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# SOLID-STATE SENSOR BEHAVIOR IN REDUCED-PRESSURE ENVIRONMENTS DEMONSTRATION USING AN EXPERIMENTAL INDIUM TIN OXIDE OZONE GAS SENSOR FOR OZONE SOUNDING

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## ABSTRACT

Sensors in reduced pressure environments exhibit largely uncharacterized behaviors. Several solid-state sensors, such as the CMOS indium tin oxide ($In_2O_3$:$SnO_2$) sensor, offer key benefits in response rates and sensitivity when compared to temperature and pressure dependent sensor technologies. The indium tin oxide (ITO) thin film sensor for the measurement of ozone, developed by the University of North Florida, represents a unique, easily produced in mass, and newly designed solid-state nanocrystalline gas sensor array (patent pending). These sensors allow detection to the parts per billion by volume (ppbv) level. Testing for ozone quantitatively agreed with calibration to within 15% and involved testing aboard a NASA high altitude balloon. This research addresses many serious unmet needs for miniature sensors capable of *in-situ* and real-time detection of ozone gas in reduced pressure and low temperature. Because these ITO sensors, developed at UNF, do not require the high operating temperatures of previous sensors and because these sensors maintain good stability under harsh atmospheric conditions, gas sensors of this type appear as good candidates for use in extraterrestrial applications, and in space flight instrumentation. These sensor archetypes should help satisfy the rigorous demands of space flight technologies and the future demands of the VSE.

## 1. INTRODUCTION

Since the early 1960s, gas-sensitive resistors have existed commercially with a demonstrated capability to detect combustible gases at the ppmv level. However, recent thin film gas sensors demonstrate great promise at reliably detecting concentrations at the ppbv (Hansford *et al.* 2004). Specifically, the use of ITO follows as the most recent solid-state gas sensor development (Patel *et al.* 2003, 2005-7).

Current conventional ozone sensors, such as the electrochemical concentration cell (ECC) ozonesonde, suffer from pressure sensitivities, great expense, and difficulty in their deployment. The uses of ITO sensors demonstrate great improvements over prior gas sensors, such as those that use tungsten tin oxide, in sensor response and sensitivity. The ability to detect such low levels of concentration is critical in both ozone atmospheric sampling and in the measurement of ground level pollution that vary on the ppbv level.

The implementation of ozone sensors, such as those developed at UNF, at high-altitudes plays a key role in understanding the current ozone situation. The structure of the ozone loss in near-space and space over time helps to test and confirm scientific understanding about the future state of the ozone. Such understanding helps also to resolve questions regarding how gas phase and heterogeneous modulation of chlorine monoxide and inorganic chlorine ($ClO/Cl_y$) partitioning affects ozone depletion. In fact, stratospheric ozone depletion depends largely on the partitioning processes that arise from the release of halogen atoms. These chemical processes in the stratospheric ozone layer help clarify effectiveness of the changes in chlorine enacted in the twentieth century (Solomon 1999).

Further, such robust and rugged sensors remain a critical need for NASA. Originally, research out of the Space Life Science Laboratory at Kennedy Space Center from Dr. Phillip Fowler and Dr. Vadim Rygalov, among others, into methods on how effectively to monitor greenhouses in reduced pressures and potentially hostile environments highlight many current sensor shortcomings. These efforts lead, in part, to the underlying research.

In all, the data suggests that sensors such as those developed at UNF help satisfy the robust and rugged demands. They represent the low power requirement, the high accuracy required, and the response criteria for space applications, such as those in environmental systems.

## 2. SENSOR OPERATING PRINCIPLES

The detection of bio-spherical ozone and other gasses remain extremely important due to their physiological relevance. Because typical concentrations of ozone remain very low, gas sensors need high sensitivity together with good selectivity in order to avoid any cross interference with other gases. Gas sensors in the form of thin films appear more promising over the pellet form or thick film form because they are low in cost and rugged. Gas sensors using oxide semiconductors remain the subject of extensive investigation after more than four decades; primarily this focus rests on tin oxide. Indium tin oxide (ITO) thin film currently in widespread use serves as a transparent conductive electrode for several optoelectronic devices and for less studied gas sensing applications. Thin film ITO gas sensors underwent development at UNF as reported earlier (Patel *et al.* 2003, 2005-7).

Gas sensors based on ITO utilize selective chemical sensitivities of their surface to different adsorbed gases. This causes changes to the electrical resistance of the sensor. Appropriate doping in ITO provides electronic defects that increase the influence of oxygen partial pressure on electrical conductivity. Because oxygen vacancies on metal-oxide surfaces exist as electrically and chemically active, these vacancies function as n-type donors decreasing the electrical resistivity of ITO. Reducing gases such as CO, $H_2$, and alcohol vapors result in detectable decreases in the electrical resistance of n-type ITO by way of producing oxygen vacancies and conduction of electrons at higher temperatures. At high temperatures, vacancies in the oxide lattice at the gas-sensor interface occur when thermal pressure ejects the oxygen atoms. Similarly, upon adsorption of charge accepting molecules at the vacancy sites, namely from oxidizing gases such as ozone ($O_3$) and $NO_2$, these electrons effectively deplete themselves from the conduction band of the semiconductor. This leads to an increase in the electrical resistance of n-type ITO.

### 2.1 Sensor Fabrication

Fabrication of gas sensors and sensors arrays utilized thermal evaporation methods. The thermal evaporation method employed a high vacuum system consisting of a metal box chamber. Cleaned glass microscope slides and alumina served as the substrates for deposition by the different ITO thin film sensors. Indium tin oxide (ITO) bulk powder from Alfa Aesar acted as the source material. The thicknesses of the films varied from 800 to 3000Å, while the substrate temperature varied from 200 to 300$^o$C. Further, gold thin film, deposited on both sides of the ITO thin film, established ohmic electrical contacts.

Each array consists of 24 sensors fabricated on one glass slide. In one processing of the deposition, 96 sensors on four glass slides simultaneously underwent fabrication. Each sensor on the array has an active area of about 25mm x 25mm. During operation, each sensor can work individually or as part of an array, and all 24 sensors generate electrical signals simultaneously.

Of the arrays, a chosen glass slide substrate interfaced with a specially designed printed circuit board. This mounts inside a small metal box with a small fan on one side in order to blow uniformly the air molecules over all 24 sensors during operation. The mounted sensor included a flexible Kapton heater (Omega) and resistive thermal device (RTD) on the backside of the glass slide in order to allow for the ability to maintain a constant temperature across the array at about 24°C during flight. It is important to note that the power consumption is minimal, as these sensors do not require the high operating temperature, approximately 530°C, reported with the tungsten oxide sensors (Hansford et al. 2004).

### 2.2 Sensor Properties

Examination of the surface morphology of the ITO thin film used a FEI Quanta 200 Environmental Scanning Electron Microscope (ESEM). Confirmation of the chemical composition used an Energy Dispersive Analysis of X-rays (EDAX).

Electron micrograph images demonstrate these morphologies and layups as seen in Figure 1 and Figure 2.

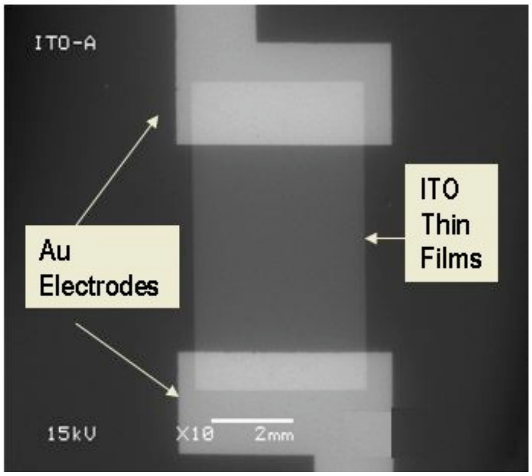


**Figure 1. Scanning Electron Micrograph of ITO Thin Film Sensor**

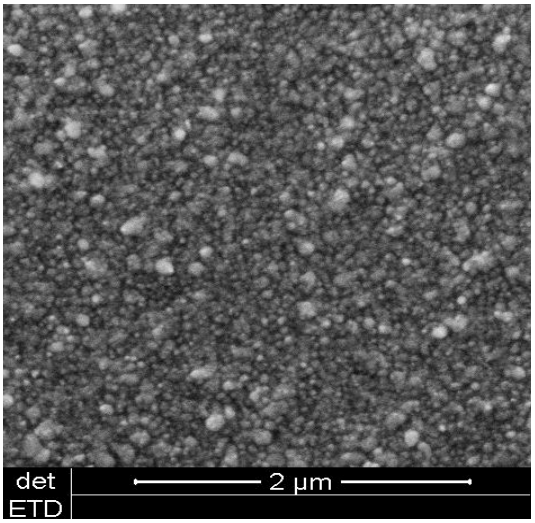


**Figure 2. Scanning Electron Micrograph of Surface of ITO Thin Film**

The first figure (Fig. 1) shows the scanning electron micrograph of a typical ITO thin film sensor with gold electrodes. The next figure (Fig. 2) shows the scanning electron micrograph surface of the ITO thin film. This micrograph revealed and confirmed that the ITO thin film has nanocrystalline grain sizes, with an average ITO grain size of 45±12 nm.

The characteristic reason for nanocrystalline ITO thin film sensors sensitivity to oxidizing ozone gas is due to this large surface to volume ratio and the large number of unsatisfied bonds in the nanocrystals. Adsorption of the electron acceptor gaseous species leads to band bending and formation of a surface depletion layer due to the capture of the free charge carrier at the surface. All available conduction electrons remain trapped in the surface states of nanocrystalline ITO, which enhances the sensor response.

## 3. HASP OVERVIEW

The NASA Balloon Program Office and the Louisiana Space Consortium at Louisiana State University support the overall High Altitude Student Platform (HASP) program. HASP enables student built payloads to fly aboard a long duration high altitude helium balloon. This zero pressure, thin-layer polyethylene (~0.8 mil), light balloon spans 400 feet in diameter and expands to 11,000,000 SCF using helium. The flight duration lasts approximately 23 hours and reaches a target altitude of 36 km (~120,000 ft). This effort provides an ideal cost-effective near space environment, offers students experience in designing and creating scientific payloads for space environments, and remains an excellent Science-Technology-Engineering-Management (STEM) development resource.

The HASP includes a standard mechanical, power and communication interface for student payloads to help prevent complexity during the integration process. Further, this system provides some flexibility to the mission requirements and launch schedules, which in turn aids the success of the student payloads.

This program fosters excitement in aerospace related careers by providing a minimal expense "space test platform" available to twelve student groups selected from respondents to the call for proposals.

### 3.1. Flight Overview

The HASP ascends at approximately 1000 feet per minute into the stratosphere. Unlike other regions of the atmosphere, the stratosphere warms with increases in altitude. During this flight, the platform experiences huge temperature variations between +40°C and -80°C as shown in Figure 3.

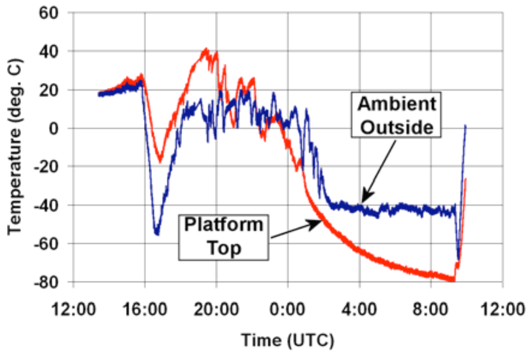


**Figure 3. Temperature Profile (Guzik *et al.* 2006)**

The stratosphere rests above the Earth's troposphere, generally considered to span from 10 km to 50 km above the surface. This temperature inversion region partially traps trace gases such as ozone, among others (*e.g.* NO2, BrO, OClO) and small particles for long periods, often for several years. This stratospheric ozone layer is within the range of the flight.

Once at altitude, the expected ozone concentration corresponds to between 10 ppm and 15 ppm as shown in Figure 4, which is in agreement with Chapman mechanism predictions.

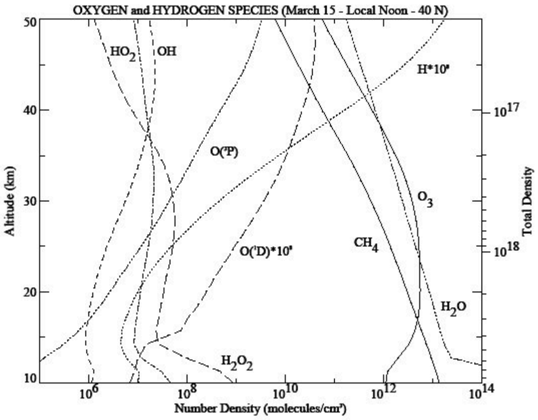


**Figure 4. Oxygen and Hydrogen Species Concentrations (Demore *et al.* 1997)**

### 3.2. Technical Overview

The HASP platform provides the BUS support for up to 12 payloads through a gondola support structure. These payloads vary in size and respective support as outlined in Table 1.

**Table 1. HASP Technical Summary**

| | **Small Student Payloads:** | **Large Student Payloads:** |
|---|---|---|
| **Available Slots:** | 8 | 4 |
| **Maximum Weight:** | 3 kg (6.6 lbs) | 20 kg (44 lbs) |
| **Maximum Footprint:** | 15 cm x 15 cm (~6"x6") | 38 cm x 30 cm (~15"x12") |
| **Maximum Height :** | 30 cm (~12") | 30 cm (~12") |
| **Supplied Voltage:** | 29-33 VDC | 29-33 VDC |
| **Available Current:** | 0.5 Amps @ 30 VDC | 2.5 Amps @ 30 VDC |
| **Maximum serial downlink (bitstream):** | 1200 bps<br>2 bytes per command | 4800 bps<br>2 bytes per command |
| **Serial interface:** | 1200 baud,<br>RS232 protocol,<br>DB9 connector | 4800 baud,<br>RS232 protocol,<br>DB9 connector |
| **Analog downlink:** | 2 channels<br>0 to 5 VDC | 2 channels<br>0 to 5 VDC |
| **Discrete commands:** | Power On,<br>Power Off | Power On,<br>Power Off |
| **Analog & discrete interface:** | EDAC 516-020 | EDAC 516-020 |

Each class of payload received their respective mounting plate as shown in Figure 5.

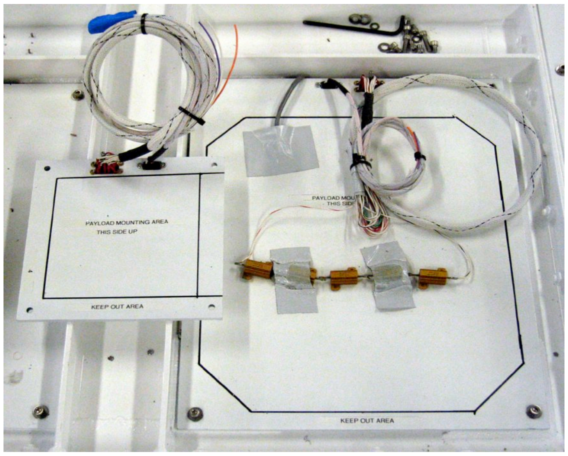


**Figure 5. Mounting Plates (Guzik *et al.* 2006)**

The HASP launches from Ft. Sumner, New Mexico in a controlled launch operation. NASA mitigates and handles technical launch procedures and issues. This includes a support launch structure as depicted in Figure 6.

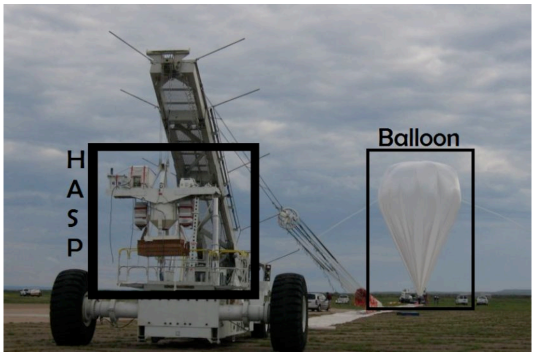


**Figure 6. HASP Pre-Launch (adapted from HASP website image courtesy of Doug Granger, Michael Stewart, Greg Guzik)**

3.3. Payload Mechanical Design

The UND and UNF payload only required the small payload having a mass of less than 2 kg (~4 lbs).

The designed support structure utilized an Al 6061-T6 alloy. The support structure includes three center holes and a corner hole to attach to the mounting plate to via carriage screws with locknuts. Similarly, in the case of the corner hole, a threaded rod runs the length of the payload and attaches at the top via a locknut to ensure the lid maintained a snug fit. Inside the supports rests a 1 cm thick R12 polyurethane rectangular housing, reducing the expected heat transfer at extremes to 0.4 W. Figure 7 illustrates the primary structure.

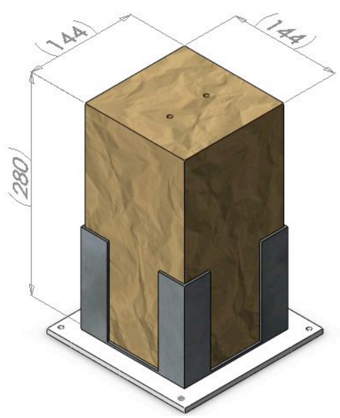


**Figure 7. Primary Structure (mm)**

The structure housed the sensor, shown on top, and the accompanying electronics, shown on bottom, via mounting screws as in Figure 8.

**Figure 8. Internal Payload**

The design accounted for thermal considerations to ensure that the payload remained within ideal operating conditions under a near zero convection state. Under these conditions, thermal insulation and heat dissipation arise as serious issues. Metalized plastic sheets (MPS), space blankets, when layered over the R12 insulation help to retain and reflect back up to 95% of the heat. The final payload structure includes Kapton (polyimide) thermally conductive tape to further support the top and side structure. The final integrated payload with HASP is shown in Figure 9.

**Figure 9. Integrated on HASP**

In addition, the heater element aboard the sensor helped to maintain the payload at the target temperature of ~24°C. The thermal design underwent validation through BEMCO vacuum and thermal testing. During these extremes, the internal temperature maximum reached and maintained 58°C. On the negative temperature swing, the internal temperature minimum reached ~11°C, whilst the heater turned off. When turned on the temperature quickly reached a temperature of 30°C whilst trying to maintain the targeted temperature of ~24°C. These conditions all fall under the well-accepted standards for the sensors' operation.

Drop testing, prior to integration, confirmed the structure design viable to withstand a vertical 10 g force and a horizontal 5 g force, as laid out in the technical requirements for HASP payloads.

### 3.4. Payload Electronics Design

The UND developed electronic circuit board measures, stores, and transmits the voltage signals from the ozone sensors as well as the two RTDs. A multiplexed Wheatstone bridge converts the resistances of each individual ozone sensor to voltage. Further, the circuit board controls the heater to maintain the targeted internal temperature. A second board supplies power for the main circuit board. The second board contains a DC-to-DC converter and a linear regulator. The power supply takes in 24-32VDC from the HASP platform and provides ±5VDC, +15VDC to the main circuit for the fan and sensor heater, respectively.

During the initial start up sequence, a solid LED indicates to the ground crew that the power is on and a blinking LED indicates the payload is actively sampling. A second LED faces the interior of the platform making it viewable from HASP's web camera, CosmoCam. HASP ground controllers initialize the payload's operation through a programmed hex command (0x7878). During normal operation, each of the 24 ozone sensors and environmental sensors sample three times every minute. Onboard memory records this data and the circuit assembles the information into a data packet that transmits the data back to the ground station. Figure 10 shows an overview of the software routines.

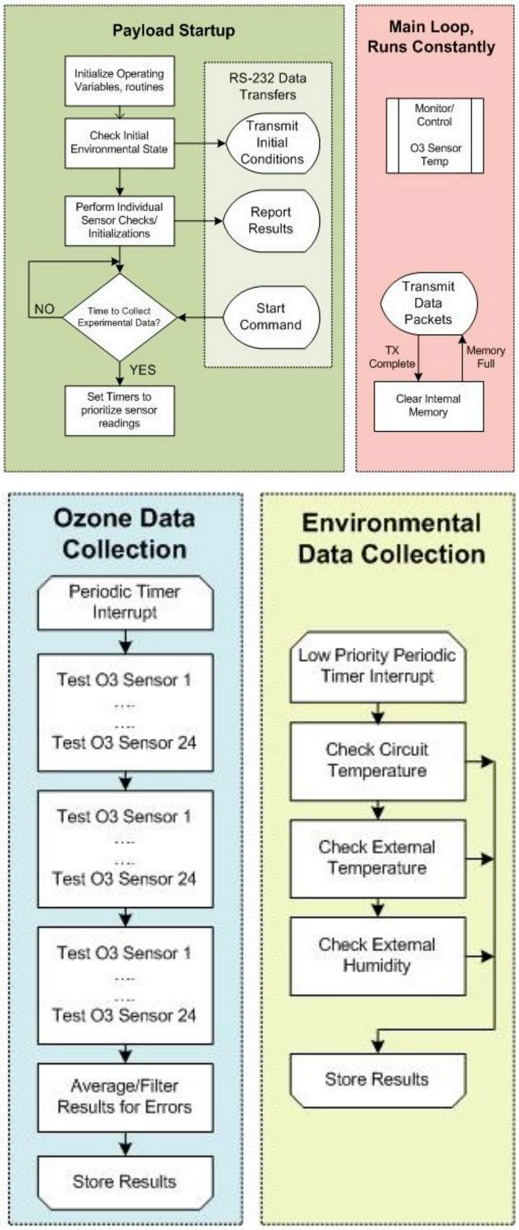


**Figure 10. Software Overview**

In the event of an anomaly, the payload operation resumes by a secondary hex command (0x7272) from HASP to the payload. In case of restart during the flight, the system checks the memory for errors and continues recording data after the last correct record.

The UND designed the printed circuit board displayed in Figure 11, and the power supply in Figure 12. After assembly, an application of conformal coating helps to protect against moisture and extreme temperatures. Figure 13 shows the coated PCB and power supply.

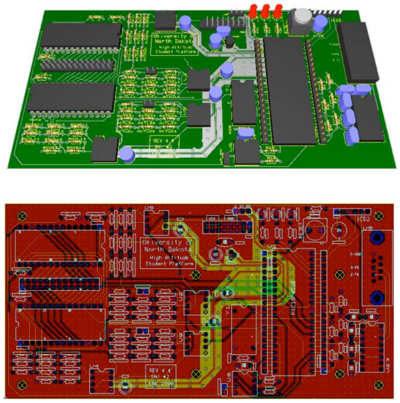


**Figure 11. PCB**

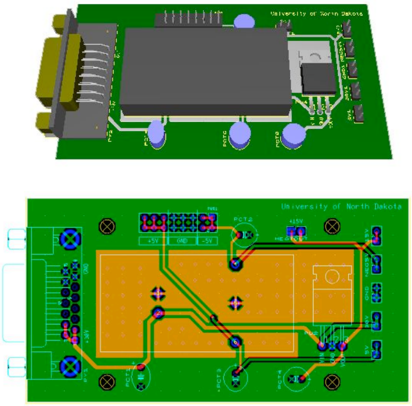


**Figure 12. Power Supply**

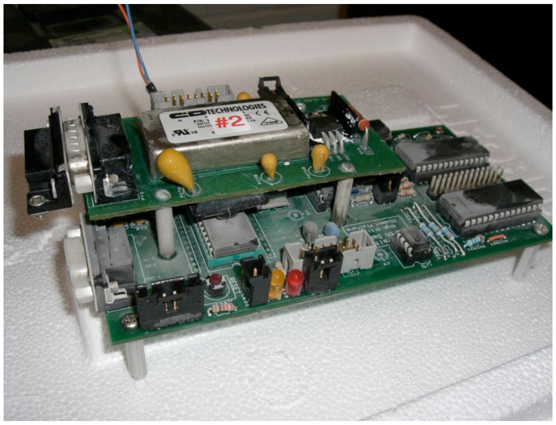


**Figure 13. Coated PCB and Power Supply**

## 4. CALIBRATION AND LAB DATA

The first step in supplementing and even replacing current ozone sensor techniques requires accurate calibration and testing. Calibration of the UNF ITO gas sensors occurred with different concentrations of ozone (0.5 ppm to 14 ppm) under different total pressures. Testing of the solid-state ITO sensors required a reduced pressure and temperature setting to mimic a near space environment. Testing occurred at the Low Pressure Test Bed at NASA's Kennedy Space Center (KSC) Space Life Science Laboratory facility, the University of North Dakota (UND), NASA's Columbia Scientific Balloon Facility (CSBF), and in the upper atmosphere aboard NASA's High Altitude Student Platform during flight. All calibration and in flight conditions remain thermally regulated to maintain the sensor at roughly 24$^{o}$C.

### 4.1 Calibration Methods

The calibration of these sensors occurred under varying ozone concentration, pressures, and ambient temperatures. This procedure required the use of a simple pressure test bed as seen in Figure 14.

**Figure 14 – UND Calibration**

The procedure at UND involved using two calibrated sources: an OMC-1108, from Ozone Solutions, and the OMZ-3400 ozone generator, from Ozone Solutions, with a known output of 3.4 g/hr from air at atmospheric pressure. The OMC-1108 was calibrated with an Advanced Pollution Instruments 401X O3 Photometric Calibrator – serial number 331. The analyzer remains actively certified under NIST standards and calibrated according to API specifications.

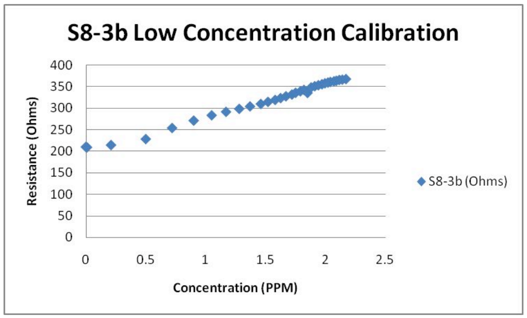
The OMZ-3400 sat internally in the 0.1058 m$^3$ chamber generating a known amount of ozone from the chamber air, and the calibrated OMC-1108 sensor further validated this calculation. Stark differences result from the high reactivity and small size of the ozone molecule, making it hard to fully contain within the chamber under steady state conditions.

Initial testing utilized the calibrated OMC-1108 to give real time readings of internal ozone partial pressure to total pressure (concentration).

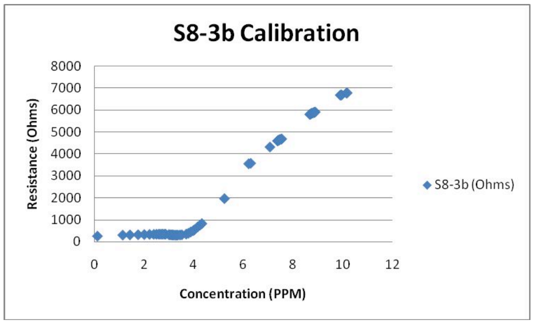
After these initial tests, the integrated payload underwent a series of tests at NASA-CSBF, Palestine, TX. Testing included a BEMCO thermal and vacuum test. During this test, the payload experienced temperatures ranging from $56^{o}$C to $-45^{o}$C and pressures from atmospheric pressure down to 1mBar pressure.

4.2 Calibration and Lab Results

Of the three sensor types tested, each underwent testing at 1.0 atm, 0.5 atm, and 0.15 atm. The chosen sensor most suited to this application is the S8-3b. There is a clear linear relationship at low concentration among all of the sensors; however, this linear relationship changes characteristic slopes at certain mid-concentrations. Graph 1 shows the clear linear relationship ($R^2$=0.99) for low concentrations of ozone, and Graph 2 demonstrates the overall general behavior of the ITO sensors.

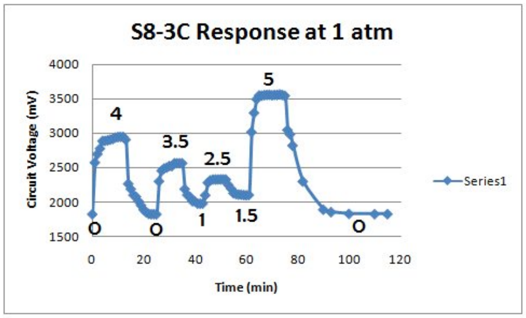


**Graph 1. S8-3b Low Concentration**



**Graph 2. S8-3b Overall Calibration**

Although the relationship is most likely a general polynomial, so far modeling includes piecewise linear slopes that correspond to the appropriate regimes. As expected, the slope (dR/dC) increases for reducing pressures.

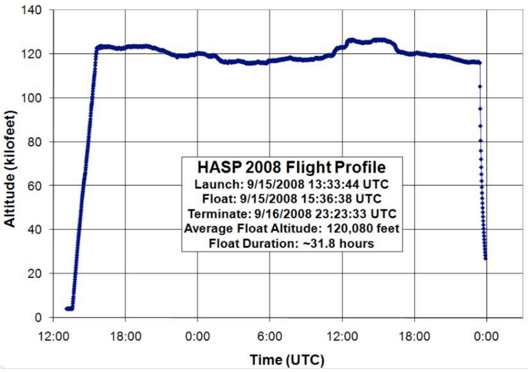


**Graph 3 – S8-3c Response**

In addition, extensive testing helps establish the expected responsiveness of the chosen S8-3b sensor. Graph 3 shows the response, where the printed numbers on each cycle represents the concentration of ozone in ppm.

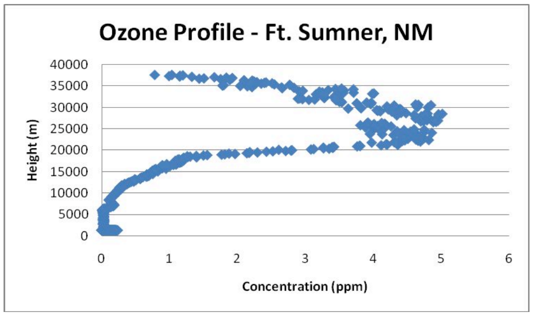
## 5. FLIGHT DATA

HASP launched on September 15, 2008 at 13:33:44 UTC from Ft. Sumner, New Mexico with operational support from CSBF. This flight had a record duration of 31.8 hours, and approximately 30 hours of flight at float. The platform maintained an average altitude at float of 36.6 km and a peak altitude of ~38.6 km. Graph 4 summarizes the HASP 2008 Flight Profile.



**Graph 4. HASP Flight Profile (courtesy of Guzik)**

An onboard test resistor measured pressure and temperature variations, these with temporal variations, once accounted for gives an accurate picture of the voltage across the sensor, and therefore, concentration. This methodology includes taking the root mean square difference between the adjusted baseline and the sensor. Graph 5 shows the resulting flight profile.



**Graph 5. Ozone Flight Profile**

Data preliminarily supports calibration and lab findings; however, the results demonstrate a lower than expected ozone concentration. Most likely explanations include Hurricane's Ike's circulation effect on the distribution and budget of ozone and other trace gasses. This is a similar phenomenon as the one noticed over East Asia in spring 2004 (Chan *et al.* 2007). Also, the model used to predict may have been prone to estimation due to its predominant dependence on Chapman mechanism predictions that fail to sufficiently capture other photolytic effects. Additional explanations could include the low-resolution bridge of the circuitry not falling within the expected values.

## 6. CONCLUSIONS AND FUTURE WORK

The lower expense and versatile nature of these ITO sensors over conventional sensors, such as ECC sensors, make them prime candidates for further research. The reusability of these sensors makes it possible to increase the frequency of gathering ground truth data regarding the upper atmosphere. Such increased frequencies could lead to a better understanding of the mechanisms of ozone depletion and help to lessen some of the potentially harmful consequences.

This research also helps to address many serious unmet needs for miniature sensors capable of in situ, real-time analysis of gases that can detect environmental factors such as contaminants in regenerative life support systems. Further, such solid-state sensors offer a method to study physical phenomena such as the mechanisms of water transfer and diffusion in reduced-pressure environments. Because of stability of sensor response with temperature, pressure, and time, gas sensors of this type are ideal candidates for use in extraterrestrial applications and space flight instrumentation. Further, the rugged nature, small size, low mass, increased response rate, and limited power demands of these sensors begin to satisfy the rigorous demands of many space flight technologies. The characterization of these sensors' behaviors, under varying conditions, remains crucial to the application of sensors in upper atmospheric applications, and space applications necessitated in the Vision for Space Exploration (VSE), including the creation of a prolonged human presence on the Lunar and Martian surfaces.

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