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Operated by Savannah River Nuclear Solutions, LLC

We Put Science To Work

Tritium Instrument Demonstration Station (TIDS)

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Tritium Focus Group Meeting

SRNL-STI-2015-00248

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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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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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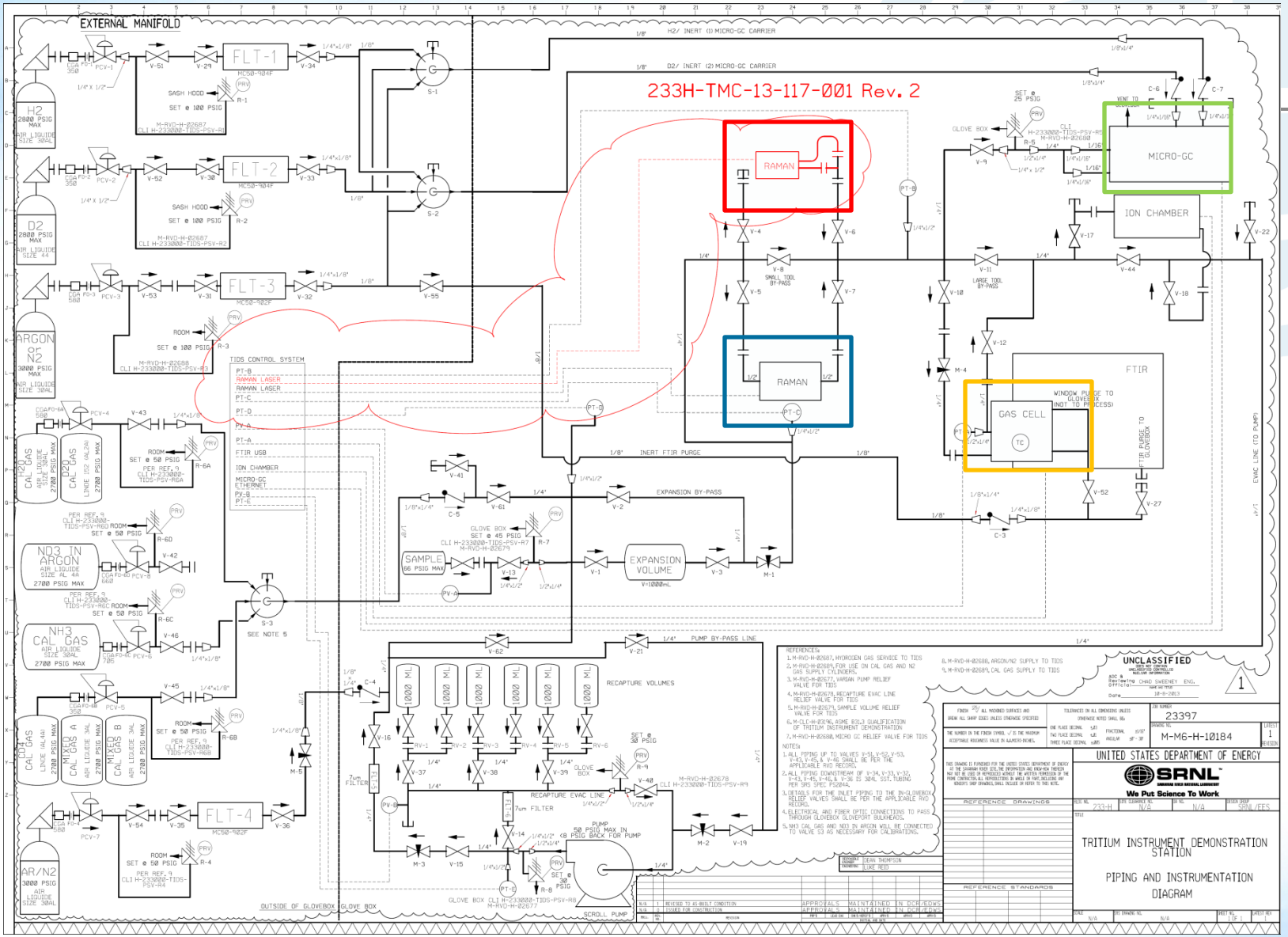

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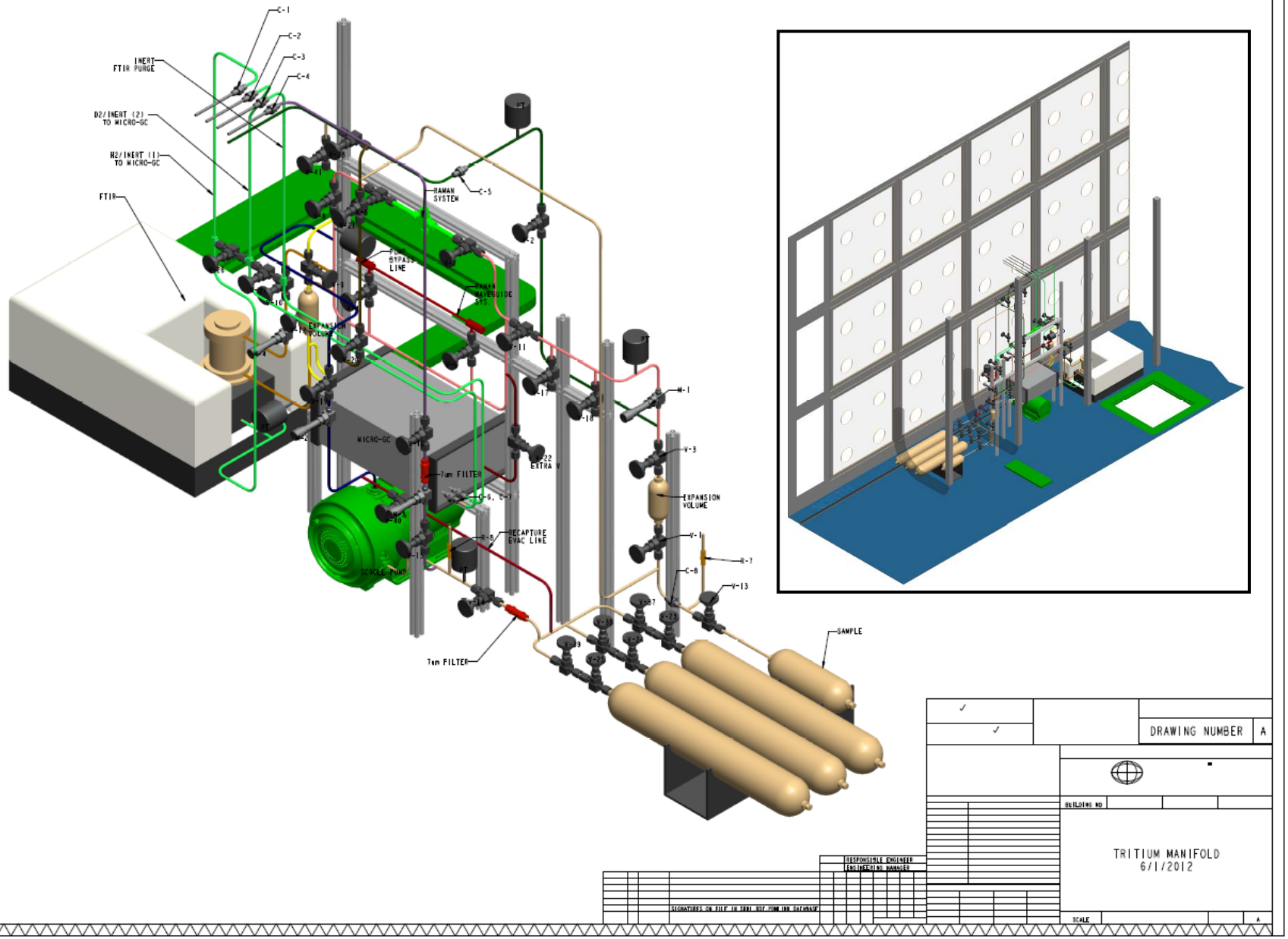

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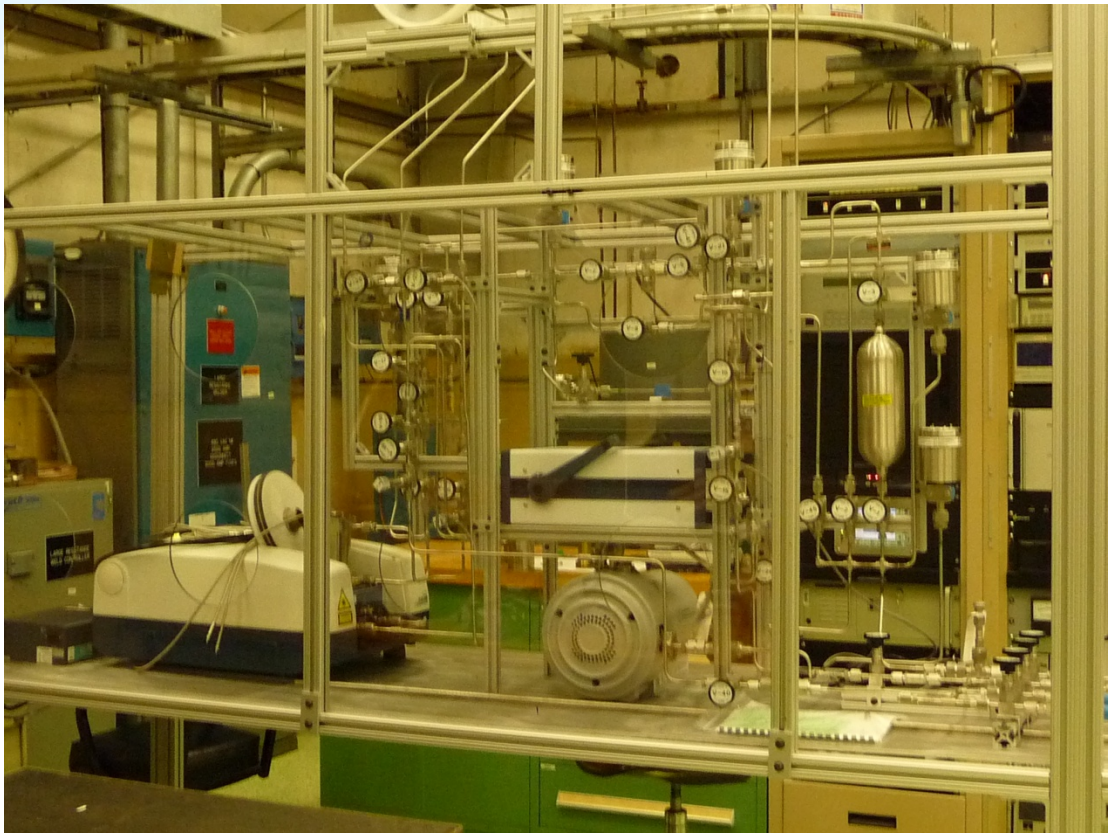
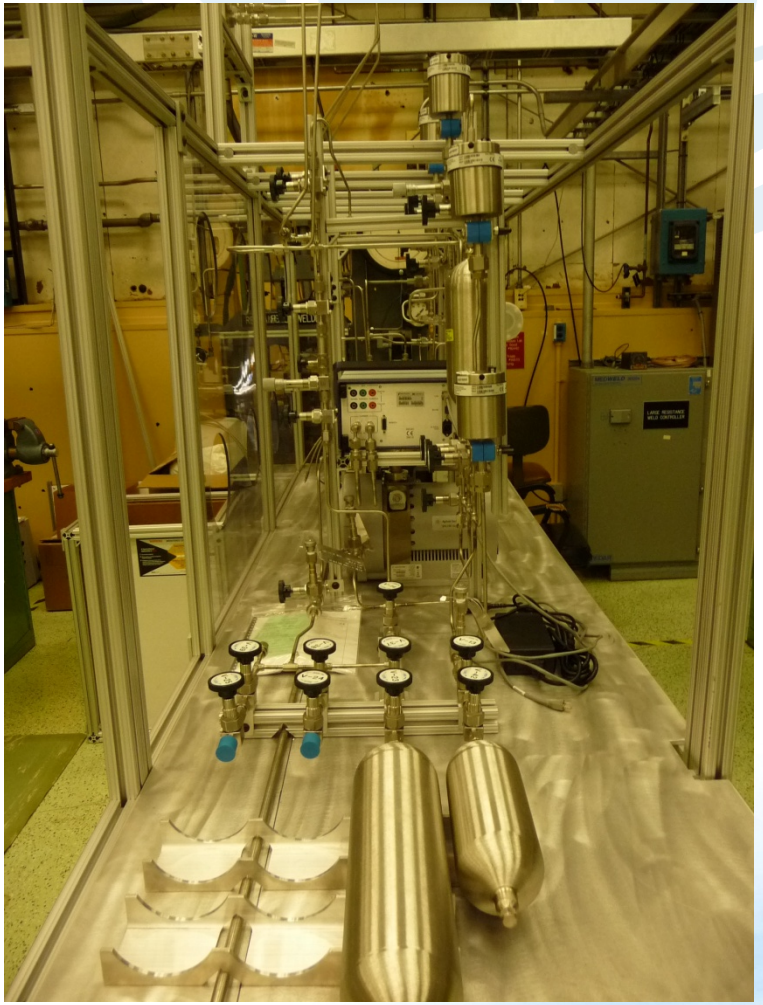
From sketch to finished product – TIDS fabrication





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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:

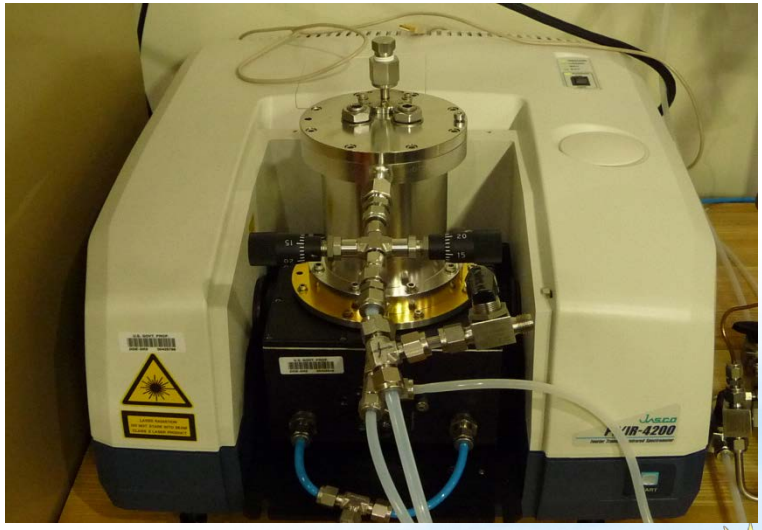
NX_3, CX_4, X_2O, CO_2, CO, C1 to C4 analytes

Sensitivity / Limit of Detection (LOD):

<1 ppm with appropriate gas cell

Uncertainty:

5% at ~ 1ppm



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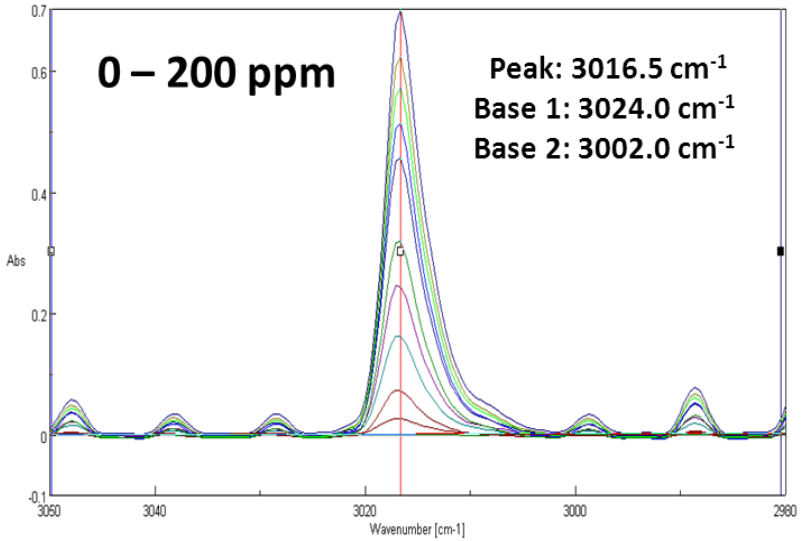
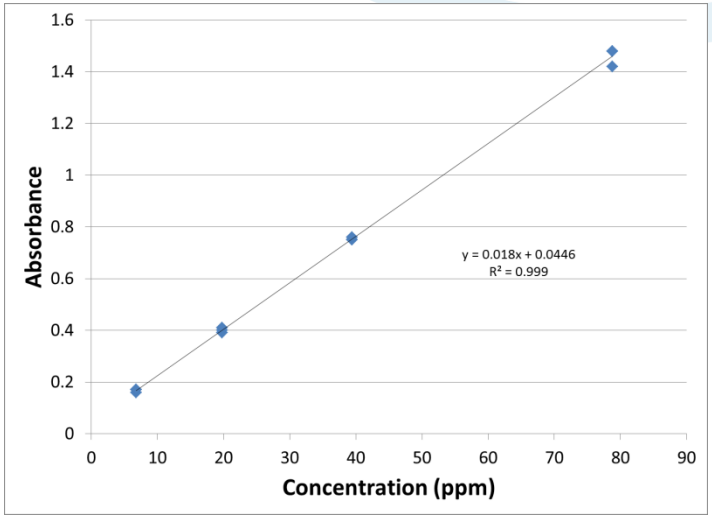
Sample FTIR Spectra: CH4

Asymmetric C-H stretching





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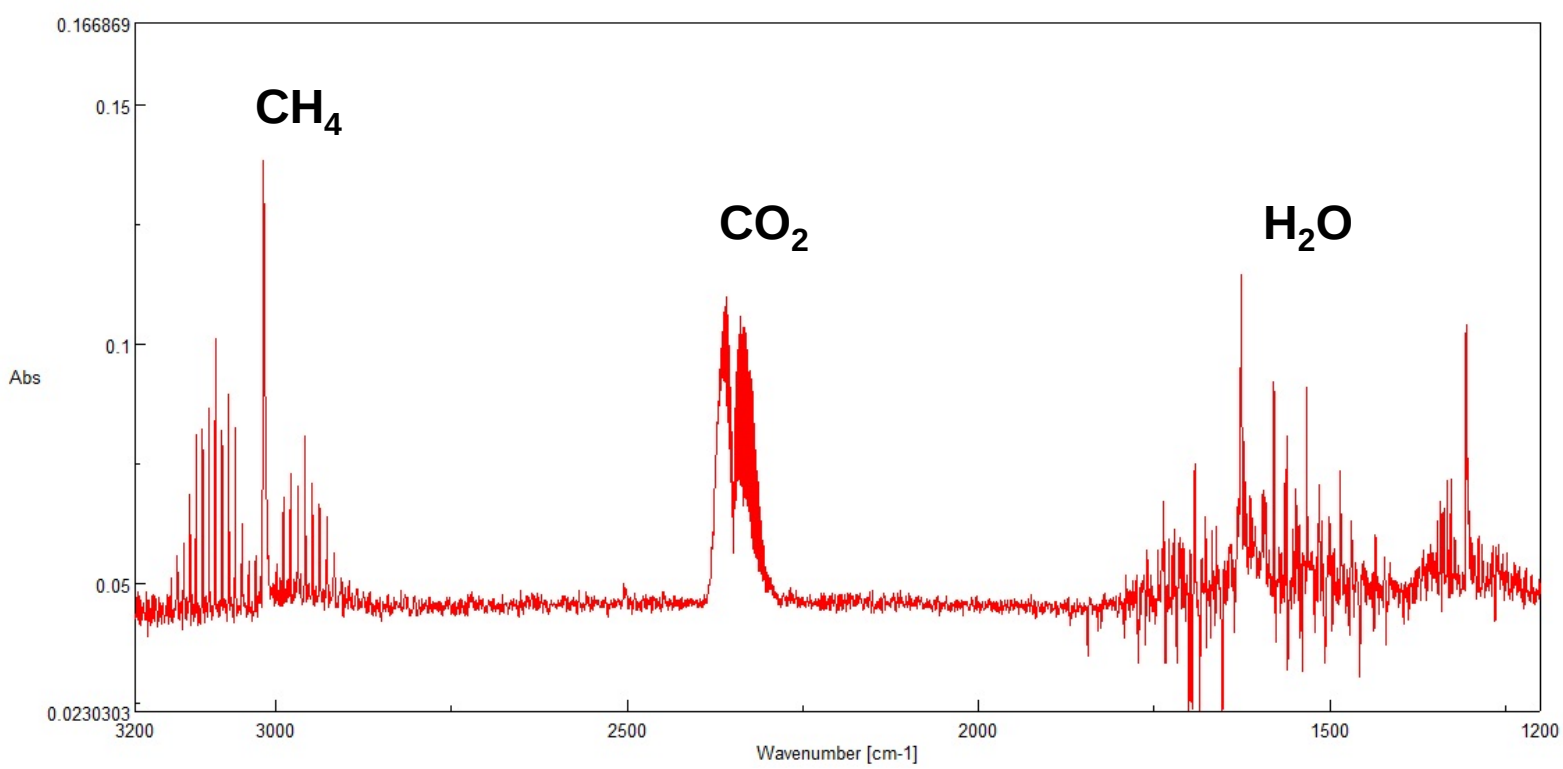
TPS Sample 1: FTIR Spectrum



~24 ppm CH_4, ~30 ppm CO_2, and 70 ppm H_2O

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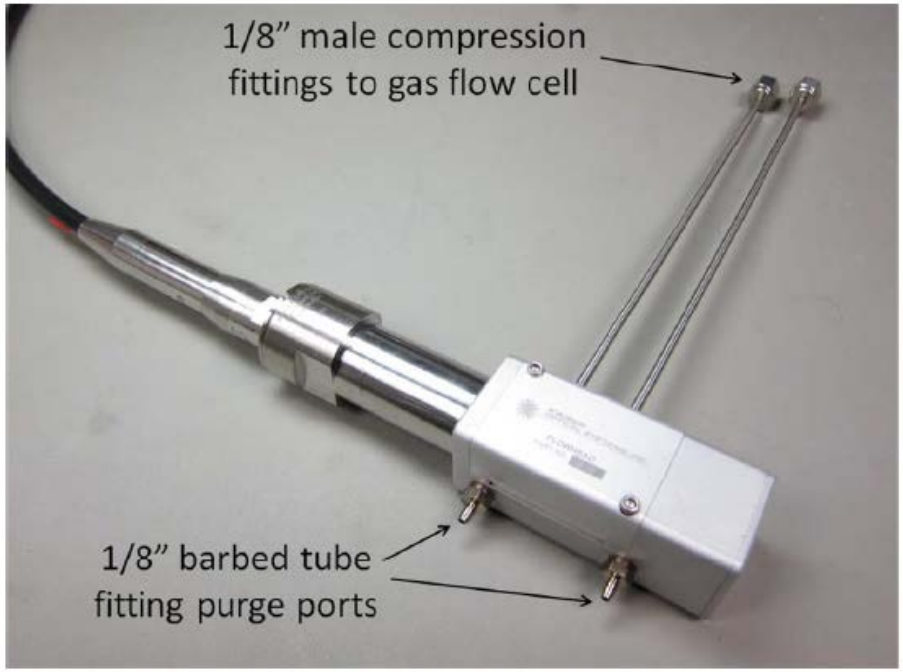
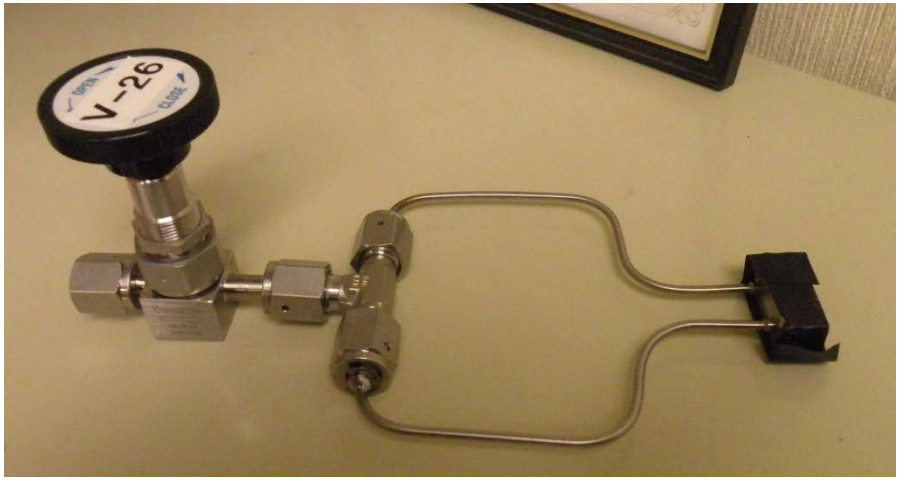
Raman Probe

- **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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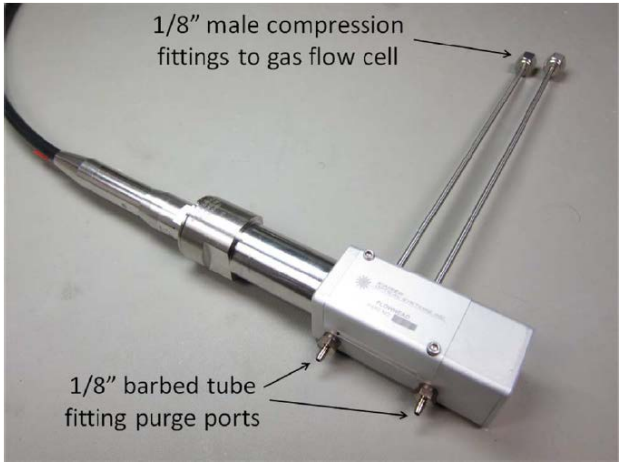
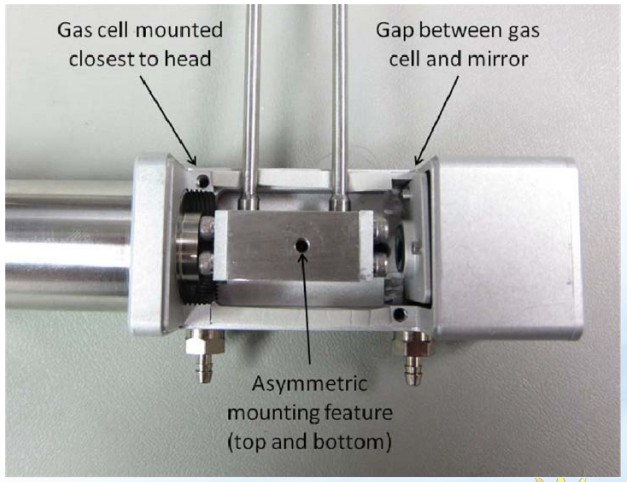
Raman Spectroscopy

- Kaiser Optical Systems (KOSI) HoloSpec
  - f/1.8 imaging spectrograph
  - Andor TE-cooled CCD
  - 532 nm, 0-4300 cm^{-1} range (5 min integration)
- DHOM DPSS laser
  - 150-200 mW

- KOSI FlowHead probe
  - Fiber-coupled imaging probe
  - Retroreflecting cavity
  - Flow cell with sapphire windows





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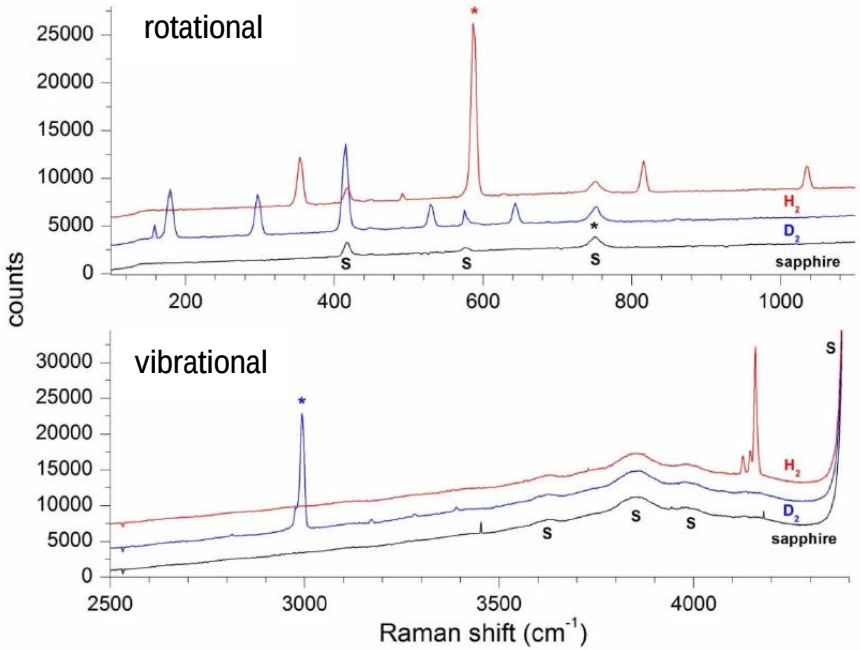
Raman Spectroscopy - Calibrations

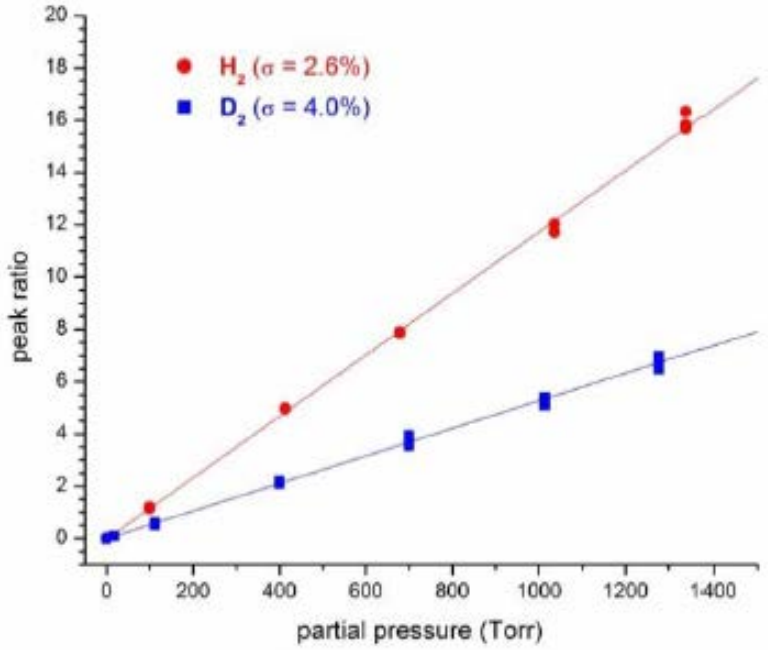
Analyte	H_2	D_2	HD, HT, DT, T_2	N_2	CD_4	ND_3	O_2	CH_4	CO_2
LOD (Torr)	5	3	~10	8	3	>20	~6	~2	~7
Calibration	Direct		Estimated	Direct		Estimated			

spectra

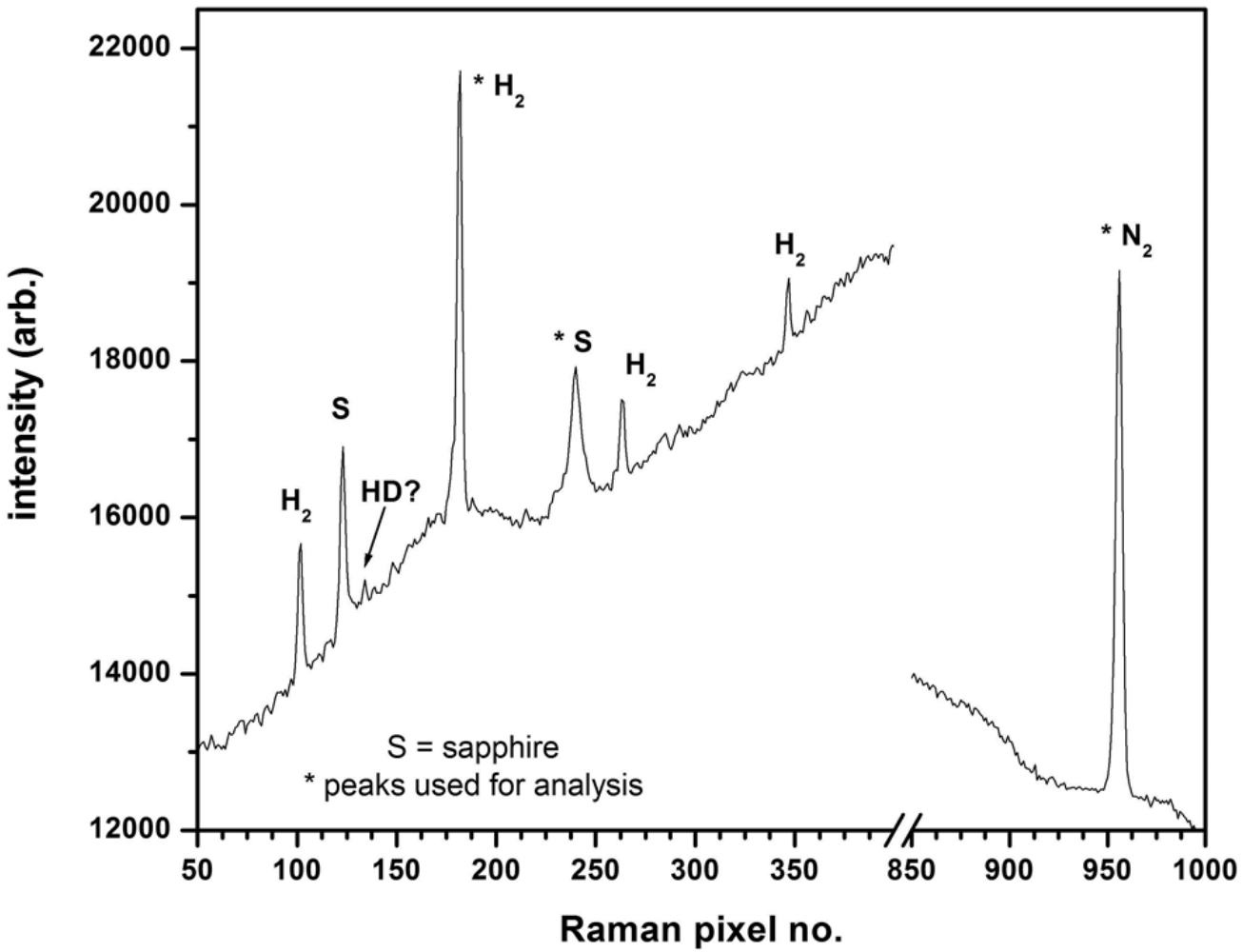
H_2, D_2



calibration curves



Sample Raman spectrum



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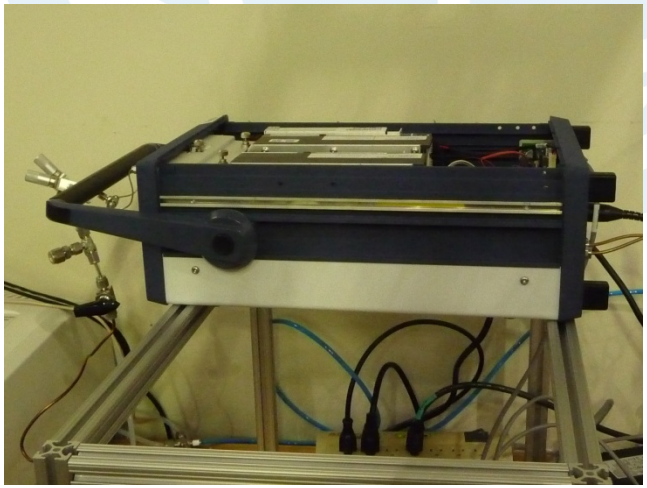
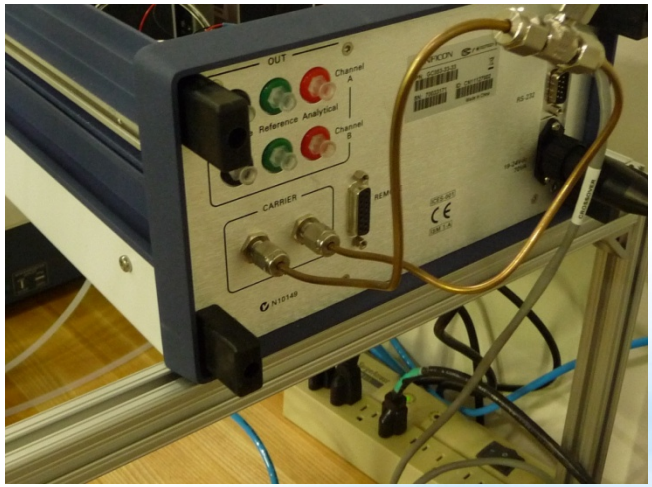
Analytical Instrumentation Specs

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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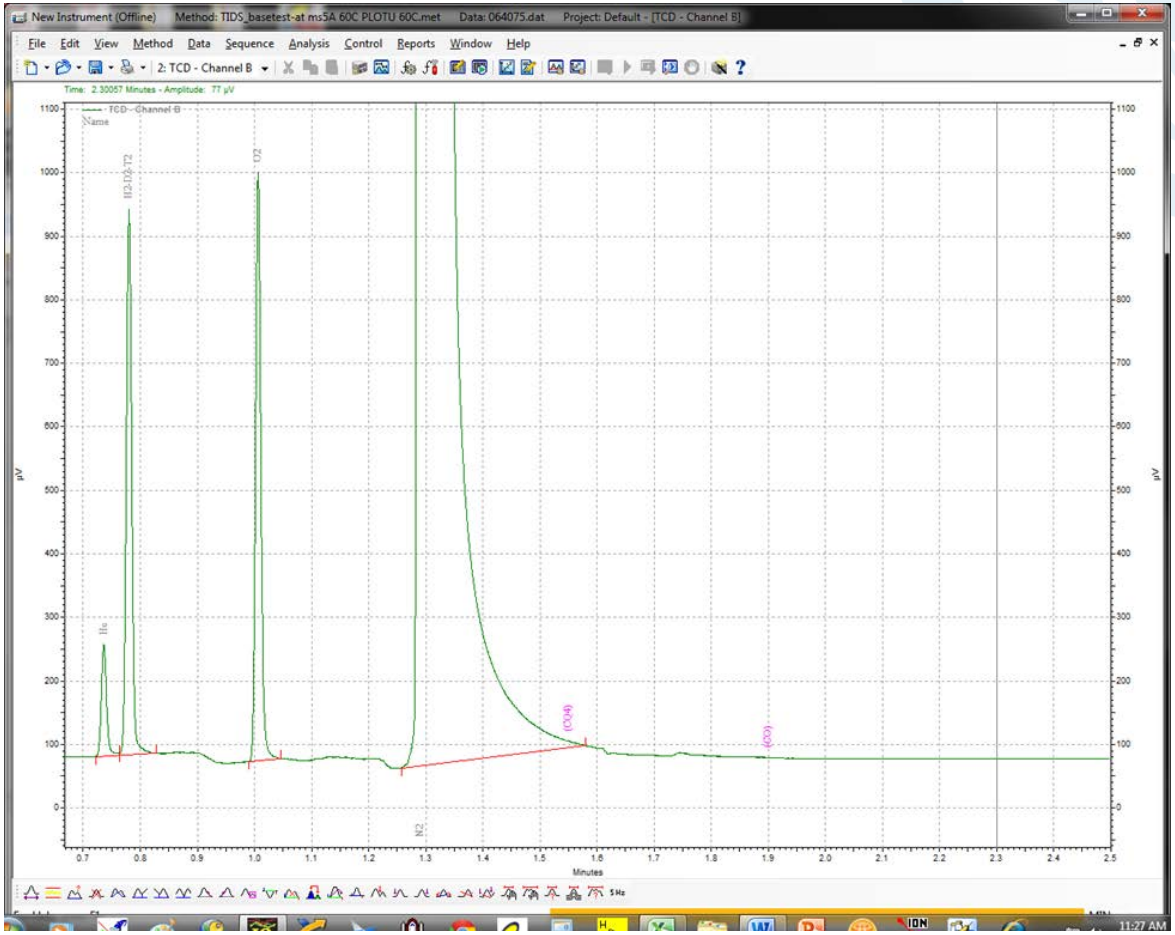
Sample Gas Chromatogram



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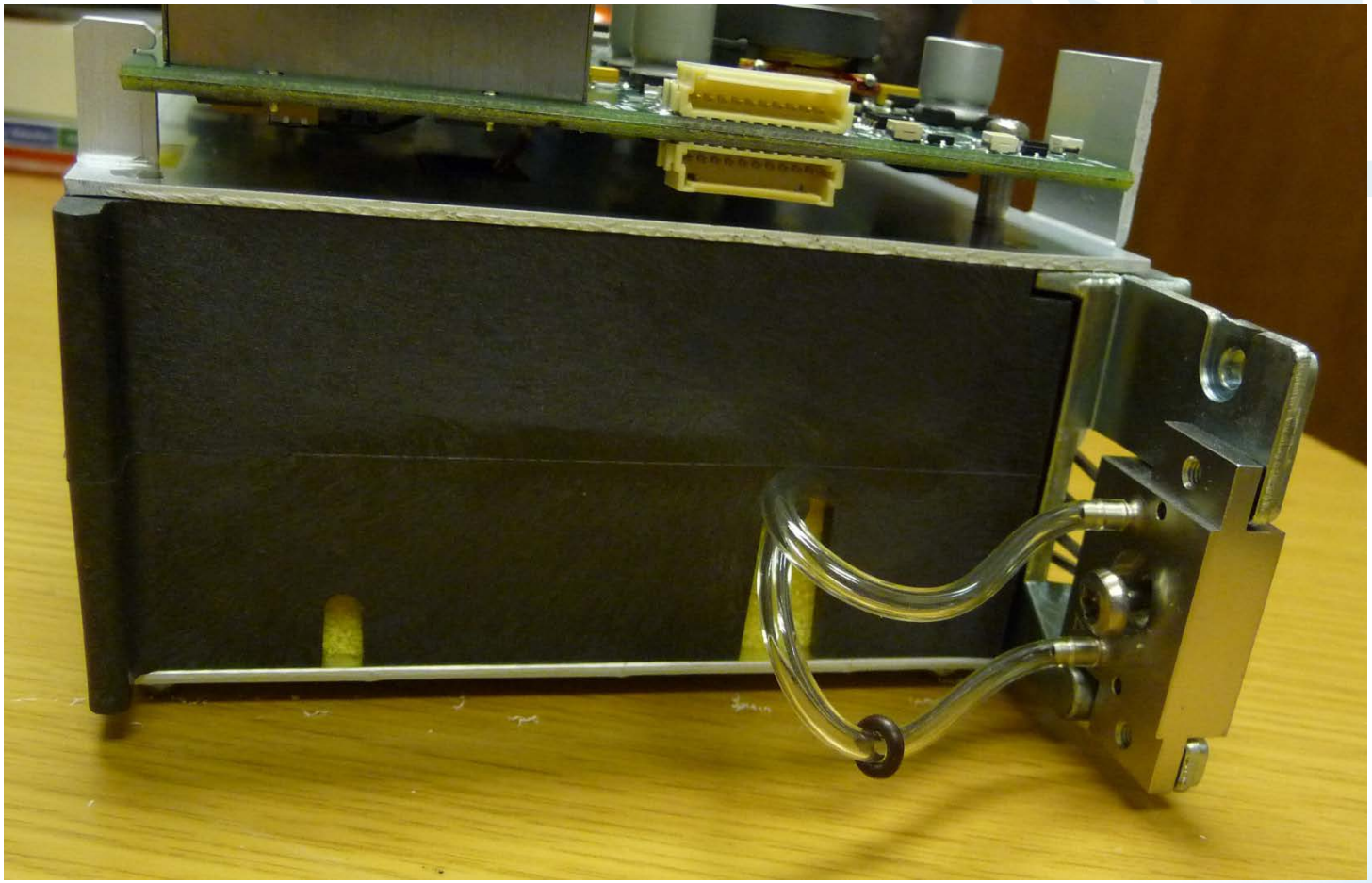
Column and Reference Exhaust Lines



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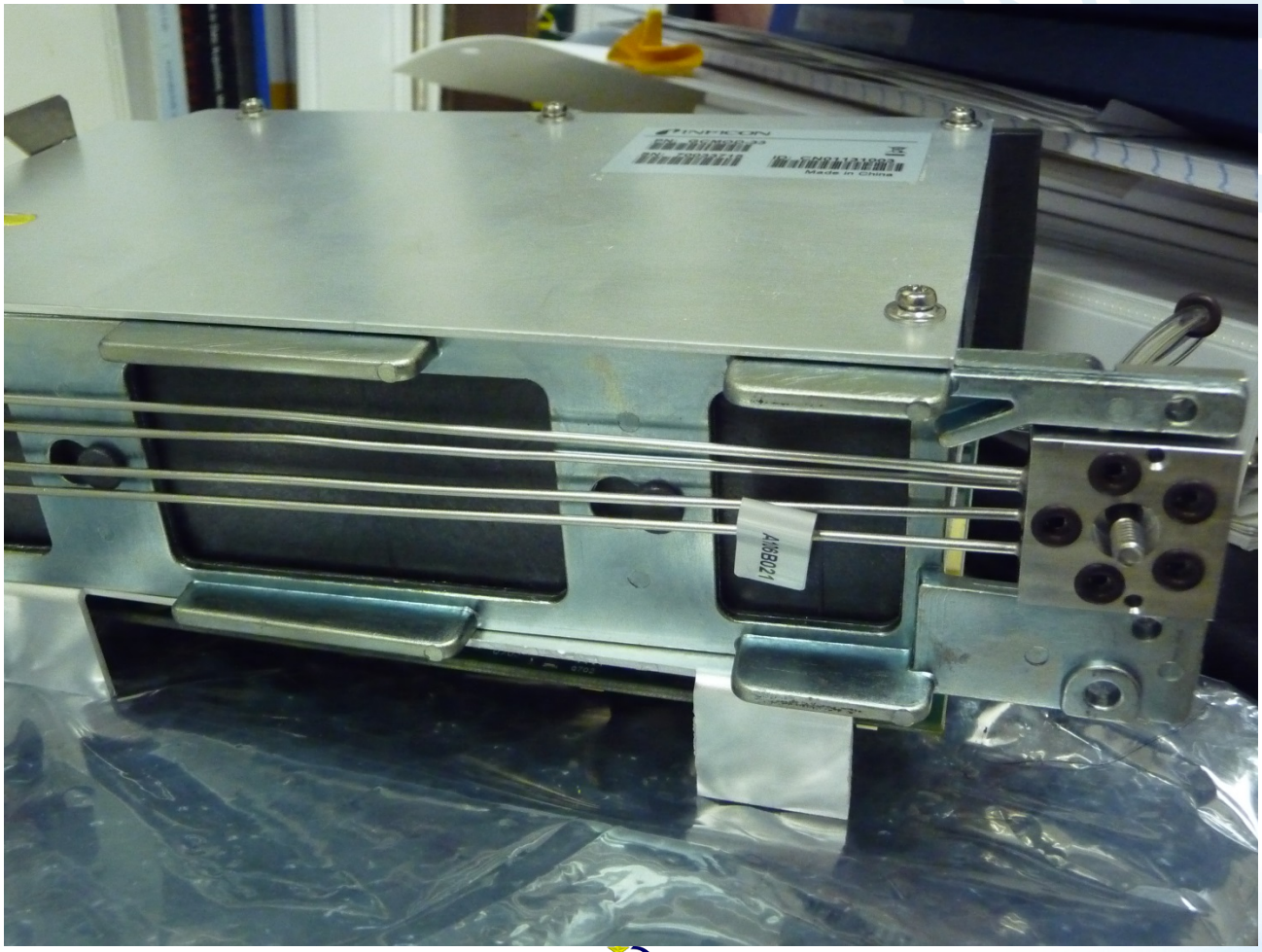
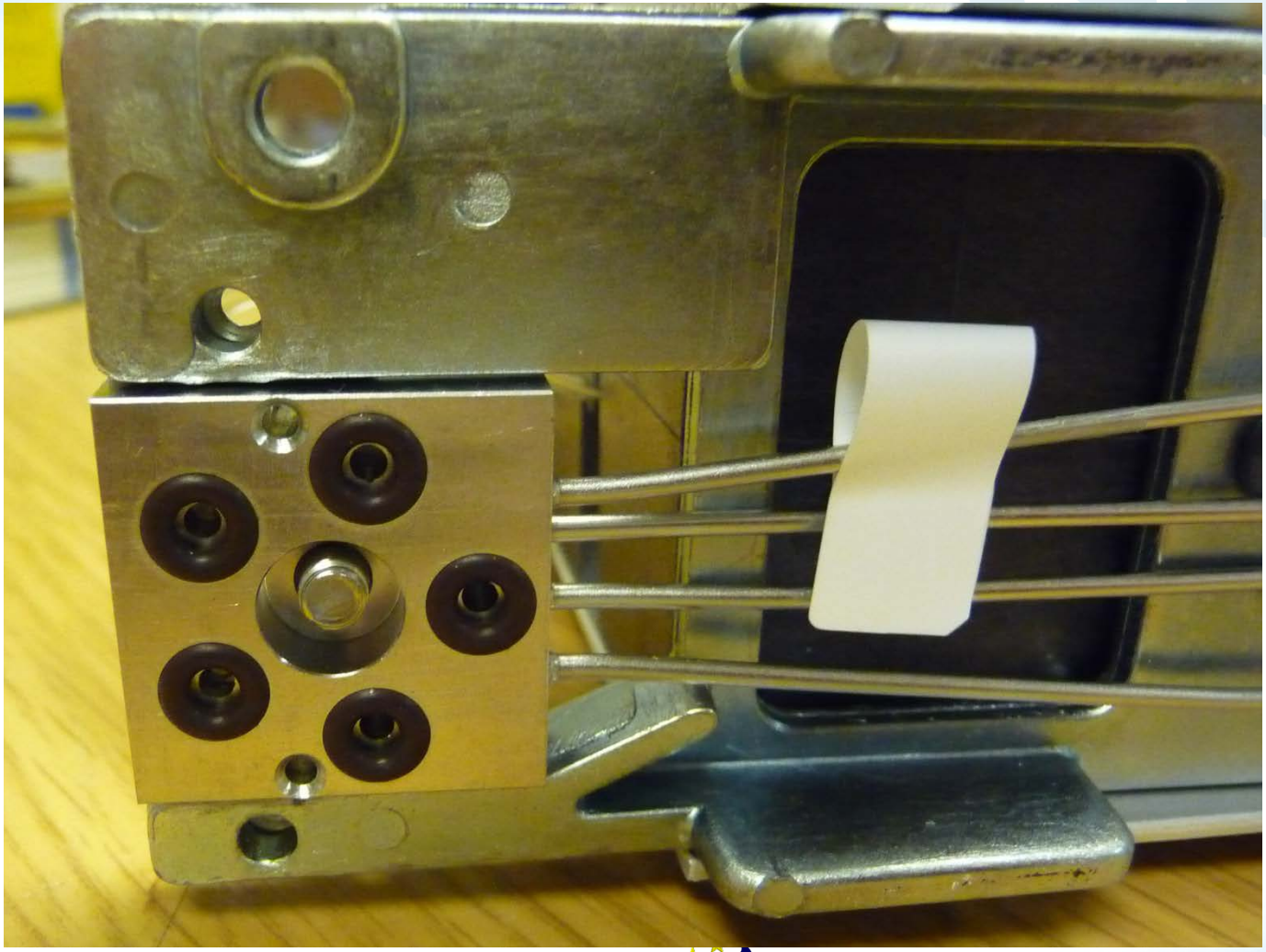
Gas Lines - Jumper



O-Ring Seals



Summary

- **TIDS provides a reliable platform for conducting analyses of impurities**
- **Analytical instruments provide complementary data to current technologies**
- **Sampling needs to be improved**
- **Calibration with other gas species are needed.**

Team Members/Contributors

- **SME's**
  - Rob Lascola
  - Bill Spencer
- **MTF**
  - Steve Murphy
  - George Thomas
- **Tritium**
  - Todd Woodsmall
  - Trey Williamson
  - Jared Clark
  - Louis Boone

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