Rotating Field Mass Spectrometer (**RFMS)**

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- The **RFMS** is placed inside a Portable Sensor packaged engineered by JPL. We have fabricated a brass board and measured both gas and liquid samples using appropriate gas or liquid ionizers.

- A portable rollerboard version has been designed and constructed to test and validate the **RFMS** in measurement of industrial chemicals and chemical threats (weapons) in air, ocean or fresh water environments.

- The task is to fabricate, test and validate the JPL developed **RFMS** and to place a prototype instrument in the “field”.

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Project Goals

- Develop low-power water ms system (<10 watts peak power)
- Attempt direct injection of liquids to include dissolved ions and compounds as well as dissolved gases and volatile compounds (via MEMS)
- Design ms system to go as deep as possible, (with full ocean depth as goal)
- Explore use of capillary electrophoresis (CE) to separate large organics, etc.
- Capitalize on unique features of RFMS
  - Rugged, compact design
  - Modest vacuum (10-100 mT) operating conditions
  - Low-power RF generation
  - High mass range (1 - >100,000 amu) with soft ionization techniques

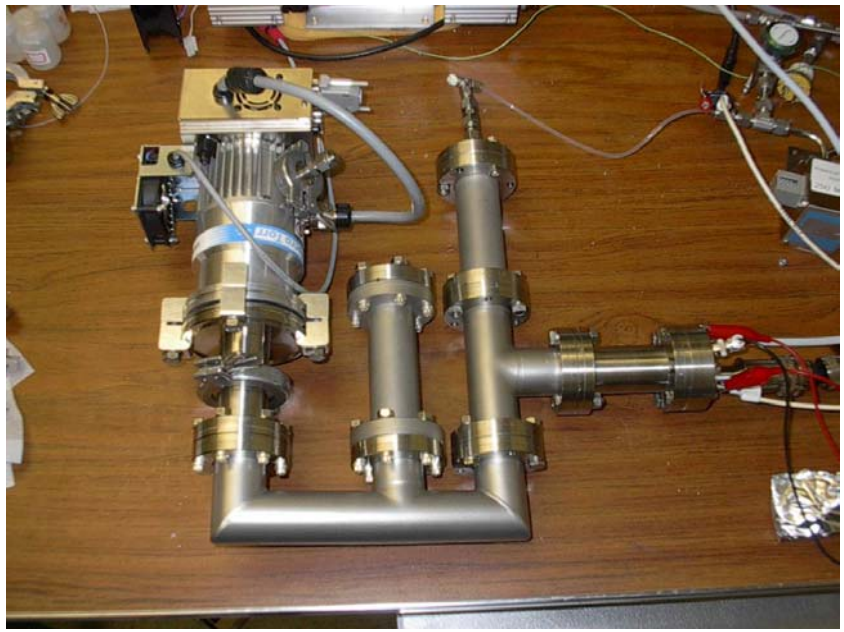
Design & Build of Mass Spectrometer

- Objective
  - Convert a laboratory instrument to a field instrument
  - Provide portability, shock and moisture resistance
  - Provide easy of sample introduction for fluid and / or gas sampling
  - Provide a simple, transparent and durable user interface
- Approach
  - Use as many off-the-shelf components as possible
  - Use manufacturing methods, such NC sheet metal forming, which are affordable.
  - Select components consistent with a conversion to 24VDC operation

Development and characterization of the mass spectrometer hardware—Dr. Steven Smith, JPL

The mass spectrometer system has two "halves". Both halves employ a separate Rotating Field Mass Spectrometer (RFMS). The water based nanospray ionizer is one half, or "leg", and the gas/air based sampler head is the other half. Gas sampling performed using using an electron filament ionizer. We have measured air and xenon gas with the gas ionizer, and MEK with the nanospray.

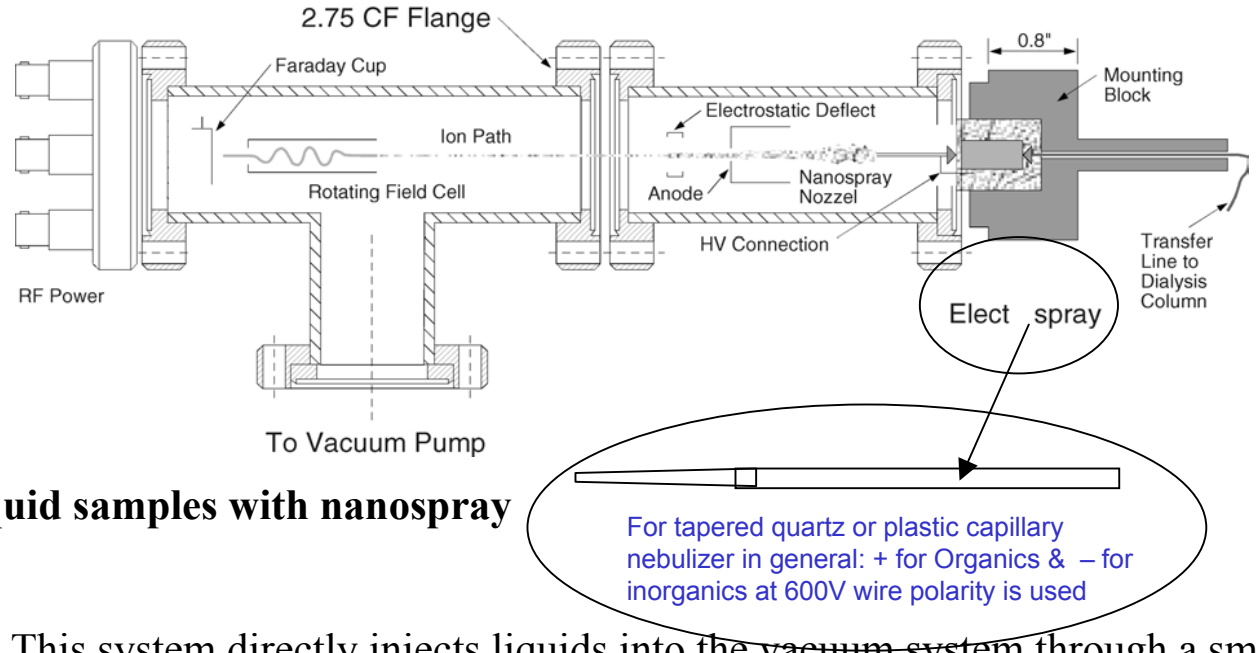
Shown below is the RFMS Mass spectrometer hardware breadboard. The pump is on the left, the nanospray leg is on the right and the filament ionizer was connected to the center leg.



JPL Technology

- Nanospray ionization
  - Developed by Dr. Steven Smith, JPL.
  - Similar to industry standard electrospray.
  - Liquid sample injected through nozzle with conductor inside directly into vacuum.
    - Voltage on wire establishes kinetic energy of ion.
    - As in regular electrospray, charge is transferred solvent carrier to dissolved solutes as the solvent flash evaporates.
  - Advantages
    - Soft—does not fragment sample.
    - Operates at moderate vacuum, 0.1-1 Torr and below.
    - Compared to electrospray.
  - Disadvantages
    - Requires ethanol as antifreeze.

Nanospray Development



Liquid samples with nanospray

- This system directly injects liquids into the vacuum system through a small nozzle with a .001 inch hole. A thin stainless wire held at 600 volts charges the solvent (water and MEK in this case), and directs a ionized flow into the mass spectrometer.

- Research grade MEK (methyl ethyl ketone) was four different concentrations ranging from 1000 to 1 ppm, dilutions with water. Spectra were obtained from the nanospray ionizer and compared with commercial NIST data taken with filament ionization.

- No sample preparation is needed, nor any surfaces need to be heated.

Liquid Samples Tested with Nanospray

Sample Concentrations range from 1000 ppm to less than 1 ppm. The in-vacuum nanospray requires no sample preparation. Direct injection is performed.

1. TCE
2. MEK We have for this task measured the following liquid samples

The previous Navy Phase I and Univ. Hawaii work included samples taken with:

1. Commercial bug spray, containing malethion
2. Large organic compounds including Lysozyme, Angiotensin, DNA PCR byproducts.
3. Important inorganic chemistries can be analyzed, such as $FeCl_2$, KNO_3, since a gas phase is not utilized.

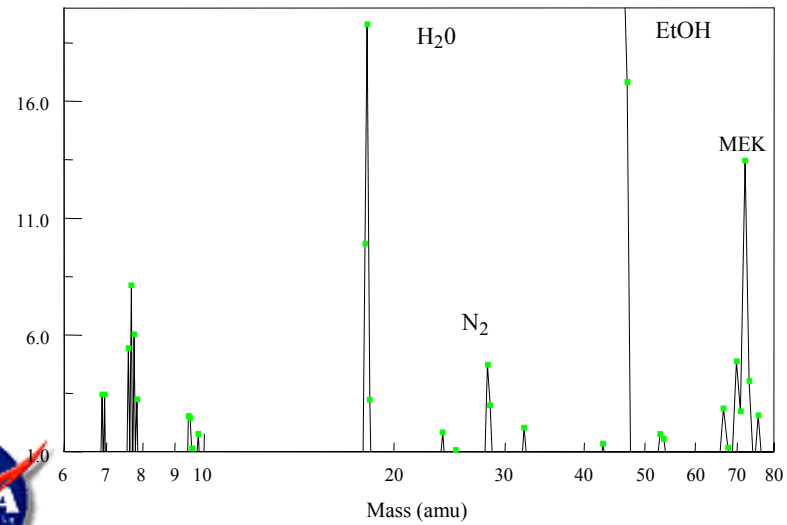
MEK Sample

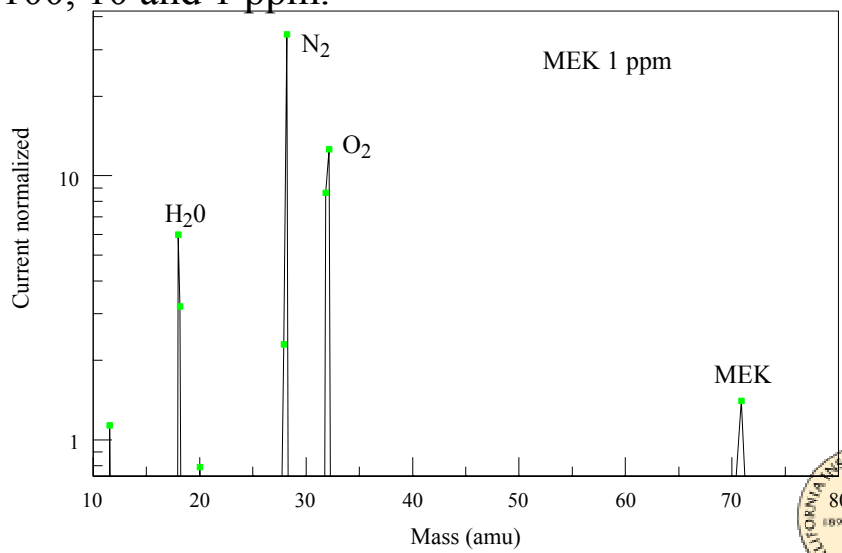
MEK is listed as a analog for certain chemical threat agents.

Molecular . Formula: C_4H_8O
Molecular Weight: 72.11

Other Names: Butan-2-one; Butanone; Ethyl methyl ketone; Ketone, methyl ethyl; Methyl ethyl ketone; MEK; C2H5COCH3; Aethylmethylketon; 3- Butanone; Butanone 2; Ethylmethylketon; Ketone, ethyl methyl; Meetco; Methyl acetone; Metiletilchetone; Metyloetyloketon; Rcra waste number U159.

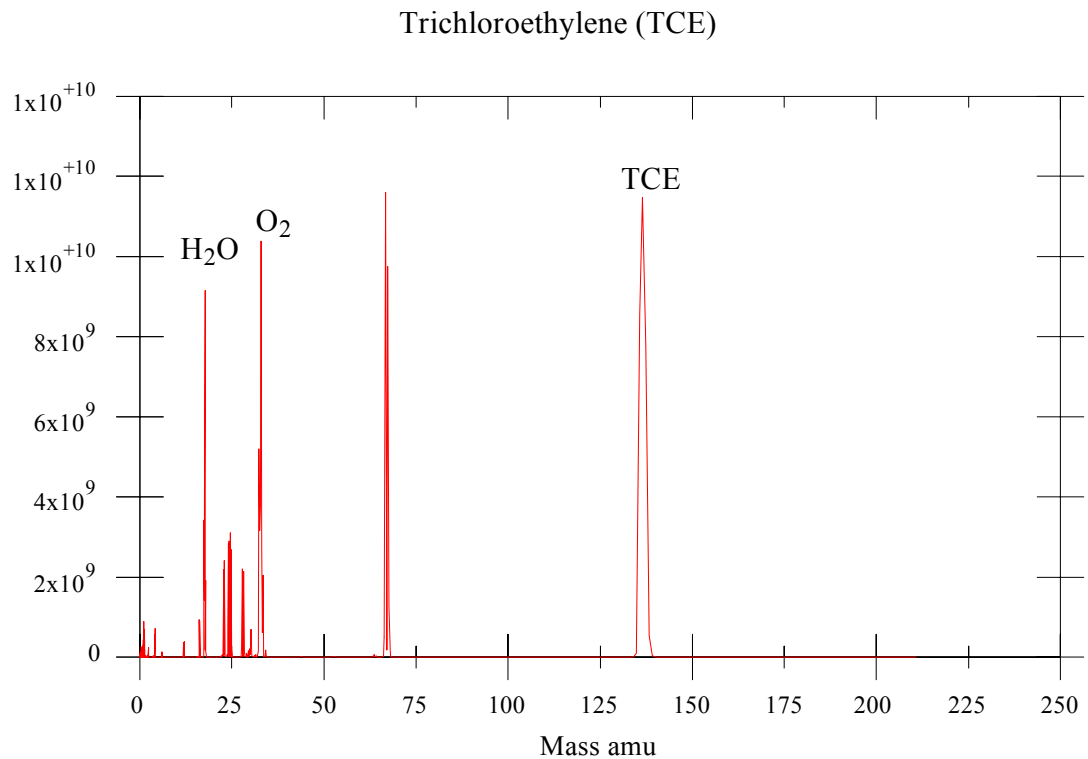
MEK was measured at concentrations of 1000, 100, 10 and 1 ppm.





TCE Sample

Shown below is trichloroethylene (chemical formula C_2HCl_3, mass 131.4).



Mass spectrum of reagent grade trichloroethylene The peak means 66 amu is likely doubly charged.

Gas Sampling with Electron Filament

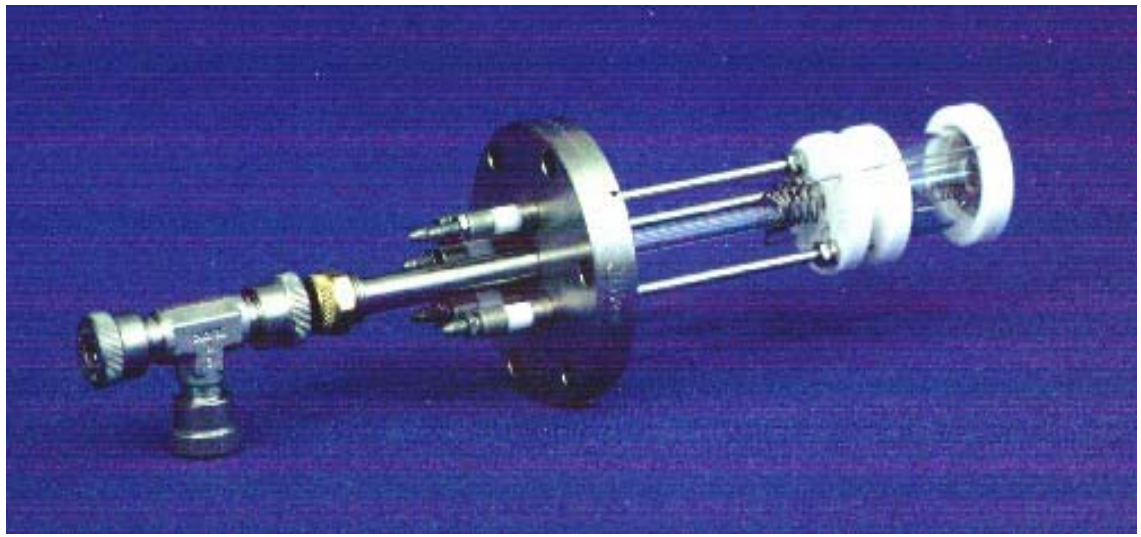
Original plan was to use field ionizing membrane. This is still the plan, but to expedite the gas sampling efforts. We simply adapted commercial ion, known as a Colutron, to do some gas sample runs.

The quartz tube is heated with a hot tungsten filament. Sample gases are introduced through a quartz tube

Ion Source Assembly

The energy spread of ion beams extracted is 0.11 eV. Extraction and ionization efficiencies range from a few percent to 30 percent.

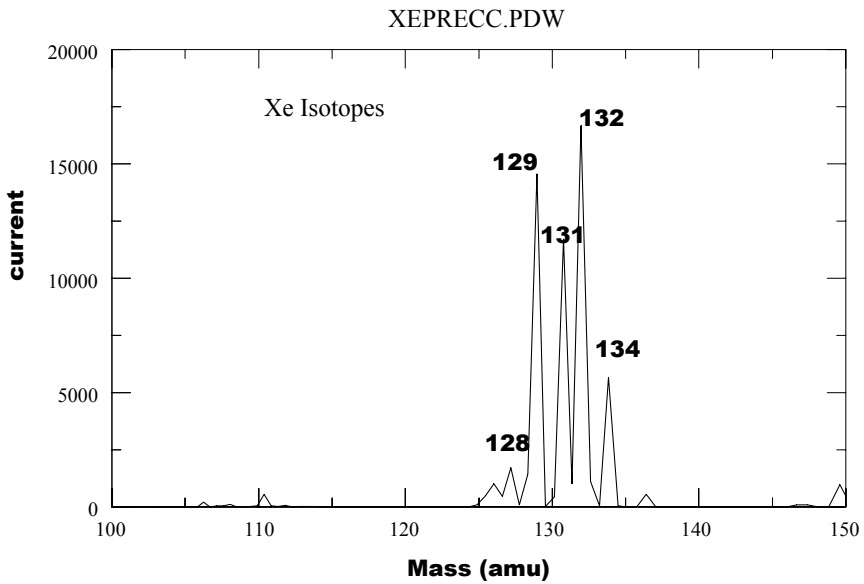
The Lens System can extract and focus beams over an energy range from 1eV to 10 keV. It consists of an extraction lens in combination with an einzel lens



Xenon Gas Tests

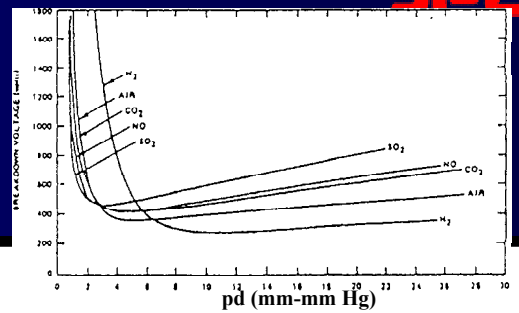
Xenon has a number of isotopes, which makes it ideal for testing mass spectrometers. Xenon has the following isotopes with natural abundance. Shown below are the known isotopes and a spectra obtained with the hot filament ionizer.

Isotope Mass	% Abundance
Xe 124	0.10%
Xe 126	0.09%
Xe 128	1.92%
Xe 129	26.44%
Xe 130	4.08%
Xe 131	21.18%
Xe 132	26.89%
Xe134	10.44%
Xe 136	8.87%



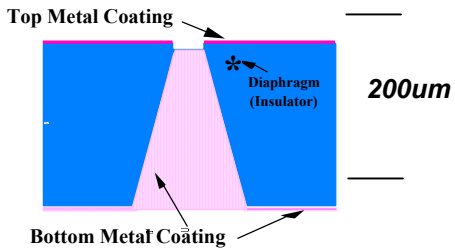
This tests shows that the ionizer and rotating field mass spectrometer are working. The qualitative features of the different Xe isotope mass peaks are seen. The actual spectrum was taken with a complete mass range between H_2 (2 amu) and 10000 amu, but only a detailed close-up of the the Xenon spectral range is shown for clarity. Mass resolution is dependent on the selected RF frequency steps, which were sufficient for this run, could be made even finer.

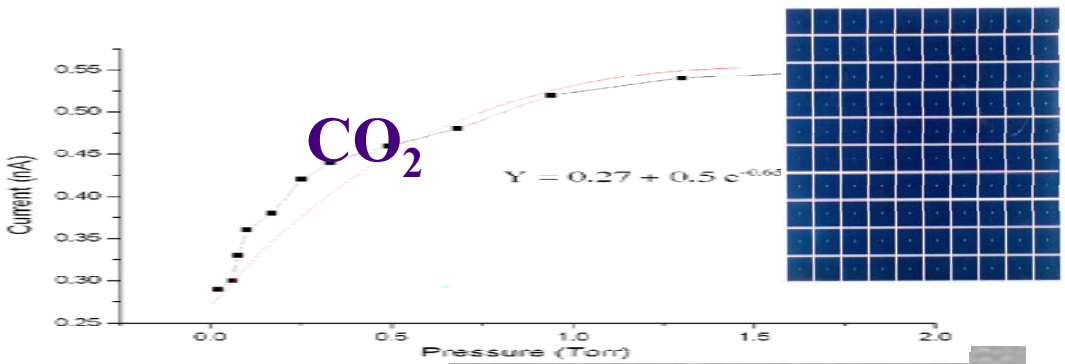
'Soft' Ionization Membrane

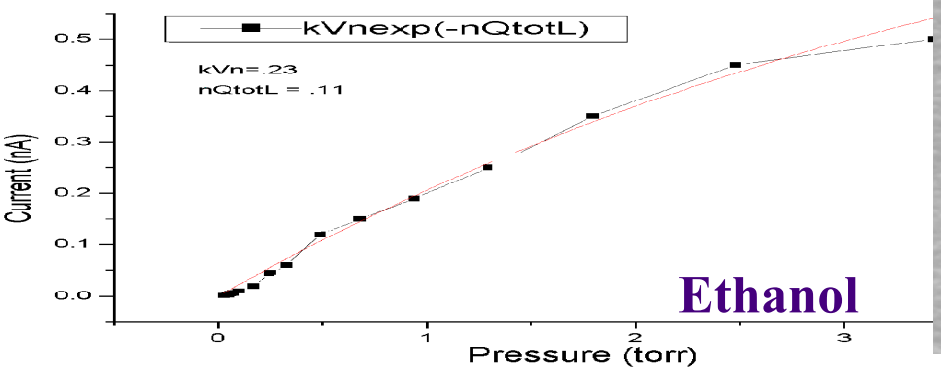


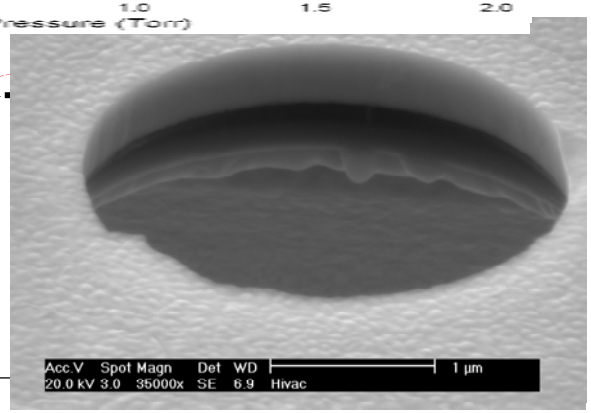
Paschen Curves for Various Gases

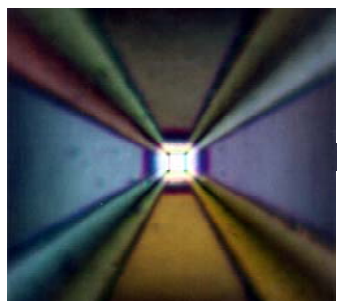
A miniature ionization device is best realized by micromachining small holes through a thin (sub-micron) membrane that has metal electrodes on each side of it. Electric field strengths in the megavolts/meter are, at sub-micron electrode separation, generated for each volt of potential difference between the metal electrodes. With electric field strengths in the tens to hundreds of MV/m any gas passing through the perforations and thus between the electrodes is totally ionized. Sub-micron thick membranes of moderate area are too fragile to sustain any pressure difference across them or to survive minor mechanical shock. A 0.1 mm thick substrate backing, back etched through to membrane, resolves these problems. The photographs present a 10x10 matrix of anisotropically etched pits, a single gold plated pit, gold covered membrane at bottom of pit, and FIB eroded hole (~2µm dia.) through 300nm Nitride membrane gold coatings (top and bottom).





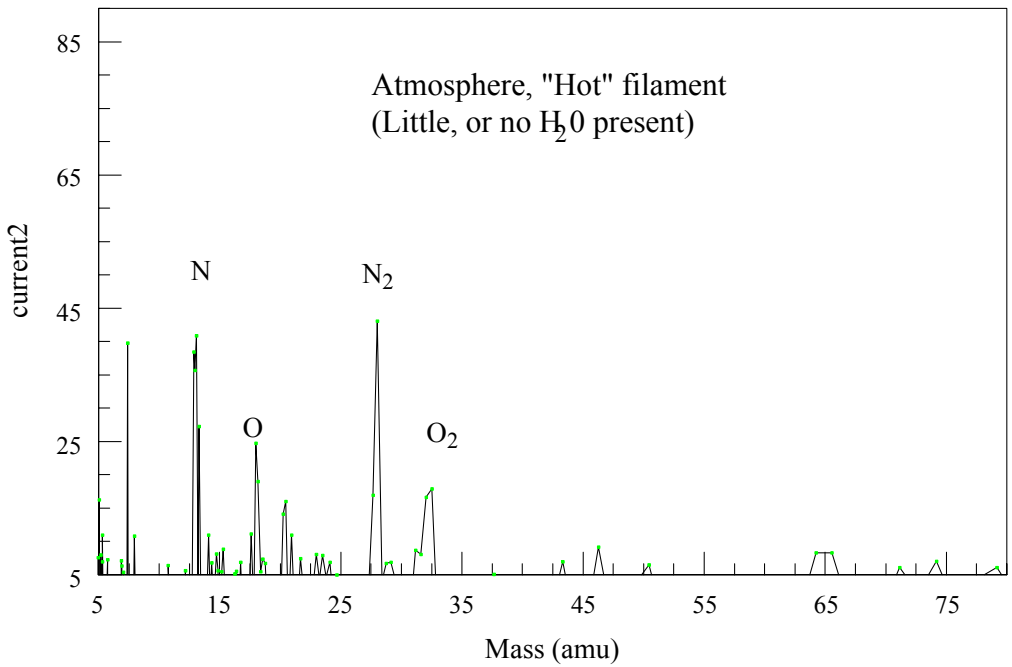






Ambient Air Sampling

We also simply measured dry ambient laboratory room air , again with the hot filament ionizer.



Explosive Analog Tests

-- We have obtained an assortment of explosive analog materials. These low concentration samples manufactured by XM division, Van Aken International, are commonly used at airports in the testing and training agents for dogs and low sensitivity pass through devices such as ion mobility detectors.

-- These analogs include chemically inert forms of RDX, TNT, PETN, etc. We plan to test our device down to about 0.1 ppm level with the current detection schemes. Further sensitivity could be achievable using a CEM ion detector that can individual ions at ppb levels

These materials are sold in a petroleum suspension. Simply washing the gel with an alcohol rinse and straight injection of the slurry into the vacuum nanospray should suffice for sampling preparation.

Summary of DNA Samples w/Nanospray Technology

Mass Analyzied to Date

Sample Types: These have included 10^{-4} to 10^{-10} molar lysozyme, angiotensin, adenosine, and most recently, large DNA fragments and 10 micromolar solutions of oligonucleotides etc. Working with samples obtained from the CITY of HOPE Medical Center, the following DNA samples have been injected into the device.

Name	#	length (mer)	MW
P21E3AS	2	20	6173
60P21E3	6	60	18283
F-N1C80	7	80	24253 + FAM (600) total = 24853
GMS2S2	8	102	31257

High Throughput Genotyping, Microbial and Biological Genotyping.

Some samples to be analyzed would contain mixture of 4-50 polynucleotides closely grouped in size from 20-100 nucleotides (6600-33000 Dalton) and would require 1 nucleotide resolution. Such analyses have been done with large state of the art MALDI-TOF instruments costing $200,000. This compact **RFMS** systems could be built and would be ideal in this application since the number of components in the mixture can typically be pre-identified, the mass values for the components of interest are known, and there is no requirement for MS/MS analysis. The potential develop a low cost system suitable for clinical or research applications.

Inexpensive RFMS units operating in parallel would allow more rapid (from hours to minutes or seconds)

Additional Sample Tests

- **Toxic Industrial Chemicals (TIC) Capability**
- **Listed are some of the Chemical agents we have obtained and are being measured with the RFMS sensor.**

Dimethyl Methyl Phosphonate – DMMP ($C_3H_9O_3P$)	1ppm – 2ppm	*Exploratory Product development in progress. This compound may prove to need to be diluted in a volatile solvent in order to manufacture a viable gas mixture, due to the low vapor pressure of DMMP.
Phosgene (Cl_2CO)	10ppm - 1%	*Exploratory Product
Phosphine (PH_3)	10ppm - 2%	
Formaldehyde (HCHO)	0.1ppm - 30ppm	

Future Tasks July-November, 2005

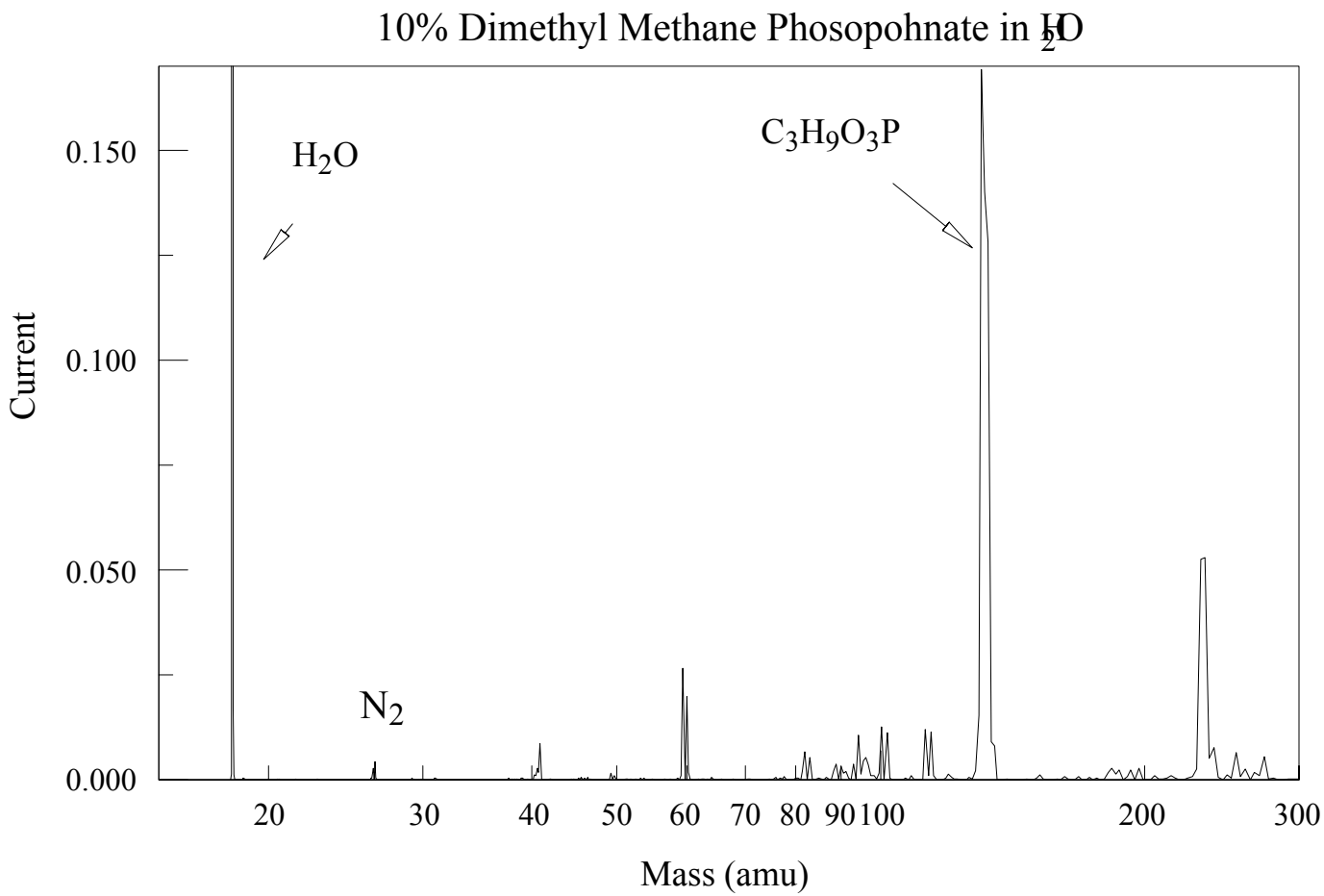
Continue brass board measurements of other chemical threat agents including

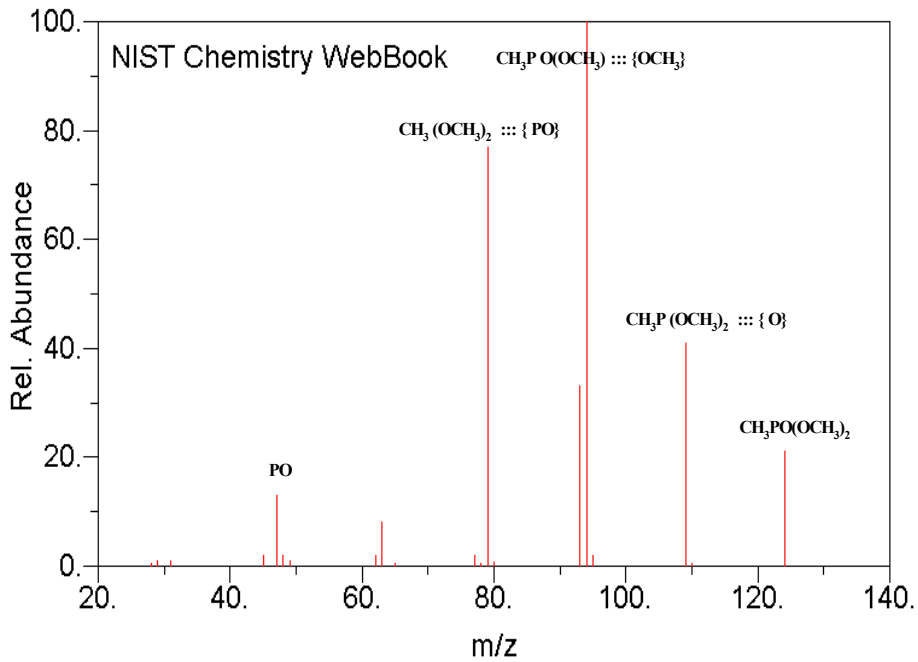
1. DMMP gas measurements at 0.5% ppm concentration, with turbopump system operating for optimum vacuum and sensitivity conditions.
2. Phosgene at 5ppm concentration.
3 Phosphine at 1 ppm concentration.

Complete electronics and plumbing details for roller case package

Calibrate flow inlet and exhaust rates for gas sampling and air sampling stages

DMMP Solution (Sarin toxin analog) demonstrates soft ionization vs NIST E bombardment





Right: – NIST mass spectrum of pure DMMP in high vacuum.

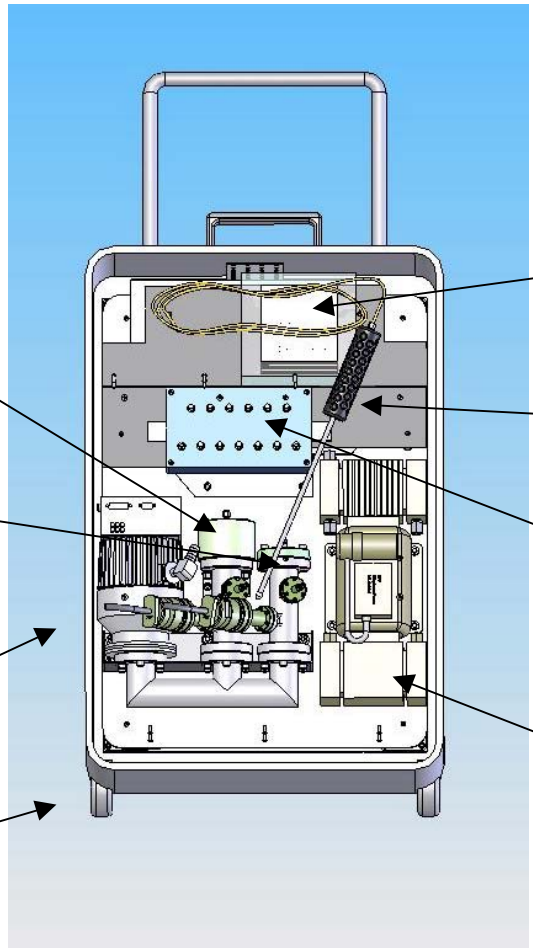
Payoff/Benefit to A/C T&E Health and Status Monitoring

The proposed gas analyzer will

- Provide real-time analysis of gas emissions (CO_2, O_2, CO, NO, NOx, total Hydrocarbons) from gas turbine engines

- Result in improved performance and reduced atmospheric emissions through active adjustment of fuel/air ratio

Package Configuration, as designed



gas channel

fluid channel

turbo pump

wheels

PC card cage

gas sample acquisition wand

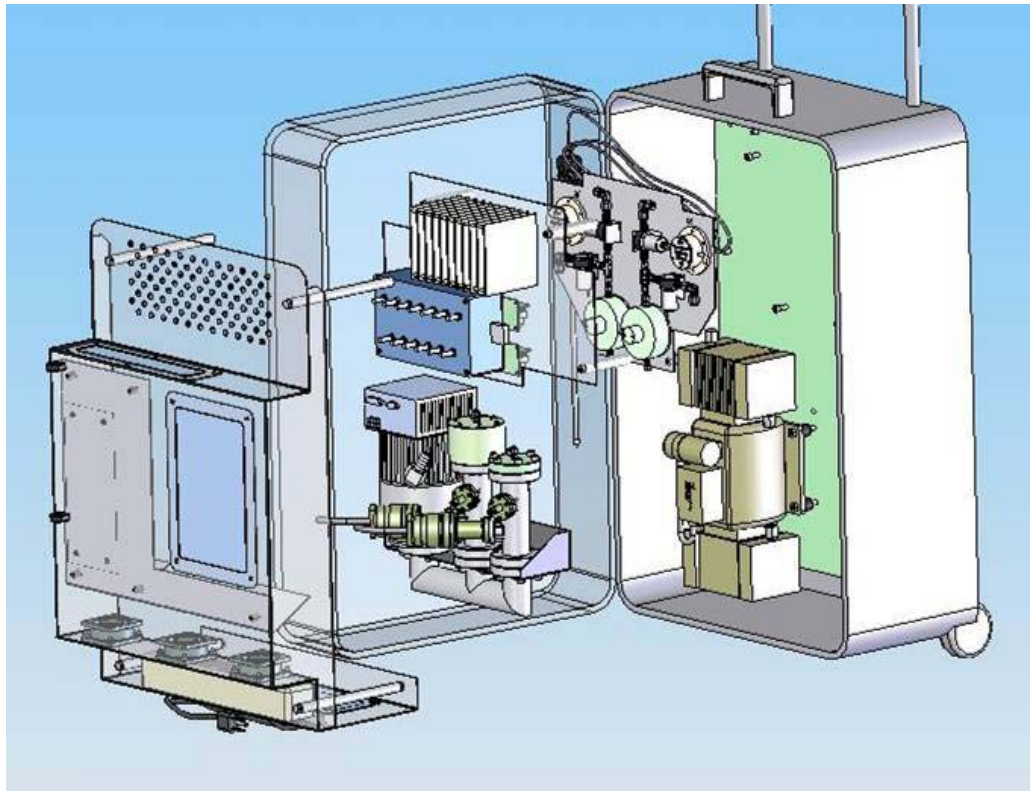
potentiometer (biasing) box

rough pump

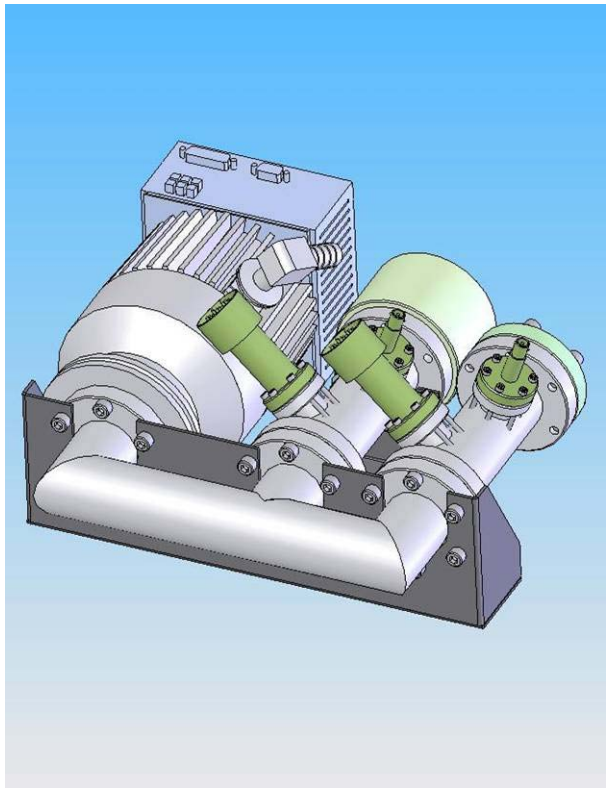
Package Configuration, as built

Detailed Design of Internal Structure

- Modular construction, with vacuum, electronic and fluid systems separated into clusters
- Stack assembly of modules, with four bolt access to any subassembly.
- Removal of four front panel bolts



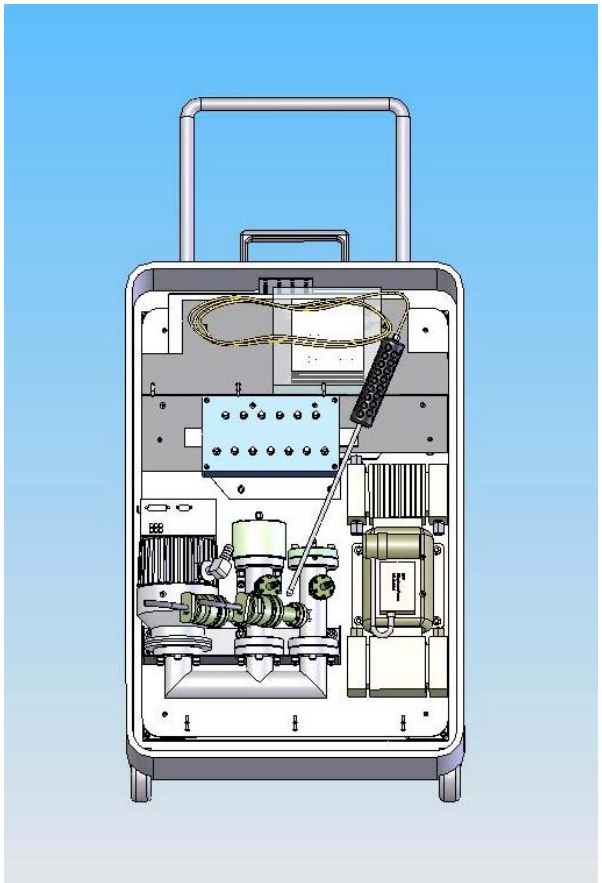
Turbo Pump & Manifold Assembly



As designed

Bench test version

As Designed & As Built

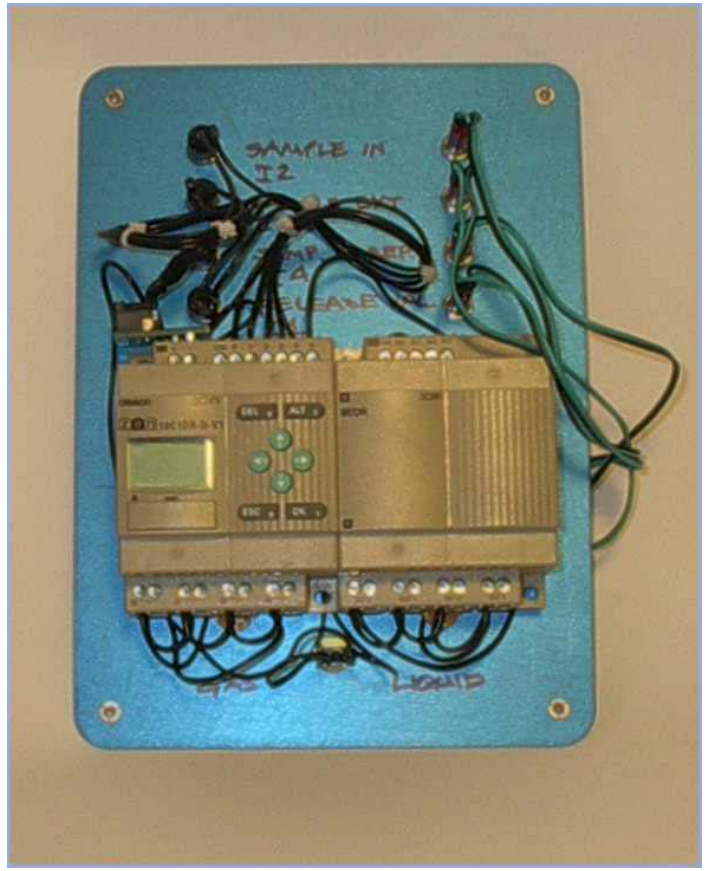


On-Going Integration Tasks

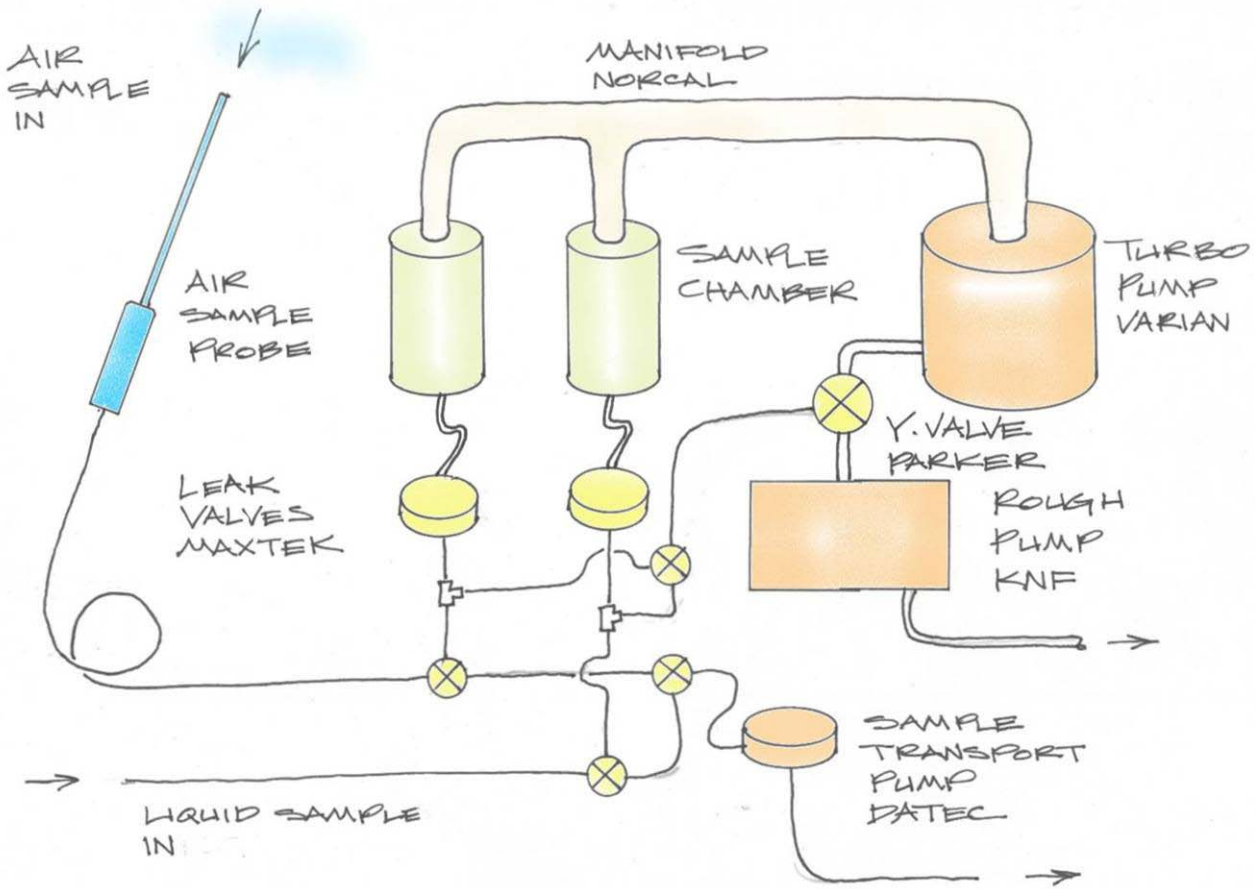
- Achieved
  - Fabrication and fit check of final internal structural supports for wet (sample hydraulic) fittings such as pumps and valves
  - Fabrication of internal structural plates for dry, PC-104 card stack, valve drivers, pump drivers, and hydraulic sequencer controller (HSC)
  - Wiring of HSC module
  - Programming of HSC, test in simulation, and test at sub-system level

Hydraulic Sequencing Controller

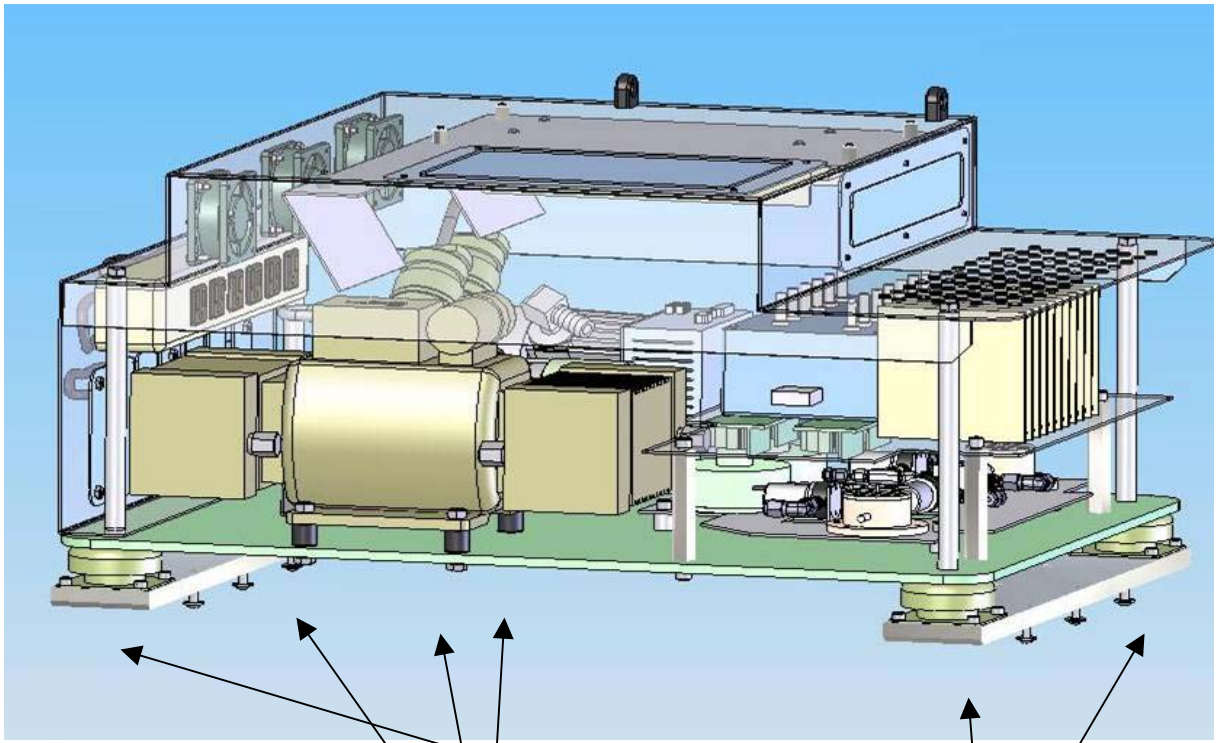
- Omron Controller
  - 6 inputs, 24VDC
  - 8 relay outputs, any voltage
- Five switch interface
  - Gas / fluid selection
  - Sample insertion
  - Sample removal
  - Stand-by mode



Vacuum & Sample Flow Block Diagram



Vibration Isolation Systems



G10 Fiberglass Subframe with good damping characteristics

Elastomeric isolation mounts for rough pump

Shock mounts

Microassembled Rotating Field Micro Gas Analyzer for Chemical Warfare Agents

Zyvex Corporation, Richardson, Texas

Program Objectives

- High sensitivity (ppt) micro gas analyzer for CWAs
- Compact (< 2 cm^3 excluding power source)
- Rapid analysis time (<4 sec)
- Low power consumption (< 1 J per analysis)
- Immunity to interferents

Team Members

Jet Propulsion Laboratory
- RFMS design
- System testing
- CWA simulants

Naval Surface Warfare Center (Dahlgren)
- CWA expertise
- System testing

Honeywell International
- MEMS Process development
- MEMS fabrication

Zyvex (Lead)
- MEMS design
- Microsystem assembly
- System integration
- Testing

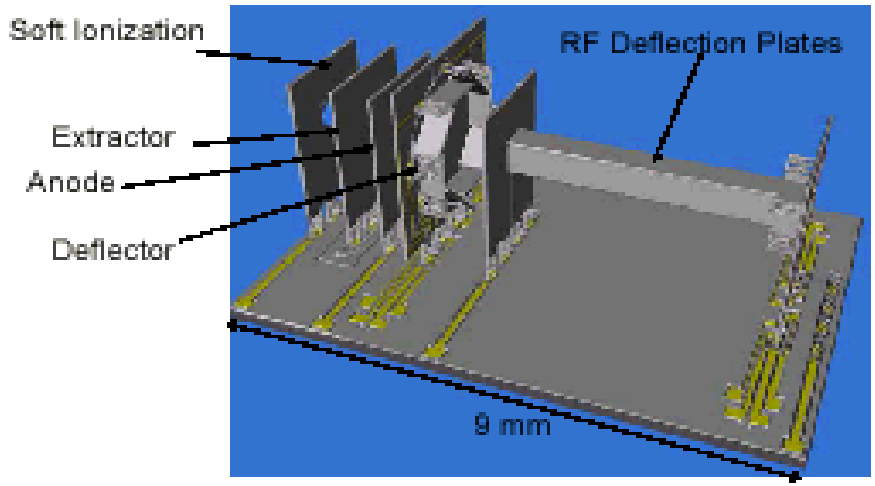
University of California Berkeley
- Control electronics
- Detection electronics
- Testing

Center for Ocean Technology USF
- MS design
- Testing

Center for Automation Technologies RPI
- Assembly automation
- Assembly sequence
- Design for manufacturing

Expected Impact

- Survival in the battlefield (information based combat)
- Perimeter protection and situational awareness
- Homeland security (explosives, bioagent detection)
- Spin-off products for numerous commercial applications



Solid Model of Proposed Microassembled Mass Spectrometer.

Key Innovations / Technical Approach

- High sensitivity Rotating Field Mass Spectrometer
- Soft ionization technology for low power consumption
- Revolutionary precision microassembly
- MEMS components designed for assembly
- Unique high sensitivity control and detection electronics
- Automated assembly for manufacturing