NASA John F. Kennedy Space Center

Internship Report

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LAVA Subsystem Integration and Testing for the RESOLVE Payload of the Resource Prospector Mission: Mass Spectrometers and Gas Chromatography

June – August, 2015

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Regolith and Environment Science & Oxygen and Lunar Volatile Extraction (RESOLVE)

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I. Acronyms

(in order of appearance in document)

RESOLVE-Regolith and Environment Science & Oxygen and Lunar Volatile Extraction

RP-Resource Prospector

ISRU-In-Situ Resource Utilization

LAVA-Lunar Advanced Volatile Analysis

GC-MS - Gas Chromatograph-Mass Spectrometer

OVEN-Oxygen and Volatile Extraction Node

LOVEN-Connection between the OVEN and LAVA subsystems

LCROSS- Lunar Crater Observation and Sensing Satellite

NS- Neutron Spectrometer

NIR-Near Infrared Spectrometer

WDD-Water Droplet Demonstration

ETU-Engineering Test Unit

SDS-Sample Delivery System

COTS- Commercial off the shelf

QMS-Quadrupole Mass Spectrometers

IonCCD- Ion Charge-Coupled Device

FSS-Fluid Subsystem

LOD-Limit of Detection

PP-Partial Pressure

RGA-Residual Gas Analyzer

MS-Mass Spectrometer

VA-Volatile Analysis

NIRVSS-Near Infrared Volatile Spectrometer Subsystem

II. Abstract

The Regolith and Environment Science & Oxygen and Lunar Volatile Extraction (RESOLVE) payload is part of Resource Prospector (RP) along with a rover and a lander that are expected to launch in 2020. RP will identify volatile elements that may be combined and collected to be used for fuel, air, and water in order to enable deeper space exploration. The Resource Prospector mission is a key part of In-Situ Resource Utilization (ISRU). The demand for this method of utilizing resources at the site of exploration is increasing due to the cost of resupply missions and deep space exploration goals.

The RESOLVE payload includes the Lunar Advanced Volatile Analysis (LAVA) subsystem. The main instrument used to identify the volatiles evolved from the lunar regolith is the Gas Chromatograph-Mass Spectrometer (GC-MS). LAVA analyzes the volatiles emitted from the Oxygen and Volatile Extraction Node (OVEN) Subsystem. The objective of OVEN is to obtain, weigh, heat and transfer evolved gases to LAVA through the connection between the two subsystems called the LOVEN line. This paper highlights the work completed during a ten week internship that involved the integration, testing, data analysis, and procedure documentation of two candidate mass spectrometers for the LAVA subsystem in order to aid in determining which model to use for flight. Additionally, the examination of data from the integrated Resource Prospector '15 (RP' 15) field test will be presented in order to characterize the amount of water detected from water doped regolith samples.

III. Introduction

NASA is developing a payload for ISRU to facilitate planetary exploration by extracting and using lunar resources to supply mission consumables. Producing life support essentials such as water, breathable air, and propellants locally during lunar and solar system exploration will reduce mission cost, enhance feasibility and safety, and extend mission duration. The Lunar Prospector mission and Lunar Crater Observation and Sensing Satellite (LCROSS) have indicated that water/ice and other volatiles may be present and accessible in the lunar regolith, particularly at the poles.

NASA's Resource Prospector mission, scheduled to launch in 2020, will include a rover hosting the Regolith & Environment Science and Oxygen & Lunar Volatile Extraction (RESOLVE) payload. RESOLVE instrumentation includes the Neutron Spectrometer (NS), Near Infrared Spectrometer (NIR), Drill, OVEN, and LAVA subsystems. If water is identified by LAVA, the volatiles will be cooled and condensed in LAVA's water droplet demonstration (WDD) assembly.

A previous version of the RESOLVE project completed an ISRU analog mission on Mauna Kea to test the integration of the payload on the rover to capture and process regolith simulant. The most recent hardware is known as RP'15. It does not include all of the hardware but rather shows the functionality of the integrated rover and payload. Various subsystems, including LAVA, are in the Engineering Test Unit (ETU) phase to assure that all vital components of the payload are space-flight rated and will perform as expected during the mission.

OVEN delivers the evolved gas into surge tank where it is quantified by measuring the resulting pressure observed. The Sample Delivery System (SDS) is then filled with the volatile gases that have been extracted from the heating of the regolith in OVEN. SDS acts as a mini reservoir where the sample gas can be diluted. The SDS transfers the gas to either the GC or directly to the MS for identification of the components present.

III A. Resource Prospector ’15

Resource Prospector (RP)’15 is different from the mission that will launch to the moon in 2020. The integrated test operates at normal gravity and atmospheric pressure, unlike lunar conditions of one sixth normal gravity and high vacuum that the actual RESOLVE payload will encounter. RP’15 contains only the GC since the MS requires pumps to operate at atmospheric pressure, which are not part of the flight-forward design. The objective of the RP ’15 demonstration is to enhance integration of the payload with the rover, not to quantify samples, thereby allowing the GC and the fluid subsystem manifold to constantly vent, rather than transfer volatiles to a MS.

Once the regolith sample is delivered to OVEN, it begins heating in order to release the volatile gases. These gases are transferred from OVEN to the LAVA subsystem through the LOVEN line.

III B. Inficon and OIA Mass Spectrometers

The LAVA subsystem currently includes a baseline mass spectrometer from OI Analytical (OIA MS). A second mass spectrometer manufactured by Inficon can also meet the speed and detection requirements of the mission. These mass spectrometers have been modified by their manufacturers in accordance with NASA spaceflight specifications and are referred to as Commercial off the shelf (COTS) instruments. Understanding the ability of the two mass spectrometer units, Inficon and OIA, to detect low molecular weight volatiles (CO, CO2, H, He, N2, H20, O2) that may be present in regolith sample under simulated lunar conditions is crucial to the success of the Resource Prospector mission. The reproducibility and accuracy of results in the MS units with identical sample gases is also a factor in deciding which mass spectrometer will be used on the payload.

The Inficon Transpector MS model is a quadrupole mass spectrometer (QMS). QMS instruments are often used for space experiments since they are smaller, lighter, and less expensive than traditional magnetic field analyzers. The three components within this type of mass spectrometer are: the ion source with electron impact ionizer and ion extraction optics, the actual quadrupole analyzer consisting of four cylindrically rods, and the ion detector or electron multiplier.

The OIA MS is a magnetic sector analyzer mass spectrometer. As an array detector, the Ion Charge-Coupled Device (IonCCD) employs two sector fields, an electrostatic analyzer and a magnetic sector, to separate and detect all ions according to their mass-to-charge (m/z) ratio simultaneously. The IonCCD includes 2126 discrete detector elements, or pixels. A positively or negatively charged ion strikes a pixel, is discharged, and the charge is counted.

IV. Procedures

IV A. Inficon Transpector MS Procedure and Testing

Inficon Transpector MS testing was performed in order to understand the behavior of the mass spectrometer electronics in vacuum. This is crucial for the LAVA subsystem since the MS electronics are not made to operate under conditions on the lunar surface. This testing was performed through FabGuard software

The experiments were conducted on different days with the use of room air. Fluid Subsystem (FSS) measurements were recorded during each test for future data analysis using a custom data recorder. Another Inficon MS was used as a residual gas analyzer (RGA) to monitor chamber health in case any anomalies occurred. The temperatures of the MS and RGA mass spectrometers were recorded to ensure that they would not exceed their 70°C limit. The chamber pressure for all tests was <5x10-5 Torr. The chiller was operated at ≈ -20°C. It is crucial to operate under these conditions in the vacuum in order to eliminate any error related to Earth's Atmosphere that will not be present on the moon.

Sample delivery was enabled through the FSS/SDS. The carrier gas was supplied to the Sample Delivery System(SDS). Once the Inficon Transpector MS had recognized the applied gas and a steady signal of the partial pressure was established, the vent roughing pump command was initiated to empty the SDS. For Room Air, the SDS was filled for 1 minute and evacuated for 3 minutes. For Helium, the SDS was filled for 30 seconds and evacuated for 30 seconds. These tests were performed with Dr. Janine Captain.

The output from FabGuard in **Figure 1** identifies the signal, or partial pressure, throughout the time interval of this test. This rise in partial pressure to form a peak occurs when pressure is applied to the SDS. The partial pressure will then become steady. Once the SDS has been evacuated, the signal will return to the baseline partial pressure. The different colors represent pressure, ionizer state, or the atomic mass units of varying elements or compounds.

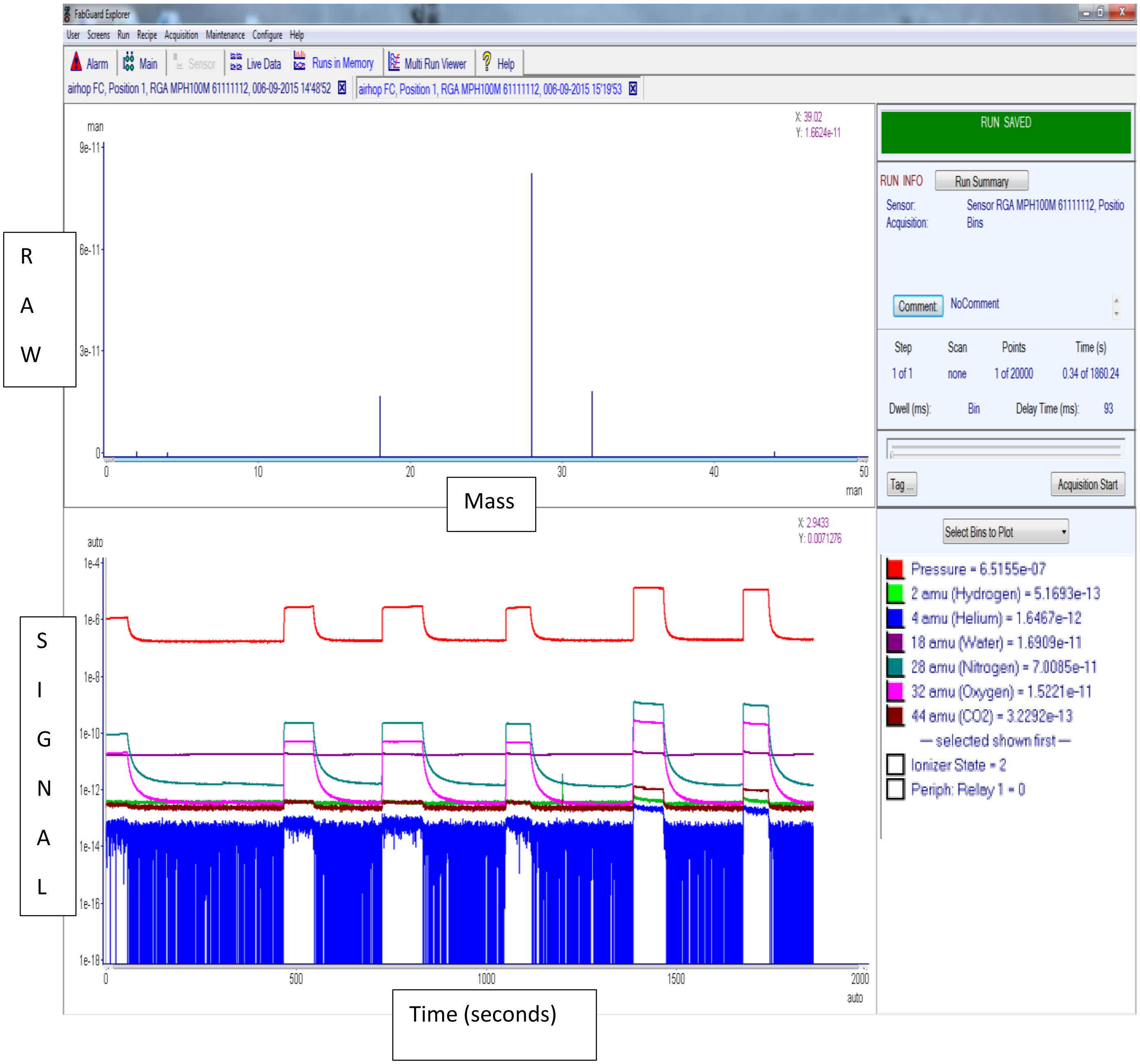


Figure 1. *Output from FabGuard during test on 6/9/15.*

For all of the test days (**Figure 2**), this information was recorded in order to properly compare the results. The "number of peaks" refers to the application of room air to the SDS and then the evacuation to result in a fall back down to the baseline signal as discussed above for **Figure 1**.

Test	***SDS***	***FSS***	***Constituent***	***Relative Humidity***	***Temperature***	***Number of Peaks for 3 psia***	***Number of Peaks for 5 psia***	***Number of Peaks for 10 psia***
6/9/2015	Not Heated	Not Heated	Room Air	38.6	24.8	3	3	4
6/10/15	Heated	Heated	Room Air	40.3	24.5	3	3	4
7/14/15	Heated	Heated	Room Air	41.5	24.2	4	4	5
7/17/15	Heated	Heated	Room Air	41.1	23.8	4	3	5

Figure 2. *Inficon MPH testing descriptions.*

IV B. GC LOVEN RP'15 Procedure and Testing

These tests were performed at either KSC or in JSC-1A High Bay with the data analysis and monitoring occurring at KSC. These GC runs occurred within payload functional checkouts that were performed post rover integration with a focus on testing Volatile Analysis.

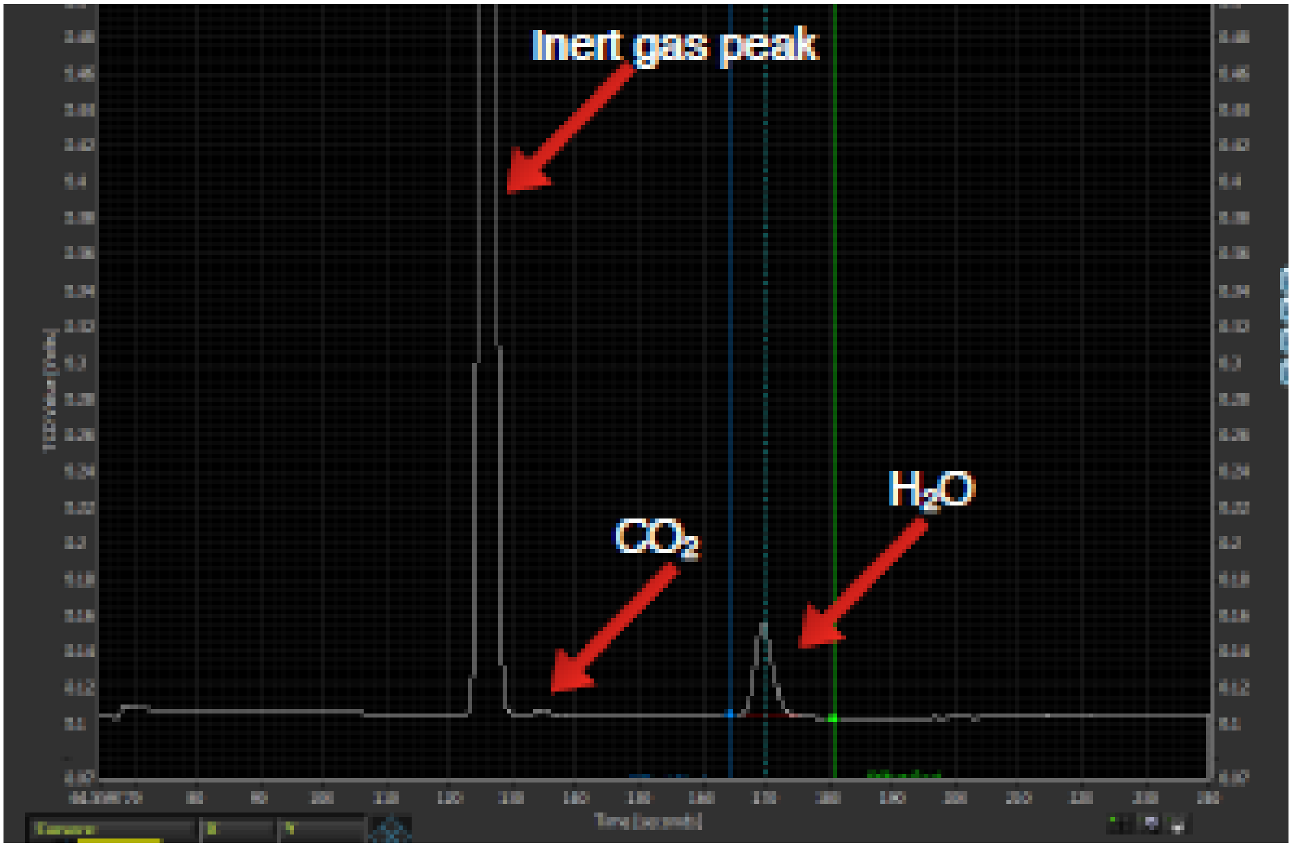
In order to perform the GC runs, the drill must be used to acquire sample to deposit into OVEN. Before drilling occurs, the GC and FSS must warm up to temperature. Once the proper temperature set points have been reached, a regolith sample equivalent of a 20 cm drill bite was taken out of simulant regolith. The simulant used was either 2% or 5% water. The drill then retracted to "Sample Capture Position" and Near Infrared Volatile Spectrometer Subsystem (NIRVSS) Sample Collection and Transfer Procedure was followed. The sample was deposited into the crucible and then sealed.

Once the crucible was sealed, the Volatile Analysis (VA) began. With the current model of RP'15, the first GC run identifies water marginally higher than what would usually be seen from a GC in atmosphere that had just turned on preparing to sample without any bake out or calibration. The initial concentration of water identified could be due to the crucible heating up and desorbing surficial water. This will be operationally addressed for flight. To quantify peak area, integration is used with the retention time, which is unique for each gas, as the limits. The peak area is identified for inert gases, CO2, and water (shown in **Figure 3**). The water peak area is used to plug into calibration curves that will help identify the concentration of water present. There are two calibration curves that make up the non-linear range.

As OVEN heats, the water peak amplitude and retention time increases. There is not a significant difference shown among the runs until Run 3. Once OVEN has reached the setpoint of 150°C, 2 more GC runs are recorded.

During these tests, the OVEN pressure is also recorded. The pressure rises while OVEN is heating, but drops once temperature has been reached. The pressure in the manifold is kept below 40 psia to avoid damaging the GC, and is accomplished by constantly venting pressure from the OVEN. For flight, the built up pressure, temperature and volume of the surge tank will be used to examine the volatiles.

Voltage



Time (seconds)

Figure 3. *GC run with Inert gas, CO2, and Water peaks*

Test	***Date***	***Simulant***	***% water vapor in atmosphere***
1	5/27/2015	Room Air	1.65
2	6/1/2015	Room Air	1.36
3	6/2/2015	5% water	1.42
4	6/4/2015	5% water	1.45
5	6/30/2015	2% water	1.42
6	7/15/2015	2% water	1.27
7	7/16/2015	2% water	1.3
8	7/17/2015	2% water	1.41
9	7/17/2015	Dry sample	1.41

Figure 4. *GC LOVEN test descriptions.*

V. Results

V A. Inficon Transpector MSResults

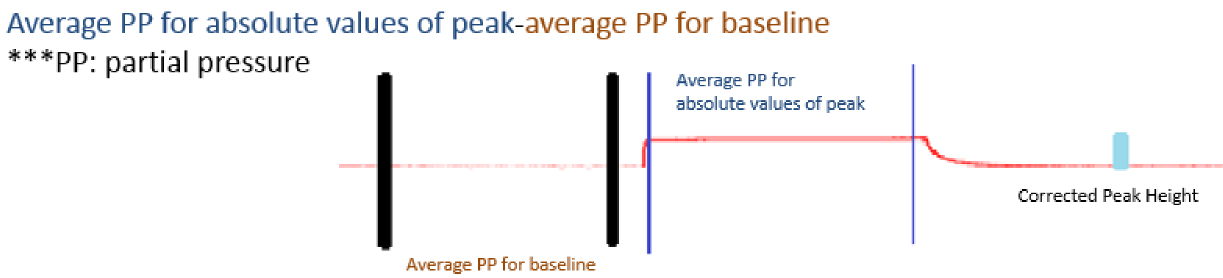
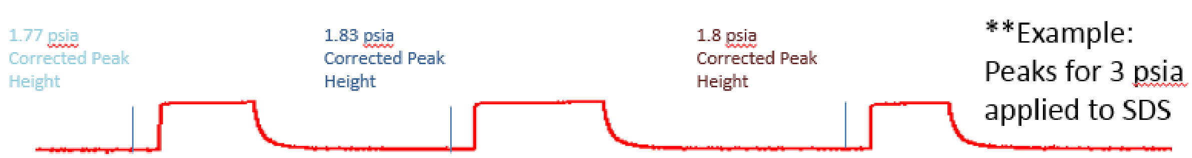


Figure 5. *In order to find the true signal of each peak, the baseline must be subtracted to get the "Corrected Peak Height".*

Once the Corrected Peak Heights has been calculated, the Relative Peak Height indicating partial pressure can be used to determine if the system is properly functional and verify that flow is linear with pressure. Relative Peak Height is measuring the actual signal that the mass spectrometer is able to identify. In order to get an accurate idea of the partial pressure of each mass, the baseline signal must be subtracted.

Calculation for actual psia: SDS Sample pressure (psia)-SDS Initial Pressure (psia)



Calculation example: (1.77 psia Corrected Peak Height) /1.77

Figure 6. *Relative Peak Heights*

	2_amu_(Hydrogen)	18_amu_(Water)	28_amu_(Nitrogen)	32_amu_(Oxygen)	44_amu_(CO2)
1: Actual psia: 2.87	Below LOD	1.5E-12	6.3E-11	1.4E-11	4.5E-14
2: Actual psia: 2.80	3.0E-15	6.5E-14	6.1E-11	1.3E-11	3.8E-14
3: Actual psia: 2.84	3.2E-15	1.7E-13	6.1E-11	1.3E-11	3.9E-14

Figure 7. *These Relative Peak Heights are from the test conducted on 6/10/15. To confirm system functionality, a comparison can be drawn for the different psia for each amu. From this test, the values for Nitrogen, Oxygen, and CO2 are close. It is also evident here that Hydrogen and Water were not identified well by the system because these constituents were not supplied. Only Room Air was being tested.*

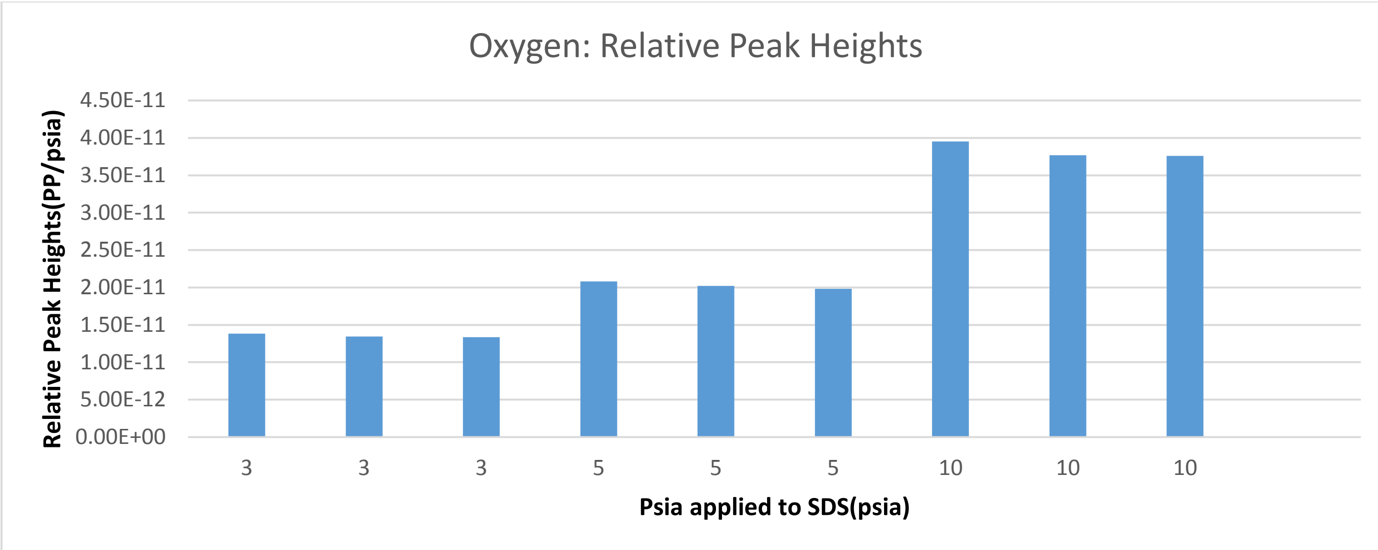


Figure 8. *Results from Test conducted on 6/10/15. The Relative Peak Heights 3, 5, and 10 psia applied to SDS indicate that the flow is linear to pressure for Oxygen and that the results are reproducible*

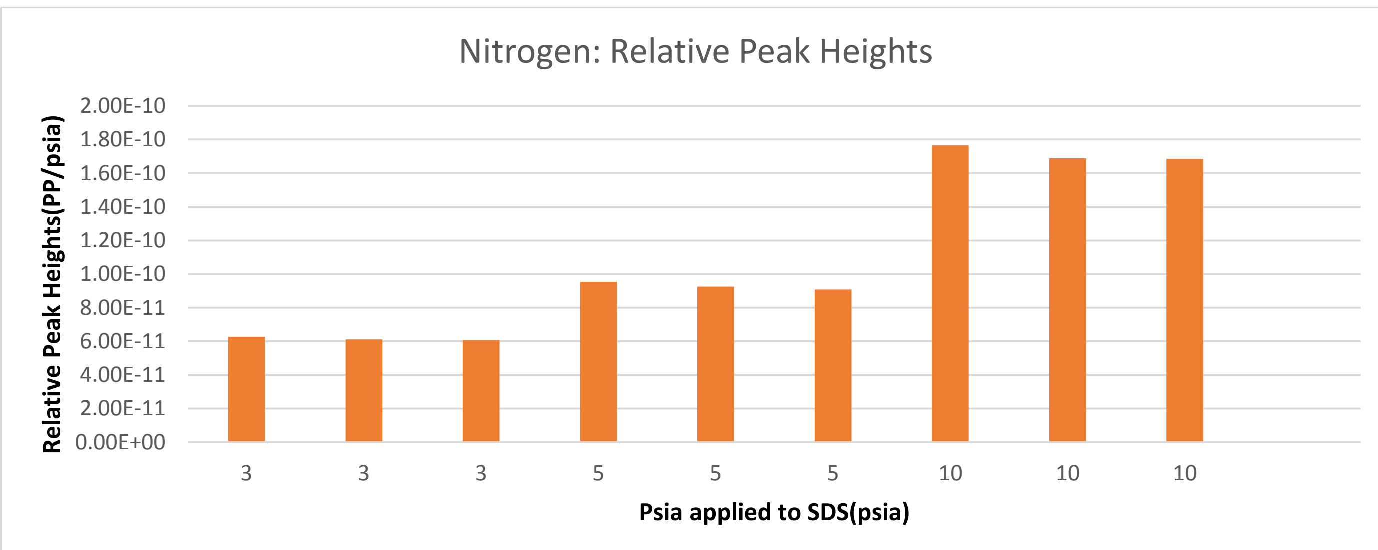


Figure 9. *Results from Test conducted on 6/10/15. The Relative Peak Heights 3, 5, and 10 psia applied to SDS indicate that the flow is linear to pressure for Nitrogen and that the results are reproducible.*

	Hydrogen	***Water***	***Nitrogen***	***Oxygen***	***CO2***
5/3 PP	Below LOD	2.9E-01	1.5	1.5	1.4
5/3 psia	1.7	1.7	1.7	1.7	1.7
10/5 PP	2.3	8.3	1.8	1.9	2.8
10/5 psia	2.0	2.0	2.0	2.0	2.0
10/3 PP	Below LOD	2.4	2.8	2.8	4.0
10/3 psia	3.5	3.5	3.5	3.5	3.5

Figure 10. *Comparing % Increase of Partial Pressure and Psia, test date 6/10/15*

Calculation: $\frac{\text{Avg. Relative Peak Heights for 5 psia}}{\text{Avg. Relative Peak Heights for 3 psia}} \approx \frac{\text{Avg. actual 5 psia}}{\text{Avg. actual 3 psia}}$

Results from the test conducted on 6/10/15 in **Figure 10**: The % increase of partial pressure and % increase of pressure should be similar if the system has a linear response. The values again for Nitrogen, Oxygen, and CO_2 show evidence that flow is linear with pressure. Hydrogen and water were not above the detection limit for the Inficon Transpector MS.

Tests conducted on 6/10/15, 7/14/15, and 7/17/15 all showed similar results. The test from 6/9/15 varies significantly since the SDS and FSS were not heated. From analysis, the system does function accurately overall and flow is linear with pressure. Additionally, without heating of the SDS and FSS, the results are not as accurate. Almost all of the signals for each of the different masses were not above the limit of detection. With the heating of the FSS and SDS, the higher pressures applied to the SDS (3 and 5) showed better signals.

The comparison of the % increase of partial pressure and sample pressure shows that repeatability is possible with the Inficon MS.

Inficon MS has not been vibration tested yet. Once the vibe test has been complete, more functional testing will occur to ensure that the Inficon MS is still operating as it should.

V B: GC LOVEN RP'15 Results

For Test 2 as shown in **Figure 11**, room air was used as a constituent to provide a baseline of the system for comparison in the future before using a simulant doped with water. Without a sample producing water, there is a decreasing water trend since no water is being evolved. In future testing, the results from test days with simulant could be corrected by subtracting this baseline.

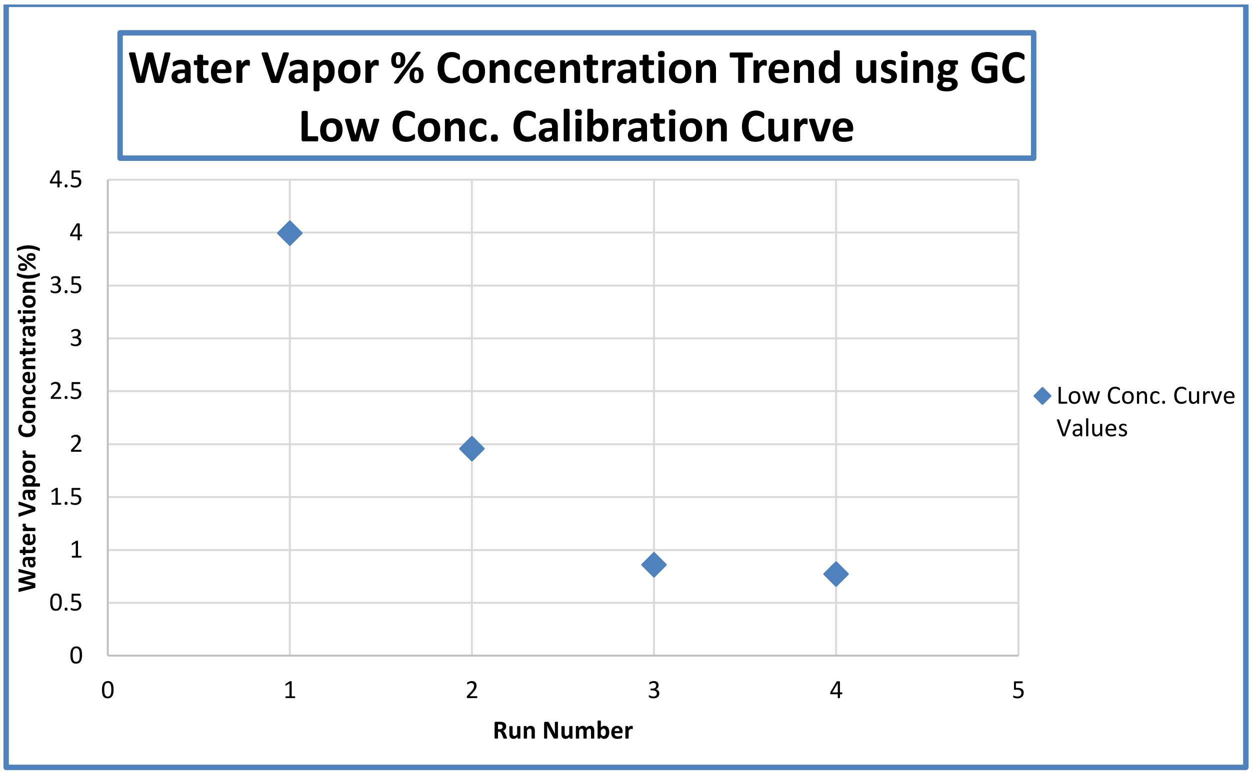


Figure 11 *Test 2 6/1/15*. Water vapor % decreases with time as runs continue to occur.

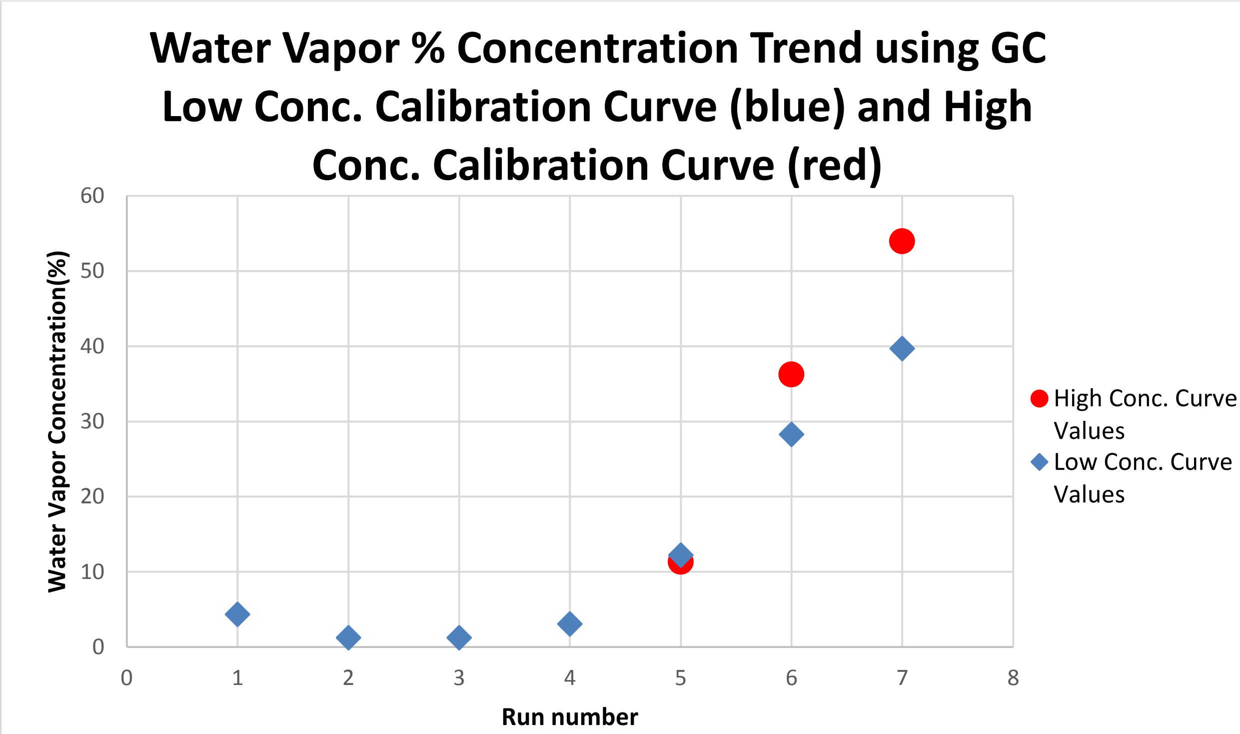


Figure 12. *Test 4 6/4/15*

As shown from **Figure 12,** the water vapor % increases with time as runs continue to occur. This trend was recognized with all tests run for 5% and 2% simulant.

The results obtained for tests with 5% and 2% simulant are reproducible as shown by **Figure 13** and **Figure 14** below.

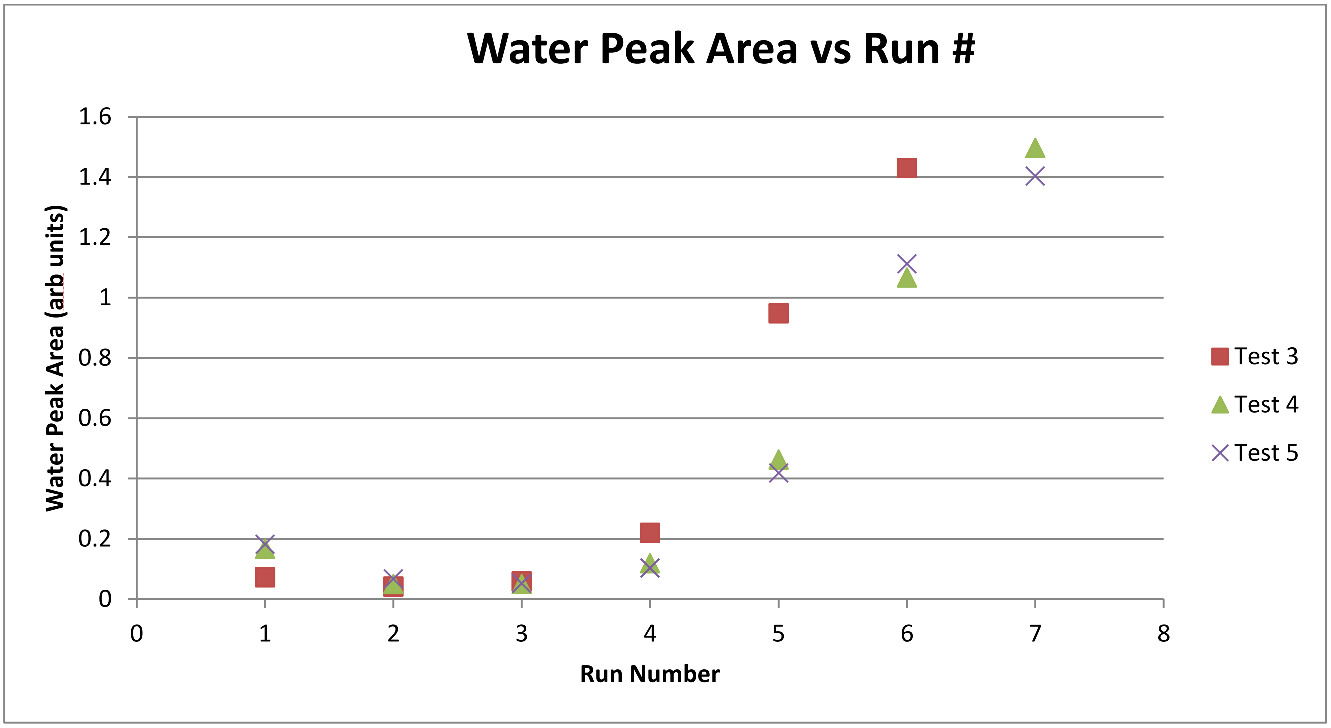


Figure 13. *Tests run with 5% simulant*

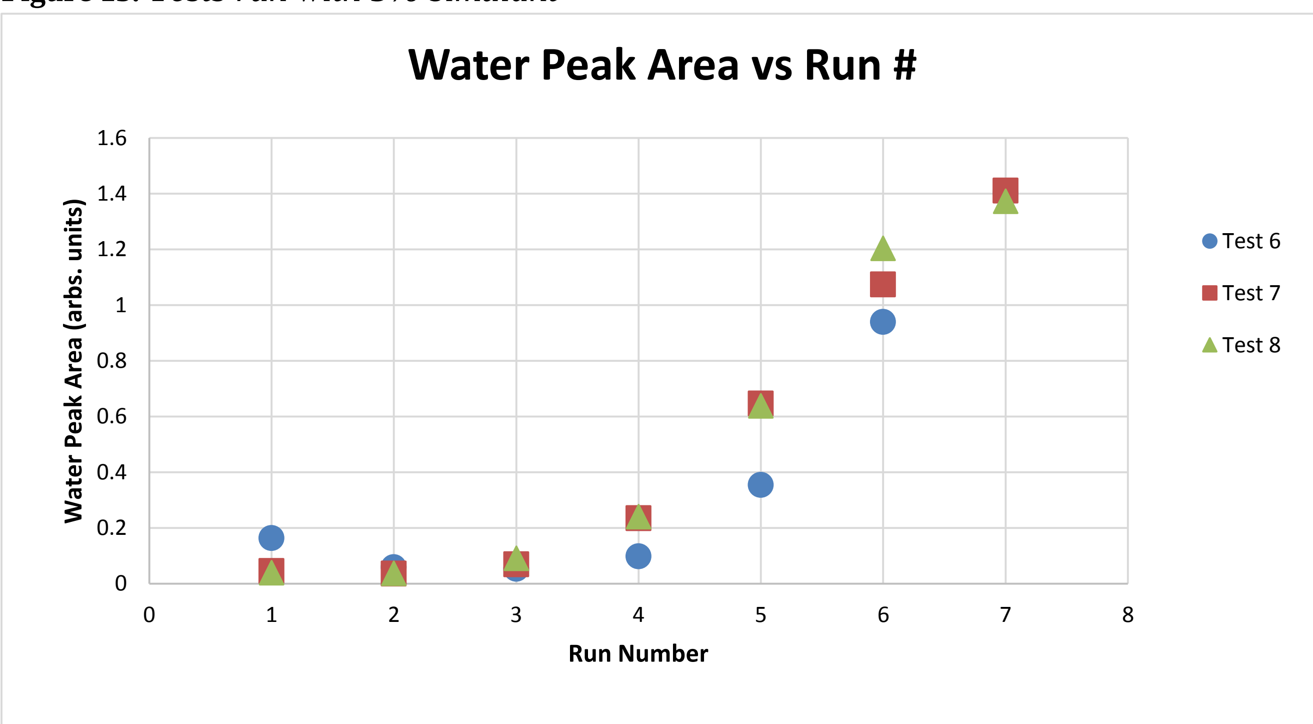


Figure 14. *Tests run with 2% simulant*

As shown in **Figure 15**, the water peak area is very similar whether 2% or 5% simulant is used.

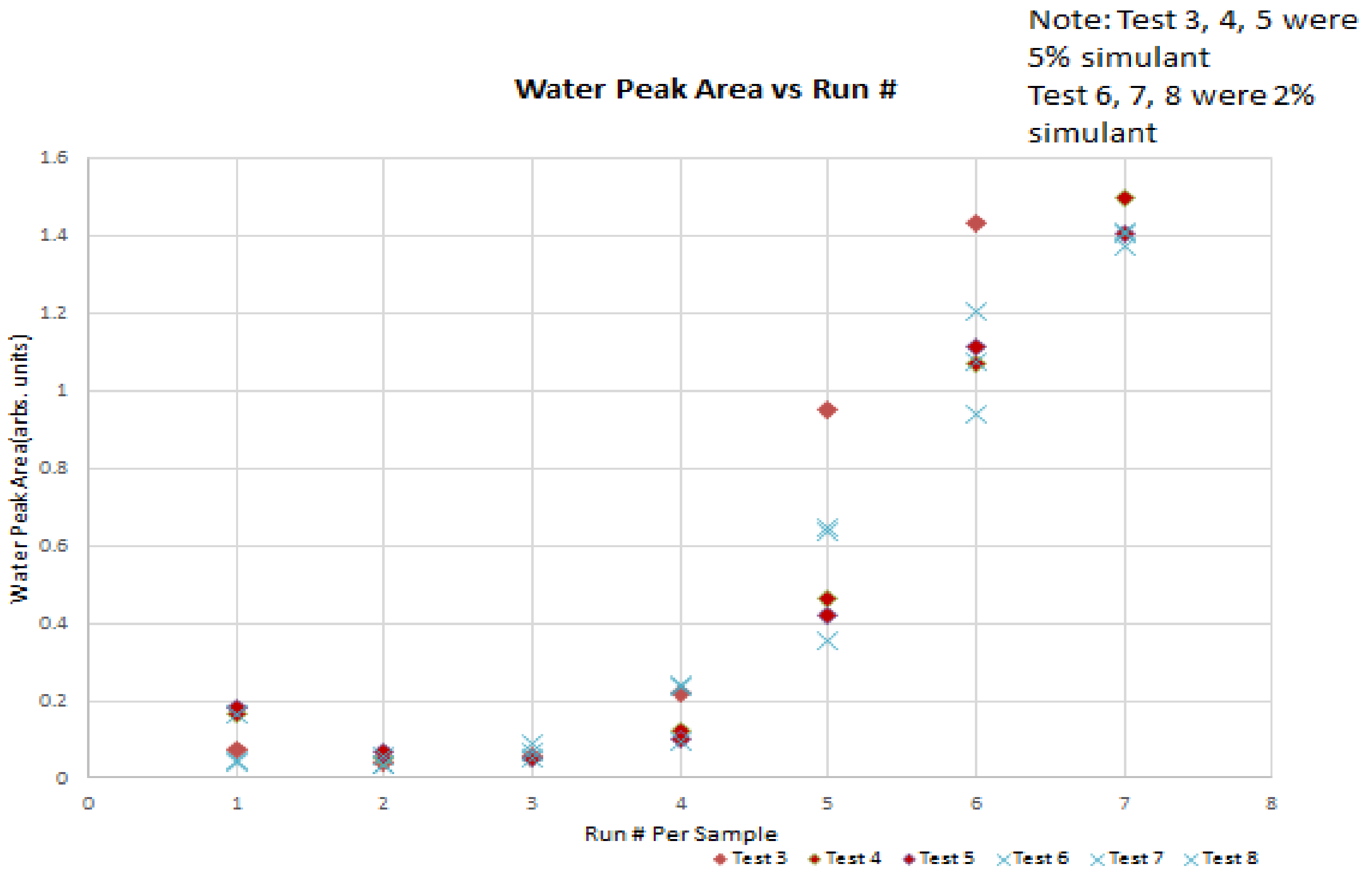


Figure 15. *Tests run with 2% and 5% simulant*

VI. OIA MS Troubleshooting

OIA MS has had issues in the past with the Integrated High Voltage (IHV) Board, Filaments, and loss to the SDS. Tests were performed at KSC with Beau Peacock, Janine Captain, and Josephine Santiago-Bond in collaboration with Evan Niedholdt from JPL and Gottfried.

Throughout many tests conducted over the past 10 weeks, the RGA or Inficon MS was used to monitor the chamber health. The use of another mass spectrometer in addition to the OIA MS aided in detecting anomalies and possible causes for them.

A cause for concern was the possibility of the electronics within either the mass spectrometer or the IHV board burning out. New recipes were created in the Inficon FabGuard software in order to select specific masses of interest to watch during the tests. With the ability to make these recipes, a method could be created to watch Carbon in Oxygen in order to determine whether components were being burnt.

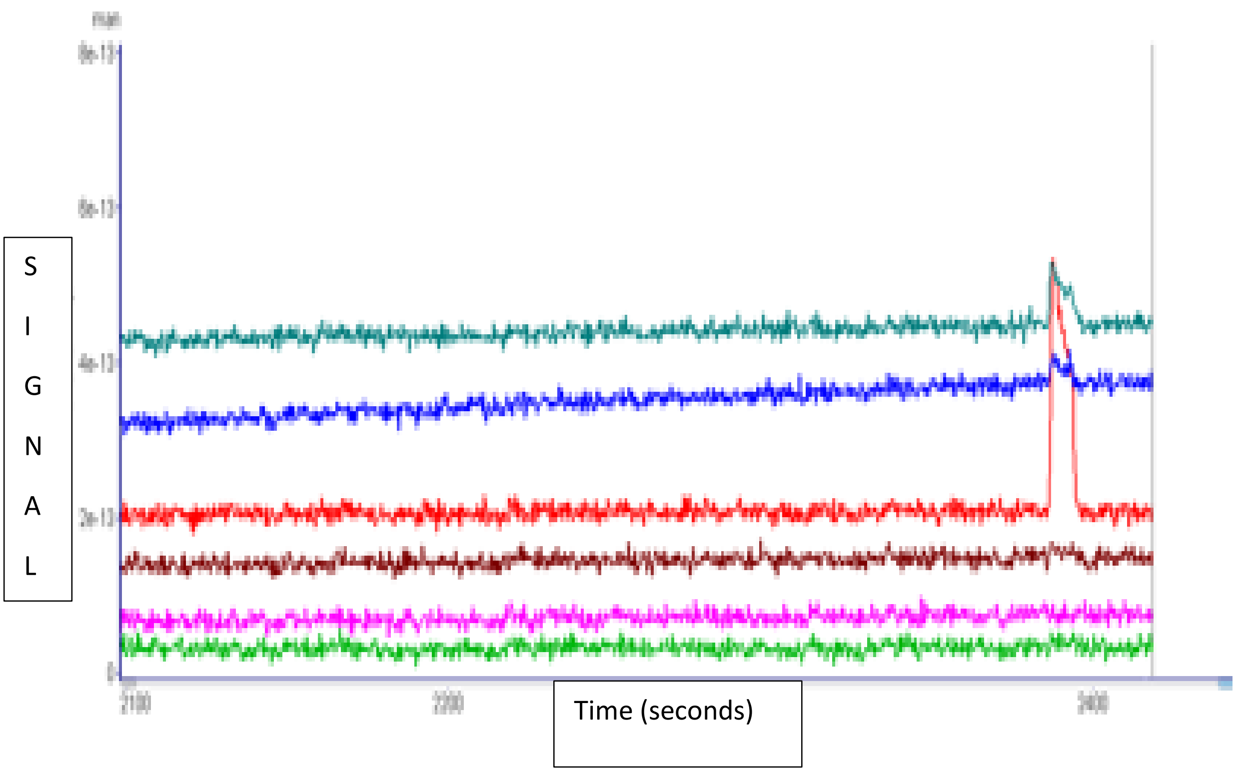


Figure 16. Example from RGA showing peaks in Nitrogen, Oxygen, and Hydrogen seen during an OIA MS test on 6/17/15

VIII. Conclusion

The Inficon Transpector MS has potential to be used as part of the GC-MS set up on the RESOLVE payload since there is data from these first tests to show that the instrumentation can perform in a vacuum. While there are many more pre-flight tests to occur before coming to a decision, this data will be referenced in the future after a vibrations test is performed.

The GC RP’15 LOVEN data shows that RP’15 has met another pre-flight objective. While there was no mass spectrometer, the lessons learned from design, development, integration and mission operations is valuable for the flight mission. There is now a better understanding of appropriate system requirements to be allocated to subsystems as the project moves forward.

As NASA continues to expand their space exploration with the goal of “pioneering” Mars, the use of ISRU becomes more prevalent. The Resource Prospector mission is fundamental to future success in space exploration. With the use of resources from planetary bodies, the goal is human civilization in our solar system is attainable. Once there is a better knowledge of components on the moon, deep solar system manned missions can be enabled.

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