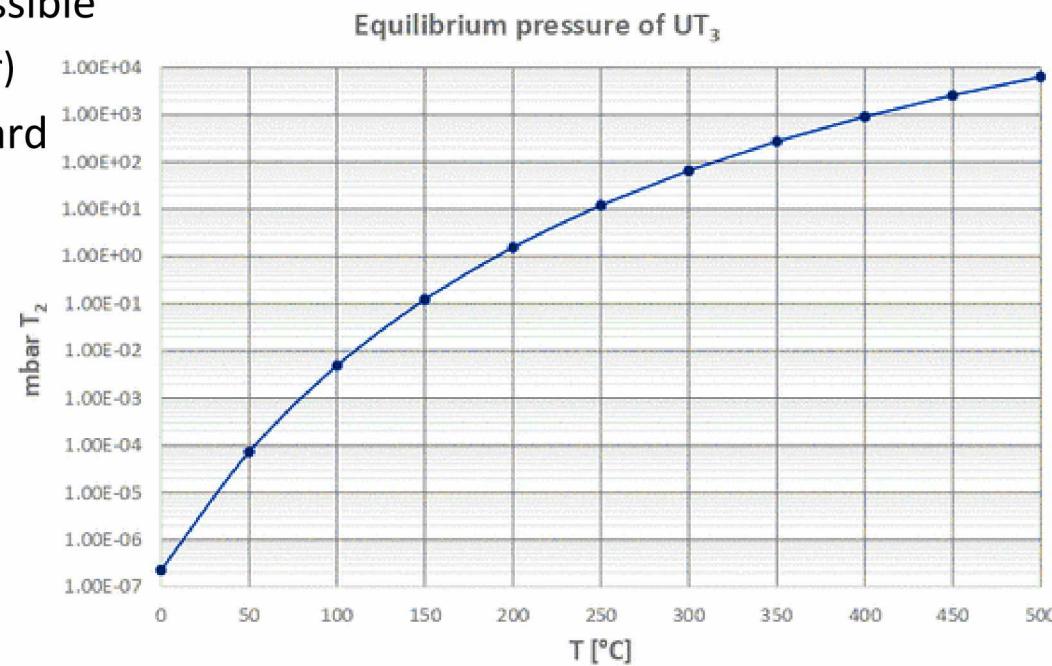


Currently used for tritium gas mixes- internal tritium contamination exists at low levels

Have secondary syringe pump system which could be built into a non-contaminated system for other customers

Pending System Updates

- Reduced volume manifold custom built from SNL-CA
 - Allows for transfer of lower quantities of tritium to the system- better transfer efficiency from the supply cylinder to the fill apparatus
- Supply of tritium gas from U-bed arrangement pending authorization and paperwork update
 - No ${}^3\text{He}$ contamination
 - Higher supply bottle pressures possible
 - At 650 C possible 1 bar (~750 Torr)
 - Currently limited by tritium standard
 - Delivered at 630 Torr
 - Continually depleted with use
 - Ingrowth of ${}^3\text{He}$ with T_2 decay
- Improved Pressure Gauge- higher fidelity with better response



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Experimental Problem Statement and Intro

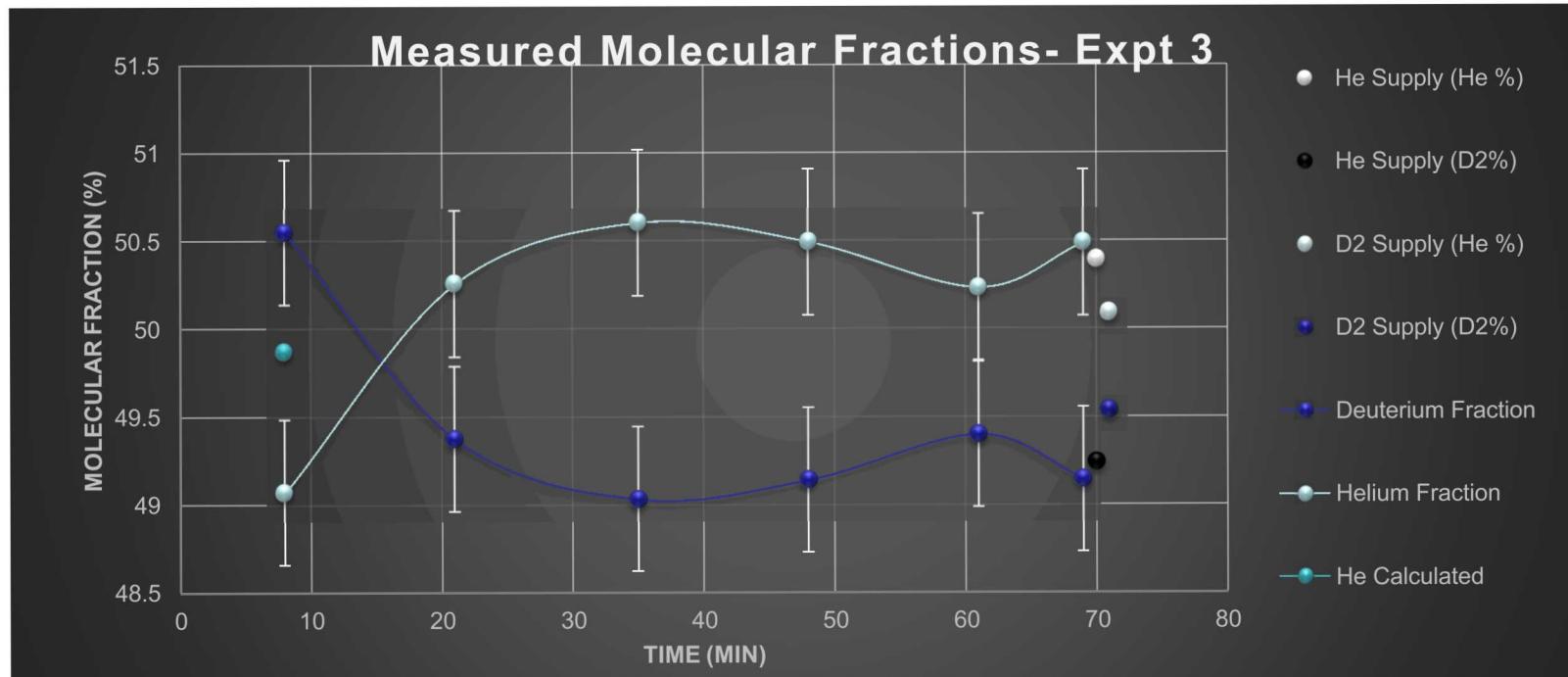
- The system was assembled and documented for use in Z-GTS fills in a short timeframe; therefore a complete characterization of the capabilities and mixing parameters of the system was never completed.
- Initial experiments* set mixing times between 15 and 24 hours for equilibration of the gas mix, even with the improved system.
- During mixing, ~3% of the gas was lost through the O-ring seal on the syringe pump piston, posing a potential hazard to the MOW when tritium gas is present in the mixing system.
- The syringe pump piston housing was refabricated to serve as a ventilated fume hood, removing the hazard to the worker and sweeping any leaked tritium to the TES.
- As tritium quantities increase, the amount of tritium released out the stack will also increase which may eventually lead to potential environmental concerns.



Better characterization of the GMM was required to continue use for increasing quantities of tritium.

Gas Mixing Characterization Results

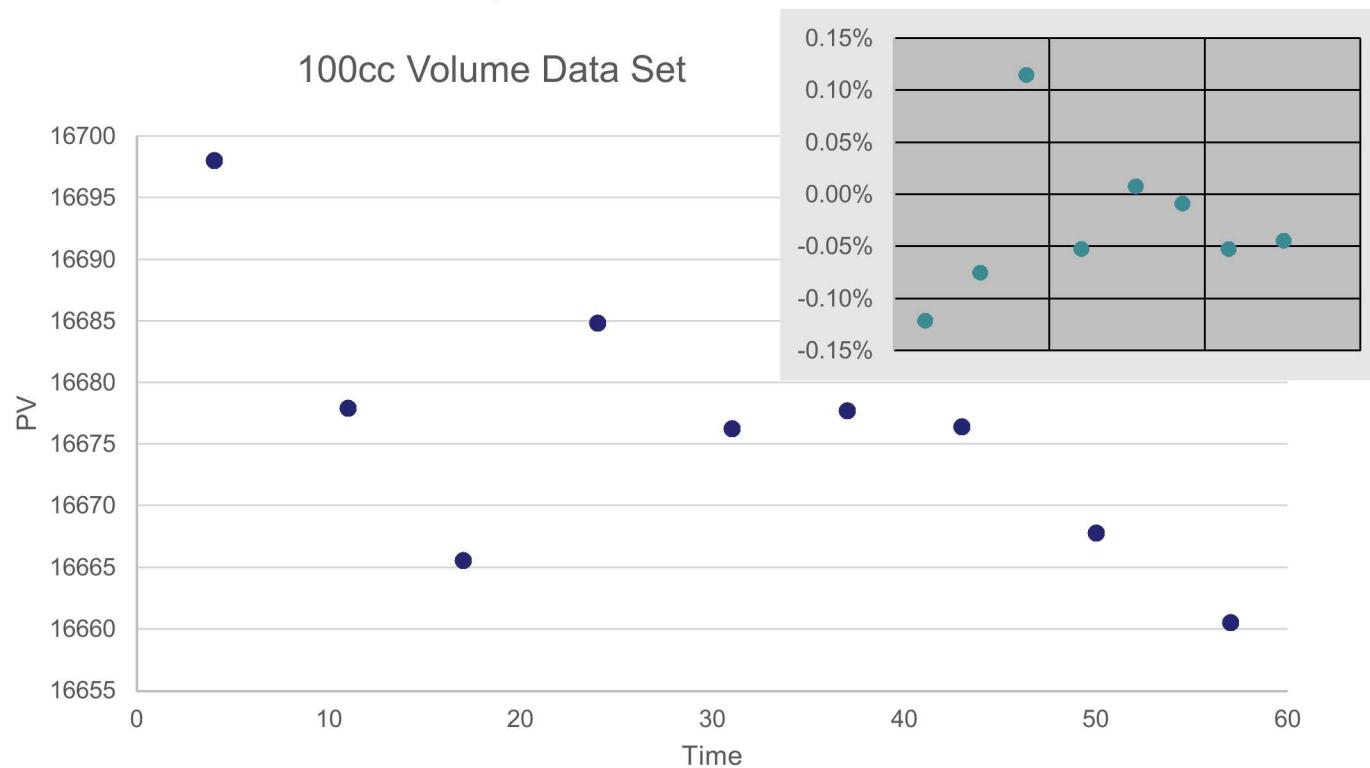
- Samples were taken at ~10 minute intervals.
- Molecular Fraction of He to D₂ compared for each interval
- Error bars generated from standard deviation from the last three data points



Based on the measurement of both initial gas supply bottles at the conclusion of the experiment, no preference for the initial supply gas was present after 1 hour of mixing. Both bottles showed similar fractions of D₂ and He.

Pressure Loss Experiment Results

- The gas mixing system was filled with D₂ to a starting pressure of 500 psig.
- The system was set to recycle at 35 cc/min and allowed to run for 1 hour (~6 minutes per complete cycle- expansion and compression)
- The volume of the syringe pump and the pressure of the system were monitored and recorded every seven minutes.



Even accounting for the volume change between each recorded pressure, no pressure loss was measured above the noise level of the gauge during the hour of the mixing experiment

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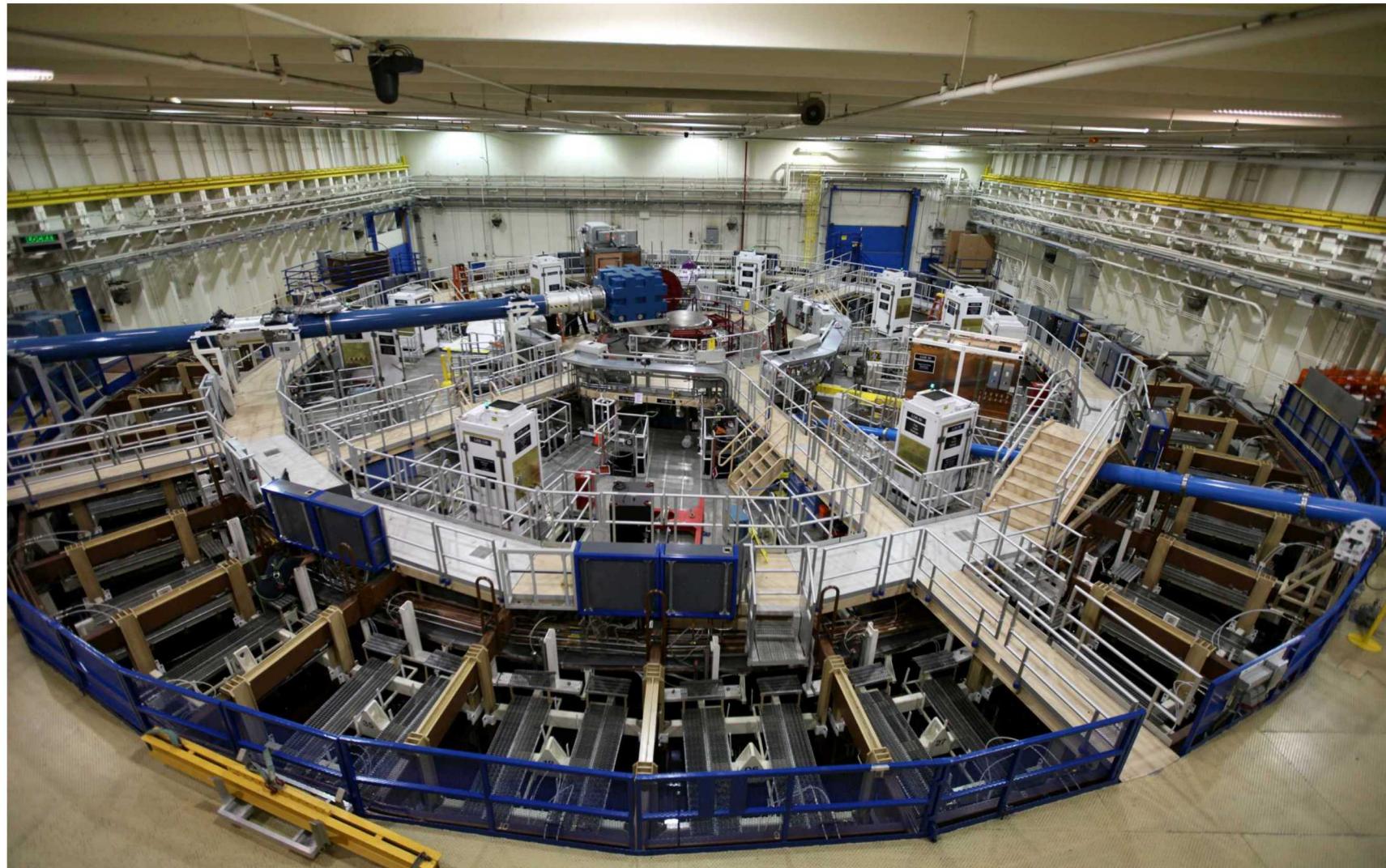
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Gas Analysis Round Robin

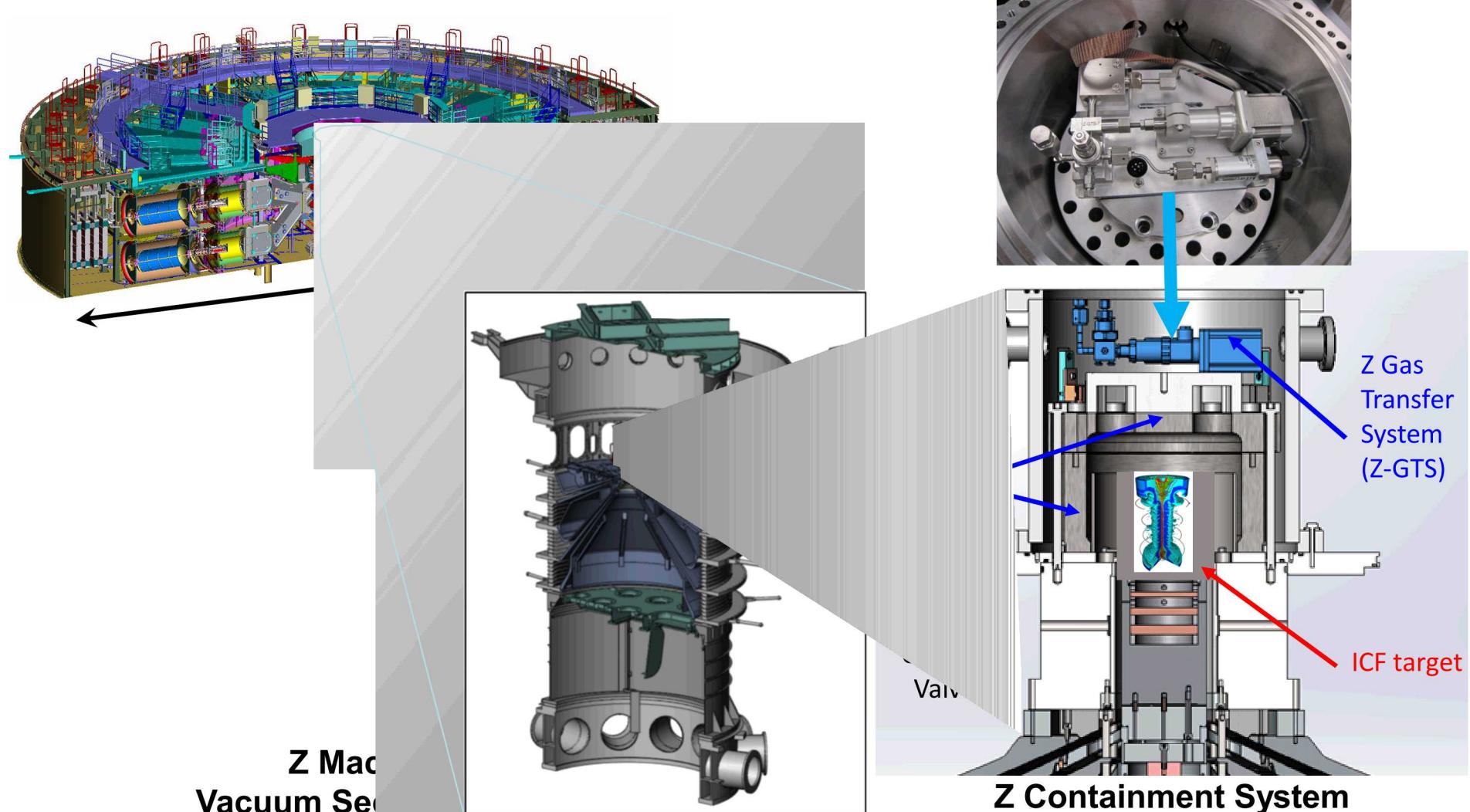
- Concept pending for 12+ years for the complex as a whole
- Intent to Round Robin to compare gas analysis techniques across the different sites
 - Cross-site validation, instrument performance comparison, identify best practices
- Decided to start the effort rather than wait for external initiation
- Good opportunity to test the mixing station concurrently
- Segregated effort into phases and started inviting other sites to participate
 - Phase 0: Analysis of 3 known gas mixtures from Matheson Tri-gas- good history
 - Phase 1: Validation and verification of SNL in-house gas mixes- gas mixing station
 - Phase 2: Analysis of tritium blends for those sites that can accept them- future initiative

Site	Mixes Sent	Pressure	Cylinder Type	Date Shipped
LANL	Phase 0	24.7 psia	50 cc	07/02/18
PNNL	Phase 0	24.7 psia	50 cc	07/11/18
Omega at LLE	Phase 0	28 psia	50 cc	8/14/18
AWE	Phase 0	12.7 psia	500 cc	Pending shipping authorization

The Z Machine at Sandia National Labs



The Z Machine at Sandia National Labs



**Z Mac
Vacuum System**

History of Z Machine Target Fills

- Have provided 9 fills for Z Machine Experiments to date- 2 more planned for FY19

Shot #	Date	Containment	Gas Fill (atomic %)	Gas Fill System	Description
2822	06/27/15	Mock	D, 50.7% ^4He , 4.7% ^3He	Z-GTS	Containment "Null Shot"
2856	09/30/15	Explosive	D, 41.4% ^4He , 12.8% ^3He	Z-GTS	Measure Containment Effectiveness
2857	10/02/15	Uncontained	D, 9% ^3He , 0.02%Kr	Z-GTS	Uncontained Torrance with 3He
2980	07/12/16	Explosive	D, 0.1% ^3He	Z-GTS	Containment Confirmatory with 3He
2987	08/03/16	Explosive	D, 0.1%T	Z-GTS	Contained trace tritium
N/A	07/31/17	N/A	^4He , 2.5% D, 0.0022%T	TH3GA	Seed and Capture Experiment
3167	11/01/17	Uncontained	^4He , 2.5% D, 0.0022%T	Z-GTS + CV	Integration Ride-Along Experiment
3178	11/30/17	Uncontained	D, 0.1%T	Z-GTS + CV	Uncontained Trace tritium
3266	06/13/18	Uncontained	D, 0.5% T	Z-GTS + CV	Chamber Confined Trace Tritium

Continually changing experiment designs and physics goals create an ever changing requirement set for the target fills and as a result the Z-GTS fill composition and pressure

Mixing for Tritium Fills on Z

A spreadsheet is used to calculate the required quantities of gas for each fill

January 19 Z-shot of 0.5% Tritium		
	$R = 0.062363 \text{ torr}^* \text{cc}/\text{umol}/\text{K}$	29017 Ci/mol T
OBJECTIVE: Inject 0.5 % T/D into target with total activity of 685 mCi.		
GTS Fill Pressure	1140 psia	58954.986 Torr
GTS volume	1.935 cc	
T supply bottle	50.000 cc	
1.721 Ci	5.93E-05 mol T	
	551.72 Torr*cc of T as T2 @ 25 C	29.673 =umol T2
TCV-69	72.00% tritium conc. (adjusted for decay per window at right) 05/30	
TCV-69	0.00% %D2 content	
766.276 Torr*cc of TCV-69 gas in FINAL SEED GAS in Z-GTS		

Multiple factors need to be considered to perform the fill calculation:

- Volume of the system, the supply cylinder, and the apparatus being filled
- The concentration of tritium at the time of the fill (accounting for tritium decay)
- The pressure required for the final fill

Potential Need for In-House Tritium Standards

- If Savannah River Site ever loses the ability to provide calibrated tritium gas standards, we will still require gas standards to continue meeting NG production requirements
 - Extend calibration life for current standards – easiest solution, most desirable
 - Less desirable contingency plan: Make standards in-house – lengthy PSL certification process, small volume fills, shorter calibration lifetimes
 - Tritium would still be needed from Savannah River along with cross calibration from some verified source.

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Future Experiments to be Done

- Recharacterize the system to measure equilibration times for the new smaller volume manifold from SNL-CA.
 - Ensure complete mixing times are well known before the fill if performed for the January 2019 experiment.
- Look into the potential of real-time sampling of the manifold mixture with the RGA set-up in the lab.
 - May need to purchase a smaller leak gasket to minimize the depletion of the gases in the system.
 - Characterizing the effect of pulling live gas samples from the mix on the equilibration will need to be considered. Each sampling changes the end state equilibrium that may be reached.
 - With effectively small samples, can this effect be ignored?

Outstanding Questions to Address

- Will a U-bed transfer of 100%T gas from the Development Loader assist with transferring the required amount of tritium for the high pressure fill in January?
 - Initial discussions with Clark Snow point to positive results. The Paro gauge on his system has shown pressures around 750 Torr from the U-beds upon heating to 600 Celsius or above (characterization of this regime for the U-beds has not been performed as pressures would likely exceed the operating parameters of the Paro gauge in the system)
 - This may require the sharing of the remaining tritium in TCV-69, though would facilitate future Z-GTS fills from the same source.
- Can Liquid Nitrogen be used to increase the transfer of tritium to the mixing station?
- How much does the lower volume manifold help increase the transfer efficiency?

Questions? Comments?

