

An Automated Oxygen Diffusion Measurement System for Porous Media in Microgravity

Scott B. Jones
Utah State University

Gail E. Bingham and T. Shane Topham
Space Dynamics Laboratory

Dani Or
University of Connecticut

Igor G. Podolsky and Oleg Strugov
Institute of Biomedical Problems

Copyright © 2003 SAE International

ABSTRACT

Liquid and gas exchange within a particulate plant-rooting medium is likely to be altered in a microgravity environment. A difference in gravitational force can result in significant offsets in control parameters developed on earth for optimum plant growth, due to the shift in hydrostatic water distribution. The experiment being developed will examine the effects of variable gravity on water distribution and gas diffusion. We are developing and testing an automated gas diffusion measurement system for use on the International Space Station (ISS). To allow comparison of μg and 1g conditions, gas diffusion cell designs were horizontally oriented to minimize gravitational effects using 1) a 'thin rectangular profile' cell and 2) a cylindrical cell design for flight. Electronic solenoid valves provide air and water flow control while pressure transducers measure water and substrate potential. Porous media water content is controlled using a porous membrane coupled with a metered pumping system. Diffusion measurements are made after purging two gas chambers separated by the porous medium with N_2 and with atmospheric air (~20% O_2). The system allows continuous measurement of oxygen concentration for fitting the diffusion coefficient to measured data at a given water content set point. The entire measurement range includes 10 set points, which can be completed in a period of about 2 weeks. The LADA control system aboard ISS will provide control and monitoring capabilities for the ORZS system that will be launched to the ISS on a Russian Progress vehicle in 2005.

INTRODUCTION

Gas exchange within porous plant growth media is a critical process for plant root and microbial growth and maintenance. The porous medium provides a root support network that also facilitates liquid and nutrient supply and storage. Root and microbial respiration generate oxygen and carbon dioxide gradients between surface concentrations and those within the profile of the porous medium. A number of factors influence the respiration rate such as temperature, nutrient availability, photosynthesis, and water content. Higher water content leads to reduced gas exchange because diffusion of gases through the water phase occurs at rates 4 orders of magnitude less than diffusion through the air phase. The gas diffusion process is air-filled porosity dependent and is often described in terms of the gas diffusion coefficient. Measurement and modeling of porous media physical characteristics are needed to design and model improved plant rooting environments (Jones and Or, 1998; Scovazzo et al., 2001). Research with plant growth systems for the microgravity environment of space has been focused on refining our understanding and control of plant-fluid-porous medium interactions (Bugbee and Salisbury, 1989; Ivanova and Dandolov, 1992; Jones and Or, 1998; Mashinsky et al., 1994; Morrow et al., 1994). Altered porous media hydrodynamics have been noted in past studies in a microgravity environment (Bingham et al., 1996; Podolsky and Mashinsky, 1994; Yendler et al., 1996).

The ORZS (optimization of root zone substrates) research proposal was developed to provide direct measurements and modeling of plant rooting media that will be used in future ALS (advanced life support) plant growth

experiments. A key part of this effort is to obtain quantifiable wet substrate oxygen diffusion measurements for model development. While the proposed measurements appear simple and well studied in agricultural soils on earth, collecting repeatable results at high water contents in the coarse textured growth media in microgravity is significantly more complex. These measurements have not been made in microgravity previously and will require almost complete automation. Designing an experiment in which both 1g and μg measurements can be compared requires careful attention to details and subtleties. Coarse-textured plant growth media are commonly used in containerized systems where plant roots are restricted to a volume much smaller than commonly available in a native soil. The relatively small and shallow volume creates two problems, i) a reduced water storage capacity (and reduced surface area for root absorption) and ii) a perched water table at the container bottom leading to potential aeration problems (Bunt, 1988). For coarse-textured materials with a narrow pore-size distribution the hydrostatic fluid distribution creates large changes in water content within a shallow sample profile. This leads to potential order-of-magnitude differences in the associated vertically distributed gas diffusion coefficients in a 1g environment. In a microgravity environment the theoretical assumption is that the water would distribute itself uniformly within the porous medium, but air entrapment and altered hydrodynamics (Jones and Or, 1999) may significantly change fluid distribution and physical processes associated with porous medium gas exchange.

The objectives of the ORZS research project relevant to this discussion are to i) design and test a gas diffusion measurement system amenable to evaluating gas diffusion in 'coarse' textured (0.25 to 2 mm) growth media at 1g and in μg , ii) determine oxygen diffusion coefficients in plant growth media as a function of water

content at 1g for eventual comparison to measurements at μg .

DUAL CHAMBER GAS DIFFUSION MODEL

For the variable pressure environment of a spacecraft (e.g., ISS or Space Shuttle), a closed chamber is preferable to avoid convective transfer of gasses and changes in background oxygen concentration during measurement. Here we describe a gas diffusion model for a sealed dual-chamber diffusion system illustrated in Figure 1. Details on the porous medium – water content relations influencing diffusion through the porous medium are described elsewhere (Jones et al., 2002; Rolston and Moldrup, 2002).

The basis for most measurement methods of soil gas-diffusivity is the application of *Fick's second law*. The 1-D diffusive gas flux, J_g , may be written as the product of the air-filled porosity (AFP) dependent soil gas diffusion coefficient, $D_s(e)$, and gas concentration gradient between corresponding positions, x_1 and x_0 .

$$J_g = D_s(e) \frac{c_1 - c_0}{x_1 - x_0} \quad (1)$$

Analytical solutions to specific diffusion problems relate Fick's law to a physical system such as a single or double chamber device (Rolston and Moldrup, 2002). Glauz and Rolston (1989) derived an analytical solution for determining the gas diffusion coefficient using a sealed dual-chamber diffusion apparatus. Assuming a constant cross-sectional area, the model was derived in terms of chamber dimensions H, K, and L (Figure 1). Parameters describing the relative lengths were omitted in the paper of Glauz and Rolston (1989) and are given as (Rolston and Moldrup, 2002);

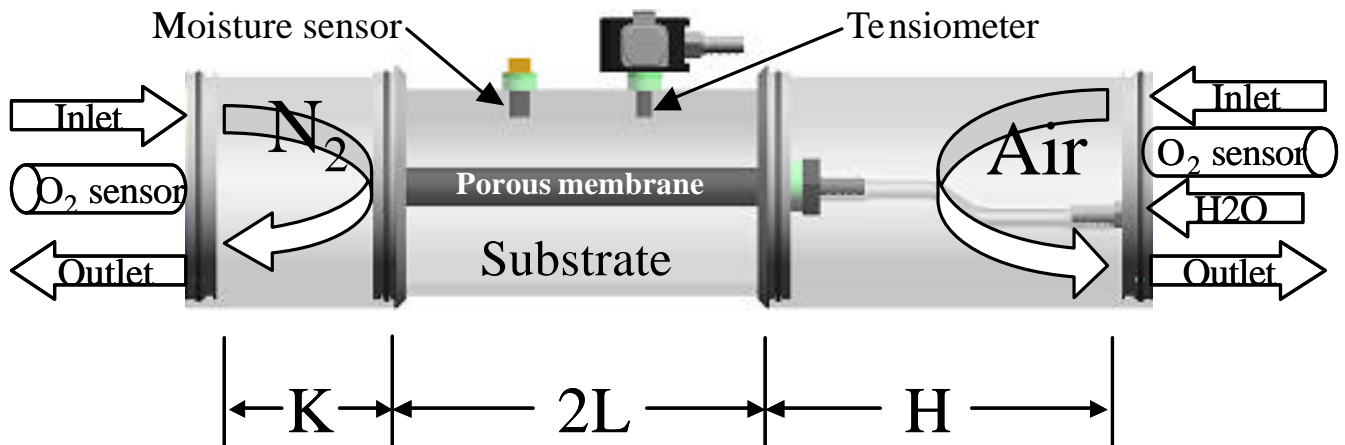


Figure 1 Flight gas diffusion chamber illustration showing a porous membrane for establishing target water content in the substrate, which is monitored with a heat-pulse moisture sensor and tensiometers. Inlet and outlet solenoid valves are actuated to prime sink (N_2) and source (Air) chambers after which oxygen sensors monitor concentration with time.

$$\mathbf{g} = \frac{H}{K}, \quad \mathbf{b} = \frac{H}{L \cdot \mathbf{e}}, \quad \mathbf{t} = \frac{D_s t}{L^2 \mathbf{e}} \quad (2)$$

Where ε is known and using measured gas concentration (c) as a function of time (t), D_s is optimized to $c(t)$ data where c_0 is the initial gas concentration.

$$c(t) = \left[\frac{1}{1 + \frac{1}{\mathbf{g}} + \frac{2}{\mathbf{b}}} - \frac{A(\mathbf{b})}{B(\mathbf{b})} \cdot \exp(-\mathbf{a}_1(\mathbf{b})^2 t) \right] - c_0 \quad (3)$$

The additional parameters $A(\beta)$, $B(\beta)$ and $\alpha_1(\beta)$, neglecting second order effects, are given as

$$\mathbf{a}_1(\mathbf{b}) = \left[\frac{1}{2 \cdot \mathbf{b}} \cdot (1 + \mathbf{g}) - \frac{1}{3 \cdot \mathbf{b}^2} \cdot (\mathbf{g}^2 - \mathbf{g} + 1) \right]^{\frac{1}{2}} + \frac{2}{45 \cdot \mathbf{b}^3} \cdot (4 \cdot \mathbf{g}^3 - 3 \cdot \mathbf{g}^2 - 3\mathbf{g} + 4) \quad (4)$$

$$A(\mathbf{b}) = \left[\mathbf{a}_1(\mathbf{b})^4 + \frac{\mathbf{g}^2}{\mathbf{b}^4} + (1 + \mathbf{g}^2) \cdot \left(\frac{\mathbf{a}_1(\mathbf{b})}{\mathbf{b}} \right)^2 \right]^{\frac{1}{2}} \quad (5)$$

$$B(\mathbf{b}) = \mathbf{a}_1(\mathbf{b})^4 \cdot \frac{\mathbf{b}}{\mathbf{g}} + \mathbf{a}_1(\mathbf{b})^2 \cdot \left(\frac{1}{\mathbf{g} \cdot \mathbf{b}} + \frac{\mathbf{g}}{\mathbf{b}} + \frac{1}{2 \cdot \mathbf{g}} + \frac{1}{2} \right) + \frac{\mathbf{g}}{\mathbf{b}^3} + \frac{\mathbf{g}}{2 \cdot \mathbf{b}^2} + \frac{1}{2 \cdot \mathbf{b}^2} \quad (6)$$

and for monitoring concentration in the sink chamber, eq. (5) is replaced with

$$A(\mathbf{b}) = -\frac{\mathbf{g}}{\mathbf{b}^2} - \frac{\mathbf{a}_1(\mathbf{b})^2}{\mathbf{g}} \quad (7)$$

DIFFUSION CELL DESCRIPTION

The ORZS diffusion cell design includes 1) a ground unit whose substrate geometry is a 'thin rectangular dimension' (Jones et al., 2002) and 2) a flight unit described in detail here. The flight diffusion cell shown in Figure 1 consists of three cylindrical chambers that are joined concentrically. Two chambers are gas filled and the center chamber is filled with substrate. The three chambers are separated at their interfaces by hydrophobic, Teflon coated screens. The screens are 74 mesh with an aperture size of 0.249 mm and have a larger open area (52.7 %) for diffusion than most substrates tested. Each gas chamber has an inlet and an outlet gas solenoid valve, which are actuated

simultaneously to prime the gas chambers. One chamber will be purged with atmospheric air (~20 % O_2) and the other with nitrogen. Each chamber also has an oxygen sensor to measure oxygen content of the gas inside the chamber over the course of measurement cycle. Both chambers are purged at the same time by opening all four valves. The gas flow rates must be similar to avoid convective transport through the substrate into the opposite chamber. Each chamber-priming event requires approximately 20 minutes at a flow rate of 65 ml/min, which was prescribed by an existing nitrogen delivery unit on the ISS. Once both gas chambers have been purged with their respective gasses the valves are closed thereby sealing the cell. The oxygen sensors in each chamber record oxygen concentration as the gasses diffuse through the substrate toward an equilibrium concentration.

WATER CONTENT CONTROL

The experiment requires testing gas diffusion through the substrate at various moisture contents. To control the moisture content of the substrate the substrate chamber contains an array of sensors and a water injection/removal system. Three sensors are used to measure water content in different ways shown in Figure 1. One of these is a heat-pulse soil moisture probe that relies on the temperature change generated by a 20 second heating period measured in the tip of the probe. Different amounts of water in the substrate influence the resulting temperature rise as heat is conducted out of the probe tip based on the thermal conductivity of the surrounding substrate. The heat-pulsed probe measurements are purely for monitoring moisture content and are not used to control moisture content.

Two versions of a tensiometer are used to measure water tension in the substrate. Both use differential pressure transducers connected to the media through a porous stainless steel interface. One tensiometer is located near the outside of the substrate chamber and uses a porous cup as its interface to the substrate. The other uses the porous tube at the center of the substrate chamber as its interface to the media. This pressure transducer doubles as a pressure limit measurement for injecting and removing water through the porous tube. During ground experiments it was noticed that atmospheric pressure changes, which were not seen inside the cell, caused offsets to the differential pressure measurements of the tensiometers. To correct for atmospheric pressure changes another differential pressure transducer is used in the air chamber of each cell to give a reference pressure difference between the inside and outside of the cell. The hydraulic schematic shown in Figure 2 illustrates water injection and removal from the substrate through the tube using a peristaltic pump. Water is delivered from a water source through a manifold to serve nine different cells at a rate of 144 μ l per revolution of the pump. Solenoid valves are opened one at a time to serve

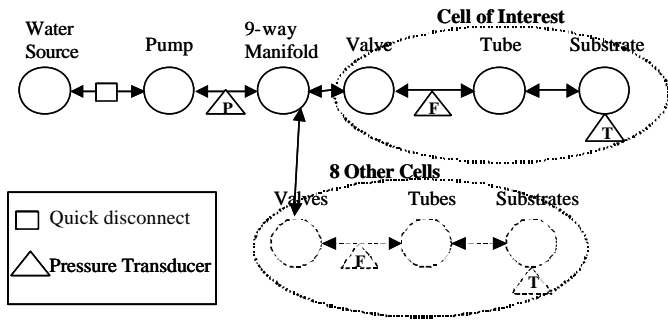


Figure 2 ORZS hydraulics diagram illustrating water flow and pressure measurement locations. Pressure transducers labeled P, F, and T refer to pump, flow, and tensiometer, respectively.

each cell. When the valve is open the tube tensiometer (pressure transducer F) measures the dynamic pressure of the water being pumped and can be used to turn the pump off if pressures get too high during pumping in or too low during pumping out. This can keep the pump from drawing air bubbles in through the tube or from pushing water too quickly allowing it to leak into the air chambers.

AIR AND NITROGEN PRIMING

A countdown timer and adjustment algorithm are used to determine timing for gas priming and diffusion. The initial time is set to the maximum time, t_{max} , which any priming or diffusion event is allowed to establish a steady-state concentration. Since the gas purge normally begins with the oxygen sensor reading difference near zero, the maximum concentration difference at the start of diffusion between the oxygen sensors, $\Delta C(t=0)$, is used as a reference. For the initial gas purge when no previous data is available, t_{max} is used for that first gas priming event. The oxygen sensor difference is monitored during priming until it crosses a threshold. The threshold for the purges is taken as a fraction, f , of $\Delta C(t=0)$, which for priming $f = 63\%$. At the time the threshold has been crossed the total purge time, t_{tot} , is calculated as $5t_p$. If the new purge time is less than the maximum time, the timer is updated with the new time. The gas purge is finished after the timer runs out. For priming, t_{max} is currently 30 minutes.

GAS DIFFUSION MEASUREMENT

Similar to priming, the diffusion measurement control algorithm uses a countdown timer to determine how long a diffusion event should last. Initially the diffusion timer is set to t_{max} , which for diffusion is currently 100 hours (4 days). The maximum concentration difference is recorded at the start of diffusion, $\Delta C(t=0)$, and the concentration difference is continually monitored and compared to a threshold value ($f \cdot \Delta C(t=0)$). At the point

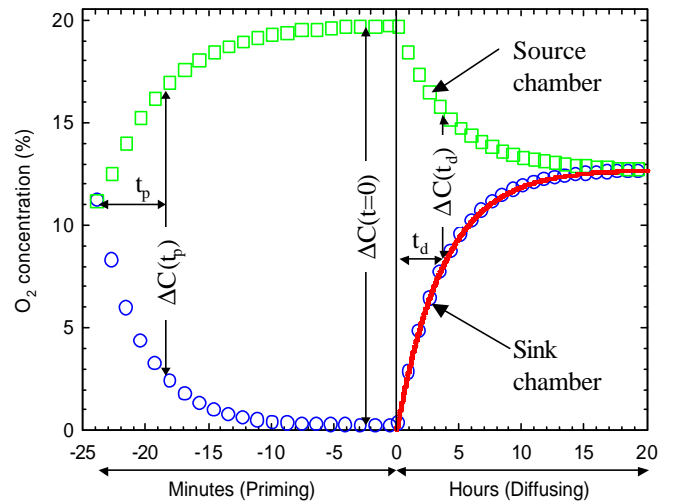


Figure 3 Measured oxygen concentration with time in the sink and source chambers. The priming process time is in minutes and diffusion in hours. The model of Glauz and Rolston (Glauz and Rolston, 1989) is used to fit the diffusion coefficient to sink chamber data shown here in 1 – 2 mm Turface particles with an air-filled porosity of 20%.

that $\Delta C = f \cdot \Delta C(t=0)$, the corresponding time, t_d , is recorded and the total diffusion time, t_{tot} is established. The diffusion experiment is terminated when the lesser of $8t_d$ or t_{max} is reached.

DIFFUSION COEFFICIENT DETERMINATION

The diffusion coefficient, D_s , was determined using measured oxygen concentration in the sink chamber from time, $t = 0$ in Figure 3 to fit the model shown in eq. (3). Data collected in the source chamber could also be used to determine D_s , providing a backup and means of double checking results. The model's ability to fit the data is dependent only upon the single fitting parameter, D_s , and measured O_2 concentrations with time. A minor offset in the measured concentrations arises from the lack of a 'gate' in the diffusion chamber, separating the substrate and sink chambers. The mathematical solution to the dual chamber diffusion problem satisfies assumptions of Fick's law where the initial boundary conditions specify a diffusing gas concentration of zero in the soil and sink (N_2) chambers and the tracer gas (O_2) concentration is initially C_0 in the source chamber. To accomplish this, a mechanical gate is typically used to seal the sink chamber during chamber priming. For an automated measurement system made up of multiple diffusion cells, a gate adds considerable complexity to the system control and maintenance. A comparison of measurements with the gate open and closed in a single chamber diffusion cell using Currie and Taylor analysis techniques (see Figure 5) revealed no significant difference in the resulting diffusion coefficients. While the lack of a gate

alters the initial conditions of the theory and may pose a short-term perturbation in the porous medium gas concentration profile, diffusion measurements were found to be well correlated with modeled results and the gate was eliminated in our design. This does require equilibrating the air priming flow rates in order to minimize convective gas transfer between air chambers and maximize the initial concentration gradient.

Galvanic oxygen sensors are used because they are inexpensive and provide a continuous monitoring capability. They act like a d.c. battery whose output voltage is linear and proportional to O₂ concentration, but whose total potential decreases under atmospheric conditions, exhibiting a shelf life of 24 months and requiring periodic recalibration. The use of oxygen as the tracer gas is a disadvantage when oxygen consumption due to microbial activity is significant and leads to over prediction of the gas diffusion rate. Oxygen concentration monitored in a 70 ml beaker showed consumption rates in Turface up to 1 % per day while additions of a carbon source (sucrose) lead to complete consumption of oxygen within 2 days.

DIFFUSION MEASUREMENT SCHEDULE

The proposed schedule for diffusion measurements begins with triplicate measurements in the substrate at the water content at which it is launched. The measurement process is illustrated in Figure 4 and depicts an initial full wetting followed by draining of the substrate to prepare it for the measurement cycle. The substrate water retention curve for the macropore water regime of Turface is shown in the upper left hand portion of Figure 4 revealing 10 measurement points, 5 on the wetting (lower) curve

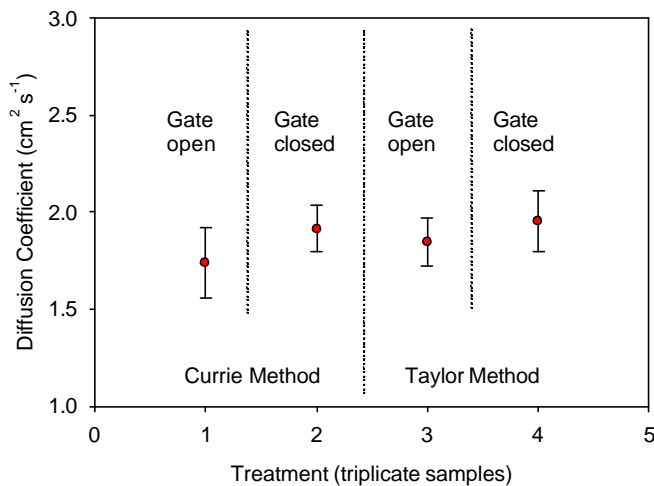


Figure 5 Mean oxygen diffusion coefficients measured in a single chamber diffusion cell showing standard deviation bars. Both Currie (Currie, 1960) and Taylor ((Taylor, 1949)) diffusion coefficients were determined from triplicate measurements (i.e. gate open and closed).

and 5 on the draining (upper) curve. With each increment of water added or removed, a new measurement cycle (Figure 3) is processed, the longest requiring 100 hours to complete. The estimated time in hours for each measurement component is listed above each symbol along the measurement cycle and one complete cycle is estimated to take about 2 weeks.

FLIGHT MODULE

The flight unit will contain nine individual gas diffusion cells capable of running independent diffusion measurements (Figure 6). All nine cells will share most resources such as water, nitrogen, air, power, and data acquisition. These resources will be managed by the LADA control module (Bingham et al., 2002), an existing piece of hardware on the ISS. Nitrogen supply and water supply will be external, and will be provided by Russian participants. The flight unit will be small enough to fit into a Space Shuttle mid-deck locker for return.

CONCLUSION

The ORZS gas diffusion measurement system incorporates nine automated dual chamber closed diffusion cells managed by the LADA control system on ISS. Substrate water content and energy status are monitored by a heat-pulse moisture sensor and by tensiometers in contact with the substrate. Water additions and removal are made using a peristaltic pump with control derived from the pressure transducer measuring the input water supply energy status. Porous stainless steel membranes interface with the substrate providing water supply and removal capability. At pre-determined water content set points, diffusion measurements are carried out in a process of priming the

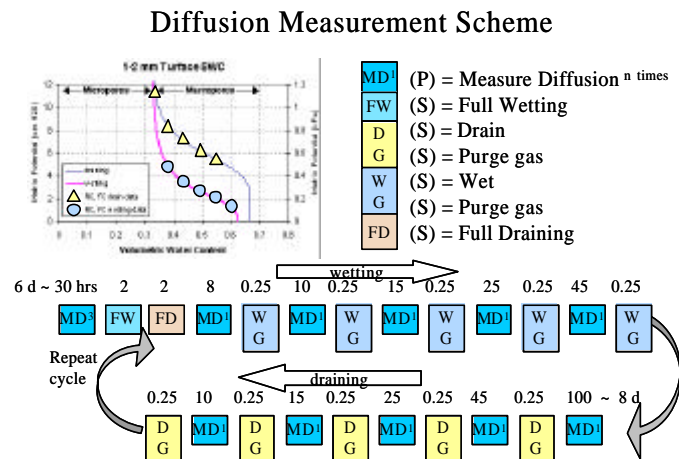


Figure 4 Predicted timeline for the flight diffusion measurements assuming available resources (water, Nitrogen) for two complete cycles. The upper-left hand figure illustrates proposed measurement points plotted on the wetting (lower) and draining (upper) substrate water characteristic curves of Turface.

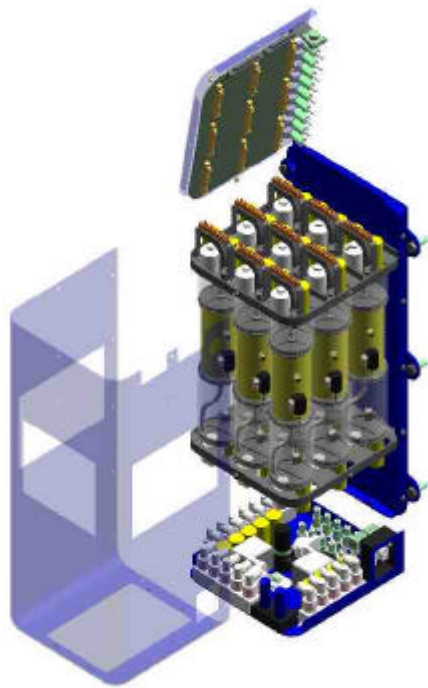


Figure 6 Flight module showing details of the nine-dual chamber diffusion cells and fluid management system which interfaces with the LADA control system.

diffusion cell air and nitrogen chambers and allowing the oxygen concentration in each to come to equilibrium. Oxygen concentration time-dependent data are used to fit the diffusion coefficient. The entire wetting and draining cycle contains 10 water content set points and requires an estimated 14 days to complete. Two complete cycles (28 days) are planned for measurement on the ISS in 2005.

ACKNOWLEDGMENTS

The authors gratefully acknowledge funding from NASA-JSC grant NAG 9-1284.

REFERENCES

- Bingham, G.E., S.B. Jones, I. Podolsky, and B.S. Yendler. 1996. Porous substrate water relations observed during the greenhouse-II flight experiment (Mir Space Station, 1995). SAE Tech. Paper 961547.
- Bingham, G.E., I.G. Podolsky, T.S. Topham, and J.M. Mulholland. 2002. Lada: The ISS plant substrate microgravity testbed. SAE Tech. Paper 2002-01-2388.
- Bugbee, B.G., and F.B. Salisbury. 1989. Controlled environment crop production, p. 107-130, *In* D. L. Henninger, ed. Lunar Base Agriculture: Soils for

- Plant Growth. ASA, CSSA, SSSA., Madison, Wisconsin.
- Bunt, A.C. 1988. Physical aspects, *In*: Media and mixes for container-grown plants:48.
- Currie, J.A. 1960. Gaseous diffusion in porous media. Part 1. A non-steady state method. *Br. J. Appl. Phys.* 11:314-317.
- Glauz, R.D., and D.E. Rolston. 1989. Optimal design of two-chamber, gas diffusion cells. *Soil Sci. Soc. Am. J.* 53:1619-1624.
- Ivanova, T.N., and I.W. Dandolov. 1992. Dynamics of the controlled environment conditions in "SVET" greenhouse in flight. *Compt. rend. Acad. Bulg. Sci.* 45:33-35.
- Jones, S.B., and D. Or. 1998. Design of porous media for optimal gas and liquid fluxes to plant roots. *Soil Sci Soc Am J* 62:563-573.
- Jones, S.B., and D. Or. 1999. Microgravity effects on water flow and distribution in unsaturated porous media: Analysis of flight experiments. *Water Resour. Res.* 35:929-942.
- Jones, S.B., D. Or, G.E. Bingham, and R.C. Morrow. 2002. ORZS: Optimization of root zone substrates for microgravity. SAE Tech. Paper 2002-01-2380.
- Mashinsky, A., I. Ivanova, T. Derendyaeva, and F. Salisbury. 1994. "From seed-to-seed" experiment with wheat plants under space-flight conditions. *Adv. Space Res.* 14:13-19.
- Morrow, R.C., R.J. Bula, T.W. Tibbitts, and W.R. Dinauer. 1994. The ASTROCULTURE flight experiment series, validating technologies for growing plants in space. *Adv. Space Res.* 14:29-37.
- Podolsky, I., and A. Mashinsky. 1994. Peculiarities of moisture transfer in capillary-porous soil substitutes during space flight. *Adv. Space Res.* 14:39-46.
- Rolston, D.E., and P. Moldrup. 2002. Gas diffusivity, p. 1113, *In* J. H. D. a. G. C. Topp, ed. *Methods of Soils Analysis: Part 4, Physical Methods*. SSSA, Madison, WI.
- Scovazzo, P., T.H. Illangasekare, A. Hoehn, and P. Todd. 2001. Modeling of two-phase flow in membranes and porous media in microgravity as applied to plant irrigation in space. *Water Resour. Res.* 37:1231-1243.
- Taylor, S.A. 1949. Oxygen diffusion in porous media as a measure of soil aeration. *Soil Sci. Soc. Am. J.* 14:55-61.
- Yendler, B.S., B. Webbon, I. Podolsky, and R.J. Bula. 1996. Capillary movement of liquid in granular beds in microgravity. *Adv. Space Res.* 18:233-237.

CONTACT

Scott B. Jones, Dept. Plants, Soils and Biometeorology, Utah State University, Logan, UT 84322-4820; Phone:(435) 797-2175, FAX:(435) 797-2117, Email: scott.jones@usu.edu.