

molecules, which may provide clues about the presence of past or extant biology. GSFC has successfully demonstrated that the microfluidic analytical chip is able to separate the amino acids glycine and leucine, as well as the chiral amino acids L-valine and D-valine.

The microscale liquid chromatography analytic column is suitable for miniaturized liquid chromatography and mass spectrometry. Its serpentine microchannel in the microfluidic chip

provides up to 100-mm length for analyte molecules interacting in the column. The 100-mm length of microchannel is compatible with commercial analytic column for better separation. It is easy to integrate other electronic devices on the chip such as a micro heater and temperature sensor to monitor and control liquid temperature. The chip can stand up to 4,000 psi ( $\approx 27.6$  MPa) pressure, which is much higher than a polymer-made lab-on-a-

chip. Silicon and Pyrex microchannels can be used in a wide range of solutions, including strong acid and base solutions. It will not contaminate the analyte molecules. Also, the cost of the microscale analytic column is much less than a commercial column.

*This work was done by Yun Zheng, Stephanie Getty, Jason Dworkin, Manuel Balvin, and Carl Kotecki of Goddard Space Flight Center. Further information is contained in a TSP (see page 1). GSC-16517-1*

## Spectroscopic Determination of Trace Contaminants in High-Purity Oxygen

**A glow discharge emission system is used to detect and quantify trace amounts of argon in pure oxygen.**

*Lyndon B. Johnson Space Center, Houston, Texas*

Oxygen used for extravehicular activities (EVAs) must be free of contaminants because a difference in a few tenths of a percent of argon or nitrogen content can mean significant reduction in available EVA time. These inert gases build up in the extravehicular mobility unit because they are not metabolized or scrubbed from the atmosphere. A prototype optical emission technique capable of detecting argon and nitrogen below 0.1% in oxygen has been developed. This instrument uses a glow discharge in reduced-pressure gas to produce atomic emission from the species present. Because the atomic emission lines from oxygen, nitrogen, and argon are discrete, and in many cases well-separated, trace amounts of argon and nitrogen can be detected in the ultraviolet and visible spectrum. This is a straightforward, direct measurement of the target contaminants, and may lend itself to a device capable of on-orbit verification of oxygen purity.

A glow discharge is a plasma formed in a low-pressure (1 to 10 Torr) gas cell between two electrodes. Depending on the configuration, voltages ranging from 200 V and above are required to sustain the discharge. In the discharge region, the gas is ionized and a certain population is in the excited state. Light is produced by the transitions from the excited states formed in the plasma to the ground state. The spectrum consists of discrete, narrow emission lines for the atomic species, and broader peaks that may appear as a manifold for molecular species such as  $O_2$  and  $N_2$ , the wave-

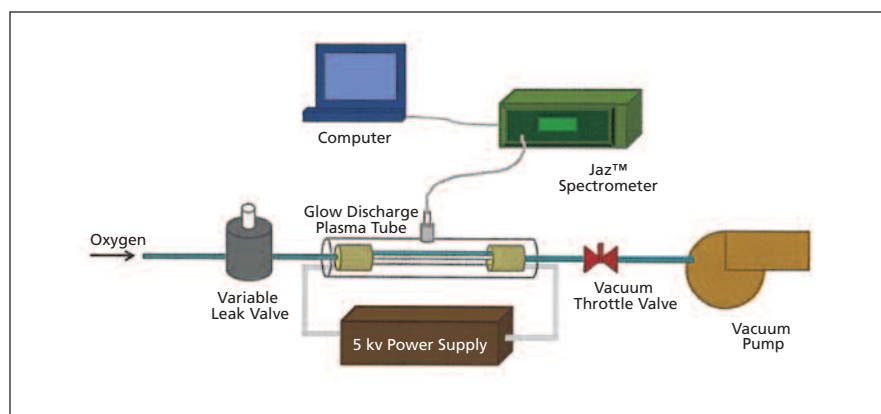


Figure 1. A schematic diagram of the **Glow Discharge System**.

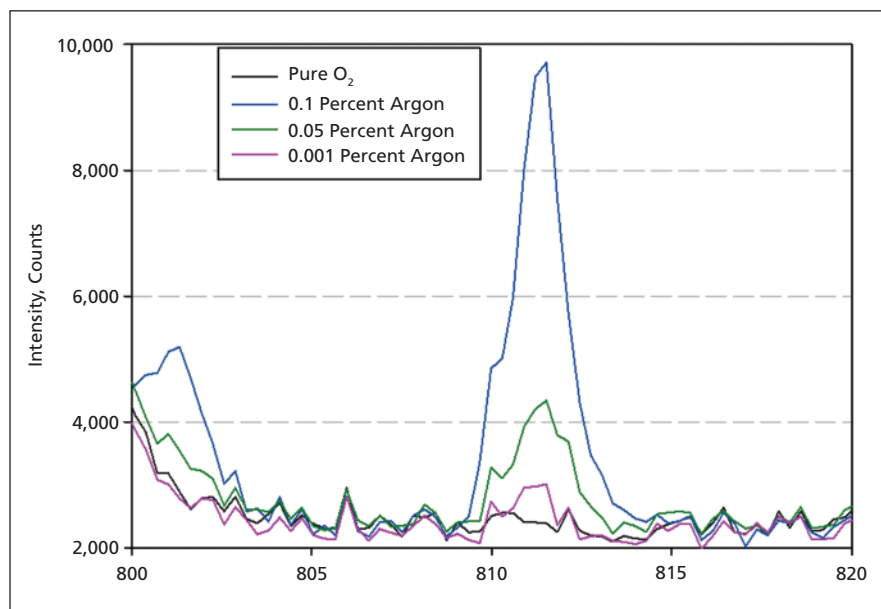


Figure 2. Region around 811-nm argon shows **Peak for Pure Oxygen** and 0.1, 0.05, and 0.01 percent argon in oxygen.

lengths and intensities of which are a characteristic of each atom. The oxygen emission is dominated by two peaks at 777 and 844 nm.

For testing, a quartz capillary tube with stainless steel end fittings forms the glow discharge tube. The sample gas is introduced into the glow discharge cell using an adjustable vacuum leak valve. From the glow discharge cell, the sample gas passes a vacuum gauge, the downstream valve, and then the vacuum pump. During operation, the pressure in the glow discharge cell is maintained between 0.5 and 10 Torr using the adjustable leak valve and the downstream valve. Light from the discharge is collected by a lens and coupled to a UV-visible fiber-optic cable. This cable directs the light from the glow discharge into a

spectrometer. The spectrometer detects in the 200- to 850-nm region with a spectral resolution of 1.5 nm using a 25- $\mu$ m entrance slit. The spectrometer is connected to a data acquisition computer via a USB cable. For this work, Ocean Optics' SpectraSuite<sup>®</sup> software was used for the data acquisition, setting parameters such as wavelength range, integration times, and scans to average.

For a peak to be at the detection limit, it must be recognizable as a peak, be resolved from other peaks, and have a peak intensity three times the standard deviation of background noise in the region of the peak. The peak corresponding to 0.01% argon is just above the baseline of pure oxygen (see Figure 2), and the signal-to-noise ratio is 2.6, indicating the detection limit is between

0.05 and 0.01%.

This work represents a proof-of-concept investigation into using a glow discharge emission system to detect and quantitate trace amounts of argon in pure oxygen. A similar analysis will need to be done for nitrogen. Optimization of experimental parameters such as operating pressure, discharge current, voltage, and spectrometer integration time needs to be further investigated. A redesigned discharge cell that will use a lower-voltage DC power supply with a higher discharge current is being designed to provide a spectrally brighter, lower-noise glow discharge.

*This work was done by Steven Hornung of Johnson Space Center. Further information is contained in a TSP (see page 1). MSC-25116*

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## Method of Separating Oxygen From Spacecraft Cabin Air to Enable Extravehicular Activities

*Lyndon B. Johnson Space Center, Houston, Texas*

Extravehicular activities (EVAs) require high-pressure, high-purity oxygen. Shuttle EVAs use oxygen that is stored and transported as a cryogenic fluid. EVAs on the International Space Station (ISS) presently use the Shuttle cryo O<sub>2</sub>, which is transported to the ISS using a transfer hose. The fluid is compressed to elevated pressures and stored as a high-pressure gas. With the retirement of the shuttle, NASA has been searching for ways to deliver oxygen to fill the high-pressure oxygen tanks on the ISS.

A method was developed using low-pressure oxygen generated onboard the ISS and released into ISS cabin air, filtering the oxygen from ISS cabin air using

a pressure swing absorber to generate a low-pressure (high-purity) oxygen stream, compressing the oxygen with a mechanical compressor, and transferring the high-pressure, high-purity oxygen to ISS storage tanks. The pressure swing absorber (PSA) can be either a two-stage device, or a single-stage device, depending on the type of sorbent used. The key is to produce a stream with oxygen purity greater than 99.5 percent. The separator can be a PSA device, or a VPSA device (that uses both vacuum and pressure for the gas separation). The compressor is a multi-stage mechanical compressor. If the gas flow rates are on the order of 5 to 10 lb ( $\approx$ 2.3 to 4.6 kg)

per day, the compressor can be relatively small [3 $\times$ 16 $\times$ 16 in. ( $\approx$ 8 $\times$ 41 $\times$ 41 cm)].

Any spacecraft system, or other remote location that has a supply of low-pressure oxygen, a method of separating oxygen from cabin air, and a method of compressing the enriched oxygen stream, has the possibility of having a regenerable supply of high-pressure, high-purity oxygen that is compact, simple, and safe. If cabin air is modified so there is very little argon, the separator can be smaller, simpler, and use less power.

*This work was done by John C. Graf of Johnson Space Center. Further information is contained in a TSP (see page 1). MSC-24806-1*

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## Atomic Force Microscope Mediated Chromatography Trace-chemical and microfluidic analyses are taken to higher precision.

*NASA's Jet Propulsion Laboratory, Pasadena, California*

The atomic force microscope (AFM) is used to inject a sample, provide shear-driven liquid flow over a functionalized substrate, and detect separated components. This is demonstrated using lipophilic dyes and normal phase chromatography. A significant reduction in both size and separation time scales is achieved with a 25-micron-length col-

umn scale, and one-second separation times. The approach has general applications to trace chemical and microfluidic analysis.

The AFM is now a common tool for ultra-microscopy and nanotechnology. It has also been demonstrated to provide a number of microfluidic functions necessary for miniaturized chromatography.

These include injection of sub-femtoliter samples, fluidic switching, and shear-driven pumping. The AFM probe tip can be used to selectively remove surface layers for subsequent microchemical analysis using infrared and tip-enhanced Raman spectroscopy. With its ability to image individual atoms, the AFM is a remarkably sensitive detector that can be