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SAVANNAH RIVER NATIONAL LABORATORY
Operated by Savannah River Nuclear Solutions, LLC

We Put Science To Work

Tritium Instrument Demonstration Station (TIDS)

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April 22, 2014

Tritium Focus Group Meeting

SRNL-STI-2014-00172

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Plant Directed R&D PDRD

What is the challenge?

Tritium Facilities is critically reliant on dated analytical technologies

Low-mass, high-resolution mass spectrometer issues:

- Near end-of-life (30+ years old)
- Spare parts not available from vendor
- Vendor support is difficult or unavailable

Lifetime extended through SRNL R&DE developed:

- Intensive electronic upgrades
- Hardware modifications

Need for alternative, accessible analytical technologies within DP for:

- Complement current analytical methods
- Greater ability to troubleshoot process issues
- Minimization of process upsets and delays
- Optimization of process performance

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Purpose of the PDRD – TIDS FY14

PDRD Fulfillment SRS Tritium Mission – Key Work Scope:

- Maintain skill set and core competencies related to Tritium Facilities
- Integrate SRNL research with Tritium Facility objectives and needs

FY14 Goals:

1. Operation of TIDS in P1 glovebox in 233-H
2. Verification of calibrations using H and D species.
3. Calibration of instrumentation with tritiated species.
4. Analysis of Process sample.

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What measurements are important for Tritium Facilities?

System	Sampling Location	Species
TPS	Inlet TPS/ Outlet ST-909	NX_3, CX_4, X_2O
HT-TCAP	Col C PCV	X_2, N_2, X_2O, CX_4, He
ZR	Tank	NX_3, CX_4, X_2O
Stripper/ ZR	Glovebox	X_2O, NX_3, O_2, sulfurs, hydrocarbons, CO_2, CO
TCAP/ HT-TCAP/ P-Evac	Feed/ Recovery Beds/ Product & Feed headers	X_2, N_2, He, X_2O
DI	Inlet/ Outlet of: FTB, DE, Diffuser Stage, Downstream from ST-198	N_2, NX_3
DI/P-Evac	DE Vessel/ Acc. Tank	NX_3, X_2O, X_2
SCLU	Possibly around the 100L tank	CX_4
Mixing	Ar and D_2 cylinders	Ar, X_2, O_2

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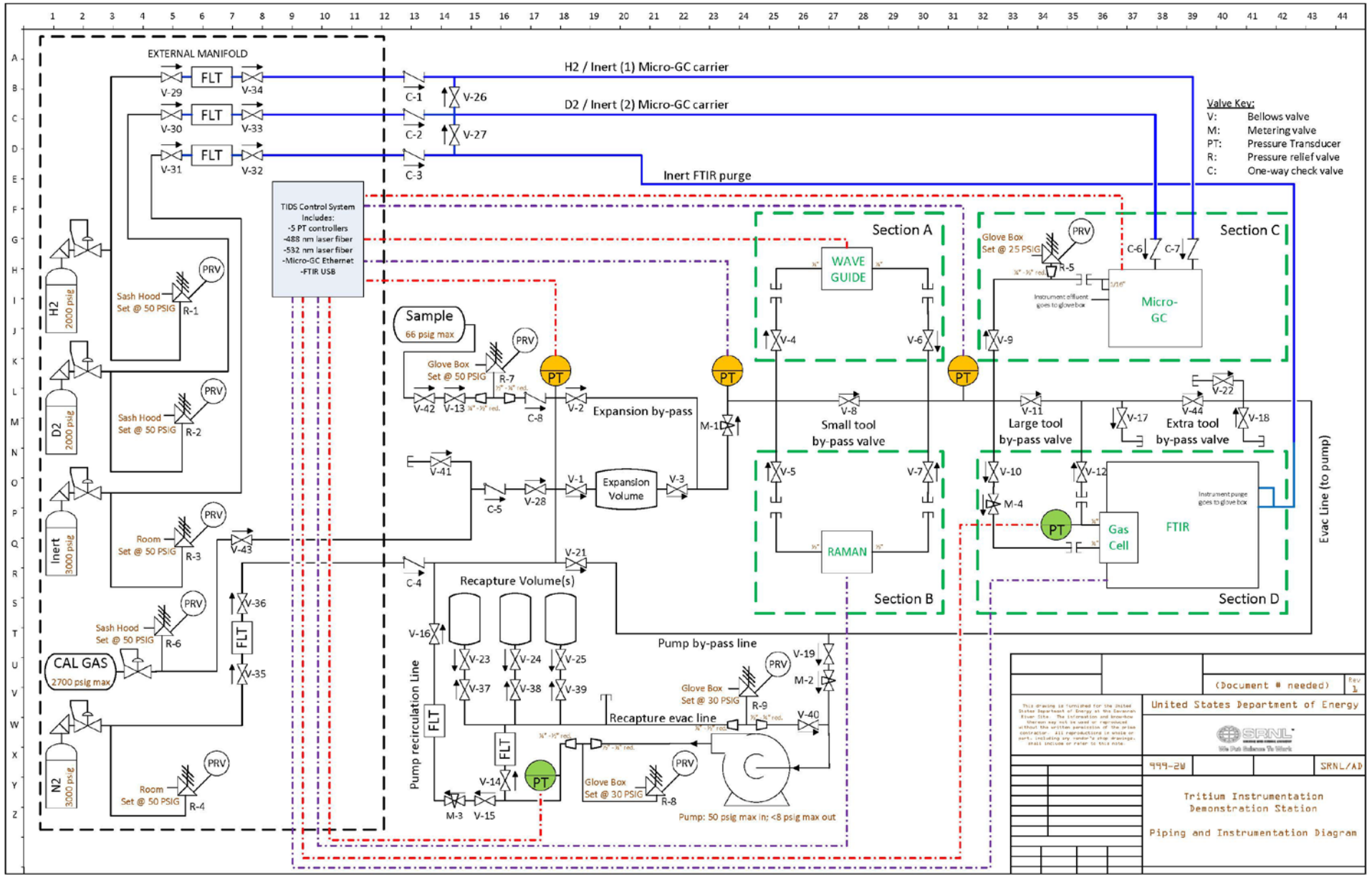

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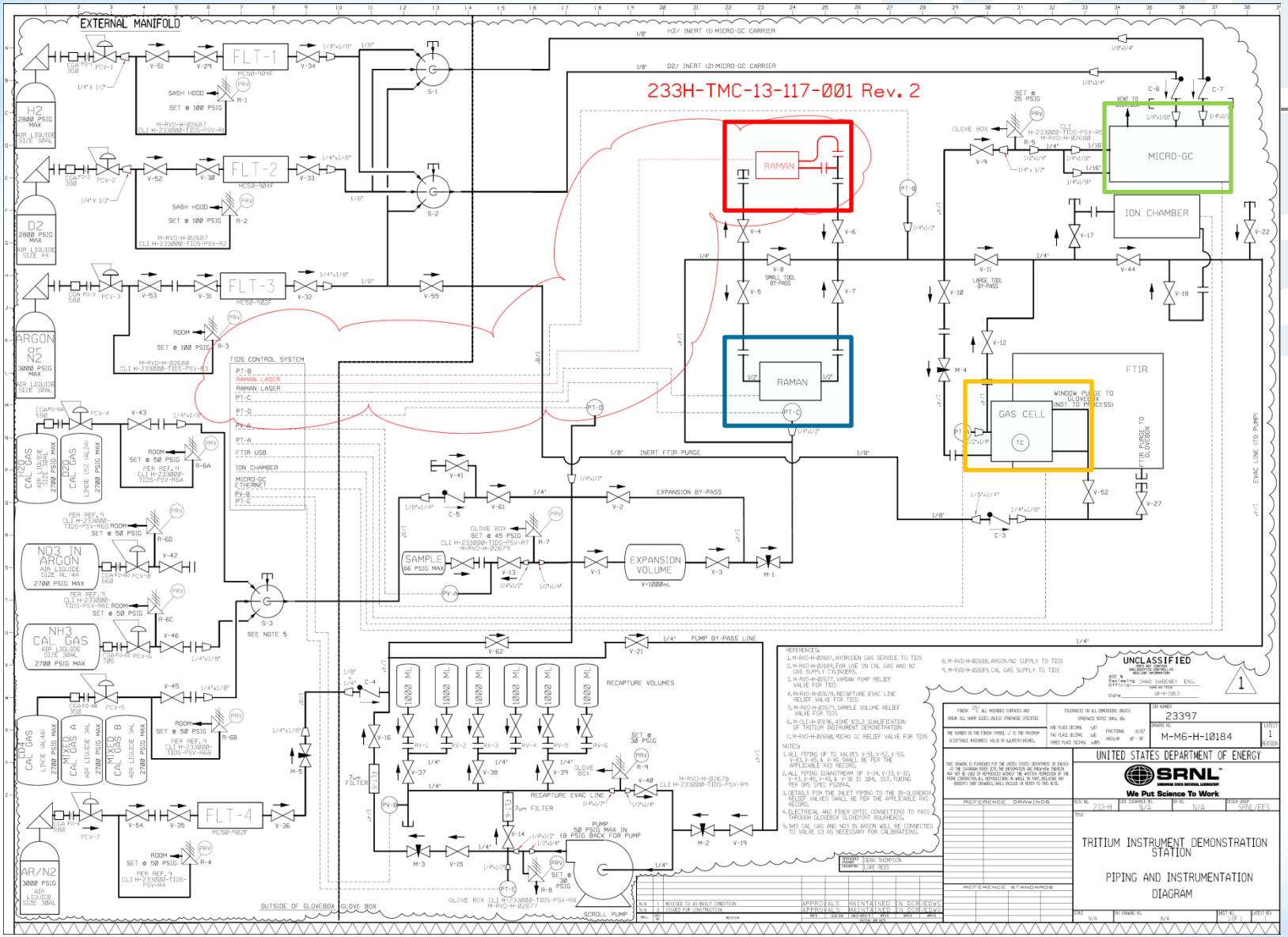

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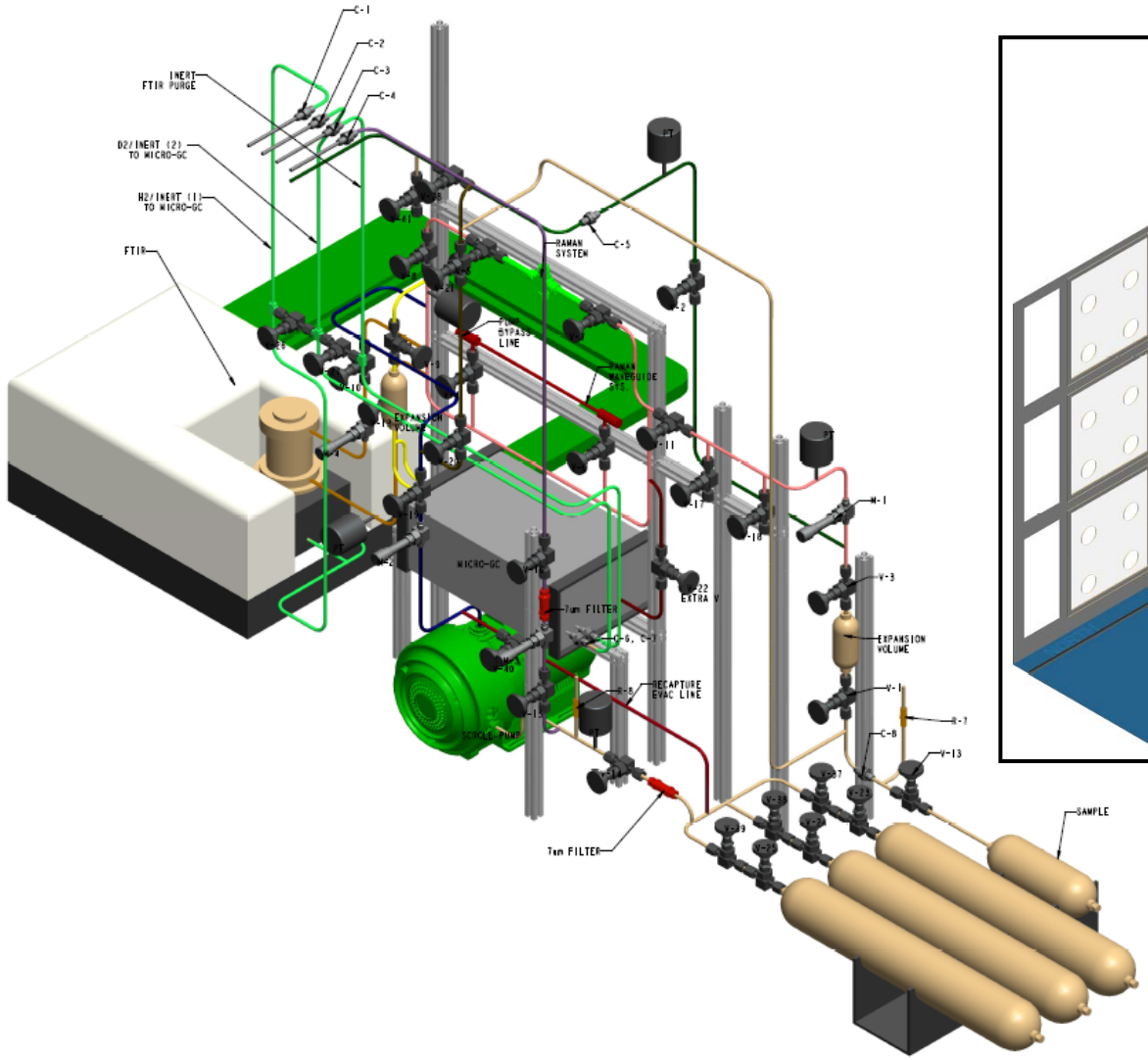

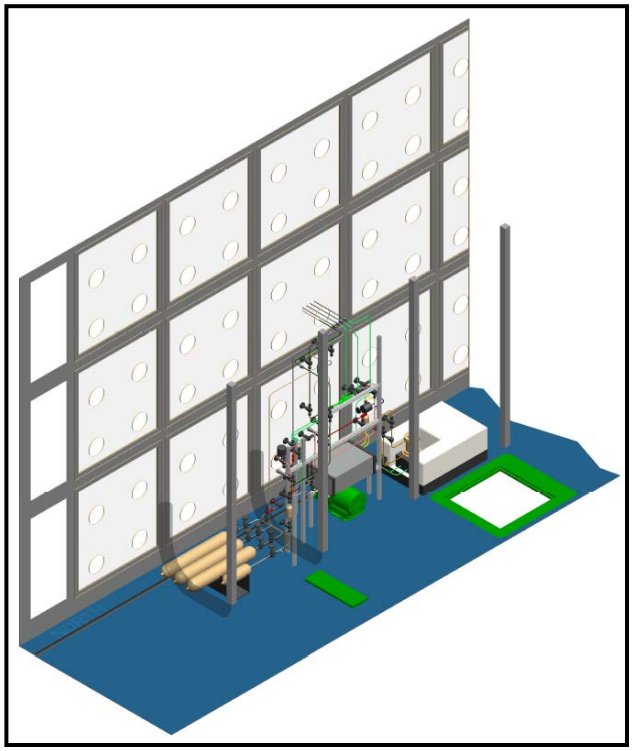

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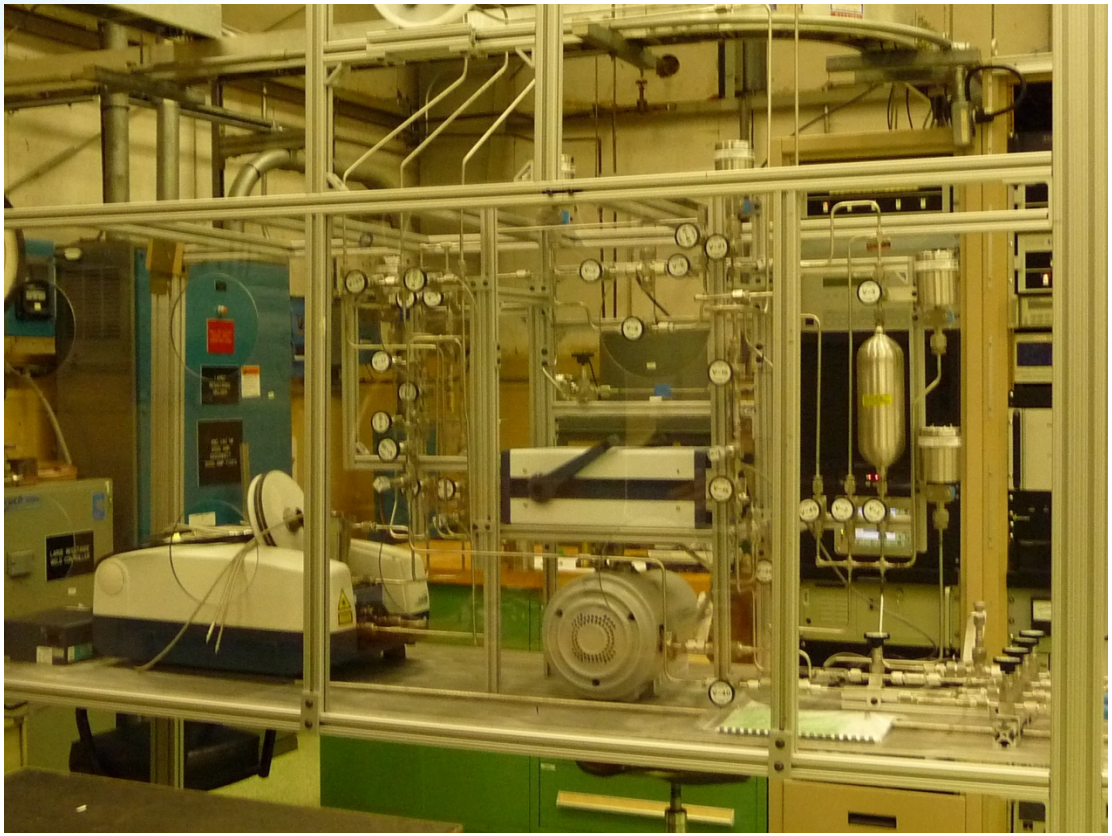
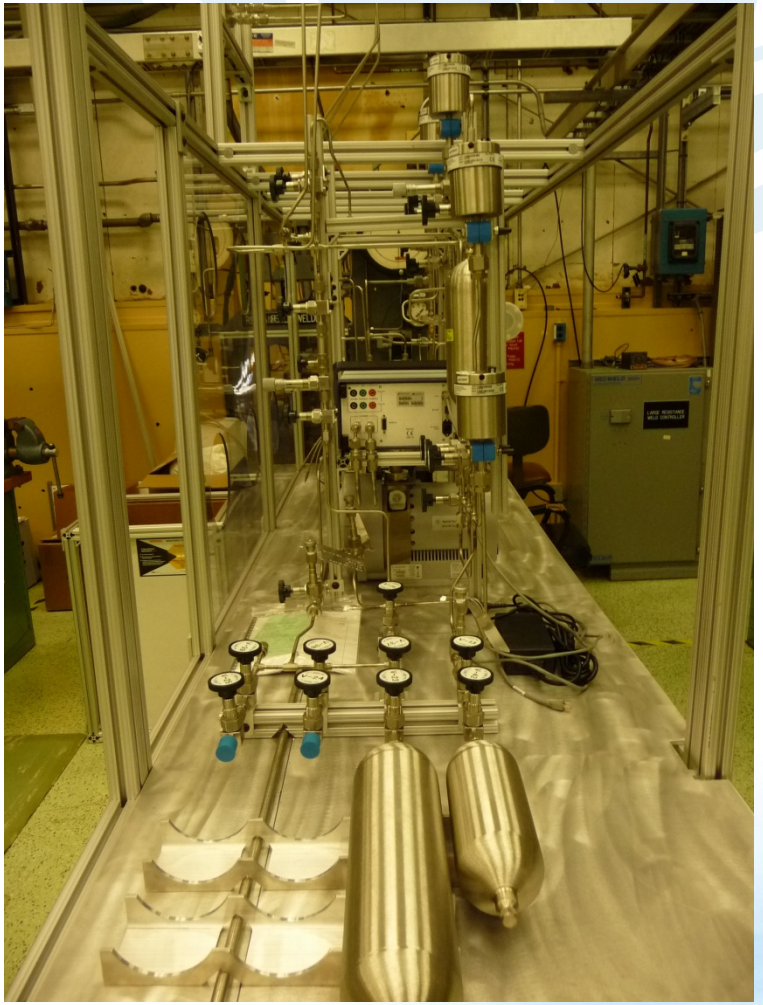
From sketch to finished product – TIDS fabrication





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Analytical Instrumentation Specs

A) Fourier Transform Infrared Spectrometer (FTIR)

Tool:

JASCO 4200

CIC Photonics 4Runner (6 meter) custom-made gas cell

Detection Purpose:

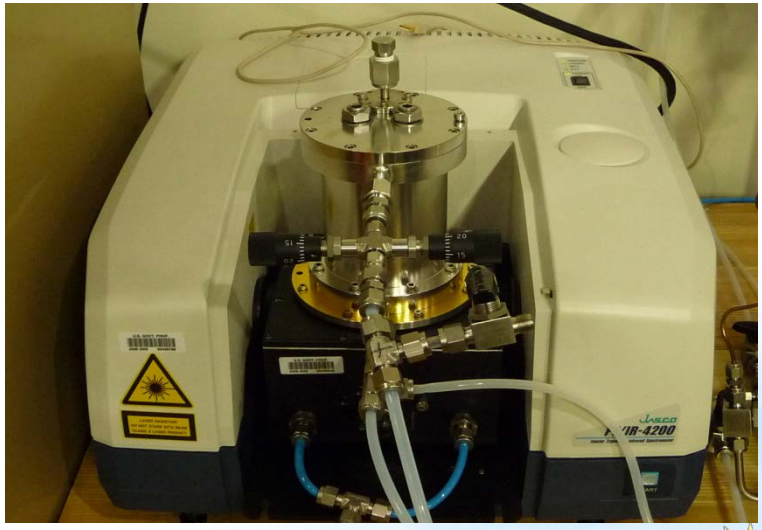
NX_3, CX_4, X_2O, CO_2, CO, C1 to C4 analytes

Sensitivity / Limit of Detection (LOD):

<1 ppm with appropriate gas cell

Uncertainty:

5% at ~ 1ppm



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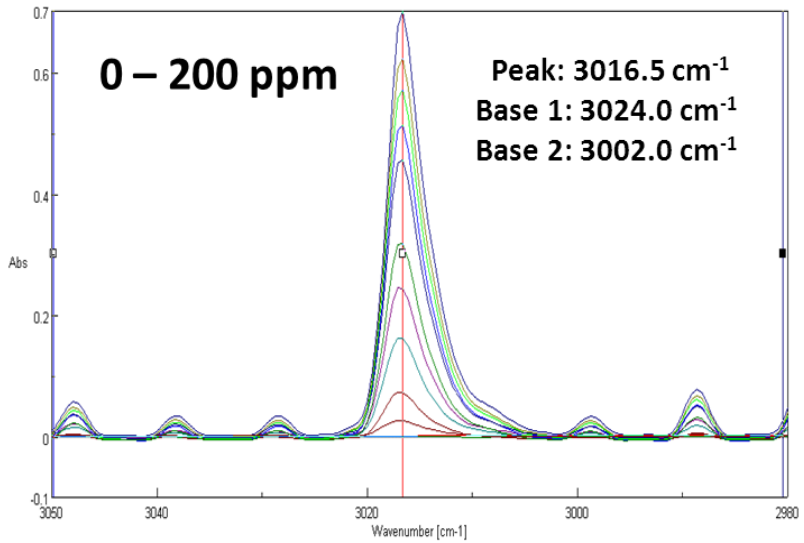
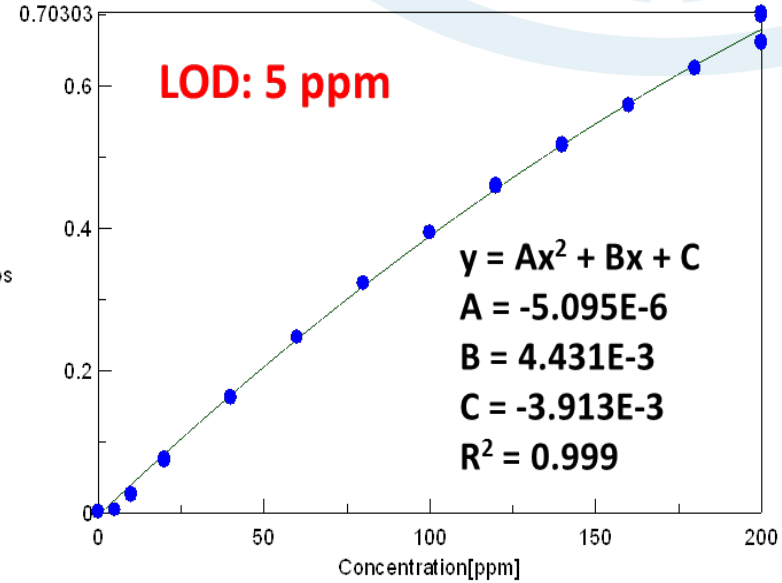
Sample FTIR Spectra: CH_4

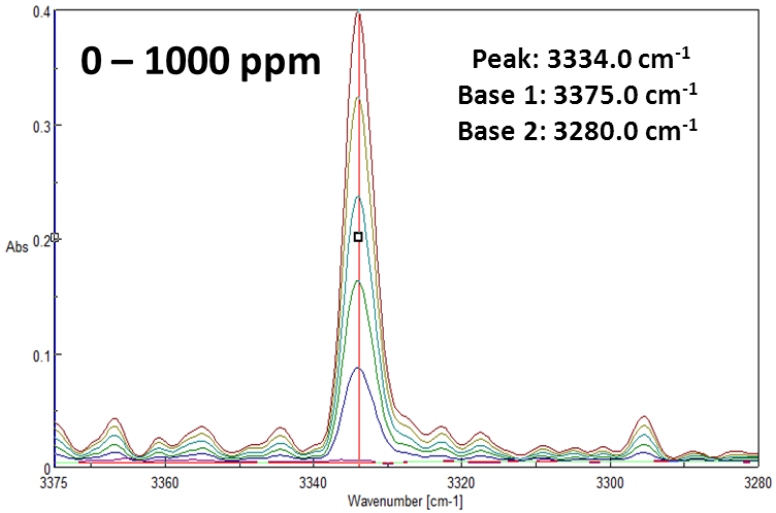
Asymmetric C-H stretching

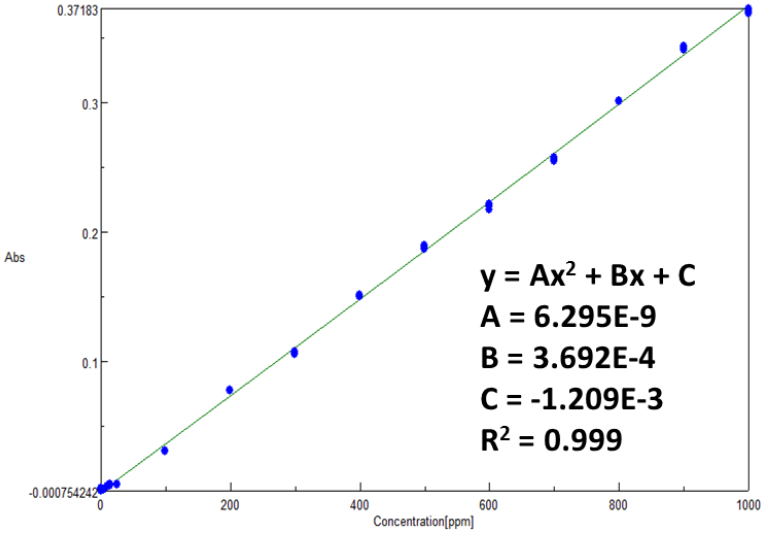




Sample FTIR Spectra: NH_3

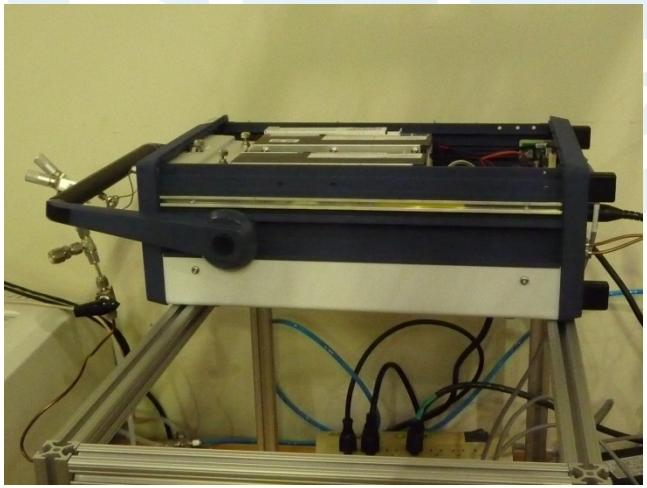
Symmetric N-H stretching

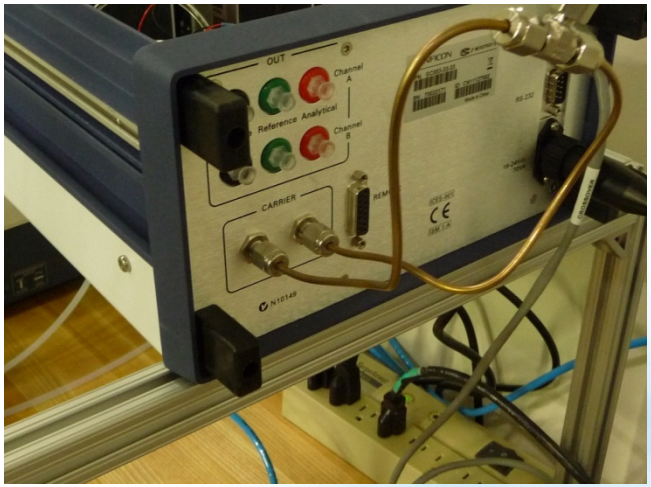




Analytical Instrumentation Specs

B) Micro Gas Chromatography (Micro GC)





Tool:

Inficon (Agilent) 3000 Micro GC

Duel 5A mol-sieve PLOT columns (10 m)

Plot U (6 m) on order

Detection Purpose:

X_2, CX_4, CO_2, N_2, He, CO

NX_3

Sensitivity / Limit of Detection (LOD):

~200 ppm for “X” isotopologues

~10 ppm for all non-X analytes

Uncertainty:

3-5% typical

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Analytical Instrumentation Specs

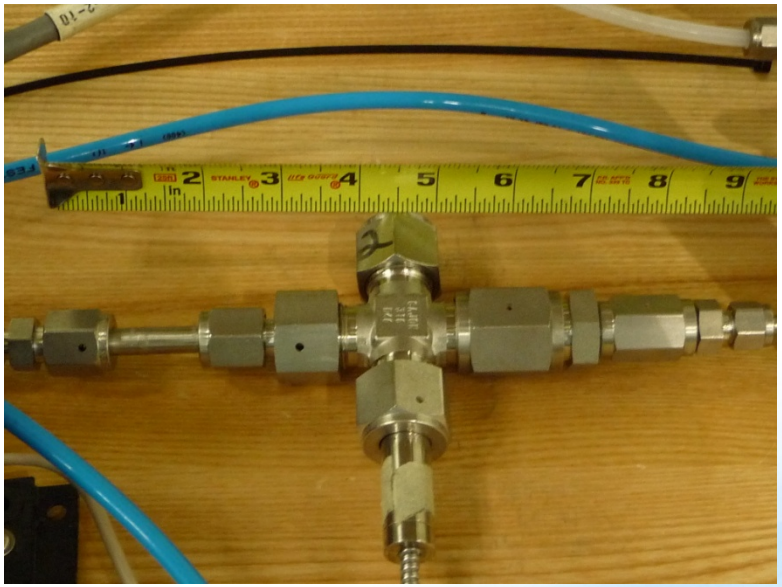
C) SRNL-developed Raman probe

Tool:

Coherent Sapphire 200 – 488 nm; 200 mW max power
RoMack Inc. 20' fiber optic (6 around 1) on ½" VCR fitting

Kaiser Optical Holospec- f/1.8 I VIS w/ 488 grating

Andor DV-420-OE CCD detector



Detection Purpose:

Primary – X_2, CX_4, N_2

Secondary – NX_3, CO_2, CO

Sensitivity / Limit of Detection (LOD):

> 1000 ppm for X_2

Unknown for other analytes

Uncertainty:

2-5% typical

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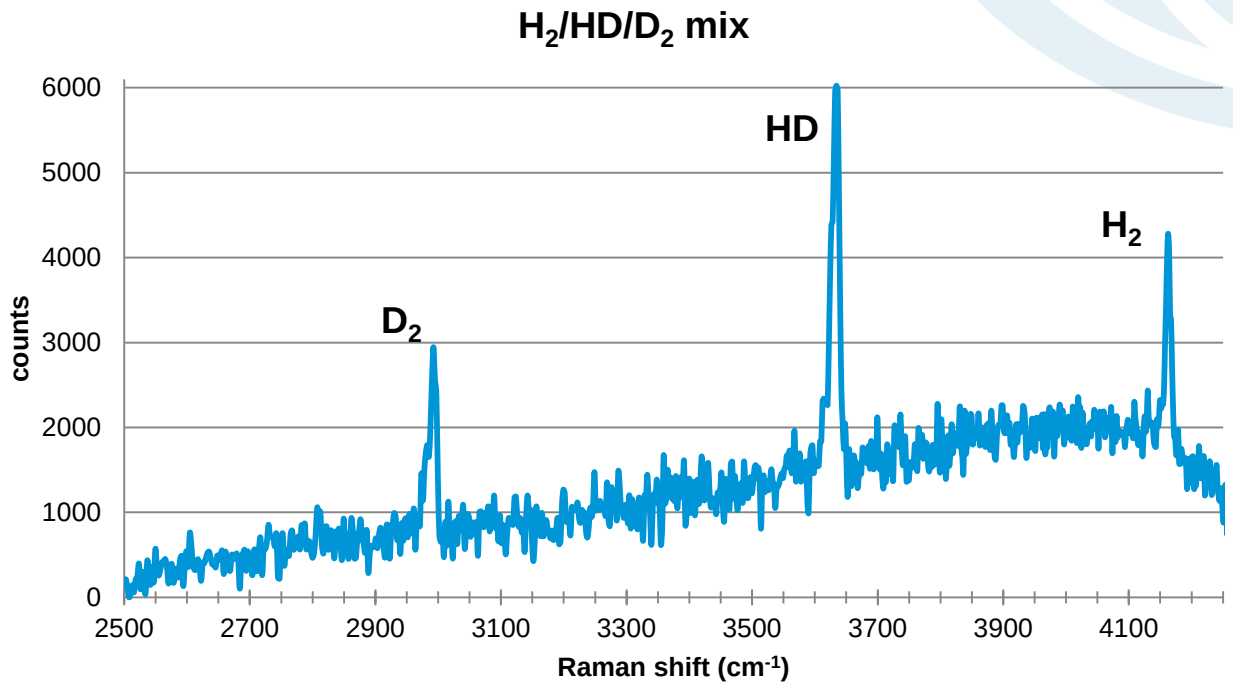
Sample Raman spectrum



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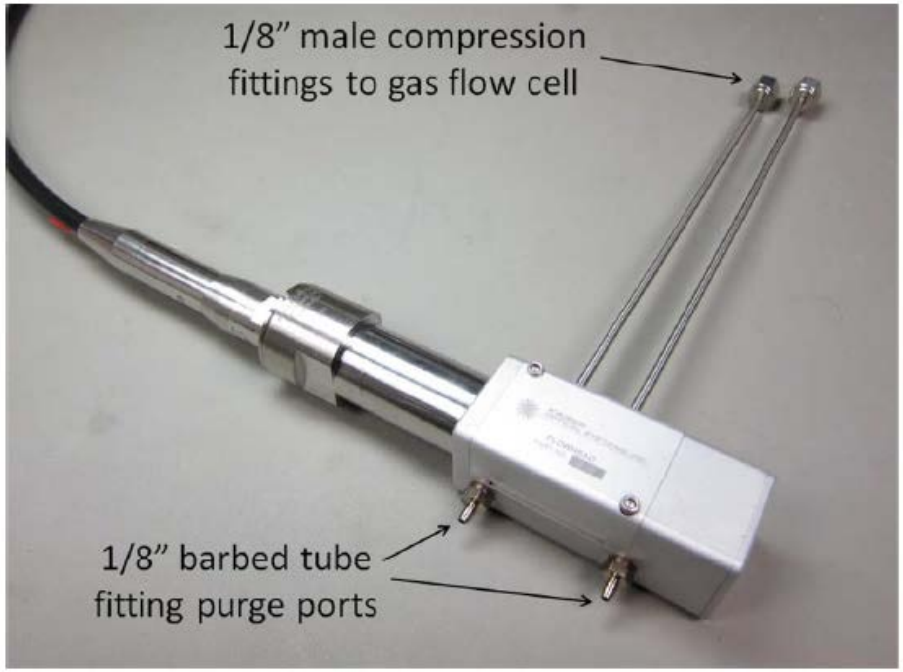
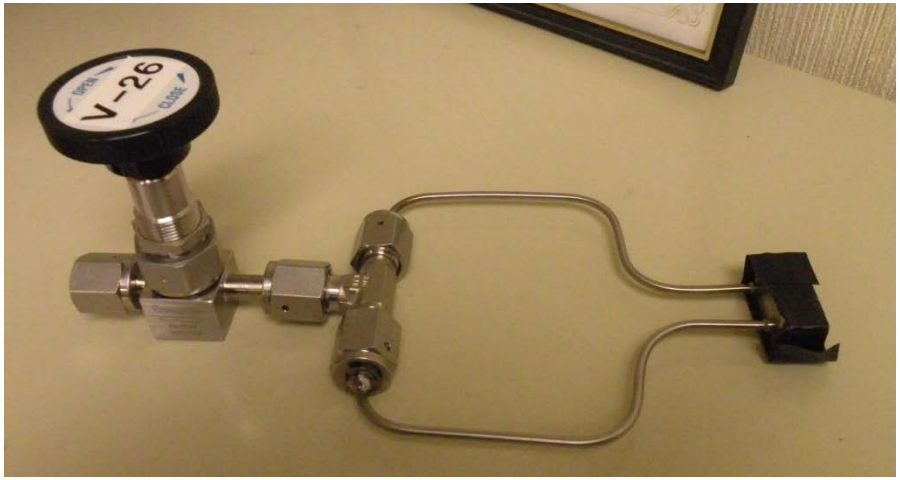
Raman Probe

- **Kaiser “AirHead” probe with “FlowHead” attachment**
  - Gas cell inserted into retroreflection cavity
  - Cell is part of manifold; reduces demands on fiber integrity





FlowHead before insertion into probe

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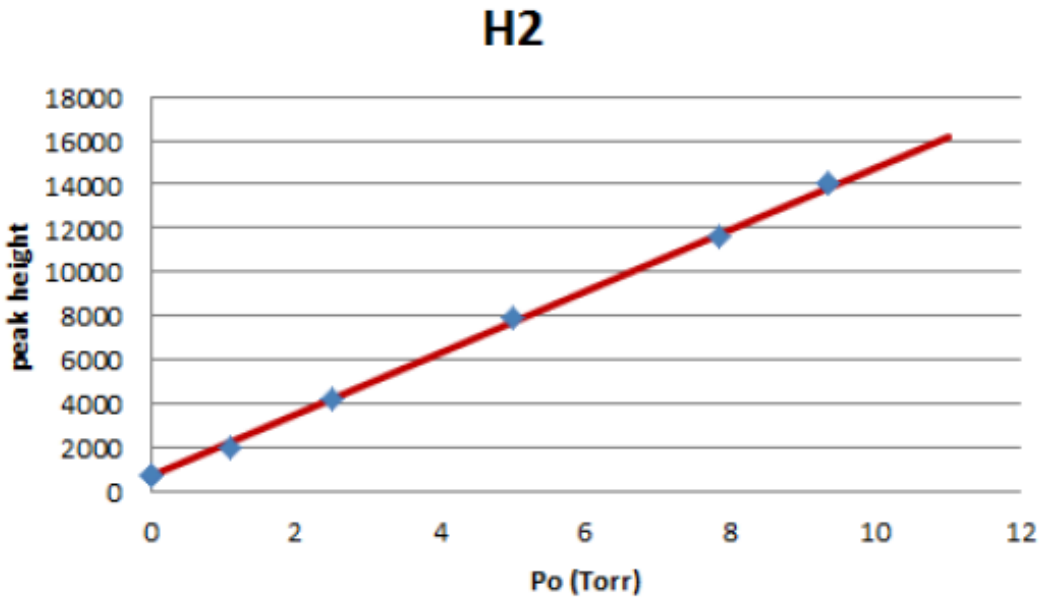
Raman Probe – lab results with FlowHead

- **Estimated LODs:**
  - H_2, $D_{2:}$ ~0.2-0.3 Torr
  - (~300 ppm for 1 atm sample)
  - CH_4: 0.1 Torr
  - CO_2: 0.4 Torr
  - O_2: 1 Torr
  - N_2: 2 Torr



- **Probe/method status:**
  - Probe installed in glove box and functional
  - Optimization of gas cell position required to increase signal
    - (decrease time required to obtain above LODs)

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Achievements

- **External manifold was fabricated**
- **Temp Mod was USQ reviewed and approved**
- **TIDS manifold and instruments operating procedures approved**
- **Temp mod generated for discharge of gases**
- **Kaiser Airhead probe installed**

Lessons Learned

- Select a PI and plant process engineer who are known and respected by the plant and the lab.
- Establish an MOA between the lab and the plant clearly establishing roles and responsibilities for design, fabrication, installation and operation
- Identify early in the project, personnel who will be needed at every step of the project.
- Schedules = Attention; Get the project on the plant schedules early.

  This helps with identification of personnel that needs to be involved.

  Integrate installation, hazard analysis, safety basis reviews and changes, procedure development and personnel training into the plant schedules even if you have to add detail later.

- Clearly identify analysis requirements/needs of the plant (Sensor Technology Roadmap)

  Develop a sensing needs and requirements questionnaire for the plant

  Develop a sensor technology capability and attributes questionnaire for the lab

  PIs and process engineers complete questionnaires

  Develop a semi-quantitative assessment to rank and prioritize plant needs and match them to sensor capabilities

Lessons Learned (cont'd)

- **Identify a host glovebox that would not affect production**
- **Design TIDS so that installation does not require a production outage**
- **Work with the Tritium process engineer to identify special requirements for equipment and work in a tritium facility**

  Conduct of Operations vs. Conduct of R&D
  Tritium compatible materials in equipment/instrumentation
  Security Plans – Data management, transfer between SRNL/Tritium
- **Fabricate the "Tritium Ready" test station in SRNL where researchers can easily access it**
- **Calibrate sensors with non-tritiated gases in a clean environment**
- **Develop operating procedure(s) simultaneous to clean calibration work**

  Allows for early identification of required changes

Lessons Learned (cont'd)

- **Remember that calibration standards containing tritium for analytical instrumentation are not commercially available and so consider options early.**

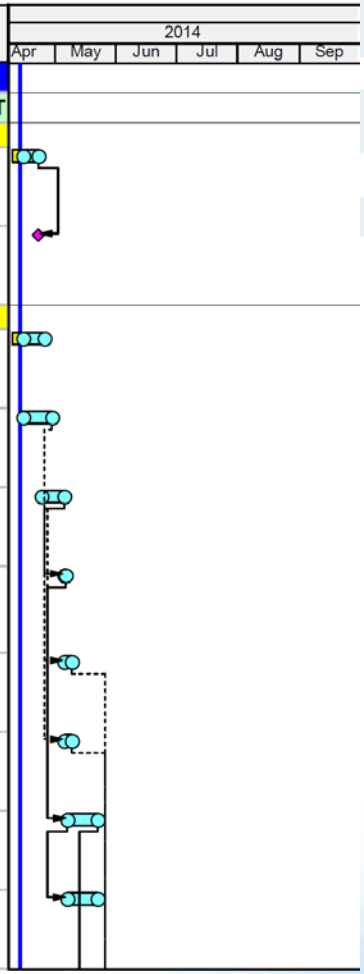
  Prepare standards on TIDS
  Procure passivated standard volumes as early as possible
  Ask Tritium Labs to prepare; plan for long lead time and they will not prepare tritiated ammonia or moisture
  Estimate/Model tritiated analytes based on protium/deuterium standards.

- **Identify line break prep conditions and test/optimize operating procedure in a clean environment**
- **Develop facility training package for SRNL researchers working on the TIDS**
- **Move TIDS to tritium plant and transition lead role to plant personnel for plant installation**
- **Transfer "ownership" back to SRNL**

FY14 Schedule

SRTE PROGRAMS | TIDS | Current Date = 16-Apr-14

#	Activity ID	Activity Name	Start	Finish	Perf Org	Comments
1		**WAEP FY14 DELIVERABLES**				
2		**3.8 - PDRD - SR12022 TRITIUM INSTR. DEMO STATION (TIDS) (Cortes-Concepcion - SRNL, Huckabee - T**				
3		**OPERATIONS ACCEPTANCE CHECKLIST (OAC)**				
4	ATIDSSU5	STARTUP - COMPLETE OAC SECTION B / MGR ACCEPTANCE	10-Apr-14 A	22-Apr-14	SU	
5	ATIDSSU7	RTO - TIDS MANIFOLD HAS BEEN HANDED OVER TO SRNL PERSONNEL		22-Apr-14	SRL	
6		**POST OUTAGE WORK**				
7	AREDV	233-H FACILITY IN REDUCED VENTILATION FOR CW COOLING COILS REPLACEMENT	10-Apr-14 A	25-Apr-14	OPS	
8	A130791909	CLEAN SWAGELOK FLEX LINES	15-Apr-14 A	29-Apr-14	TMO	STAGED
9	A130640101	SRNL TO PERF CHARACTERIZATION OF TIDS VOLUME(S)	24-Apr-14*	05-May-14	SRL	
10	A130487601	INSTALL 233H-TMC-13-150 TO SUPPORT TIDS CALIBRATION	06-May-14*	06-May-14	TMO	REQ149512 & 144850 STAGED, POC - L BOONE & M. THOMAS
11	A130791902	INSTALL M-DCF-H-12618; FLEX H-233000-DI-FCON-DI.31	06-May-14*	09-May-14	TMO	
12	A130791903	INSTALL M-DCF-H-12618; FLEX H-233000-DI-FCON-DI.113	06-May-14*	09-May-14	TMO	
13	A130640103	SRNL TO PERF TIDS CAL - NON-AMMONIA	07-May-14*	22-May-14	SRL	GB DISCHARGE
14	A130640102	GLOVEBOX DISCHARGE OF TIDS CALIBRATION GASES	07-May-14*	22-May-14	SRL	



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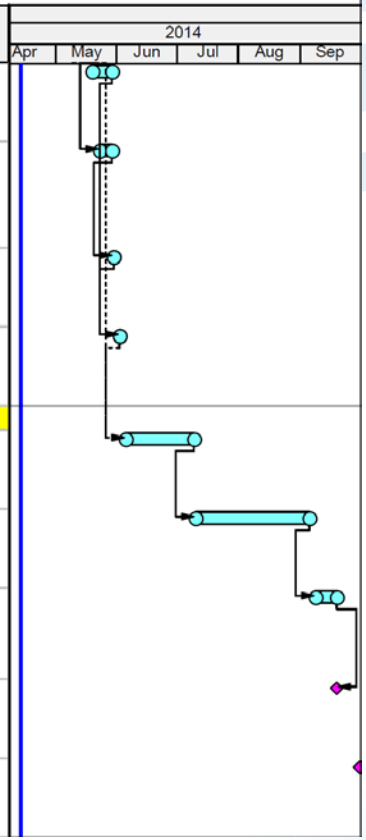
FY14 Schedule (cont'd.)

SRTE PROGRAMS | TIDS | Current Date = 16-Apr-14

#	Activity ID	Activity Name	Start	Finish	Perf Org	Comments
15	A133627801	CLEAN / FUNC CHECK SPARE ION CHAMBER ON TIDS MANIFOLD	19-May-14*	29-May-14	TMO	MAR - REQ#168026 - PSD - 5/15 / P1 GB
16	A130640104	SRNL TO PERF TIDS CAL - AMMONIA (METHANE)	23-May-14*	29-May-14	SRL	SASH HOOD DISCHARGE, ALT MONITORING REQ, OPS BYPASS FORMS MONITOR
17	A130487602	REM 233H-TMC-13-150 TO SUPPORT TIDS CALIBRATION	30-May-14*	30-May-14	TMO	154871 STAGED
18	A133627802	INSTALL SPARE ION CHAMBER ON TIDS MANIFOLD	02-Jun-14*	02-Jun-14	SRL	G SIDES
19	WAEP 3.8 DELIVERABLE					
20	ATIDSDEMO	DEMONSTRATION - DELIVERY OF FIRST PROCESS SAMPLE (DI Sample)	05-Jun-14*	09-Jul-14	SRL	PERFORM OPS PROCEDURE 233-52200
21	ATIDSOPS	OPERATE TIDS TO ANALYZE TRITIUM PROCESS SAMPLES	10-Jul-14*	05-Sep-14	OPS	
22	ATIDSRP	WRITE & ISSUE REPORT OF ANALYSIS EVAL AND RECOMMEND PATH FORWARD FOR TRIT PROCESS OPTIMIZATION	08-Sep-14*	19-Sep-14	SRL	
23	ATIDSRP5	FORECAST COMPLETE / CLOSE CODES		19-Sep-14	OPS	
24	DEL3.8	MILESTONE - OPERATE TIDS, ANALYZE SAMPLES & GASES, & ISSUE REPORT		30-Sep-14*	MTF	Murphy



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Plant Directed R&D PDRD

Team Members/Contributors

- **SME's**
  - Rob Lascola
  - John Young
  - Bill Spencer
- **MTF**
  - Steve Murphy
  - George Thomas
- **Tritium**
  - Jon Wright
  - Don Appel
  - Joey Huckabee
  - Stephen Douglas
  - Raymond Lott
  - Brad Gentry
  - Rebecca Floyd
  - Louis Boone

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