Chemical sensors for monitoring gas composition, including oxygen, humidity, carbon dioxide, and trace contaminants, are needed to characterize and validate spacesuit design and operating parameters. This paper reports on the first prototypes of a non-intrusive gas sensing technology based on flexible sensitive patches positioned inside spacesuit prototypes and interrogated via optical fibers routed outside the suit, taking advantage of the transparent materials of the suit prototypes. The sensitive patches are based on luminescent materials whose emission parameters vary with the partial pressure of a specific gas. Patches sensitive to carbon dioxide, humidity, and temperature have been developed, and their preliminary laboratory characterization in Mark III-like helmet parts is described. The first prototype system consists of a four-channel fiber optic luminescent detector that can be used to monitor any of the selected target gases at four locations. To switch from one gas to another we replace the (disposable) sensor patches and adjust the system settings. Repeatability among sensitive patches and of sensor performance from location to location has been confirmed, assuring that suit engineers will have flexibility in selecting multiple sensing points, fitting the sensor elements into the spacesuit, and easily repositioning the sensor elements as desired. The evaluation of the first prototype for monitoring carbon dioxide during washout studies in a spacesuit prototype is presented.

**Nomenclature**

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<th>Acronym</th>
<th>Definition</th>
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<td>avalanche photodiodes</td>
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<td>Intelligent Optical Systems, Inc.</td>
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<td>emission lifetime</td>
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<td>$p_{CO_2}$</td>
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<td>PLSS</td>
<td>portable life support system</td>
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<td>RH</td>
<td>relative humidity</td>
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<td>standard deviation</td>
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<td>SMTA</td>
<td>Suited Manikin Test Apparatus</td>
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I. Introduction

Efficiently developing a system as complex as a spacesuit requires extraordinary ground-based testing and space-based operational capabilities. Spacesuit development and testing require significant investments in sensing and analytical instrumentation, and spacesuit prototypes are equipped with dozens of physical and chemical sensors to validate their design and functionality. Chemical sensors for monitoring gas composition, including oxygen, humidity, carbon dioxide, and potential trace contaminants are needed to characterize and validate a spacesuit design and operating parameters (pressure, gas flow rate...). While miniature thermosensors measure reliably at low cost, and can be incorporated all around spacesuit prototypes, incorporating gas sensors at locations of interest inside a spacesuit has been a significant challenge – in particular for human subject tests – because of the size and cost of available instrumentation. The sensor probes and cables must not restrict the suit or human subjects’ mobility, and must not disturb the gas flow. Relocating gas sensors, when possible, requires modifying the laboratory models, and can be time and resource consuming.

This paper presents (1) a sensor technology based on luminescent flexible sensing patches designed to nonintrusively monitor critical life support gas constituents and trace contaminants in spacesuits in situ, and in real time, and (2) the results obtained with the first demonstrator system. The sensing patches are positioned inside spacesuit prototypes, and their luminescence is interrogated via optical fibers routed outside the suit. The hemispheric dome of the helmet is transparent (usually made of polycarbonate), as are the entire spacesuit models used at Johnson Space Center for testing and characterization with mannequins (usually made of urethane). This makes it practical to put luminescent sensor coatings (patches) inside the prototypes and detect the luminescent signal through the plastic walls, via thin optical fibers. The patches can be used to monitor multiple locations; they are easy to reposition, do not interact with the air flow, and do not disturb the subject, if there is one.

The sensor element comprises a luminescent indicator whose emission lifetime depends on the target gas concentration, supported in a chemically and mechanically passive multi-layer polymer film. Since measurements that rely on emission lifetime are insensitive to fluctuations in the intensity of the excitation light, to orientation of the optical fiber, to and light reflections in the spacesuit material, the measurements are reliable and stable. Each sensitive material is specifically sensitive to one target gas, so interference among gases is avoided.

In characterizing the composition of gas in spacesuits, the primary interest is in carbon dioxide levels, particularly in the helmet, in order to meet gas level requirements established by NASA. Removal of metabolic CO₂ is critical to the development of advanced spacesuit technologies, and significant investment has gone into improving the current system in the ISS. Characterization of advanced spacesuits and portable life support subsystems involves detailed and extensive evaluation of CO₂ removal under all operating conditions (supply air/oxygen flow rate, metabolic workload...). Carbon dioxide monitoring instrumentation is critical to this. Two types of monitors are typically used: (1) sensor probes such as the Vaisala GM Series, and (2) gas analyzers such as the Picarro G2301/G2401 units, the AEI Technologies CD-3A model, and the more economical LI-COR instruments. There are significant limitations on fitting sensor probes inside a suit, even with mannequins, and particularly during human subject testing. Accurate gas samplers and analyzers are costly, limiting the number of simultaneous monitoring locations. Nevertheless, monitoring CO₂ at multiple locations inside the suit and more importantly inside the helmet is necessary to assure that it is not accumulating, and to understand CO₂ washout and removal. Thus, carbon dioxide-sensitive patches have been the focus of our initial development.

II. Phase-Resolved Luminescence Detection

The main challenge for interrogating luminescent sensor coatings through a transparent material with an optical fiber is maintaining repeatable, well-calibrated readings. Emission intensity measurements are affected by the dimensions of the optical fibers, mechanically-induced variations in fiber transmission, and variation in the thickness of the sensor element. For this application, intensity measurements also depend on the alignment of the optical fiber and the sensitive patch, and on the optical transmission of the material of the spacesuit prototype, limiting repeatability and reliability.

In contrast, time domain measurements, which rely on the fluorescence lifetime (the delay between the arrival of the excitation light and emission of photons by the indicator dye), are insensitive to these variables (as long as a minimum level of light is transmitted), making it a reliable, stable measurement. In our work we have used phase-resolved luminescence measurements as an indirect means of determining the mission lifetime of the optical sensor patches. We used a compact four channel phase-resolved luminescence measurement system initially developed for gas monitoring in the portable life support system. This instrumentation measures phase shift between the excitation signal and the sensor signal, when the excitation light is modulated at a particular frequency following a sinusoidal waveform.

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For phase-resolved measurements, the instrument generates a continuous sinusoidal waveform at a programmable known frequency that modulates the light illuminating the indicator on the patch. As a result, the luminescence signal from the indicator dye is intensity modulated at the same frequency as the excitation source. However, because of the finite lifetime of the dye’s excited state, there is a phase delay between the excitation signal and the sensor signal (Figure 1). An estimate of the fluorescence lifetime of the indicator can then be computed by measuring the phase ($\phi$) shift between the excitation and sensor signals ($\tan\phi = 2\pi f\tau$), where $f$ is the modulation frequency and $\tau$ is the emission lifetime of the probe.

![Figure 1. (left) Excitation signal modulated in intensity, emission signal modulated at the same frequency, and phase shift resulting from the luminescence kinetics; (right) fiber optic phase-resolved luminescence detectors for sensor interrogation.](image)

IOS has developed sensors based on indicator chemistry whose emission – and the variations therein caused by the interaction with the target gas – can be determined by phase-resolved luminescence measurement.

### III. Fabrication of Sensor Patches and Optical Cables

#### A. Fabrication of Sensor Patches

Sensor patches for carbon dioxide, relative humidity, oxygen, ammonia, and temperature were fabricated, using sensor materials previously developed by IOS for gas monitoring in the portable life support system (PLSS). In this paper, only the results obtained with the CO$_2$, relative humidity, and temperature patches are shown; those obtained with the sensors for oxygen and ammonia will be included in future papers. The sensor patch design consists of three layers: (1) an external layer, which is gas permeable and opaque, and incorporates a ring of adhesive to attach the patch to the helmet; (2) sensitive film incorporating luminescent chemistry reactive with the target gas; and (3) plastic backing that supports the flexible sensitive film. Figure 2 shows a CO$_2$ sensor patch attached to a plastic square about as thick as a Mark III helmet.

![Figure 2. CO$_2$ sensor patch attached to a Mark III helmet-like plastic part.](image)

#### B. Fabrication of Optical Cables

Our purpose in this initial study was to demonstrate proper signal collection through flexible optical cables. The optical cables are bifurcated fiber optic bundles, as are those typically used for fluorescence measurements. In order
to guide light efficiently between patches flat on the surface and fiber cables also parallel to the helmet surface, we attach prisms on the tips of the common end of optical cables to maximize reflection of light at 90°, optimal for patch illumination and luminescence collection. Figure 3 shows two optical cable prototypes that we fabricated and evaluated.

Figure 3. Optical cable prototypes fabricated with 10 mm and 14 mm prisms placed on the common end of the bifurcated fiber optic bundles.

C. Repeatability Among Patches and Among Sensor Locations

In order to determine the gas levels, it is necessary to establish a correlation between the luminescence signal (phase shift) and the concentration of gas, which determines the calibration function. A critical aspect of this technology is designing the optical cables and patches to assure that the sensor signal recorded, for a set level of gas, is repeatable when different patches are used and when the patches and optical cables are relocated on the helmet. That enables us to establish the sensor calibration function, and thus to calculate the gas concentration.

In order to evaluate this repeatability, we fabricated two Mark III-simulating helmet parts with the same dome diameter and wall thickness as the Mark III helmet. One of these parts was designed as a chamber with input and output ports for flowing gases to determine sensor analytical characteristics (Figure 4). This design enabled us to evaluate the effect of the curvature of the helmet on the coupling of the optical cable with the sensor patches.

Figure 4. Mark III helmet-like parts fabricated for optical cable testing and sensor evaluation.
To validate the optical cable performance, we fabricated sensor patches and placed them inside the helmet-like parts. Tests were conducted to evaluate the repeatability of the phase measurements when: (1) the optical cables are installed and reinstalled multiple times for reading a particular patch, and (2) a particular sensor patch is relocated multiple times around the surface of the helmet.

1. Test 1: Optical Cable Placement Repeatability – Single Measurement

To perform this first study, the system was completely set up in the helmet-like part with CO₂ sensor patches affixed to the inside of the helmet prototype and a fiber optic cable attached on the outside corresponding to each patch. Initially, the optical cable was installed on top of the sensor patch, removed, and reinstalled multiple times, with phase shift measured every time at ambient conditions. The standard deviation between measurements was 0.24° when the cable equipped with the 10 mm prism was used, which corresponds to a deviation of 0.1 mmHg of pCO₂ at 8 mmHg pCO₂ and 0.3 mmHg of pCO₂ at 16 mmHg, at ambient temperature and 50% RH.

2. Test 1b: Optical Cable Placement Repeatability – Full Calibration Test

A standard test was then run at ambient temperature under varying humidity (30%-50%-80% RH) with the patches being exposed to 0–0.2%–0.5%–1%–2% CO₂. CO₂ sensor patches and relative humidity sensor patches were used to perform this study. After the test, the fiber optic cables were completely removed and stored, and the system shut down. The following day the fiber optic cables were reattached and the test was rerun. Figure 5 shows the results of this test, displaying accuracy better than 1.5% between days, with the variation attributable to a slight variation in ambient temperature. Temperature monitoring was not conducted during these tests, but was implemented later as recorded in the calibration tests below.

3. Test 2: Sensor Patch Placement Repeatability

A CO₂ sensor patch was placed on the helmet part, and then the optical cable on the top of it, and phase measurements were taken. The sensor and cable were then relocated multiple times to other locations inside the helmet, and measurements were taken each time. The standard deviation between measurements was 0.26° or lower (similar to Test 1), which corresponds to a deviation of around 0.1 mmHg of pCO₂ at 8 mmHg pCO₂ and 0.3 mmHg of pCO₂ at 16 mmHg, at ambient temperature and 50% RH.

Table 1 records the results of Tests 1a and 2. The sensor was relocated to five spots: base plate, bottom right, bottom left, top right, and top left; and phase and amplitude were recorded five times at each spot, using two different optical cables.

Figure 5. (left) Response profile of two sensor patches for CO₂ and two sensor patches for humidity when exposed to three levels of humidity and five levels of CO₂, recorded on 11/06/2014 and 11/07/2014, with the optical cables dismantled and reassembled between tests. (right) Comparison of the calibration functions for a CO₂ patch at three levels of humidity (phase vs. pCO₂) during tests conducted on two consecutive days.
Table 1. Repeatability of Optical Cable and Sensor Patch Placement*

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* Phase in degrees of angle; amplitude in arbitrary units. SD: standard deviation. MED: median.

The repeatability determined for these three tests validates the optical cable design and assures that the calibration functions (phase measurement vs. CO₂ partial pressure) determined in the laboratory on a helmet part will be relevant later when the sensor patches are placed in the helmet in multiple locations, and when the optical cables are installed and reinstalled multiple times.

D. CO₂ Sensor Patch Repeatability

A set of CO₂ sensor patches were fabricated and tested for reproducibility. Good reproducibility in the calibration function among 12 sensor patches was observed, as shown in Figure 6, with slight variations in the offset and slope of the calibration curves. Based on these results, calibration functions established under a range of conditions of temperature, humidity, and pressure for a particular patch and unit can be applied to all carbon dioxide sensor patches and units, applying a three-point recalibration (conducted at ambient conditions) to compensate for offset and slope differences, in order to maintain accuracy.

Figure 6. Comparison of the calibration functions (phase vs. pCO₂) for 12 CO₂ patches at ambient temperature and 50% RH.

IV. Demonstrator Unit Assembly and Calibration Tests

Two demonstrator system were assembled, each of them including a four-channel luminescence phase-resolved detector (developed by IOS for the gas sensors for the PLSS), and optical cable, with two CO₂ sensor patches, one humidity sensor patch, and one temperature sensor patch. The readout units are powered at 125 V DC and connected.
with a laptop via a standard USB cable. The LabVIEW user interface was developed for the gas sensor system for the PLSS, and through it we can record phase shift and amplitude (emission intensity).

Each set of four sensor patches was set up in a flow-through cell and connected to the corresponding readout unit through a four channel optical cable. Calibration tests were conducted over a range of relative humidity levels and temperatures, with the flow-through cell in a climate chamber. At each environmental condition, CO₂ levels from 0 to 2% (0.0, 0.2%, 0.5%, 1.0%, and 2.0%) in nitrogen were generated by volumetric mixing of carbon dioxide and nitrogen, and the sensors were exposed to them twice. The flow rate of gas components was controlled precisely by programmable digital mass flow controllers. The phase shift measurements recorded for a set of sensors during a typical full calibration test is shown in Figure 8. The sensitivity of the CO₂ patches varied as expected with both temperature and humidity. Each sensor signal was acquired for 250 ms per s. Data was only recorded once every 5 s to avoid collecting an excessive number of data points and to avoid unnecessary sensor exposure to excitation light.

Based on the data recorded during the full calibration tests and data previously recorded with twelve CO₂ sensor patches, four humidity sensor patches, and four temperature sensor patches at ambient temperature and 50% RH,
generic calibration algorithms were developed for each of these three parameters. A LabVIEW-based calibration application was created to validate the algorithm. Using the generic calibration algorithm and one (for the temperature patches), two (for the humidity patches) or three (for the CO2 patches) experimental data points (i.e., phase vs pCO2) taken with each individual sensor patch, calibration tables for each patch were created and used to calculate temperature, humidity, and partial pressure of CO2 from the phase shift measurements. The result of applying the calibration algorithm to the data plotted in Figure 8 is shown in Figure 9. The algorithms apply temperature compensation to the humidity patches, and temperature and humidity compensation to the CO2 patches.

Figure 9. (top) Calculated CO2, humidity, and temperature applying the calibration algorithms to the phase shift data recorded during a full calibration test; (bottom) detail of the data calculated at 18°C, expanded for clarity.
V. Validation Tests in a Mark III Spacesuit Prototype

The two demonstrator systems were taken to the JSC for validation in the Suited Manikin Test Apparatus (SMTA). The SMTA is a sophisticated system designed for developing and testing advanced spacesuit systems and portable life support systems and elements included in both systems. It is designed to evaluate gas flow and composition under a wide range of operating conditions. Part of the SMTA is a Mark III spacesuit prototype fabricated in polyurethane.

A test protocol including steady state conditions of humidity, carbon dioxide, and pressure was generated, with the double objective of demonstrating the technology operation and of finding practical limitations of the initial design, which could be corrected in future versions. Conducting actual CO₂ washout studies was not part of the testing plan.

The sensor patches were installed in the helmet-like part of the SMTA. The optical cables were attached and connected to the readout units, and proper signal recording was confirmed. The installation of the sensors did not require any modification of the SMTA, and the entire deployment and checking of the two demonstration systems was conducted in approximately two hours by one person.

Both sensor sets were placed near the gas outlet in front of the mouth of the manikin: one set at the right side and one set at the left (Figure 10). All sensor patches were installed in that area to facilitate rapid gas level stabilization so that as many tests as possible could be performed during the two days allocated for testing.

![Figure 10. (left) Sensor patches before we attached the optical cables (four patches at center) and after we attached the optical cables (right) on the Mark III helmet prototype. (right) View from inside the helmet after we placed the sensor patches. In the center of the helmet are a temperature sensor and corresponding cables, part of the SMTA.]

Both the sensor patches and the prisms at the ends of the optical cables were protected from ambient light to avoid saturating the photodetector, a photomultiplier tube. An advanced generation of the readout units has been equipped with avalanche photodiodes (APD) instead of photomultipliers in order to avoid saturation by ambient light. Nevertheless, as shown in Figure 10 (right), even with the first version of the system demonstrator the sensors and cables can be placed in the helmet without significantly disrupting the view of a human subject. In addition, in the next system generation, the diameter of the optical cables could be reduced to improve the use of the sensor patches in human subject tests. In future human subject tests, the flexible optical cables will be directed towards the back of the helmet and fixed to the suit so as not to interfere with subject movement.

Tests were conducted (Figure 11) according to a pre-defined test protocol designed to demonstrate proper sensor operation and precision under a range of environmental conditions. According to our established protocol for the demonstrator system, we adjusted the temperature, humidity, and CO₂ sensor calibration using one, two, and three experimental data points, respectively.
The results collected in one of the tests conducted in the humidity range from 30% to 50% RH, together with the readings of the probes of the SMTA, are recorded in Figures 12 and 13. The correlation between the measurements taken by the sensor patches and the data collected by the SMTA probes is excellent, as is the repeatability of the CO₂ reading throughout the test. Because IOS and SMTA probes record data at different frequencies, the initial synchronization between the data taken with IOS and NASA instrumentation is not optimal, as seen in the graphs. Note that the SMTA probes and the sensor patches are located in different areas and may reflect actual differences in the gas composition at different times.
the synchronization between the data collected by IOS and NASA instrumentation is not perfect, and conclusions should not be drawn about the kinetics of the process.

Figure 13. CO₂, humidity, and temperature recorded by IOS sensor system (Blue unit) and by SMTA probes during one of the tests. Amplification of the data over a 30minute period is shown for clarity. Note that the synchronization between the data collected by IOS and NASA instrumentation is not perfect, and conclusions should not be drawn about the kinetics of the process.

Figure 14 shows the readings for pCO₂, RH, and temperature recorded by the IOS sensor system and by the SMTA CO₂ and RH probes when the tests were conducted at low humidity (RH <30% RH). There was excellent correlation between the measurements taken by the IOS system and by the instrumentation in the SMTA.
Figure 14. CO₂, humidity, and temperature recorded by IOS sensor systems (Green and Blue units) and by SMTA probes during one of the tests conducted. Note that the synchronization between the data collected by IOS and NASA instrumentation is not perfect, and conclusions should not be drawn about the kinetics of the process.

With the validation of the two system demonstrators in the SMTA and the Johnson Space Center, the feasibility of the technology was demonstrated.

VI. Conclusion

A novel approach for chemical monitoring in spacesuit prototypes based on luminescent patches has been developed and demonstrated for the first time in a spacesuit prototype. This approach exhibits significant advantages for spacesuit development in comparison with current off-the-shelf instrumentation. Flexible sensitive patches inside prototype spacesuits, interrogated via optical fibers, do not disturb the gas flow or the human subject, making it easier to select multiple sensing points, fit the sensor elements into the spacesuit, and cost effectively relocate the sensor elements as desired.

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Sensor patches for monitoring partial pressure of CO₂, partial pressure of water (or relative humidity) and temperature are presented in this paper. Additional sensors for monitoring partial pressure of oxygen and ammonia have also been demonstrated, and the results will be presented in future papers.

Two first demonstrator systems have been tested in a Mark III spacesuit prototype fabricated in polyurethane, and the reading of the CO₂, humidity, and temperature sensor exhibited good correlation with the data from established sensor instruments in a set of tests conducted at steady state conditions.

These initial tests have served to demonstrate the feasibility of the technology and to identify limitations of the first generation design, which will be corrected in advanced versions of the system.

Acknowledgments

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