

Air Monitoring for Hazardous Gas Detection

C Richard Arkin¹, Timothy P. Griffin², Fredrick W. Adams², Guy Naylor¹, William D. Haskell¹, David P. Floyd¹, Charles H. Curley¹, Duke W. Follistein²

¹Hazardous Gas Detection Lab, ASRC Aerospace, ASRC-14, Kennedy Space Center, FL 32899

²NASA, YA-D7-E1, Kennedy Space Center, FL 32899

The Hazardous Gas Detection Lab (HGDL) at Kennedy Space Center is involved in the design and development of instrumentation that can detect and quantify various hazardous gases [1-7]. Traditionally these systems are designed for leak detection of the cryogenic gases used for the propulsion of the Shuttle and other vehicles. Mass spectrometers are the basis of these systems, which provide excellent quantitation, sensitivity, selectivity, response times and detection limits. Table 1 lists common gases monitored for aerospace applications. The first five gases, hydrogen, helium, nitrogen, oxygen, and argon are historically the focus of the HGDL.

Hydrogen	Cryogenic fuel, leak may cause hazardous condition due to flammability
Helium	Common purge gas, and leak test gas
Nitrogen	Common purge gas
Oxygen	Cryogenic fuel, leak may cause hazardous condition due to reactivity
Argon	Used to identify oxygen source as fuel or air leak
Ammonia	Common refrigerant
Carbon Dioxide	Leak test gas (larger leaks produce visible spray)
Hydrazines	Hypergolic fuels
N ₂ O ₅	Hypergolic oxidizer

Table 1 – Common gases monitored for aerospace applications.

Mass Spectrometry

In its most basic form, a mass spectrometer operates by producing ions of the components of interest, separating these ions in an electromagnetic field, and detecting the ions. There are numerous ways to perform these three steps, giving rise to a large number of unique mass spectrometer systems. The mass spectrometer is operated under vacuum, one of the main reasons being that the ions have to travel the length of the analyzer without colliding with molecules. This high vacuum is achieved by specially designed vacuum pumps. All modern mass spectrometers are coupled to computer data acquisition systems. A general block diagram of a mass spectrometer system is shown in Figure 1.

Mass spectrometry is ideally suited for qualitative as well as quantitative analysis. The qualitative ability (determine what is present) stems from the uniqueness of the spectra for specific compounds. The pattern of the mass spectra for a specific compound is used to determine what compounds are present. Mass spectrometers have the potential to differentiate between any two chemical species. This specificity also includes the ability to measure helium, which few instruments can accomplish. Essentially no other instrument can identify and quantify helium simultaneously with other components. Mass spectrometers also provide quantitative information and typically have linear dynamic ranges of 5-orders of magnitude or more. Mass spectrometers also have rapid response times, with sub-second response and recovery times being common.

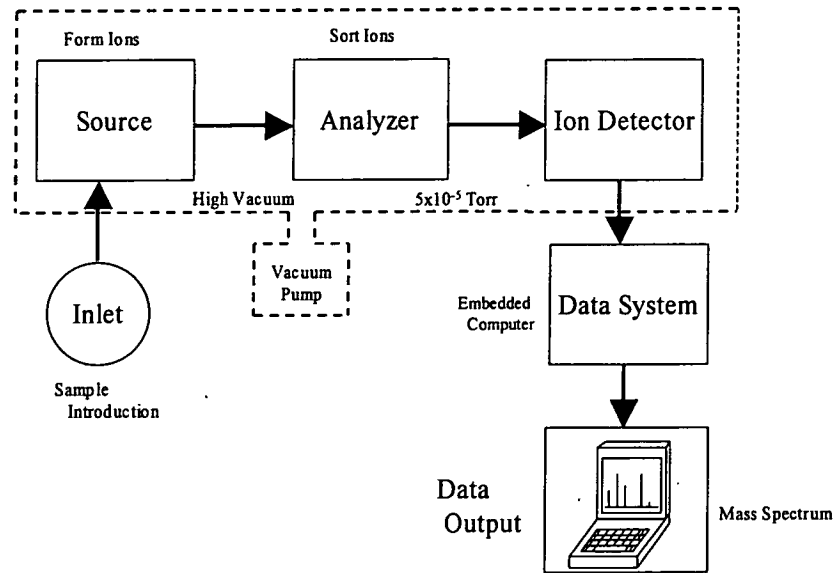


Figure 1 – A General Mass Spectrometer Block Diagram

Mass Spectrometry at Kennedy Space Center

Mass spectrometer systems have been used at KSC since the beginning of the space program. The primary reasons mass spectrometers are used are their excellent limits of detection (LOD), response time, recovery rate, accuracy, and capability to monitor and differentiate several species. A primary function of the HGDL is to develop systems that monitor the cryogenic fuels, liquid hydrogen, and liquid oxygen used for launching the Space Shuttle. A buildup of gaseous hydrogen or oxygen during fueling or launch creates a hazardous environment. For this reason, areas of potential hazard are purged with nitrogen and analyzed for hydrogen, helium, oxygen, and argon. (Hydrogen and oxygen indicate a leak of the cryogenics, argon indicates an air leak, and helium is used for leak checking prior to fueling.) Currently, a large mass spectrometer (MS) system (see Figure 2) performs this task, which has several undesirable attributes. Long transport lines (up to approximately 115 m [370 ft]) are used to deliver the sample to the analyzer which means the sample being analyzed is actually 15 to 30 s old. Among the various problems that this delay in response poses, one of the most serious is that monitoring for leaks during the last fraction of a minute prior to launch is precluded. Sequential, round-robin sampling of the lines causes additional delays. This system is also very large ($\sim 3.65 \times 10^6 \text{ cm}^3$), heavy ($\sim 770 \text{ kg}$ [1700 lbs]), and expensive ($\sim \$1\text{M}$). Last, if more sampling points were added, more transport lines would be needed, thus adding to the size and weight of the system and creating even longer delays between consecutive readings.

There is interest in replacing this stationary system with several small (about the size of a shoe box), inexpensive ($\sim \$20 \text{ k}$, US), mobile systems that provide ease of operation and maintenance and can be used for most, if not all of the needs of NASA. The new portable, rugged mass spectrometers would act as point sensors that could be placed at the sampling locations. Such a small, lightweight system would provide several advantages. First, since it is a point sensor, there is no need for long transport lines, thus eliminating the delay between sample uptake and analysis. Second, with multiple sensors, several locations can be monitored simultaneously. (The current systems use multiple sample lines with a single sensor.) Additional sampling points would not create a delay between consecutive scans. Third, small instruments tend to cost less than their larger counterparts. Fourth, in the event one system fails, several entire systems can be on the shelf available for installation as needed. Currently, when an instrument fails, that instrument is evaluated and parts are repaired or replaced. As a result, numerous parts must be stocked, qualified personnel must spend valuable time involved in repairs and the potential

exists for a costly delay in vehicle processing. And, if one system fails, the other sampling points continue to be monitored. Also, with the systems being light-weight and portable, there is the potential that these systems can remain functional during the launch, ascent, orbit, and descent.

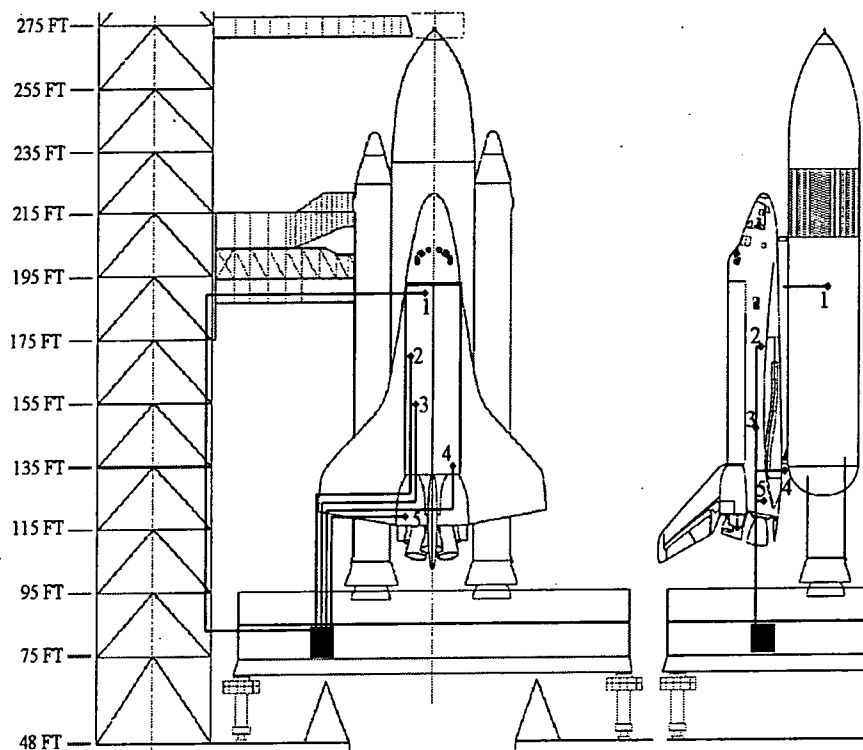


Figure 2 – Diagram of the Shuttle with the approximate location of the existing MS system (black square), the long sample lines, and the primary sampling points (numbered).

Small MS Project Description

The HGDL is developing these small MS systems for Shuttle leak detection, as well as other applications such as air quality analysis within the International Space Station (ISS), automated environmental analysis and long-term, unattended, remote sampling. In order to meet these goals, it is necessary to research, design and develop mass analyzers, sample transport systems, low and high vacuum pumps, and control circuitry that are small, lightweight, rugged, and provide rapid, accurate, and component specific information. These goals are being accomplished in three phases – component identification and evaluation, iterative prototype design and testing, and development of the final system. The project is currently in the beginning of the second phase, having completed the first design and test iteration.

Evaluation Phase of Components

A cross section of small mass spectrometer systems were evaluated, with analyzers such as linear quadrupole, quadrupole ion trap, time of flight, and sector. Under the conditions similar to those required for the Shuttle, various figures of merit were determined [12]. A variety of rough vacuum pumps were evaluated for mass flow, power, heat dissipation, weight, size and contamination production. The vacuum technologies included scroll, diaphragm and roots. A number of other components have been investigated for reduced size, weight and power usage as well as improved performance. These components include flow control devices, pressure transducers, power conversion devices, embedded computers and other electronic control components.

Iterative Design Phase – First Prototype

It was identified that an appropriate approach to develop the desired system with the resources available is to proceed in discrete steps where progress can be quantified on a regular basis, experience can be gained in a short time scale, and development and/or modification of existing prototypes allows for laboratory testing and field testing. The field testing is of vital importance, since it provides unique challenges that are difficult to anticipate based on laboratory simulations. An additional benefit of the discrete development method is that systems are available on a regular basis for application purposes. There are many benefits gained by including application efforts on a regular basis during this long-term project: (1) realistic field testing is performed, (2) the system is influenced by realistic needs of scientists and engineers, (3) application scientists obtain valuable data while valuable system design data is obtained, (4) application scientists have access to a system that they generally could not afford, (5) applications can supplement the development funding, the ultimate cost.

The first prototype system [22-23], named AVEMS as a result of the scientific application it was initially used, was intended as a proof-of-concept unit that served to better understand the aspects involved in integrating all of the components evaluated in the first phase of this project. The scientific application that supplemented the field testing was to evaluate and quantify the gaseous emissions from volcanoes in Costa Rica. AVEMS was designed to function aboard aircraft such as the WB-57 high-altitude (>65,000 ft) research plane. Flight-tested in the fall of 2002, this system is (to the authors' knowledge) the first mass spectrometer based instrument capable of autonomous, quantitative gas detection aboard an airplane.

During the development of AVEMS there were a number of ground tests including altitude simulations, vibrational and stress analysis, along with stress calculations. Typical characterization of analytical instrumentation such as quantitative accuracy, detection limits, and response time was also performed. Two environmental chambers were used to simulate the effects of altitude. The first chamber, capable of controlling temperature between -34 °C (-30 °F) to 85 °C (185 °F) and relative humidity between 5 – 95%, was used for thermal testing. This system was used first since the operator had access to the equipment during the testing. The second chamber, capable of controlling temperature between -73 °C (-100 °F) to 121 °C (250 °F) and pressure from 1 torr to ambient, was used for more realistic altitude testing. Tests performed at varying pressures were used to determine the optimal test points required for the internal operating pressure of the gas transfer system. The temperature tests indicated that special attention should be paid to the soft goods used for the vacuum seals. In addition to these operating factors, design issues for operating over the wide ranges were fine-tuned. The design included an insulation bag to keep the heat generated close to the system, small heaters to heat specific areas of concern, and temperature sensors to monitor the actual temperature of various parts of the system. The tests also showed that in the range of 50 to 100 torr certain electronic would become damaged placing an altitude ceiling of approximately 45,000 feet. Vibration tests were performed to ensure that no harmonic of the aircraft vibration caused catastrophic damage to the system. This was important not only for the life of the system but to make sure no items would break off and damage the aircraft. Ground isolation and EMI filtering were two important aspects that varied significantly from a laboratory-based system. AVEMS was analyzed in an electromagnetic interference (EMI) test chamber. Initial tests demonstrated potential EMI problems; additional filtering allowed the system to meet accepted standards.

AVEMS was also tested for chemical figures of merit. The system was shown to quantify permanent gases as well as some common volatile organic in the low part-per-million concentration range. The system also showed acceptable values for accuracy, precision, drift and response time. However, hydrogen and carbon dioxide were difficult to measure soon after the system was powered. The primary reason for this is the presence of water. These tests are initiated within 1 hour of system start-up and as a result, there is still significant amounts of water in the vacuum and on the chamber walls.

However, the tests were performed this way (contrary to an overnight, or 24 hour, bake-out period which is common) since the system is intended for field analysis and is expected to perform analysis within an hour of power up. Investigating the rapid power-up, water issue will be an important area of focus in the future, since any practical system must have a reasonable "warm-up" time, perhaps 30 minutes. (Although 30 minutes is a long time, most laboratory mass spectrometer systems require several days to be peak operating condition.)

Following exhaustive laboratory and ground testing, AVEMS was flight tested several times. Figure 3 shows the result of the system flying over an industrial area near Houston, Texas. This data set identifies the presence of a hydrocarbon-based pollutant above an area with several petroleum refineries. The component monitored is a characteristic marker for several simple hydrocarbons, such as isooctane (gasoline). As indicated in Figure 3, the sample was acquired at an altitude of 4000 feet. Experience has indicated that the altitude monitoring ceiling (for detection of chemicals originating from ground sources) for this system is approximately 5000' above the source. AVEMS has been shown to operate reproducibly at 44,000 feet; however, chemical information from ground sources has not been obtained at that altitude.

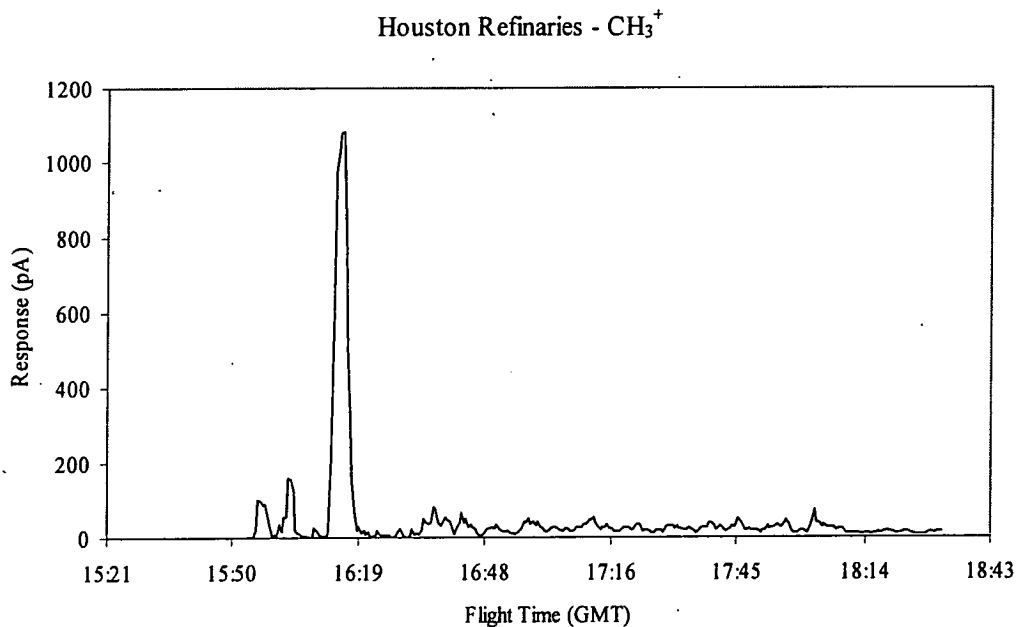


Figure 3 – Identification of Hydrocarbon Pollutant over Houston Area

Iterative Design Phase – Second Prototype

Although still in the design phase, work on the second prototype has begun. There are several goals for this system. Compared to AVEMS this system will (1) have a weight reduction of 35%, (2) have a size reduction of 25%, by volume, (3) increase the scan rate by 100%, (4) improve the average duty cycle by reducing when calibration is required and calibration duration, (5) exceed the 45,000 ft. altitude ceiling of AVEMS and (6) be 25% more power efficient. The system is expected to be completed by August 2004, with preliminary characterization complete by September 2004. Although no formal plans are complete, a number of field test applications have been identified for this unit.

Future Project Efforts

Significant progress has been made toward developing small mass spectrometers for Shuttle applications. Future efforts will generally be in two areas – the reductions of commercial (COTS) components used in the system and ruggedization of the system. The first two prototype systems rely

heavily on COTS components, primarily due to the cost and availability. However, combining COTS components is inefficient for several reasons. COTS items are self-contained causing significant, and unwanted redundancy in many areas such as power supplies, data conversion and computer communications. These redundancies result in added weight, size and power. And, often times COTS equipment create integration issues with other components. Elimination of such equipment allows for the development of an optimized system. The current technologies associated with mass spectrometers cause them to be very fragile. Since almost all such systems are used in a laboratory environment, the generally is not a problem. But since, the system under development here must withstand the rigors of space flight and similar situation, significant effort will be directed toward ruggedization.

Potential Applications

Small, rugged mass spectrometer systems have a number of potential applications for both NASA and industry. For NASA, such tasks include the monitoring of hazardous gases around the Shuttle, and other launch vehicles, the detection of toxic propellant vapors such as hydrazine derivatives, process control of a fuel production plant on Mars, and air monitoring on the International Space Station and other long-term manned facilities. Other applications are given in Table 2. In general, system cost is lowered as size is reduced, and with a low cost air analysis system, several systems could be utilized for monitoring large areas. These networked systems could be deployed at job-sites for worker safety, throughout a community for pollution warnings, or dispersed in a battlefield for early warning of chemical or biological threats.

Table 2 – Potential Applications for Systems Designed at HGDL

<p>Air Quality</p> <ul style="list-style-type: none"> • Environmental • Workplace 	<p>Medical Analysis</p> <ul style="list-style-type: none"> • Blood Analysis • Liver Analysis
<p>Leak Detection</p> <ul style="list-style-type: none"> • CRT Industry • Refrigeration Industry • Automotive Industry • Food Industry 	<p>Battlefield Threat</p> <ul style="list-style-type: none"> • Chemical Weapons • Biological Weapons • Land Mines
<p>Process Monitoring</p> <ul style="list-style-type: none"> • Semiconductor • Petrochemical • Cross-country Pipeline 	<p>Contraband Detection</p> <ul style="list-style-type: none"> • Explosives • Drugs
	<p>Geological Prediction</p> <ul style="list-style-type: none"> • UV Hazards • Volcanic Eruption

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Hazardous Gas Detection Monitoring

C Richard Arkin, Ph.D.
ASRC Aerospace
Kennedy Space Center, FL

Overview

- HGDL
 - Brief History
 - Mass Spectrometry
 - Need for Miniature Gas Detection System
 - General Component Evaluation Phase
 - Iterative Prototype Design Phase
 - Future
-

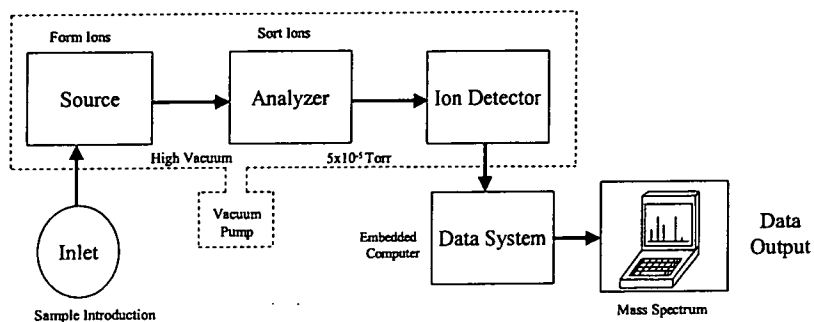
Hazardous Gas Detection Lab

- Real-time Quantification of Hazardous Gases in the Field
- Instrument Development
- Method Development
- Evaluate Commercial Components

Work Performed by Laboratory

- | | |
|---|---|
| ■ Systems Used Since Beginning of Shuttle Program | ■ Explosives & Fuels <ul style="list-style-type: none">□ Hydrogen & Oxygen□ Hydrazines |
| ■ Developed and Built GSE Systems | ■ Toxins <ul style="list-style-type: none">□ Hydrazines□ Volatile Organic Compounds (VOCs) |
| ■ System & Component Evaluation | |

What is Mass Spectrometry?

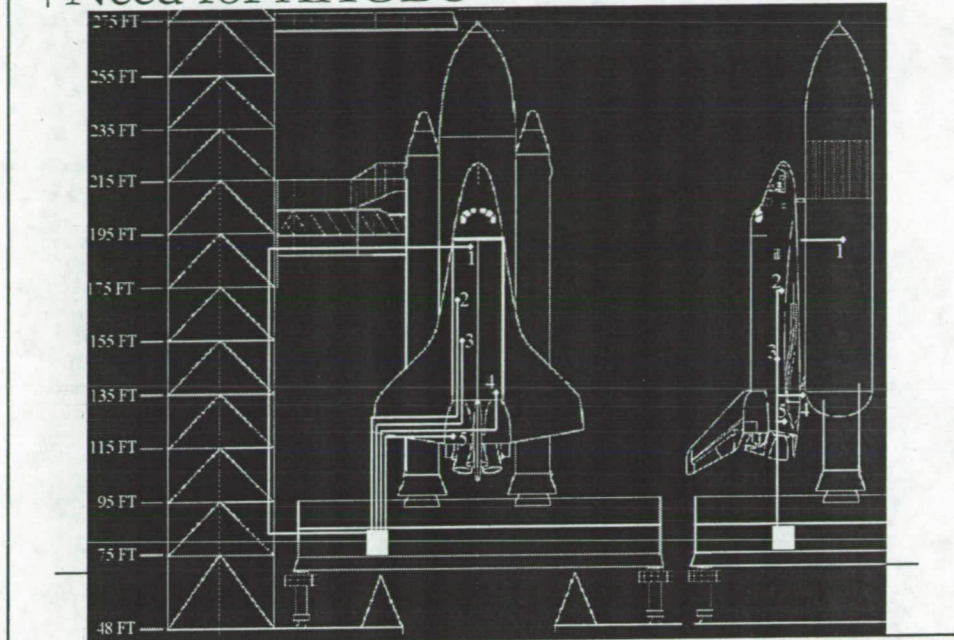


- Chemical analysis by transferring a charge to the molecule; electromagnetic separation then detection

Why Mass Spectrometry?

- | | |
|-----------------------|---------------------|
| ↑ Extremely Specific | ↓ Ruggedness |
| ↑ Sample Variety | ↓ Weight |
| ↑ Qualitative | ↓ Size |
| ↑ Quantitative | ↓ Cost |
| ↑ Rapid Response | ↓ Power |
| ↑ Large Dynamic Range | ↓ Operator Training |

Need for AHGDS



Motives to Reduce System Size

- Cost
- Mobility
- Reduced Sample Contamination
- Reduced Analysis Time
- Multiplexing Capability
- Increased Applicability
- Sample Throughput

Ultimate Goals

- Small
- Light Weight
- Power Efficient
- Rapid Analysis
- Good Dynamic Range
- Gas Identification & Quantitation
- Easy to Operate & Interpret

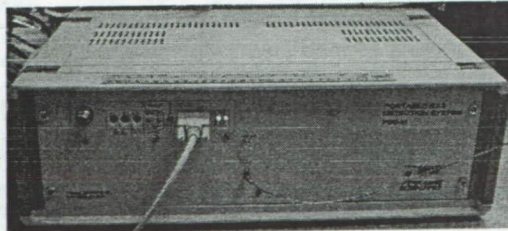
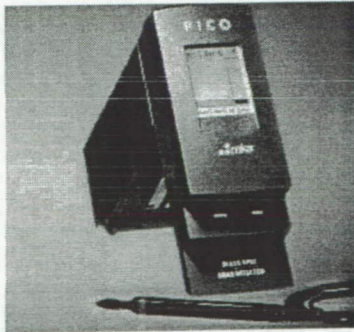
System Comparison



Project Phases

- Market Survey
 - Systems
 - Components
- Evaluation
 - Systems
 - Components
- Iterative Development/Field Tests
 - Develop System
 - Lab Test System
 - Field Test System

Market Survey

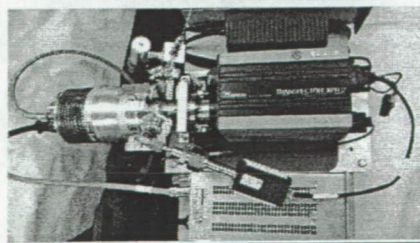
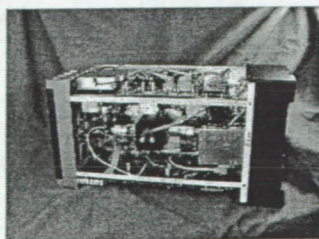
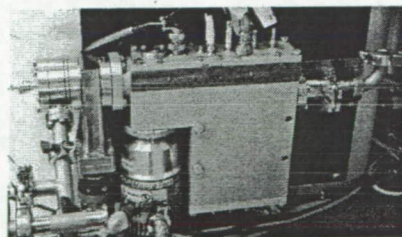
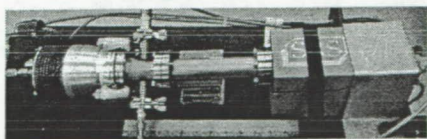


- Several Companies in Development
- Some Companies in Market
- No System meets the needs of NASA

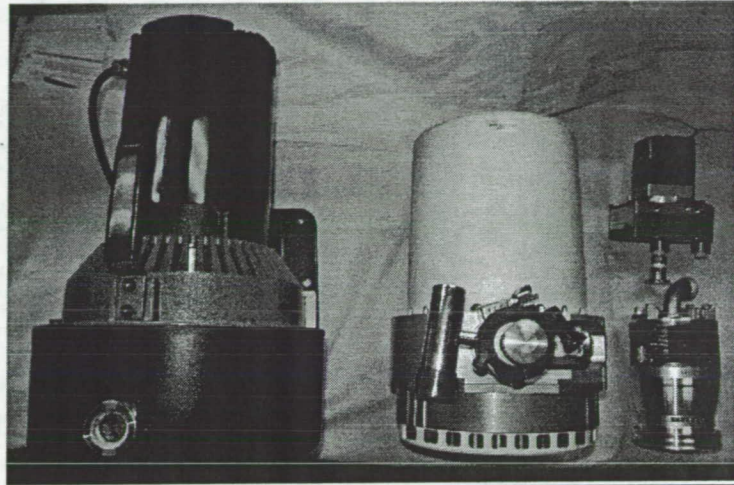
Evaluation

- Mass Spectrometers
- High Vacuum Pumps
- Backing/Rough Pumps
- Control Components
- Sensor Components
- Power Components
- Computer & DAQ Components

Mass Spectrometers



Vacuum Pumps

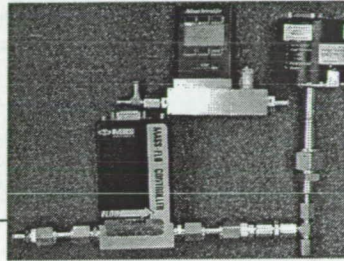
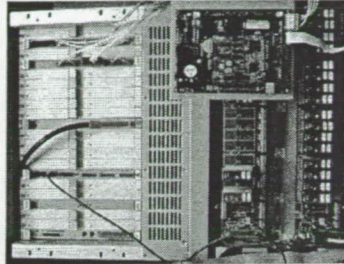
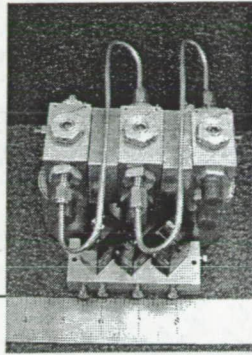


Other Components Evaluated

- Embedded Computers
- Solid State Relays
- Thermal Controllers
- Fluid Valves
- Flow Controllers
- Low Pressure Gauges
- High Vacuum Gauges
- Voltage Conversion
 - Inputs: 240 & 115 Vac, 28 Vdc
 - Outputs: 24 & 5 Vdc
- EMI Shielding
 - Emissions
 - Susceptibility

Subsystem Development

- Sample Transport
- Calibration
- Control



Iterative Development & Testing

- Develop Prototype
 - Lab Tests (Characterization & Simulation)
 - Field Tests (Application)
- Redesign Prototype / New Prototype
 - Lab Tests (Characterization & Simulation)
 - Field Tests (Application)
- Final Product
 - Will have significant experience
 - Applications benefit during development
 - Distribute Cost

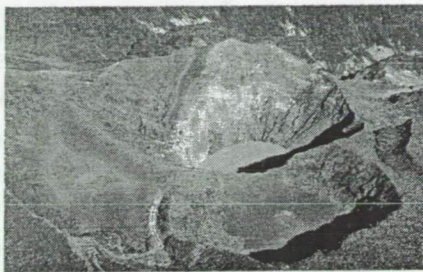
Scientific Goals

■ Monitor Volcanic Gas Emissions

- Helium (He)
- Carbon Dioxide (CO₂)
- Sulfur Dioxide (SO₂)
- Hydrogen Sulfide (H₂S)
- Multiple Organic Compounds

■ Correlate to Volcanic Cycles

- Compare to Ground Based Systems
- Determine Concentration Gradients



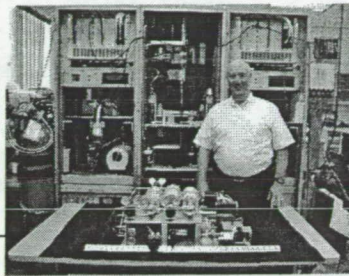
Engineering Goals

■ System Miniaturization

- Reduce Size
- Reduce Weight
- More Power Efficient

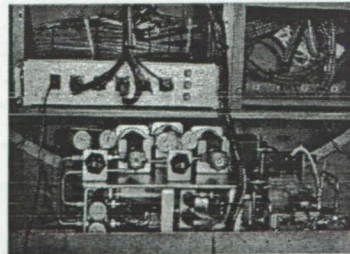
■ System Ruggedization

- Altitude
- Stress
- Vibration
- EMI

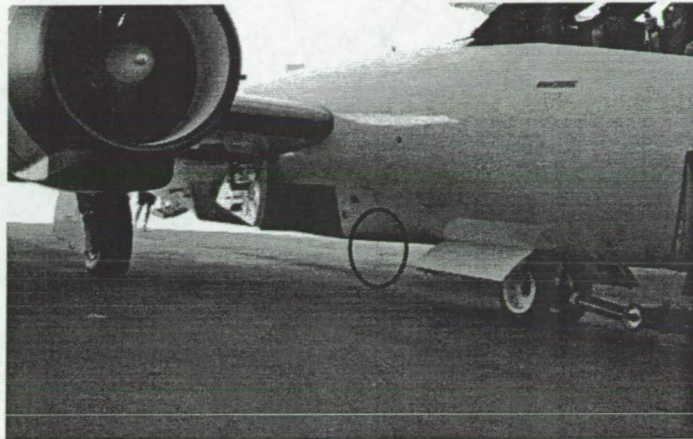


Aircraft Deployed Air Analysis

- Linear Quadrupole
- 90,000 cm³
- 47 kg (105 lb)
- 350 W (steady state)
- 6 s Scan Time
- 30 s Response Time
- Rugged (25 to -60°C; 760 - 50 torr)
- Autonomous
- 20 ppm LOQ

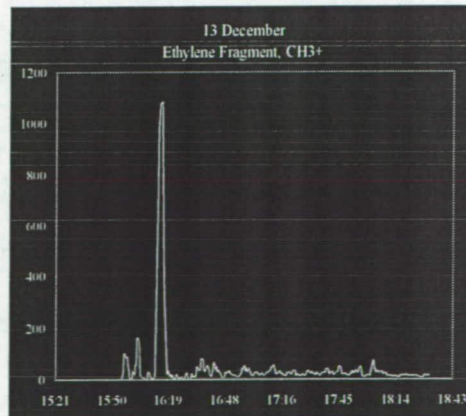


Sample Inlet on WB-57



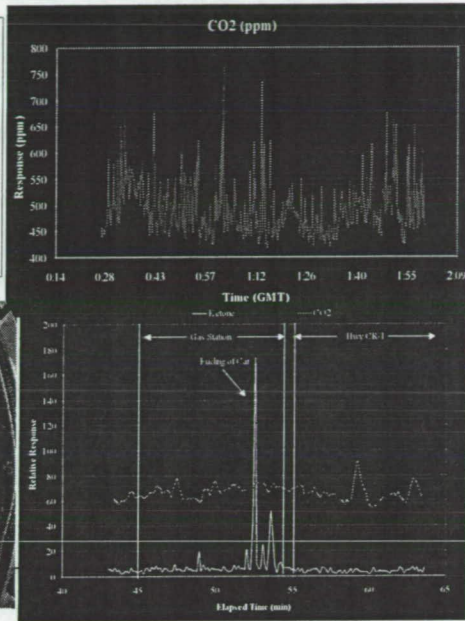
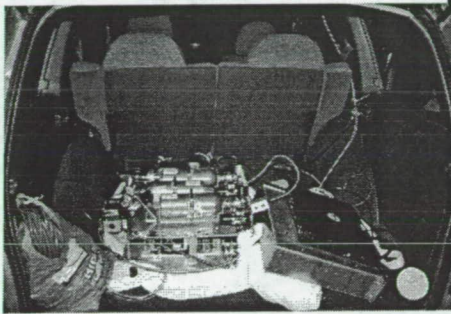
Tests in Houston

- 3 Successful Tests
- Water Problems
- Ethylene fragment
- 5000 ft.

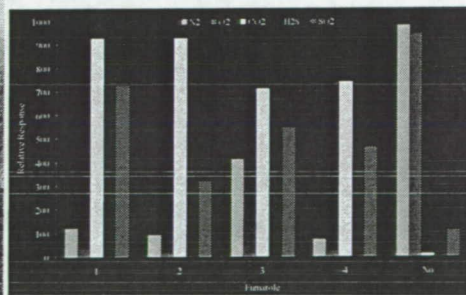


AVEMS in Back of 4x4

- Mounted System in Car
- Drove Around City
 - San Jose
 - Highway
 - Alajuela



Turrialba Volcano



History: New Installation

- Airborne Volcanic Emission Mass Spec
 - Preparation for Shuttle flight mass spec
 - Capable of monitoring Shuttle gases
 - Tailored to volcanic gases
 - Autonomous operation
 - In-situ calibration
 - Altitude from ground to 45,000 feet
 - System demonstrated in WB-57 flights over JSC and Costa Rica

AVEMS Test Flights, JSC, Costa Rica

- Requested by University of Costa Rica
- Partner with others to share flight vehicle
- Successful aircraft integration
- Successful test flights in most cases
- Demonstrated autonomous operation
- Significant operational experience acquired
- Data reduction in process

Current System – Weight & Size Reduction

- Small Autonomous Mass Spectrometer (SAMS)
- Next Generation System, More Applications
 - Lighter (~70 lbs)
 - More Stable Analysis
 - Smaller (~60,000 cc)
 - Greater H₂O Tolerance

Future Work

- Minimize COTS Components
 - More power efficient
 - Reduce weight & volume
- Methods to improve figures of merit
 - Calibration Techniques
 - Introduction Techniques
- Ruggedize

Potential NASA Applications

- Shuttle Processing
- International Space Station (ISS) Assembly Processing
- ELV Processing
- Environmental Monitoring
- ISS Crew Area Air Quality Monitor
- Next Generation Vehicle Air Quality Monitor

Potential Commercial Applications

- | | |
|--------------------------|-------------------------|
| ■ Air Quality | ■ Medical Analysis |
| □ Environmental | □ Blood Analysis |
| □ Workplace | □ Liver Analysis |
| ■ Leak Detection | ■ Battlefield Threat |
| □ CRT Industry | □ Chemical Weapons |
| □ Refrigeration Industry | □ Biological Weapons |
| □ Automotive Industry | □ Land Mine |
| □ Food Industry | ■ Contraband Detection |
| ■ Process Monitoring | □ Explosives |
| □ Semiconductor | □ Drugs |
| □ Petrochemical | ■ Geological Prediction |
| □ Cross-Country Pipeline | □ Volcanic Eruption |
| | □ UV Hazards |

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