

***DIRECT SYNTHESIS OF AEROGEL FROM
SUPERCRITICAL FLUID:
A NEW ONE-STEP PROCESS FOR WEIGHTLESS
AEROGEL PRODUCTION AND A NOVEL METHOD FOR
SUPERCRITICAL FLUID ANALYSIS***

TOPIC AREAS:

Chemistry/Weightless Materials Processing/Fluid Physics

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TABLE OF CONTENTS

Flight Week Preference	4
Advisor/Mentor Request	4
Abstract	5
I. TECHNICAL	
1. Introduction	7
1.1 What is Aerogel?	7
1.1.1 Applications of Aerogel	7
1.2 Preparation of Aerogel	7
1.2.1 Preparation of Precursor Gel	8
1.2.2 Supercritical Drying	8
1.3 Problems With Aerogel Production	9
1.4 Effects of Gravity on the Formation of Gels and the Impact on Properties of Derived Aerogels	9
1.5 Direct Synthesis of Aerogels from Supercritical Fluid	11
1.6 Introduction to Supercritical Fluids	11
1.6.1 Near-Critical Point Effects	11
1.7 Effects of Gravity on Supercritical Fluids	12
1.8 Problems With Analysis and Prediction of Supercritical Fluids	12
2. Test Objectives	13
2.1 Objectives of the Experiment	13
2.2 Not a Follow-up Experiment	14
2.3 Hypothesis	14
3. Test Description	16
3.1 Description of Chemical Reaction	16
3.2 Equipment Design	16
3.2.1 Overview	16
3.2.2 Reaction Cylinders	17
3.2.3 Delivery and Injection System	18
3.2.4 Containment Box	22
3.2.5 Automated Controls, Sensors, Computer, and Electronics	24
3.2.6 Safety Features	25
3.3 Procedures	25
3.4 Expected Results and Measurements	27
3.5 Need for Reduced Gravity	28
4. Not a Follow-up Flight	30
5. Bibliography	31
II. SAFETY EVALUATION	
6. Flight Manifest	32
7. Experiment Description/Background	33

8. Equipment Description	34
9. Structural Design	35
9.1 Overview	35
9.2 Centers of Gravity	35
9.2.1 9 G's Forward	36
9.2.2 3 G's Aft	36
9.2.3 6 G's Down	37
9.2.4 2 G's Lateral	37
9.2.5 2 G's Up	37
9.3 Summary of Apparatus Attachment to Fuselage of KC-135A	37
9.4 Structural Integrity of Internal Components Inside the Box	38
9.5 Structural Integrity of Containment Box Inside Aluminum Frame	39
10. Electrical System	41
11. Pressure/Vacuum System	42
11.1 Overview	42
11.2 Pressure Relief Strategies	42
12. Laser System	43
13. Crew Assistance Requirements	44
14. Institutional Review Board (IRB)	45
15. Hazard Analysis	46
16. Tool Requirements	56
17. Ground Support Requirements	57
18. Hazardous Materials	58
19. Procedures	59
19.1 Shipping Equipment to Ellington Field	59
19.2 Ground Operations	59
19.3 Loading	59
19.4 Pre-Flight	59
19.5 In-Flight	59
19.6 Post-Flight	60
19.7 Offloading	60
III. OUTREACH	
20. Outreach Plans	61
20.1 Outreach Objectives	61
20.2 Planned Outreach Activities	61
20.2.1 TechTV	61
20.2.2 Expand Your Horizons Program	62

20.2.3	Big Brothers/Big Sisters of Madison Field Trip	62
20.2.4	www.zerogaerogel.com	63
20.2.5	espanol.zerogaerogel.com	64
20.2.6	All-Day Presentations to Science Classes at Nicolet High School	64
20.2.7	Presentations to Science Classes at Madison Memorial High School	64
20.2.8	Engineering Saturday for Tomorrow’s Engineers at Madison Program	65
20.2.9	ASPIRE	65
20.2.10	Wisconsin Space Conference Presentation	66
20.2.11	American Institute for Aeronautics and Astronautics Presentation	66
20.2.12	Press Plan	66

IV. ADMINISTRATIVE REQUIREMENTS

21. NASA/JSC HUMAN RESEARCH CONSENT FORM 68

22. IUCAC 68

23. FUNDING/BUDGET STATEMENT 68

23.1	Itemized Budget	68
23.2	Current Sources of Funding	69

24. PARENTAL CONSENT FORMS 69

Figure 1-1:	Polycondensation of Silicon Alkoxide	8
Figure 3-1:	Reaction Cylinder	17
Figure 3-2:	Volumetric Piston	19
Figure 3-3:	Delivery and Injection System 21	
Figure 3-4:	Interface for Reaction Cylinders	22
Figure 3-5:	Containment Box	23
Figure 3-6:	Door Schematic	24
Table 9-1:	Summary of FS calculations for attachment to fuselage	38
Table 9-2:	Containment summary of internal components inside the inner box	39
Table 9-3:	Summary of FS calculations for shear stress of the outer frame under loading imposed from the inner box	39
Table 9-4:	FS associated with bending of outer aluminum frames from load induced from inner box	40
Table 10-1:	Electrical Components	41

FLIGHT WEEK PREFERENCE

First Choice: Flight Group 5 July 8 to July 17, 2004

Second Choice: Flight Group 6 July 22 to July 31, 2004

Third Choice: Flight Group 2 March 18 to March 27, 2004

ADVISOR/MENTOR REQUEST

No advisor or mentor is requested.

ABSTRACT

The experiment is testing a new, simplified process for producing aerogel in a weightless environment, analyzing the impacts of weightlessness on the nanostructure of aerogels produced through this method, and analyzing the effectiveness of using this method as a means for analyzing near-critical point density fluctuations in a supercritical fluid.

Production of aerogel is a time-consuming process typically involving, at a minimum, three separate diffusion-controlled steps and several days processing time.

Goal 1: Establish a Method of Weightless Materials Processing

Loy et al demonstrated a method of consolidating these steps into a single process. Instead of forming a gel, exchanging solvents, and then supercritically drying to produce aerogel, they conducted sol-gel polymerization in supercritical fluid to produce a gel, requiring only depressurization of the gel to produce aerogel. The entire process was conducted within an 18-hour time period. The process leads the way to many new possible applications for aerogel, including future on-demand production of aerogel in space for repairing thermal protection systems, generating insulation for satellites, and self-assembly processes for spacecraft.

We have innovated on this process and are developing a method for conducting direct synthesis of aerogel from supercritical fluid in weightlessness with the goal of establishing the first methodology for practical production of aerogel in a weightless environment.

Goal 2: Gauge the Effects of Gravity on the Nanostructures of Aerogels Produced through Direct Synthesis

We have shown from previous experiments aboard NASA's KC-135A that gels produced in a weightless environment result in aerogels possessing different nanostructures and different physical properties than aerogels derived from gels formed in an acceleration field. We believe this will translate to aerogels produced through direct synthesis in as well, with potentially more pronounced differences since a greater proportion of production is conducted in weightlessness.

We are developing a direct synthesis process with stoichiometry that will allow us to form a gel from supercritical fluid in under 20 seconds. This will allow us to fully form a gel within the amount of time that the KC-135A can produce microgravity. Subsequent depressurization of the supercritical gels will yield aerogels. We propose producing a set of aerogels through direct synthesis in

weightlessness and comparing their nanostructures with aerogels produced through a similar process in 1 G.

Goal 3: Trap the Density Structure of a Supercritical Fluid in a Solid Structure

Supercritical fluids exhibit unusual phenomena close to their critical points. One such phenomenon is called supercritical opalescence in which the supercritical fluid scatters visible light, caused by nanoscopic density variations in the supercritical fluid. Another unusual phenomenon is the extreme compressibility of the fluid near the critical point. As a result, the weight of a supercritical fluid near its critical point pushing down on itself induces a macroscopic vertical density gradient, subsequently affecting nanoscopic density gradients throughout the fluid. Because of this pronounced weight-induced macroscopic density variation in near-critical fluids, it has proven difficult to accurately measure and understand the nature of the nanoscopic density variations in near-critical fluids. The ZENO experiment attempted to do this in weightlessness without the weight-induced macroscopic density variation.

Production of a gel from a near-critical solvent may be a way to “trap” these nanoscopic density fluctuations in a solid structure. Depressurization of such a gel to produce aerogel would then allow for detailed analysis of such density fluctuations. We propose producing a set of aerogels through direct synthesis from near-critical fluid in weightlessness and comparing their nanostructures with aerogels produced through a similar process in 1 G.

I. TECHNICAL

1. INTRODUCTION

The experiment combines three concepts—the effects of gravity on gel formation, the effects of gravity on the density and homogeneity of a supercritical fluid, and a process for the direct synthesis of aerogel from gel formed in supercritical fluid.

The following sections will introduce these three concepts separately, followed by a description of how they intersect in our experiment.

1.1 What is Aerogel?

Aerogel is a nanoporous solid material composed of 50 to >99.8% air by volume. It boasts the lowest density (ranging from 0.1 g cm^{-3} to 0.0011 g cm^{-3}) and highest internal surface area (ranging from $250 \text{ m}^2 \text{ g}^{-1}$ to $2\,500 \text{ m}^2 \text{ g}^{-1}$) of any solid material. Because of its extreme low density and high internal surface area, aerogel is the best solid insulation that has ever been developed (with a typical thermal conductivity of $<0.016 \text{ W m}^{-1} \text{ K}^{-1}$) and possesses impressive impact-dampening properties. Despite its extreme low density, a piece of aerogel can withstand nearly 2,000 times its weight in applied force. Aerogels can be made of a variety of materials, including silica, metal oxides, carbon, and organic polymers. Many aerogels, including silica aerogel, can be made optically transparent.

1.1.1 Applications of Aerogel

Aerogel has potential to revolutionize applications ranging from kitchen appliances to spacecraft. Applications for commercial products include transparent insulation for windows, insulation for refrigerators and ovens, acoustic dampening, desalination filters, and computer chips. Some aerospace applications include lightweight thermal insulation for satellites, insulation for sensitive electronics, insulation for cryogenic and nuclear systems, and thermal protection systems for spacecraft reentry.

Aerogel has tremendous potential for use in space-based applications. The future nuclear space initiatives being developed by Boeing and NASA make aerogel more important than ever. Developing methods for improving aerogel properties and production are essential. Additionally, exploring possibilities for producing aerogel on-demand in space could enable more sophisticated spacecraft with abilities to perform self-assembly in space.

1.2 Preparation of Aerogel

Although many types of aerogels can be produced, we will be discussing the preparation of silica aerogel, which is the focus of our experiment.

1.2.1 Preparation of Precursor Gel

Aerogels are produced from gels. A gel is a colloidal system in which a network of interconnected solid particles spans the volume of a liquid medium. A familiar gel is the gelatin dessert Jell-O™. A gel consists of two components—a solid particle matrix and a liquid filling the volume of that matrix.

Gels are generally formed through the sol-gel process. In this process, a suspension of nanoscopic solid particles in liquid (a sol) is prepared and induced to polymerize to form a solid particle network spanning the liquid (a gel).

Silica gels can be formed by polycondensation of silicon alkoxide with water (Figure 1-1). Since silicon alkoxides and water are not miscible, a cosolvent such as ethanol is required for the reaction to occur in the same phase. The three reactions in Figure 1 can occur rather slowly at room temperature and so a basic or acidic catalyst such as ammonium hydroxide or hydrochloric acid can be added to increase reaction rate.

R=CH₃ or C₂H₅

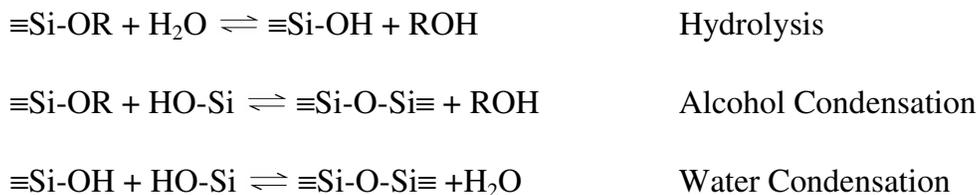


Figure 1-1: Polycondensation of Silicon Alkoxide

Gelation begins as silicon alkoxide molecules interconnect to form small, oligomeric species of a few monomer units. These oligomeric species further expand and/or interconnect with other monomeric and oligomeric species to form mesoscopic clusters. These mesoscopic clusters eventually interconnect to form a bulk-scale gel matrix that spans the entire volume of the liquid medium. Looking at a gel's solid particle matrix under a microscope, it can be seen that it is comprised of interconnected, but discrete, clusters. Gel time can be anywhere from a few seconds to several hours depending on the amount of reagents and catalyst used.

Once a gel has formed it is soaked under solvent to remove excess alkoxide, unreacted species, water, and catalyst from the gel.

1.2.2 Supercritical Drying

If the liquid in a gel is removed by evaporation, the gel's solid particle matrix will collapse to form a dense solid called xerogel. This is caused by capillary forces exerted on the pores of the gel matrix by the evaporating liquid, pulling the branches of the matrix inward. These branches subsequently stick together by hydrogen bonding.

It is possible, however, to remove the liquid from a gel without causing its solid particle matrix to collapse by eliminating the presence of capillary action in the gel matrix. One way to do this is by supercritically extracting the liquid from the gel. *Supercritical extraction* is a process in which the liquid in a gel is heated past its critical point, converting it into a supercritical fluid (supercritical fluids are discussed in more depth in Section 1.6). Supercritical fluids have the density and thermal conductivity of liquids, but unlike liquids, expand to fill the volume of their container and do not exert capillary forces. When the liquid in a gel is heated past its critical point, the liquid becomes a supercritical fluid that can diffuse out of the gel's solid particle matrix without pulling the solid particle matrix inward. Once the fluid in the gel has equilibrated with its surroundings, the system can be depressurized, converting the fluid to a gas phase. The result is replacement of liquid in the gel with gas.

Supercritical extraction typically requires the use of a high-pressure autoclave. Most solvents used in making silica gels, however, are dangerously explosive at their critical points. Instead of extracting the solvent from a gel directly, a solvent exchange can be performed exchanging the solvent with a safer, non-flammable liquid—carbon dioxide. The critical point of carbon dioxide is 304.13 K and 72.786 atm, much lower than most organic solvents. This technique, called the Hunt process, provides safe, low-temperature supercritical extraction. Since liquid carbon dioxide does not condense at atmospheric pressure, a pressure vessel is required for both soaking and supercritical extraction.

1.3 Problems With Aerogel Production

There are several problems with current aerogel production methods that make aerogels expensive. One major problem is the length of time required to make aerogels. Multiple diffusion-controlled steps are required to purify precursor gels and perform solvent exchange with carbon dioxide. Even small samples of aerogel take, at a minimum, several days to prepare for supercritical drying. Larger samples take even longer because of diffusion rates. In addition to time, multiple soakings require a significant amount of resources and allow more opportunity for gels to be damaged in the process. Improper purification prior to supercritical drying can also lead to dense and opaque aerogels with lower surface areas and higher thermal conductivity. It is therefore desirable to develop a procedure which can simplify aerogel production to reduce the amount of time and resources required to produce aerogels.

1.4 Effects of Gravity on the Formation of Gels and the Impact on the Properties of Derived Aerogels

Gravity is almost always ignored when it comes to most chemical systems, simply because its effects are so much lesser in magnitude than the quantum interactions that govern most chemical behavior. Systems involving molecular-sized particles experiencing Brownian motion typically experience only a negligible effect from gravity. Contrapositively, bulk-scale systems are heavily influenced by gravity,

and experience only negligible effects from the forces that govern Brownian motion. But at what point do we say something is “bulk scale” and no longer “molecular” in size? Where do Newtonian forces like weight and buoyancy “switch on” and does Brownian motion “switch off?”

Really there is no clear threshold differentiating the two realms. Somewhere between “big molecule” and “microparticle” lies the mesoscopic domain, where both Brownian motion and gravity have a say. Particles in the mesoscopic realm are molecular in nature, but large enough to experience weight and buoyancy. They are governed generally by Brownian motion, diffusing through a medium like a small molecule would, but much slower and more impeded. Mesoscopic particles experience drag as they barge through smaller molecules in solution, and do not have the free, unhindered ability to diffuse like small molecules. At the same time, mesoscopic particles will not rapidly settle to the bottom of their containers like bulk-scale particles, and do not sort themselves out according to relative densities. But their motion is still affected by these tendencies, and thus mesoscopic particles in solution behave and interact much differently than smaller molecules in solution, as they are governed by both Brownian and Newtonian forces.

Vanderhoff et al. demonstrated that emulsion polymerization of latexes in microgravity results in better monodispersity, increased uniformity, and reduced coagulation.¹ Noever et al. have proposed that buoyancy-driven fluid flow dramatically affects micro- and nanostructures formed in sol-gel systems just before gelation, and that these eddies are “frozen” as imperfections in the gel’s matrix.^{2,3} It would be expected then that formation of sol-gel structures in microgravity would result in more uniform gel matrix.

Smith et al. conducted a space-flight experiment on the growth of Stober particles and found space-grown samples of Stober particles resulted in low-density gels where ground-based samples remained in suspension.⁴ This implies that gravity hinders ideal formation of gel substructures, and results in a growth pattern that ultimately terminates the gel clusters’ size affecting their structure in such a way that their surfaces are less able to interconnect to form a gel structure.

Indeed our previous research in forming gels in microgravity has shown that the growth of nanostructures is affected by forces induced by gravity. Chain length and pore sizes, for example, are affected. This translates to aerogels as well, resulting in different physical properties between aerogels produced from gels formed in zero-gravity and aerogels produced from gels formed in 1 G.

¹ Vanderhoff J. W.; El-Aasser, M. S.; Micale, F. J.; Sudol, E. D.; Tseng, C. M.; Sheu, H. R. *Polym. Prepr.* 1987, 28, 455; *Mater. Res. Soc. Symp. Proc.* 1987, 87, 213.

² Noever, D. A. *Microgravity Sci. Technol.* 1994, 3, 14.

³ Zhu, J. X.; Li, M.; Rogers, R.; Meyer, W.; Ottewill, R. H.; STS-73 Space Shuttle Crew; Russel, W. B.; Chaikin P. M. *Nature* 1997, 387, 883.

⁴ Smith, D. et al. “Effect of Microgravity on the Growth of Silica Nanostructures,” *Langmuir*, 2000, 16, 10055-10060.

1.5 Direct Synthesis of Aerogels from Supercritical Fluid

Loy et al. demonstrated a method of producing monolithic silica aerogel directly from supercritical fluid. Instead of preparing a gel, purifying the gel, performing solvent exchange with carbon dioxide, and then supercritically drying, they formed a gel with supercritical carbon dioxide as the solvent, only requiring depressurization to produce aerogel. This process resulted in opaque, but monolithic, silica aerogel with lower surface area than aerogels produced by preparing a precursor gel and then supercritically drying with carbon dioxide, but still produced aerogels. The process is much more efficient and can be conducted within a few hours instead of several days!

In direct synthesis, tetramethoxysilane is polymerized in supercritical carbon dioxide to form a gel. However, since water is not miscible in either tetramethoxysilane or supercritical carbon dioxide, a soluble catalyst, such as formic acid, must be used. Loy et al used a 1.9 M TMOS in supercritical carbon dioxide solution with a TMOS to formic acid ratio of 3 or 4.

1.6 Introduction to Supercritical Fluids

A supercritical fluid is a substance past its critical point, the temperature and pressure beyond which liquid will not condense despite further increase in pressure. Technically a supercritical fluid is a gas, however near the critical point, supercritical fluids are much denser and have much higher thermal conductivity than what is usually considered to be a gas. Molecules in a supercritical fluid have so much kinetic energy that, despite their proximity to each other, their speeds overcome the quantum interactions experienced from other molecules that would otherwise cause liquid cohesion. The result is a fluid that has the approximate density and thermal conductivity of a liquid but that expands and compresses like a gas. Supercritical fluids can dissolve substrates like liquids can and can be used as solvents. Supercritical carbon dioxide, for example, has the uncanny ability to dissolve fluorinated hydrocarbons.

1.6.1 Near-Critical Point Effects

Near the critical point, supercritical fluids exhibit unusual behavior. Supercritical fluids are extremely compressible at the critical point, meaning that small pressure changes result in dramatic density changes. As a fluid approaches the critical point it exhibits something called *supercritical opalescence*, where the fluid scatter visible light and turns opaque white. Above the critical point the fluid will become clear again. This is a result of nanoscopic density fluctuations in the fluid that form as some molecules have sufficient kinetic energy to escape liquid cohesion while others still binding to other molecules cluster into nanosized domains. These nanoscopic density variations are large enough to scatter visible light, making the supercritical fluid white.

1.7 Effects of Gravity on Supercritical Fluids

As mentioned, near the critical point compressibility is extremely high, meaning small pressure changes result in dramatic density changes. As a result, the weight of the fluid pushing down on itself causes a macroscopic density gradient in a column of supercritical fluid, simply because the bottom of the column is being compressed more than the top. This gradient in pressure subsequently affects nanoscopic density variations in the fluid, meaning nanoscopic density variations in the bottom of the fluid are different from nanoscopic density variations at the top.

1.8 Problems With Analysis and Prediction of Supercritical Fluids

Understanding the nature of these density variations is important for understanding supercritical fluids. Nearly every equation that has been developed to model gases, both from empirical and first-principle approaches, falls apart at the critical point. Measuring supercritical properties gives more information to base equations on. Understanding how to calculate critical points for example, especially of mixtures, would often be useful, however, it is nearly impossible to do this with any accuracy. In preparing this experiment, for example, there were several critical points that needed to be determined that could only be determined by experiment. Being able to model supercritical fluids at the critical point and being able to make measurements about their nanodynamics is important.

Measuring the distribution of sizes of nanoscopic domains would be useful, for example. Unfortunately, it is difficult to measure these density variations. Not only are they constantly fluctuating, but gravity-induced density gradients further complicate things by distorting measurements in a terrestrial laboratory.

The University of Maryland conducted a space flight experiment to measure the lifetimes of these nanoscopic density variations with a light-scattering experiment called ZENO.⁵ Although this and other experiments have gathered useful information about the decay rates of nanoscopic density variations in supercritical fluids, there is much about the nanodynamics of supercritical fluids that is not understood.

⁵ R. W. Gammon, "Critical Fluid Light Scattering (Zeno)", in Microgravity Science and Applications Program Tasks and Bibliography for FY 1992, NASA Technical Memorandum 4469 (NASA Office of Space Science and Applications, Washington, D.C., March 1993), p. II-69.

2. TEST OBJECTIVES

2.1 Objectives of the Experiment

Our experiment proposes to accomplish three objectives. We propose the first method for direct synthesis of aerogel from supercritical carbon dioxide for production of silica aerogel in a weightless environment aboard NASA's KC-135A Reduced Gravity Laboratory. We are developing a new direct synthesis process along with engineering to adapt the process to a weightless environment (we will describe this process further in the following sections). This will enable us to produce silica aerogel by direct synthesis within the 23-second time periods the KC-135A can produce microgravity. In doing so we have three experimental goals:

Goal 1: Establish a Method of Weightless Materials Processing

With this new, simplified process for aerogel production, there is potential for developing a method of producing aerogel on-demand in weightlessness. This kind of weightless materials processing lends itself to many applications, including spacecraft self-assembly and weightless engineering for future space stations and space flights with on-board materials production capabilities.

Although gels have been formed previously in weightlessness and aerogels have been produced on Earth from gels produced in weightlessness, aerogel itself has never been produced in weightlessness.

We aim to develop and carry out a method for conducting direct synthesis of aerogel from supercritical fluid in weightlessness with the goal of establishing the first methodology for practical production of aerogel in a weightless environment. To do this, we will use a modified polymerization reaction of tetramethoxysilane by formic acid in supercritical carbon dioxide performed in a system we have developed for specifically conducting this kind of chemistry aboard the KC-135A. Recognizing potential safety hazards with high-pressure systems, we are working with the Reduced Gravity Office to ensure safety of design and operation as we build the system. Our design and construction will be monitored by engineers at the University of Wisconsin Space Sciences Engineering Center who have successfully designed and conducted high-pressure experiments aboard space shuttle flights in the past.

Goal 2: Gauge the Effects of Gravity on the Nanostructures of Aerogels Produced through Direct Synthesis

We have shown from previous experiments aboard NASA's KC-135A that gels produced in a weightless environment result in aerogels possessing different nanostructures and different physical properties than aerogels derived from gels formed in an acceleration field. We believe this will translate to aerogels produced through direct synthesis as well, with potentially more pronounced differences since a greater proportion of production is conducted in weightlessness.

We are developing a direct synthesis process with stoichiometry that will allow us to form a gel from supercritical fluid in under 20 seconds, within the amount of time the KC-135A can produce microgravity. Subsequent depressurization of the supercritical gels will yield aerogels. We propose producing a set of aerogels through direct synthesis in weightlessness and comparing their nanostructures with aerogels produced through a similar process in 1 G.

Goal 3: Trap the Density Structure of a Supercritical Fluid in a Solid Structure

As mentioned, supercritical fluids near their critical points exhibit supercritical opalescence caused by nanoscopic density variations in the supercritical fluid. The extreme compressibility of a fluid near its critical point causes extreme density variation in the fluid with only slight changes in pressure. As a result, the weight of the fluid pushing down on itself induces a macroscopic vertical density gradient, subsequently affecting nanoscopic density gradients throughout the fluid. Because of this pronounced weight-induced macroscopic density variation in near-critical fluids, it has proven difficult to accurately measure and understand the nature of the nanoscopic density variations in near-critical fluids.

Production of a gel from a near-critical solvent may be a way to “trap” these nanoscopic density fluctuations in a solid structure. Depressurization of such a gel to produce aerogel would then allow for detailed analysis of such density fluctuations. We propose producing a set of aerogels through direct synthesis from near-critical fluid in weightlessness and comparing their nanostructures with aerogels produced through a similar process in 1 G.

Using the advanced facilities available at the University of Wisconsin Materials Research Science and Engineering Center, we will be able to analyze these materials by transmission electron microscopy, Brunauer-Emmett-Teller surface area, skeletal density, and thermal conductivity.

2.2 Not a Follow-up Experiment

This experiment is not a follow-up to any previous experiment and is fundamentally different from our previous experiments.

2.3 Hypothesis

We believe that we will successfully and safely be able to form silica aerogel through direct synthesis from opalescent supercritical carbon dioxide in weightlessness. We also believe that this will be an effective method for producing aerogel in a weightless environment and that we will be able to produce valuable results from 23-second periods of weightlessness. We believe that the properties of aerogels produced through this method will exhibit slightly different physical properties than aerogels produced through a similar process in 1 G. We believe these differences will be due to quantifiable differences in nanostructure. We also believe that we will be able to observe some nanodynamic variations of supercritical fluid

“frozen” in the structure of aerogels produced through this process, although likely only the larger density variations. We believe that there will be a marked difference in these observable variations between aerogels produced in weightlessness and aerogels produced in 1 G.

3. TEST DESCRIPTION

3.1 DESCRIPTION OF CHEMICAL REACTION

Direct synthesis utilizes supercritical solvent to form a gel, eliminating the need for multiple diffusion-controlled soakings to prepare for supercritical drying. The chemical reaction being performed is a sol-gel polycondensation of tetramethoxysilane by formic acid in supercritical carbon dioxide. The supercritical carbon dioxide serves as a solvent. The reaction results in the formation of a gel that upon depressurization will yield aerogel.

The production of the supercritical fluid mixture requires high pressure and elevated temperatures (slightly above room temperature). The exact pressure and temperature is the critical point of a solution of carbon dioxide, tetramethoxysilane, and formic acid. This critical point is highly dependent on the concentration of the components. We are still determining the exact critical point that will be used for the reaction, but know that it will be in the neighborhood of 1,200 psi and 100°F or so.

The gels will have dimensions of approximately 1" in diameter x 8" tall, with a volume of approximately 6.28 cubic inches (102.96 mL).

3.2 EQUIPMENT DESIGN

3.2.1 Overview

We recognize the potential hazards involved with both high pressure systems and weightlessness. We are working closely with the Reduced Gravity Office and the University of Wisconsin Space Sciences Engineering Center in designing our equipment to ensure that the system we develop is safe and will accomplish our scientific goals. UW SSEC has flown high pressure experiments (3,500 psi) aboard space shuttle flights before and has many experts who have offered to work with us in designing our experiment. Additionally, we have the resources of the University of Wisconsin Engine Research Lab for developing components of our system.

The equipment we propose in this proposal is admittedly a first-generation design and is currently under development. We have included redundant safety systems to ensure safety even in the event of a primary system failure. In case of emergency, relief systems automatically maintain the system at safe pressures. In the event a relief system member fails, a secondary relief component will take over its operation.

The equipment is designed to withstand at a minimum 1.5 times the pressures expected in the experiment and will be pressure tested to 1.5 x MAWP with the help of UW SSEC. All high-pressure pipe components, valves, actuated valves, gauges, and sight windows are commercially obtained.

Our primary objective is safety. We feel that we have the resources, expertise, and dedication to make sure our experiment will be safe.

3.2.2 Reaction Cylinders

The reaction cylinders are removable components that serve as molds. The cylinders are composed of 304 stainless steel rated for 3,000 psi, well above the working pressure of 1,200 psi. Each cylinder is fitted with a cylindrical Teflon jacket inside in which the supercritical gel will form and be depressurized to produce aerogel. This is so that the aerogels can be removed from the reaction cylinders without damage. The cylinders interface with the injection system with two male quick connects (see Figure 3-1) which plug into two corresponding female quick connects on the injection system. The quick connects are also rated for 3,000 psi.

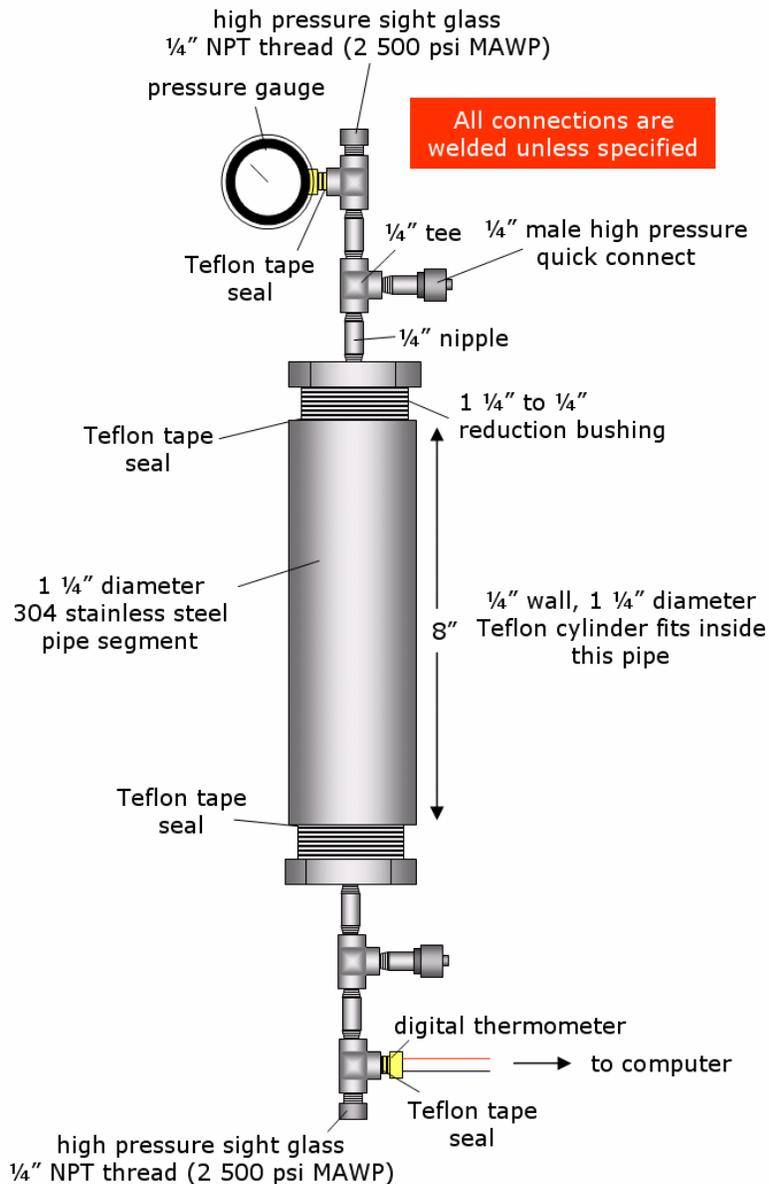


Figure 3-1: Reaction Cylinder

Cylinders are not attached or removed during flight, only during setup prior to flight. Each cylinder is equipped with a digital pressure gauge and digital thermometer interfaced with the computer. Each gauge also has a visible display. In the event of a sensor error, the cylinder is depressurized and abandoned. On the top and bottom of the cylinder are two 1/4" high pressure sight glass windows rated for 2,500 psi. These are used to shine a laser through the cylinder onto a cadmium sulfide cell to measure the clarity of the supercritical solution during flight. This is to determine when the solution is supercritically opalescent.

All pipe components of the cylinders are standard 304 stainless steel NPT components. All components of the top and bottom assemblies with the exception of the gauges will be welded in-place by a professional welder. The gauges will be sealed into the assemblies with Teflon tape. The assemblies will seal into the steel cylinder with Teflon tape so that the aerogel formed in the cylinder can be removed after flight.

3.2.3 Delivery and Injection System

The delivery and injection system is used to pressurize the reaction cylinders with carbon dioxide. The system also selects, measures, and injects two liquids into the reaction cylinders (tetramethoxysilane and formic acid). The system is comprised of two subsystems: a pair of volumetric pistons that aspirate the two liquids and inject them into the reaction cylinders, and a line selection system which selects what to inject and where to inject it. The line selection system has 10 ports for connecting to reaction cylinders. Nine reaction cylinders can be interfaced with the injection system at the same time, with the tenth port connected to a port in the containment box that connects directly to the off-board vent for delivery line waste.

Volumetric Pistons

The volumetric pistons (see Figure 3-2) are similar in design to an engine piston. The top of the piston has a screw-adjustable jam to set the volume of the piston barrel. The piston and barrel are composed of 304 stainless steel. The piston has sealing o-rings to seal against the barrel. The bottom of the barrel is threaded for a 10/32 nipple. The piston is coupled to a 1,500-pound, 48VDC electromechanical actuator that pulls it up and down. The pistons are used to aspirate liquids and inject them into pressurized reaction cylinders.

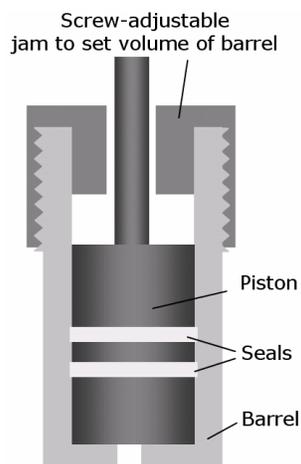


Figure 3-2: Volumetric Piston

Chemical Storage and Delivery

The pistons are connected to collapsible polypropylene intravenous delivery bags, which serve as liquid reagent storage bags. The pistons draw liquid in from these bags through a one-way check valve. The check valve is rated for 3,000 psi backflow so that pressurized liquid cannot be pushed into the storage bag. When the piston is pulled up, liquid is drawn through 1/16" fluorinated ethylene propylene (FEP) plastic tubing interfaced with the check valve by a standard plastic tubing-to-male NPT adapter. Such an adapter is commonly used in high pressure liquid chromatography (HPLC) equipment. Liquid is drawn through the check valve into high pressure steel tubing and into the piston until the piston is full. The actuator then pushes the piston down with force sufficient to inject it into a pressurized (about 1,200 psi) reaction cylinder. The liquid then travels through steel tubing, through another check valve (also rated for 3,000 psi) and into the liquid selection subsystem for delivery into a reaction cylinder. Steel tubing, adapters, and 4-way hubs are standard HPLC components.

In the event of a clog or equipment malfunction, two emergency relief valves are connected to the pistons through which pressurized liquid can be released. The first relief valve (set to open with an extra 10 psi pressure buildup) is connected through a port in the containment box to the off-board vent. The second relief valve (set to open with an extra 20 psi pressure buildup) will open in the event the first relief valve malfunctions or has a problem with the connection to the off-board vent.

Liquid Selection Subsystem

The liquid selection subsystem is comprised of two valves: a 10-way 11-port actuated valve manufactured by Rheodyne and a 3-line 4-port actuated selector valve. The 3-line valve is connected to the volumetric piston subsystems through check valves (see Figure 3-3) and to a liquid carbon dioxide siphon tank through a port in

the containment box. The liquid carbon dioxide tank will be set up and connected by NASA officials. The valve is connected to the intake port on the 10-way valve by a 304 stainless steel 10/32 thread nipple. The 10-way valve then directs intake from this valve to one of 10 ports, 9 of which will be connected to reaction cylinders with the last port connected to the off-board vent. The valve is controlled by the computer.

The system operates to select liquid carbon dioxide, tetramethoxysilane, or formic acid for injection into a reaction cylinder.

Carbon dioxide is delivered from the pressure tank outside the containment box, through a high-pressure hose, into a port in the side of the box and in through a second hose connected to an actuated ball valve that controls whether or not carbon dioxide can go into the 3-line valve.

Interface for Reaction Cylinders

Radially extending from the 10-way valve will be 9 pipe segments with female high-pressure quick connects on the end. The pipe segments are sealed into the 10-way valve with Teflon tape.

Secured to the floor of the containment box are 9 individual valve assemblies (see Figure 3-4). These assemblies consist of a female quick connect welded to a pipe segment connected to two valves. One valve is an adjustable check valve set to the critical pressure of the solution in the reaction cylinder. When the reaction cylinders are pressurized, the initial pressure from the liquid carbon dioxide is only 750-900 psi and the temperature is approximately 75°F. The critical point of the solution is above 1,200 psi and 100°F. To reach the critical point, a reaction cylinder is heated by an external heating coil wrapped around it. The reaction cylinder will reach critical pressure before it reaches critical temperature, and so the check valve will automatically release pressure until the heating coil stops heating. In the event of overpressurization, the check valve serves as a safety release. In the event of a malfunction in the check valve, a manual release ball valve is installed in each assembly which can be opened to release pressure in the cylinder. Both valves feed to ports in the containment box that connect to the off-board vent.

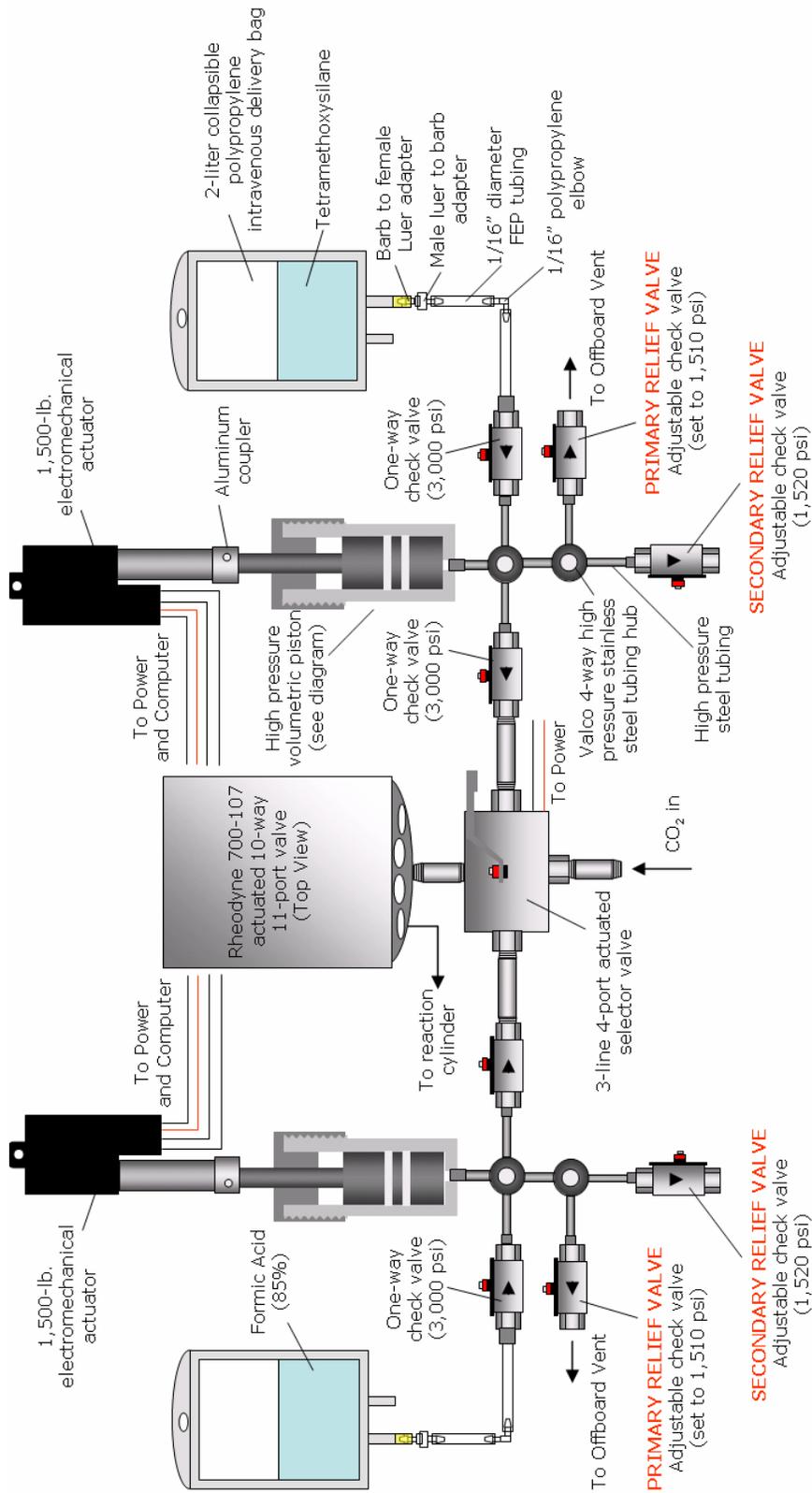


Figure 3-3: Delivery and Injection System

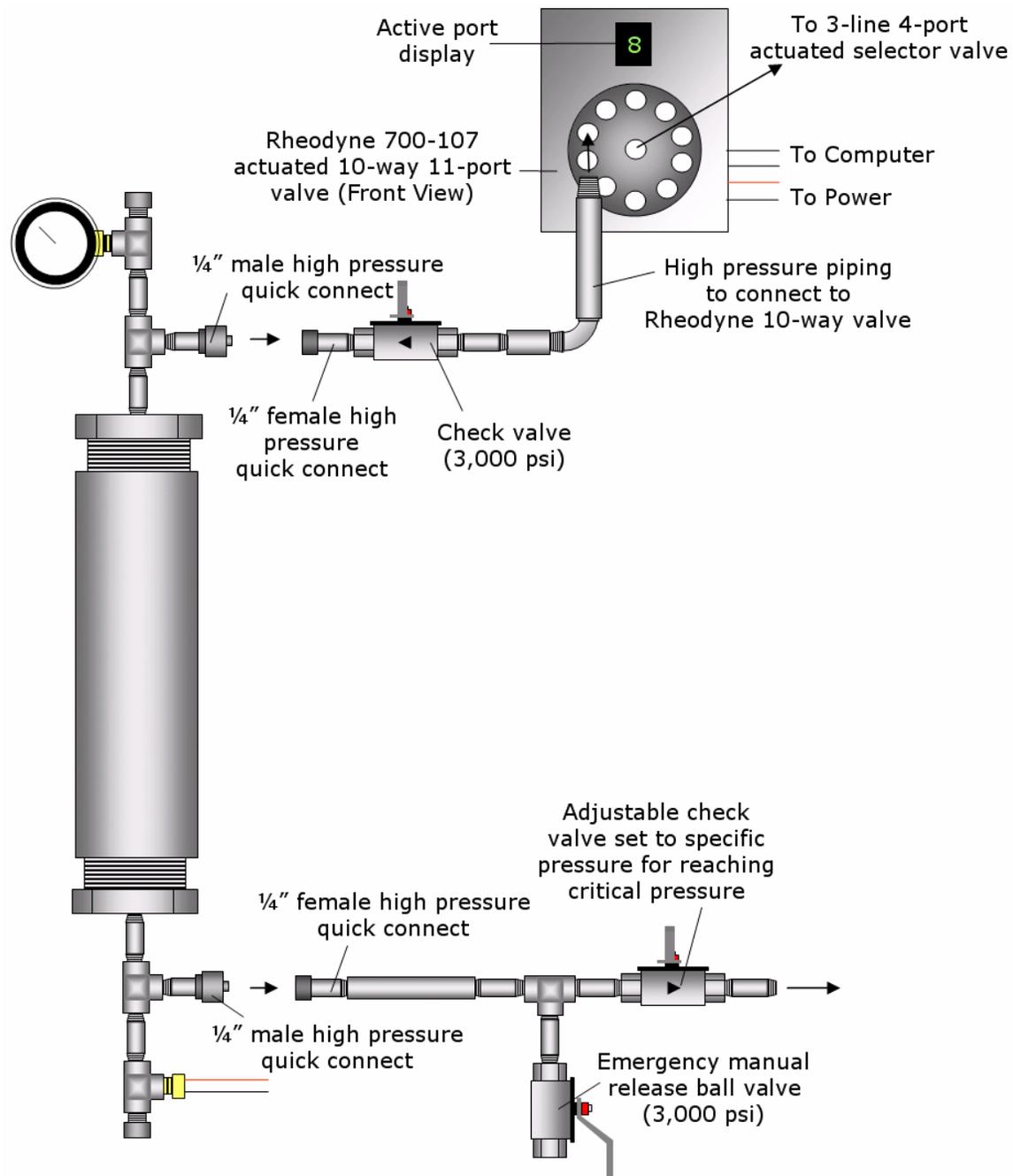


Figure 3-4: Interface for Reaction Cylinders

3.2.4 Containment Box

The containment box is designed to contain any leaks, gas or liquid, that may occur during flight. It is composed of an aluminum frame with two separate layers of

1/8” impact-resistant polycarbonate sheets. With equipment installed, the box provides three layers of containment for chemicals. All seams are sealed with silicone caulk. The box has data ports sealed in its side for interfacing electronics inside the box with equipment outside and several connectors for connecting hoses to the off-board vent of the KC-135A. The box also has an open connector connected to the off-board vent to vent the atmosphere of the box in the event of a liquid or gas leak. The box was used in a previous experiment conducted by our team on the KC-135A and has been proven safe and leak-proof for the KC-135A.

The front of the box is a hinged door with refrigerator-style gaskets. Behind the door is a pressure-fit gasketed removable panel to maintain two layers of containment. A set of butyl rubber gloves penetrates the outer door and inserts into a second skin of latex inside the second panel. This is to allow manual access of equipment in the box without opening the box to the cabin.

Equipment is installed in the box by a series of aluminum L-beams bolted to the box’s frame.

The box is secured to the KC-135A by straps over its top.

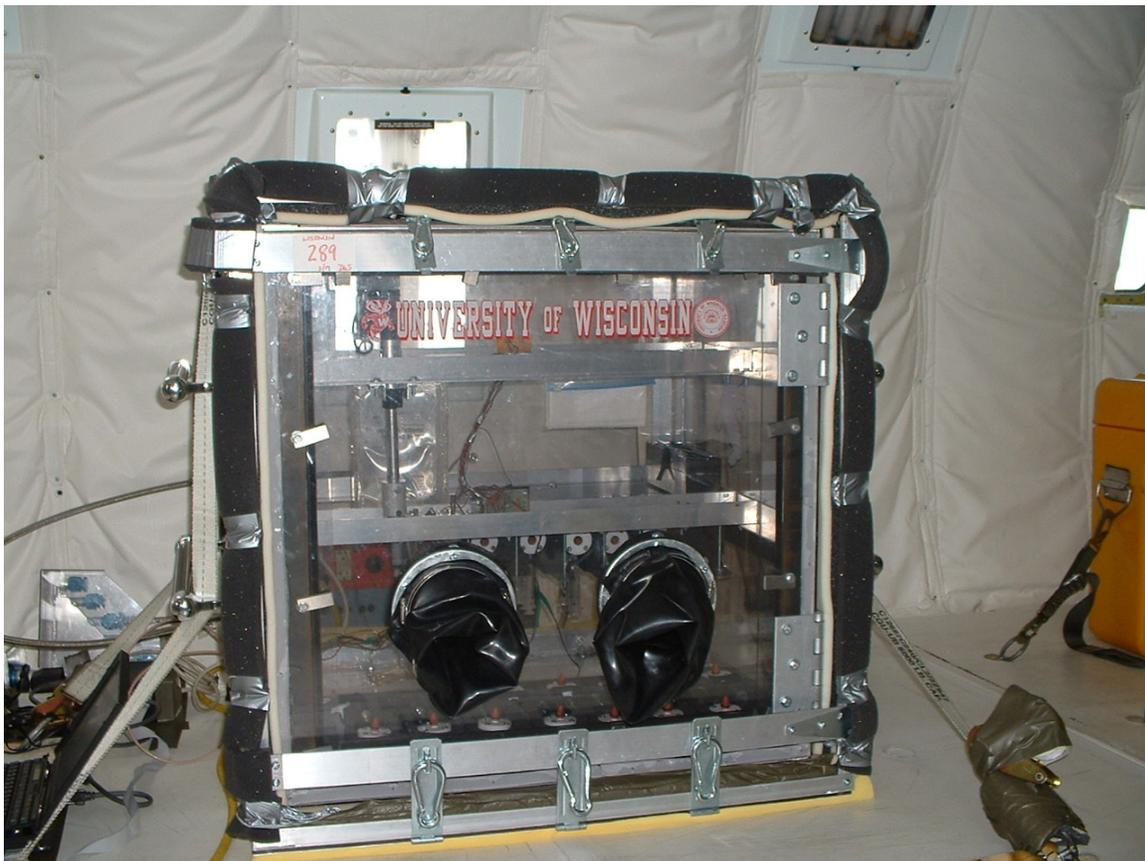


Figure 3-5: Containment Box (Containing Equipment from Previous Experiment)

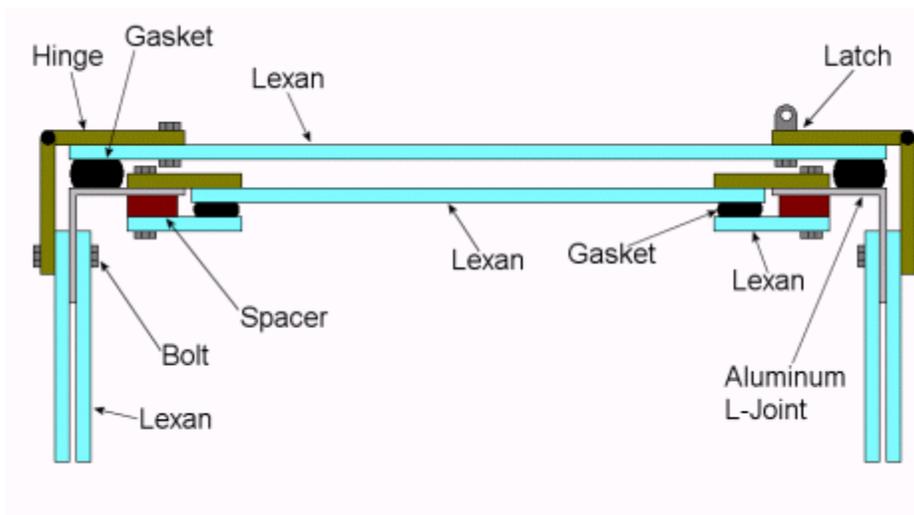


Figure 3-6: Door Schematic

Vibrational Isolation Frame

The entire containment box fits inside a foam-padded, open aluminum frame. This provides vibrational damping for the entire system. The frame is constructed of bolted aluminum L-beams and aluminum flats. There is no ledge on the front side of the frame in order to allow the doors to be opened while the box is mounted in the frame. To prevent the box from being able to slide out the front of the frame (despite the tight clamp from the frame and the tight fit from the foam), steel bars can be mounted across the front of the frame. On the sides of the front of the frame are supports for holding a foam-padded steel bar. The steel bar is drilled through on the ends for clipping carabineer clips through.

3.2.5 Automated Controls, Sensors, Computer, and Electronics

All turning valves necessary to run the experiment are electrically actuated. This allows for hands-free operation of the equipment. All other valves are either check valves that automatically open at a designated pressure or emergency manual release valves.

Temperature of the reaction cylinders is done by external heating coils wrapped around the cylinders. The voltage of these cylinders is controlled by the computer through a solid-state controlled variac. Each cylinder has its own heating coil.

Pressure, temperature, and optical transparency of the reaction cylinders are monitored electronically through a Measurement Computer 16-bit digital-to-analog converter (DAQ). The data from these sensors are then recorded by a computer.

The DAQ also controls actuation of the valves through an array of CMOS-triggered relays.

All controls are operated through a custom-designed computer program running on a Pentium 166 laptop.

3.2.6 Safety Features

We have included numerous design elements to ensure safety.

- All components are rated for at least 3,000 psi, more than twice the pressure expected during the experiment
- All materials have been carefully chosen for chemical compatibility
- Pressure buildup any where in the system can be relieved at any time through one of two sets of relief valves
- Pressure and chemicals can be flushed through one of several outlets to the off-board vent
- In the event all pressure outlets fail, pressure released into the containment box can be vented out through the box's general off-board vent
- The containment box features two independent, sealed layers of impact-resistant polycarbonate to contain any leak or dislodged solid matter
- The containment box has been tested on the KC-135A previously and adequately contained both gas and liquid leaks
- All components used in the system, with the exception of the volumetric pistons, are commercially obtained. The volumetric pistons will be made with the supervision of the University of Wisconsin Space Sciences Engineering Center and the University of Wisconsin Engine Research Lab
- Pressure and temperature are monitored at all times during operation
- The pressure tank used for the experiment will be provided and certified by NASA
- NASA personnel will assist in the setup of pressure tanks and hoses on the KC-135A

3.3 PROCEDURES

Ground Preparations

- 1) Preparation of Reaction Cylinders. Each cylinder is fitted with a Teflon jacket for aerogel to form in. Top and bottom assembly bushings are wrapped with several layers of Teflon tape and tightly pipe-wrenched into the steel cylinders. Seals are checked. Gauges are tested. Valves are checked. The completed cylinder is then snapped into a steel support disc and into the delivery and injection system installed inside the containment box.
- 2) Installation of Heating Coils. Heating coils are slipped onto reaction cylinders and connected to control circuit. Coils are tested.

- 3) Loading of Chemical Reagents. The liquid storage bags are filled with chemical by injection from a Luer-lock syringe. The bags are connected to the plastic delivery lines in the containment box and are secured.
- 4) Computer and Electronics Check. The computer program is loaded and sensors, valve actuation, and heating coils are tested.
- 5) Pressure Test. The system is pressurized and the injection system is tested. Safety relief valves are thoroughly tested.
- 6) Final Check. Equipment is inspected. Box is locked.
- 7) Installation of Experiment. The box will be strapped to the floor of the KC with help from a Test Flight Director. The carbon dioxide pressure tank will be installed by a Test Flight Director as well. Off-board vents will be connected to the ports on the experimental equipment and the equipment will be tested for sealing.

Pre-Flight

- 1) Computer is turned on. Operation program is loaded.
- 2) Liquid carbon dioxide tank is turned on.

In-Flight

- 1) A reaction cylinder is selected by the 10-way valve.
- 2) The 4-way valve is opened to carbon dioxide.
- 3) The reaction cylinder is pressurized.
- 4) The reaction cylinder is slowly heated. As it is heated pressure will increase. When pressure reaches the pressure setting on the adjustable check valve, pressure will automatically be released through the off-board vent. Heated is disengaged when the cylinder is at the proper temperature and pressure.
- 5) The waste cylinder is selected by the 10-way valve and the delivery line is depressurized.
- 6) Tetramethoxysilane is drawn out of an IV bag by the injection piston.
- 7) The 4-way valve is opened to tetramethoxysilane.
- 8) The injection piston pushes tetramethoxysilane through the delivery line into the waste port and aspirates more tetramethoxysilane.
- 9) The 10-way valve selects the original reaction cylinder.
- 10) The injection piston pushes tetramethoxysilane into the reaction cylinder.
- 11) The 10-way valve selects the waste cylinder.
- 12) The four-way valve is opened to carbon dioxide, flushing excess tetramethoxysilane out of the delivery line.
- 13) The 4-way valve is closed, depressurizing the delivery line.
- 14) Formic acid is aspirated by the other injection piston.

- 15) The 4-way valve is opened to formic acid.
- 16) The injection piston pushes formic acid through the delivery line into the waste cylinder and aspirates more formic acid.
- 17) The 10-way valve selects the original reaction cylinder.
- 18) Right before the start of a zero-g phase, the injection piston pushes formic acid into the reaction cylinder initiating the formation of a gel. A laser shining through the piston will be obscured when the cylinder reaches its critical point, detected by a cadmium sulfide cell.
- 19) After 20 seconds, a gel will have formed.
- 20) The process is repeated 9 times during flight.

Post-Flight

- 1) Manual releases on the reaction cylinders are opened. Cylinders slowly depressurize over the course of 2 to 4 hours.
- 2) Liquid carbon dioxide tank is shut off.
- 3) The 10-way selects the waste port and excess line pressure is vented.
- 4) After the reaction cylinders have fully depressurized, they are removed from the system and disassembled. Aerogels are removed. Reaction cylinders are cleaned and prepared for the next flight.

3.4 EXPECTED RESULTS AND MEASUREMENTS

It is expected that we will be able to do the following in weightlessness.

- Safely and successfully prepare supercritical carbon dioxide
- Prepare solutions of tetramethoxysilane and formic acid in supercritical carbon dioxide
- Form silica gels from solutions of tetramethoxysilane and formic acid in supercritical carbon dioxide

These expectations alone meet our first goal, which is to establish methodology for producing aerogel by direct synthesis in weightlessness. To meet our second and third goals, we will be testing for the following:

- Observable differences in the nanostructure or physical properties of aerogels produced by direct synthesis in weightlessness compared with aerogels produced by direct synthesis in 1 G
- Effectiveness of aerogels produced by direct synthesis in weightlessness as insulating and high surface area materials
- Observable macroscopic density gradient reflected in the aerogels' microstructure from aerogels formed in 1 G
- Absence of a macroscopic density gradient in the microstructure of aerogels formed in weightlessness
- Observable nanoscopic density variations in aerogels from opalescent supercritical carbon dioxide

Measurements made during the flight will include:

- Acceleration data taken with a MEMS accelerometer
- Changes in pressure and temperature during forming of supercritical gel taken with pressure gauge and thermometers attached to reaction cylinders
- Clarity of gel during gelation (measured by transmission of 635 nm laser light through the reaction cylinders onto a CdS cell)

Data will be collected with a Measurement Computing 16-bit digital-to-analog PCMCIA card.

Correlating clarity of gel during gelation and changes in pressure and temperature will allow us to ascertain shifts in the critical point of the solution as a result of gel formation.

Measurements taken after the flight will include:

- Bulk density of aerogels formed
- Thermal conductivity of aerogels formed
- Brunauer-Emmett-Teller nitrogen adsorption surface area of aerogels formed
- Dissected 1/4-inch disc bulk density (to determine presence of macroscopic density gradient) of the 8-inch aerogel cylinders
- Nanostructural statistics (cluster chain length, cluster size, and pore diameters) from BET adsorption/desorption isotherms and transmission electron micrographs

From these measurements we expect to see comparable physical properties between aerogels formed in 1 G and aerogels formed in zero-gravity with the exception of a vertical density gradient in aerogels formed in 1 G. We also expect to see differences in nanostructures and pore sizes.

3.5 NEED FOR REDUCED GRAVITY

The experiment requires reduced gravity to show how a method for producing aerogel could be done in the weightless environment of space, to understand the impact gravity has on the nanostructure of aerogels formed through the direct synthesis process, and to attempt to trap nanoscopic density fluctuations present in supercritically opalescent fluid without the distortion caused by the weight of the supercritical fluid pushing down on itself.

The first goal, establishing methodology for production of aerogel in space, requires that a system be designed and tested for operation in a weightless environment, which cannot be done in 1 G.

The second goal, analyzing the effect of gravity on the nanostructure of aerogels produced through direct synthesis, requires that a control set of aerogels produced without the effects of gravity be formed. Our previous research in forming gels (not aerogels) in weightlessness and supercritically drying such gels on Earth has

shown that aerogels produced from gels formed in weightlessness have different nanostructures than aerogels produced from gels formed in 1 G. Since direct synthesis of aerogel from supercritical fluid has never been attempted in weightlessness, it is not certain how the nanostructures of aerogels produced through this process are impacted by gravity, if at all.

The third goal, attempting to trap the nanoscopic density variations in supercritically opalescent fluid, requires that the supercritical fluid be free from the influence of gravity. At the critical point, small variations in pressure result in dramatic changes in density of the fluid, so much so, that just the weight of the fluid pushing down on itself causes a macroscopic density gradient in the fluid. This in turn distorts nanoscopic density fluctuations in the fluid. Understanding how gravity distorts these fluctuations, however, is not easy to gauge. Being able to model and understand supercritical fluids is of great importance to physical chemists, as most equations of state developed to model gas behavior fall apart at the critical point. We believe we may be able to trap some of the nanoscopic density fluctuations of a supercritical fluid in an aerogel with direct synthesis, but in order to understand how gravity affects these nanoscopic density fluctuations, we must be able to trap them without the effect of gravity influencing them and compare them with nanoscopic fluctuations in supercritical fluid in 1 G.

4. NOT A FOLLOW-UP FLIGHT

This is not a follow-up flight to a previous experiment.

5. BIBLIOGRAPHY

Atkins and de Paula in *Physical Chemistry*, 7th ed. (2002)

Chemical Rubber Company, *CRC Handbook of Chemistry and Physics*, 83rd ed. (2002)

Gammon, R. W. "Critical Fluid Light Scattering (Zeno)", in *Microgravity Science and Applications Program Tasks and Bibliography for FY 1992*, NASA Technical Memorandum 4469, NASA Office of Space Science and Applications, Washington, D.C., p. II-69. (March 1993)

Hefter, G. T. and Tomkins, R. P. T. eds., *The Experimental Determination of Solubilities*, John Wiley & Sons. (2003)

Hrubesh, Lawrence W. and Poco, John F. "Processing and Characterization of High Porosity Aerogels," *Lawrence Livermore National Laboratory Pre-print*. (1994)

Jessop, P. and Leinter, W. eds., *Chemical Synthesis Using Supercritical Fluids*, Wiley-VCH. (1999)

Noever, D. A. *Microgravity Sci. Technol.*, Volume 3, p. 14. (1994)

Smith, D. et al. "Effect of Microgravity on the Growth of Silica Nanostructures," *Langmuir*, Volume 16, pp. 10055-10060. (2000)

Tillotson, T. M. and Hrubesh, L. "Transparent Ultralow-density Silica Aerogels Prepared by a Two-Step Sol-Gel Process." *Reports from Third International Symposium on Aerogels, Warzburg, Germany*. (1991)

Vanderhoff J. W.; El-Aasser, M. S.; Micale, F. J.; Sudol, E. D.; Tseng, C. M.; Sheu, H. R. *Polym. Prepr.* 1987, 28, 455; *Mater. Res. Soc. Symp. Proc.* Volume 87, p. 213. (1987)

Zhu, J. X.; Li, M.; Rogers, R.; Meyer, W.; Ottewill, R. H.; STS-73 Space Shuttle Crew; Russel, W. B.; Chaikin P. M. *Nature*, Volume 387, p. 883. (1997)

II. SAFETY EVALUATION

6. FLIGHT MANIFEST

Flyers

Stephen Steiner, Flyer March 2003, Flyer April 2002, Flyer June 2001

Mark Schneider, Flyer March 2003, Ground Crew April 2002

Emily Prewett, Alternate Flyer March 2003

Ben Longmier, No Previous Experience

TechTV Journalist, TBA

Alternate Flyers

Tim Swenson, No Previous Experience

Ben Jaeger, No Previous Experience

Ground Crew

Nick Hanson

7. EXPERIMENT DESCRIPTION/BACKGROUND

Please see Sections 1 and 2 for Experiment Description and Background.

8. EQUIPMENT DESCRIPTION

Please see Section 3 for Equipment Description.

9. STRUCTURAL DESIGN

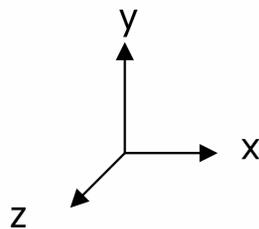
This section is based on our calculations made for the containment box adapted from a previous experiment. The interior of the box will change with installation of the new experiment, but the structural design calculations should stay the same.

9.1 OVERVIEW

The experiment equipment has been designed and built to withstand all G-load specifications required by NASA's Reduced Gravity Student Flight Opportunities Program. Calculations are first performed to show that the apparatus is securely fastened to the floor of the KC-135A. This portion also includes a floor load analysis describing how the equipment will not exceed the allowable load in flight. Next, a structural analysis of the inner box indicates that the internal components will be isolated from the fuselage of the aircraft. Lastly, calculations are shown proving that the inner box will be fully contained within the outer aluminum frame. For purposes of G-load calculations, it will be assumed that the right-hand side of the equipment will face towards the front of the aircraft. Free-body diagrams (FBD's) and induced G-loads are included for all calculations. Critical factors of safety (FS) show that the equipment is safe for all persons and aircraft components aboard the KC-135A.

9.2 CENTERS OF GRAVITY

Center of gravity calculations use a 3-D right-handed coordinate system with the "origin" located at the bottom-left-rear corner of the outer aluminum frame. Individual component weights and positions relative to the origin are used to calculate the center of gravity (CG) for the inner box and the entire system. All dimensions are shown in inches.



The total weight (at 1 G) of the inner box with internal components is 100 lbs. The outside edges of the inner box have dimensions 26 x 32 x 32 (width, height, depth). After considering left-right and front-back symmetry as well as the 2-inch foam layer between the inner box and the outer frame, it can be seen that the CG_x and CG_z of the inner box with all internal components are located at 15 ($26/2 + 2$) and 18 ($32/2 + 2$) inches, respectively. CG_y is a slightly more complicated calculation. The inner box has more weight towards the top due the 10-way valve and other equipment installed at the top. It is assumed that these components will weight 5 lbs with an average height of

about 25. The rest of the internal components (5 lbs.) have relative top-bottom symmetry. The vertical center of gravity is calculated as follows:

$$CG_y = (5*25 + 5*18 + 90*18) / 100 = 18.35$$

Hence, the CG of the inner box with all internal components is (15, 18.35, 18).

The center of gravity of the entire apparatus is required for calculations to show that the equipment will be securely fastened to the aircraft under all G-load specifications. The weight of the outer frame including foam is 40 lbs. with dimensions 30 x 36 x 36 (w, h, d). After considering total symmetry of the outer frame, the CG is (15, 18, 18).

Below are the calculations of the CG for the entire system (*i.e.*, outer frame + inner box + internal components):

$$CG_x = [15*100 + 15*40] / 140 = \mathbf{15 \text{ inches}}$$

$$CG_y = [18.35*100 + 18*40] / 140 = \mathbf{18.25 \text{ inches}}$$

$$CG_z = [18*100 + 18*40] / 140 = \mathbf{18 \text{ inches}}$$

Here it can be seen that the center of gravity for the entire system is located at (15, 18.25, 18).

The weight of the equipment will be assumed to be concentrated at the center of gravity for all moment and factor of safety calculations.

9.2.1 9 G's Forward

Under an induced gravity of 9 G's forward, the entire system will experience a weight of 1,260 lbs. (140*9). The reaction force to this weight in the horizontal direction will be provided by two 2-inch wide cargo straps attached to the handles of the outer frame (bolted to the aircraft with 3/8-inch steel bolts). Summing the forces in the x-direction it can be seen that the straps will each need to provide a reaction force of 630 lbs. in the negative x-direction. Each strap is capable of supplying $5,000 * \cos(72^\circ) = 1,545$ lbs. This results in a factor of safety of 2.45 for the entire apparatus in 9 G's forward in the horizontal direction. Taking the moments about the point (0, 0, 18) it can be seen that the reaction moment for each strap will need to be $1,260 * 18 / 2 = 11,340$ in-lbs. The straps are each capable of providing a moment of $5,000 * \sin(72^\circ) * 5 = 23,780$ in-lbs. This results in a factor of safety of 2.1. Thus, the equipment will remain static under 9 G's forward.

9.2.2 3 G's Aft

Under an induced gravity of 3 G's aft, the entire system will experience a weight of 420 lbs. (140*3). The reaction force to this weight will be provided by two 2-inch wide cargo straps attached to the handles of the outer frame (bolted to the aircraft by 3/8-inch steel bolts). Summing the forces in the x-direction it can be seen that the straps will each need to provide a reaction force of 210 lbs. in the positive x-direction. Each strap is capable of supplying $5,000 * \cos(72^\circ) = 1545$ lbs. This results in a factor of safety of 7.36 in the horizontal direction for the entire apparatus in 3-G's aft. Taking moments about the position (36, 0, 18) it can be seen that each strap will need to provide a reaction

moment of $420 \cdot 18 / 2 = 3,780$ in-lbs. Each strap is capable of supplying a $5,000 \cdot \sin(72^\circ) \cdot 5 = 23,780$ in-lbs. This results in a factor of safety of 6.29.

9.2.3 6 G's Down

While experiencing an induced gravity of 6 G's down, the equipment will experience an induced weight of 840 lbs. The area of the bottom of the equipment is 7.5 ft^2 . This maximum in-flight stress on the fuselage would be 112 lbs./ft^2 . This is well below the maximum allowable in-flight floor loading specification of 200 lbs./ft^2 . This is a factor of safety of 1.79.

Therefore, our experiment does not need to be provided floor shoring to satisfy the g-load specifications in the 6 G's down situation.

9.2.4 2 G's Lateral

The equipment will experience an induced weight of 280 lbs. in the lateral direction. To avoid translational motion in the z direction, the outer frame will need to be ratcheted down with enough force so that the frictional force generated against the aircraft foam is significantly greater than the 280 lbs. of induced force. If the case were to rotate, the cargo straps would need to counter the moment induced about the position (18, 0, 0). The moment that needs to be countered would be $280 \cdot 18 = 5,040$ in-lbs. The tether strap going over the top of the outer frame would need to provide a force of $5,040 / 36 = 140$ lbs. Since the strap is capable of providing a force of up to 5,000 lbs., the factor of safety for 2 G's in the lateral direction is 35.7.

9.2.5 2 G's Up

Under an induced gravity of 2 G's up, the weight of the equipment will be 280 lbs. This weight will be countered by reaction forces provided by the straps over the top of the outer frame. Each strap is capable of providing up to $5,000 \cdot \sin(72^\circ) = 4,755$ lbs. of force when in tension. This results in a factor of safety of $4,755 \cdot 2 / 280 = 34$.

9.3 SUMMARY OF APPARATUS ATTACHMENT TO FUSELAGE OF KC-135A

Factor of safety calculations for the entire system shows that the equipment will remain in static equilibrium for all G-load specifications. The use of two cargo straps will provide sufficient reaction forces to secure the apparatus to the fuselage of the KC-135A.

Table 9-1: Summary of FS calculations for fastening the equipment under all G-load specifications. Forces and moments shown are the reactions that need to be provided by one cargo strap.

Case	Force (lbs.)	Force FS	Moment (in-lbs.)	Moment FS
9 G's forward	630	2.45	11,340	2.10
3 G's aft	210	7.36	3,780	6.29
6 G's down	420	1.79	N/A	N/A
2 G's lateral	Need more info (see text)		5,040	35.7
2 G's up	140	34	N/A	N/A

9.4 STRUCTURAL INTEGRITY OF INTERNAL COMPONENTS INSIDE THE BOX

The inner glove box is designed to sufficiently withstand any loads imposed by the internal components on its frame. Three of the four sides are bolted to the aluminum framework by a series of 18 1/4-inch steel bolts. The cross-sectional area of each bolt is 0.0312 in² and the effective bearing area being stressed in any of the horizontal directions (forward, aft, lateral) is 0.562 in². The shear strength of polycarbonate is 7,000 psi. Therefore, these sides will be able to withstand 3,930 lbs. from internal loading.

The outer polycarbonate door on the inner box is fastened to the aluminum framing by 2 steel hinges and 4 steel locks. It will be assumed that the critical bearing stress for the outer door of the inner box will be held by the steel locks which are attached by significantly smaller bolts than the hinges (which use 8 1/4-inch steel bolts). Each lock has two 1/8-inch diameter steel bolts. Therefore, there is effectively 8 bolts (1/8-inch diameter each, effective total bearing area = 0.125 in²) holding the outer front panel on. The total weight (at 1 G) of all internal hardware components is 10 lbs.

The inner door panel of the inner box is held by four aluminum latches that utilize mechanisms similar to lavatory stall doors. The total effective bearing area of these latches is 0.25 in². Yield strength for the aluminum is 19,000 psi.

It should be noted that there are two layers of polycarbonate along the sides of the glove box. Ultimate structural failure would require both layers to break. This is extremely unlikely to happen, even under 9 G's.

The top and bottom of the inner box each consist of two 1/4-inch sheets of polycarbonate fastened to the aluminum framing with 12 1/4-inch steel bolts. The effective bearing area is 0.375 in².

A table is included that describes the factors of safety for loading induced from internal components to the walls of the inner box.

Table 9-2: Containment summary of internal components inside the inner box.

Case	Induced Force (lbs.)	Structural Strength of Box (lbs.)	Factor of Safety
9 G's forward	90	3,930	43.6
3 G's aft	30	3,930	131
6 G's down	60	2,625	43.8
2 G's lateral left	20	3,930	196
2 G's lateral right outer door	20	8,75	43.8
2 G's lateral right inner door	20	4,750	237
2 G's up	20	2,625	131

Factor of safety calculations indicate that the inner glove box is capable of containing all possible internally induced loads. All internal components will be isolated from the fuselage of the KC-135A.

9.5 STRUCTURAL INTEGRITY OF CONTAINMENT BOX INSIDE ALUMINUM FRAME

Factor of safety calculations show that the inner box will be fully contained within the outer aluminum frame under all necessary G-load conditions. The aluminum angles for the outer frame have 2.5 x 2.5-inch legs and will provide sufficient support to contain the inner box.

$$\text{Shear Stress} = F/A$$

Table 9-3: Summary of FS calculations for shear stress of the outer frame under loading imposed from the inner box.

Case	Induced Stress (psi)	Structural Strength of Outer Frame (psi)	Shear Factor of Safety
9 G's forward	200	19000	95
3 G's aft	66.7	19000	285
6 G's down	185	19000	103
2 G's lateral	44.5	19000	427
2 G's up	44.5	19000	427

$$\text{Bending Stress} = M*Y/I$$

M = maximum bending moment = Induced load * 1/2 max. length = F * 18

Y = 1/2 width of bending area = 0.5 inches

I = moment of inertia = 1/12 bh³ = 0.25 in⁴

Table 9-4: FS associated with bending of outer aluminum frames from load induced from inner box.

Case	Induced Stress (psi)	Structural Strength of Outer Frame (psi)	Bending Factor of Safety
9 G's forward	8,100	19,000	2.35
3 G's aft	2,700	19,000	7.04
6 G's down	5,400	19,000	3.52
2 G's lateral	1,800	19,000	10.6
2 G's up	1,800	19,000	10.6

10. ELECTRICAL SYSTEM

All operations in the containment box are electrically automated and computer controlled.

The computer is a Pentium 166 MHz laptop equipped with a Measurement Computing 16-bit PCMCIA digital-to-analog converter (DAQ). The DAQ interfaces with a relay box which controls motion of valves and the linear actuators in the box. The DAQ also controls power to the laser diodes shining through the reaction cylinders. The DAQ is connected to digital pressure gauges and thermometers on the reaction cylinders and measures pressure and temperature. The DAQ also measures the resistance of cadmium sulfide cells opposite the laser diodes which measure the optical clarity of the reaction cylinders.

Two power strips will be used connecting to two separate 120VAC outlets on the KC-135A Power Distribution Panel. This is so that there will be sufficient power to run all equipment since the actuators require 15 Amps each.

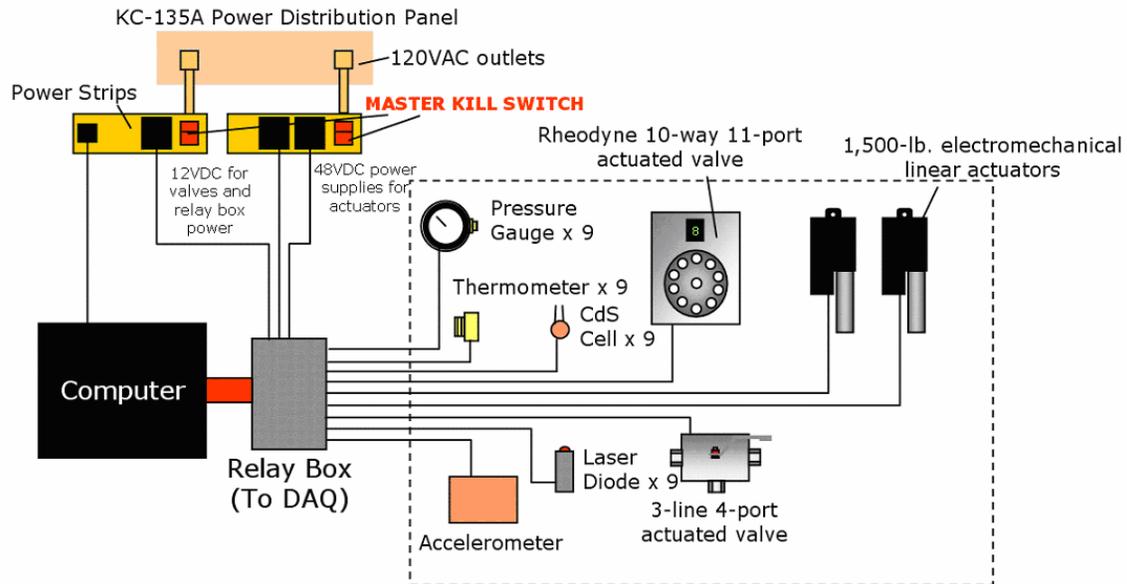


Table 10-1: Electrical Components

Name	Voltage	Current	Function
Computer	12VDC	2.0 Amps	Control/data collection
Relay Box	12VDC	500 mA	Controls valves and actuators
10-way Valve	12VDC	2.0 Amps	Selects reaction cylinder
3-line Valve	12VDC	1.0 Amps	Selects line
Linear Actuators	48VDC	15.0 Amps each	Drive chemicals into pressurized cylinders
Pressure Gauge	5VDC	200 mA each	Measures pressure in reaction cylinders
Thermometers	5VDC	200 mA each	Measures temperature in reaction cylinders
Laser Diodes	5VDC	60 mA each	Shines laser through reaction cylinder
Voltage Dividers	5VDC	200 mA	Measures resistance of CdS photocells
Accelerometer	5VDC	50 mA	Measures acceleration

11. PRESSURE/VACUUM SYSTEM

See Section 3 for more detail about the high-pressure equipment.

11.1 OVERVIEW

The entire experimental apparatus is a pressure system designed to conduct a chemical reaction with supercritical carbon dioxide. The experiment requires pressurized carbon dioxide in each reaction cylinder. Each cylinder has a volume of approximately 6.3 cubic inches and is pressurized separately throughout the flight. Total volume of carbon dioxide present in the system at any one time will not exceed 60 cubic inches. Carbon dioxide is introduced into the system as pressurized liquid, which boils until it reaches its vapor pressure of 750-900 psi, depending on ambient temperature. Supercritical carbon dioxide is prepared in pressurized reaction cylinders by external heating from a heating coil. As heating occurs, the pressure of the carbon dioxide in the pressurized reaction cylinder will increase rapidly. Pressure beyond the critical pressure is released through a check valve out to the off-board vent until the temperature of the cylinder reaches the critical temperature. Pressure in the reaction vessels should not exceed 1,200 psi (the exact pressure is dependent on the critical point of a solution of carbon dioxide, tetramethoxysilane, and formic acid, which is yet to be determined.) Pressure elsewhere in the system will be the vapor pressure of carbon dioxide at room temperature, between 750 and 900 psi.

11.2 PRESSURE RELIEF STRATEGIES

The system has numerous pressure relief strategies. They include:

- Check valves set to open automatically upon pressure buildup
- Redundant check valves in the event primary check valves fail
- Check valves and manual ball valves on every reaction cylinder
- Off-board vent connections to all check valves and manual release valves
- A general off-board vent in the containment box to vent any liquid or gas leaks

12. LASER SYSTEM

Three shielded laser diodes will be used to monitor the status of supercritical opalescence in the reaction cylinders.

- a) Lasers are Class II, red laser diodes (similar to those used in laser pointers), 6 mm dot @ 3 meters, <1 mW optical power, 60 mA maximum current, made by National Semiconductor for the Tandy Corporation.
- b) Lasers are used to shine light through the reaction cylinders onto photocells to electronically monitor the status of supercritical opalescence.
- c) Lasers will be active during the entire flight.
- d) Lasers are shielded in opaque plastic casings and can be deactivated by a switch in the box or by the master kill switch on the power strip outside of the box.

13. CREW ASSISTANCE REQUIREMENTS

Crew assistance is requested for setting up a liquid carbon dioxide pressure cylinder on the KC-135A and connecting it to the experimental equipment. Crew assistance is also requested to ensure proper interfacing with the off-board vents and that all high-pressure connects are properly sealed.

14. INSTITUTIONAL REVIEW BOARD (IRB)

The experiment does not require approval by an institutional review board.

15. HAZARD ANALYSIS

Hazard Number: 1

Hazard Description: There is a pressure leak in one of the pressurized cylinders where the gel is formed.

Hazard Causes:

- 1) Insufficient sealing at the bushing
- 2) Structural flaw such as a crack
- 3) FODD in box causes damage to cylinder
- 4) Impact to cylinder in transportation

Hazard Repercussions:

- 1) Pressure build up in box opens door.
- 2) Toxic vapors enter cabin.
- 3) Explosion inside box.

Hazard Controls:

- 1) Teflon tape seals. Inspect and ensure the seal is maintained prior to flight or pressurization.
- 2) Inspect all cylinders prior to flight or pressurization to ensure no damage has occurred.
- 3) Pressure test all cylinders to ensure they are “go for flight” prior to flight.
- 4) In the event that previous precautions fail, cylinders will depressurize through a check valve to an off-board vent.
- 5) If there is a failure with a check valve a manual ball valve can be opened.
- 6) All chemicals and vapors are able to be vent via the off-board vent.
- 7) There are multiple off-board vents, one connected to the cylinders and one to the box itself, to ensure venting ability is maintained if one of the vents fail.
- 8) There are three layers of containment, including two sealed doors that will not be able to open. The box would be depressurized through the general off-board vent.
- 9) All materials are certified for pressures well beyond our maximum working pressure.

Hazard Number: 2

Hazard Description: Structural components inside box become insufficient to support experiment device.

Hazard Causes:

- 1) Damage to supports due to FODD inside box.

- 2) Bolts come loose.
- 3) Free-floating objects inside box obstruct function of supports.

Hazard Repercussions:

- 1) Pressure cylinders that are insufficiently supported could fall over.
- 2) Pressure cylinder could break if it falls, releasing toxic chemicals.

Hazard Controls:

- 1) Numerous securely bolted components.
- 2) L-beams are directly bolted to frame of box.
- 3) Multiple supports for each part of experiment component in case one component fails.
- 4) Reaction cylinders are snapped in place by quick connects and fastened in a support disc.
- 5) Inspect and tighten bolts prior to flight.
- 6) Box has two layers of impact resistant polycarbonate.
- 7) Any leak would be contained in the box and then vented via the off-board vent, so there is no threat to the cabin.
- 8) All materials involved in pressurization are certified well beyond our maximum working pressure.

Hazard Number: 3

Hazard Description: The IV bags containing toxic chemicals come loose.

Hazard Causes:

- 1) Loose bolts.
- 2) Plastic holding bag up breaks unexpectedly.

Hazard Repercussions:

- 1) Release of hazardous chemicals into the box.
- 2) Bag could come into contact with heating coils, melting plastic and coming into contact with chemicals.
- 3) Chemicals are flammable. Fire ignites.

Hazard Controls:

- 1) IV bags are placed far away from heat source in box.
- 2) The IV bags have a polycarbonate shield around them.
- 3) The IV bags are insulated to minimize movement. This minimizes the chance of a bag breaking off its support.
- 4) The electric heating coils can be turned off if the chemicals are in danger of coming into contact with the coils.
- 5) There is a clean up kit inside the box, equipped with baggies and paper towels to clean up spills prior to opening box once on the ground.
- 6) The off-board vent will clear out vapors while in flight.

- 7) The box has three layers of containment so that the cabin is not exposed if a leak or spill occurs.

Hazard Number: 4

Hazard Description: Problems with the off-board vents

Hazard Causes:

- 1) Leaks or damage to hoses.
- 2) Insufficient connection to box.
- 3) Foreign object clogs vent.

Hazard Repercussions:

- 1) Pressure buildup in box or system.
- 2) Toxic vapors not vented outside cabin.

Hazard Controls:

- 1) Safety inspection check prior to flight.
- 2) Carefully designed system to minimize leaks and need for off-board vent. The off-board vent is a safety feature; it should not need to be used during our experiment.
- 3) Design allows for any leaks to be contained in box until landing, when box can be removed from aircraft.
- 4) There are multiple off-board vents in our system. It is unlikely that all would fail.
- 5) All materials used in delivery systems are inert to the chemicals used. The rest of the materials in the box that could potentially be exposed to chemical due to a leak are also inert.

Hazard Number: 5

Hazard Description: The electric coils overheat and cause over-pressurization of cylinders. Also see Over-Pressurization of Cylinders.

Hazard Causes:

- 1) Electrical failure causes coils to behave uncontrollably. Heaters are no longer regulated.

Hazard Repercussions:

- 1) Over-pressurization of cylinders.
- 2) Ignition of fluids if extremely hot and fluids come in contact with coils.
- 3) Could cause fire or explosion inside box.
- 4) Damage to equipment.

Hazard Controls:

- 1) Manual disengagement of heat source.

- 2) Manual depressurization of cylinders using relief valves.
- 3) The box is equipped with a port to an off-board vent.
- 4) The system can be flushed with CO₂ to douse fire if such should occur.
- 5) The box is made with two layers of impact-resistant material.
- 6) Thermometer in box to manually monitor temperature.

Hazard Number: 6

Hazard Description: The actuated piston fails.

Hazard Cause:

- 1) Electrical failure/computer failure
- 2) Mechanical failure

Hazard Repercussions:

- 1) Uncontrollable/unexpected high pressure injection of chemicals.
- 2) Seal in delivery line bursts, toxic chemicals released.

Hazard Controls:

- 1) A check valve connected to the volumetric pistons would direct pressurized liquid to an off-board vent.
- 2) A redundant check valve connected to the volumetric piston would work if the primary check valve failed.
- 3) Any leak would be vented via the off-board vent in the containment box so the cabin is in no danger.
- 4) Any spill could be cleaned up with a clean up kit placed inside containment box.
- 5) Three layers of containment are in place.
- 6) Power source can be manually deactivated.

Hazard Number: 7

Hazard Description: The actuated valves fail.

Hazard Cause:

- 1) Electrical/Computer failure

Hazard Repercussions:

- 1) Unexpected pressurization/depressurization of cylinders.
- 2) Over-pressurization of cylinders.
- 3) Leakage of chemicals into box.

Hazard Controls:

- 1) Depressurization of cylinders is not hazardous.
- 2) Relief valves have a manual release button in case of over-pressurization.

- 3) Each reaction cylinder has a manual ball valve to vent pressure as well.
- 4) Any leak can be cleaned up without opening box with clean up kit located inside box.
- 5) The off-board vent in the containment box will get rid of any hazardous vapors.

Hazard Number: 8

Hazard Description: Damage occurs to CO₂ tank valve.

Hazard Causes:

- 1) CO₂ tank becomes loose in cabin and strikes another object
- 2) Foreign object impacts CO₂ tank causing valve failure

Hazard Controls:

- 1) NASA flight crew will prepare tank for flight
- 2) NASA flight crew will secure tank to wall of plane
- 3) The tank will be flight certified
- 4) Tank valve can be shut off from experiment

Hazard Number: 9

Hazard Description: Damage occurs to hose connecting CO₂ tank and equipment box..

Hazard Causes:

- 1) Failure due to over pressurization
- 2) Failure due to foreign object severing tube
- 3) Tube connection leak

Hazard Controls:

- 1) Hose will be test prior to flight
- 2) Hose will be flight certified by NASA ground crew
- 3) The hose will be secured to the floor and/or walls of the plane during flight to avoid impact of foreign objects
- 4) Hose connections will use Teflon tape to ensure proper seals

Hazard Number: 10

Hazard Description: Pressure leak in hose connecting port in containment box to the internal experiment cylinders.

Hazard Causes:

- 1) Leak from joints or connections
- 2) Hose failure
- 3) Overpressurization of connecting tube

Hazard Controls:

- 1) All joints and connections will be sealed with Teflon tape
- 2) All hoses will be pressure certified for 3,000 psi (more than three times the pressure of the CO₂ tank).
- 3) Pressure relief valves throughout entire system and off-board vent port in box will ensure that over pressurization does not occur
- 4) Entire pressure system will be tested prior to flight

Hazard Number: 11

Hazard Description: KC-135AA's venting abilities cease.

Hazard Causes:

- 1) Venting valve malfunction
- 2) Ventilation tube becomes blocked

Hazard Controls:

- 1) Venting valves and connecting tubes will be testing by NASA ground crew prior to flight
- 2) Experiment can be shutoff either by computer or manual controls
- 3) All experiment pressure tanks and hoses can be sealed

Hazard Number: 12

Hazard Description: Electrical system draws too much current.

Hazard Causes:

- 1) Overloaded/stuck actuator causes current to actuator to rise.
- 2) Actuated valve becomes stuck and causes more current to be drawn by the valve.
- 3) Insulation of wires is worn, wires short circuit.
- 4) Improper instructions from computer allow for too many devices to operate at once, increasing the current.

Hazard Repercussions:

- 1) Overheating of electrical components can lead to malfunction of system.
- 2) Overheating can burn operator or start a fire.
- 3) Dangerous levels of current could cause sparks and shock/electrocute the operator.

Hazard Controls:

- 1) The system has breakers and fuses on all devices that have potential to draw large amounts of current.
- 2) Manual shut off of entire electrical system by the use of MASTER KILL SWITCH.
- 3) CO₂ could be used to put out any possible fires inside the box.
- 4) All wiring used will be of proper gauge to handle the specified maximum currents.
- 5) The main power strip has a built-in 20 Amp breaker.
- 6) The operator will be wearing two layers of rubber gloves to protect from the possibility of burns and electrical shock.
- 7) The electrical system is designed to prevent too many devices to be operating at once, and the software will also be programmed to prevent the operation of too many devices.
- 8) All electrical connections will be checked and the system will be tested prior to flight.

Hazard Number: 13

Hazard Description: Electrical system/computer crashes or becomes unstable.

Hazard Causes:

- 1) Poor design of system allows feedback to become uncontrolled and makes the system unstable.
- 2) Program crashes due to improper commands or unsuitable programming.
- 3) A power spike disrupts the system.
- 4) Short circuit due to poor electrical design or wear on wires.
- 5) Computer program sends improper signals to system, producing unstable response.
- 6) Loss of power from the KC-135.

Hazard Repercussions:

- 1) Loss of automated control of the system.
- 2) Unpredictable operation of the system, actuator dispenses liquid, coils heat up, actuated valves turn, or LEDs blink.
- 3) Pressure builds due to heating or improper actuator movement.
- 4) Unexpected operation of system may cause injury to operator or cause a chemical spill.

Hazard Controls:

- 1) Manual shut off of system will allow the operator to stop equipment if it is not operating properly.
- 2) Three layers of containment will contain any chemical leaks and provide protection for the operator.

- 3) Individual manual and automated control of the heating coils and actuator will allow the operator to shut down these systems if they are malfunctioning.
- 4) Relief valves will allow the system to vent off any extra pressure anywhere due to a malfunctioning electrical system to the off-board vent.
- 5) Rugged programming that will help prevent the system from becoming unstable.

Hazard Number: 14

Hazard Description: Malfunctioning sensors or loss of feedback.

Hazard Causes:

- 1) Sensors hooked up improperly.
- 2) Sensors become disconnected or shorted.
- 3) Sensors give inaccurate readings due to damage or error in the system.
- 4) Poor electrical design leads to operation of sensors beyond their specified ratings.

Hazard Repercussions:

- 1) Inaccurate data from sensors.
- 2) Inaccurate data leads to overheating of cylinder which leads to overpressurization.
- 3) If sensors completely fail then the conditions of the cylinders will be unknown which may lead to overpressurization or overheating.
- 4) Overheating may cause injury to operators.

Hazard Controls:

- 1) The system will be designed to operate within the specifications of all the devices.
- 2) The sensors will be checked and tested prior to flight to verify functionality and accuracy.
- 3) The system can be manually shut down and depressurized if the sensors are inaccurate or malfunctioning.
- 4) All sensors are backed up by analog gauges.
- 4) Off-board venting and relief valves will protect against overpressurization due to inaccurate readings of the sensors.

Hazard Number: 15

Hazard Description: Exposed electrical wires inside or outside of the containment box.

Hazard Causes:

- 1) Sharp edges of box or other materials cut the wires.
- 2) Wires are worn or broken during operation and construction.
- 3) Device becomes disconnected exposing electrical contacts.
- 4) Wires/connections are broken due to pulling of wires/leads.

Hazard Repercussions:

- 1) Possibility of electrical shock or electrocution.
- 2) Sparks due to exposed wires cause fire.
- 3) Wires become shorted by grounding producing a large current.
- 4) Loss of system functionality and control.

Hazard Controls:

- 1) Fuses and breakers will be used to prevent overdrawn currents that may be caused by shorts.
- 2) Fully insulated wires of the appropriate gauge will be used in the entire system.
- 3) The wires will be inspected and tested prior to the flight.
- 4) All sharp corners/edges will be covered by a protective layer of foam.
- 5) A manual shut off switch will allow a quick and safe way of shutting off the electricity to the system if an exposed wire is found.
- 6) The operator will be wearing rubber gloves that will protect from shock from any exposed wires inside the box

Hazard Number: 16

Hazard Title: Lasers cause damage to operator's retina.

Hazard Cause:

- 1) Laser is bumped causing it to be pointed into the operator's eye.
- 2) Laser becomes loose inside the box allowing it to point into or reflect a beam of light into the eye of the operator.

Hazard Repercussions:

- 1) Operator may be injured by temporary or permanent eye damage.
- 2) Operator may react in a potentially hazardous way, making the system malfunction.

Hazard Controls:

- 1) The lasers will be firmly mounted inside the box and point away from the operator or any other observer.

- 2) Lasers used will be class 2 with power output of 20 mW meaning that they are less likely to seriously injure the operator.
- 3) If the lasers do become free floating they can be manually shut off by the operator.

16. TOOL REQUIREMENTS

Tools required for equipment maintenance will primarily be borrowed from the tool chests at Ellington Field. Any tools brought by us will be contained in a composite tool kit and labeled “UW” for identification. This should provide a quick means of discovering missing tools and minimize risk of tools causing foreign object damage (FOD) to aircraft.

The following is a list of items we will likely bring with us to Ellington Field.

- Crescent wrench set
- Standard flathead and Phillips screwdrivers
- Duct tape
- Needle-nose pliers
- Teflon tape
- Electric screwdriver/drill kit
- Spare bolts, washers, and nuts for equipment

17. GROUND SUPPORT REQUIREMENTS

Power Requirements:

Standard 120 VAC 60 Hz power is required for testing equipment.

Hazardous Substance Requirements:

Storage for tetramethoxysilane, formic acid solution (85%), and ethanol is required, which are all flammable liquids.

Access to Building 993 During Non-Business Hours:

No access to Building 993 during non-business hours is requested.

Pressurized Gas Requirements:

The experiment will require the use of a liquid carbon dioxide siphon tank, which will be required for testing on the ground prior to flight.

General Tools Requests:

No general tools requests.

18. HAZARDOUS MATERIALS

Material safety data sheets (MSDS) will be provided with the TEDP.

Tetramethoxysilane

Use: Chemical injected to form supercritical gels in apparatus.

Hazards: Toxic, lachrymator (eye irritant), targets lungs, blood, kidneys, flammable

Amount Used: Up to 2 L

Containment: Stored in sealed glass bottles in flammables storage chest

Formic Acid Solution

Use: Chemical injected to catalyst supercritical gel formation in apparatus

Hazards: Toxic, corrosive to some materials, flammable

Amounts Used: Up to 1.5 L

Containment: Stored in polypropylene bottles in flammables storage chest

Ethanol

Use: Cleaning equipment

Hazard: Flammable

Amount Used: 500 mL

Containment: Stored in polypropylene carrying bottles in flammables storage chest

19. PROCEDURES

19.1 Shipping Equipment to Ellington Field

Equipment will be brought to Ellington Field by car.
Chemicals will be ordered from Sigma-Aldrich and delivered to Ellington Field by UPS.

19.2 Ground Operations

The equipment will be set up and tested as it would be run on the KC. This will require set up of a liquid carbon dioxide siphon tank. The system will be pressurized, liquids will be aspirated and injected in pressurized reaction cylinders, and test gels will be formed. Electronics and actuators will be tested thoroughly.

19.3 Loading

The containment unit containing the experimental apparatus will be loaded onto the KC by forklift.

19.4 Pre-Flight

The box will be strapped to the floor of the KC with help from a Test Flight Director. The carbon dioxide pressure tank will be installed by a Test Flight Director as well. Off-board vents will be connected to the ports on the experimental equipment and the equipment will be tested for sealing.

19.5 In-Flight

- 1) A reaction cylinder is selected by the 10-way valve.
- 2) The 4-way valve is opened to carbon dioxide.
- 3) The reaction cylinder is pressurized.
- 4) The reaction cylinder is slowly heated. As it is heated pressure will increase. When pressure reaches the pressure setting on the adjustable check valve, pressure will automatically be released through the off-board vent. Heated is disengaged when the cylinder is at the proper temperature and pressure.
- 5) The waste cylinder is selected by the 10-way valve and the delivery line is depressurized.
- 6) Tetramethoxysilane is drawn out of an IV bag by the injection piston.
- 7) The 4-way valve is opened to tetramethoxysilane.
- 8) The injection piston pushes tetramethoxysilane through the delivery line into the waste cylinder and aspirates more tetramethoxysilane.
- 9) The 10-way valve selects the original reaction cylinder.
- 10) The injection piston pushes tetramethoxysilane into the reaction cylinder.

- 11) The 10-way valve selects the waste cylinder.
- 12) The four-way valve is opened to carbon dioxide, flushing excess tetramethoxysilane out of the delivery line.
- 13) The 4-way valve is closed, depressurizing the delivery line.
- 14) Formic acid is aspirated by the other injection piston.
- 15) The 4-way valve is opened to formic acid.
- 16) The injection piston pushes formic acid through the delivery line into the waste cylinder and aspirates more formic acid.
- 17) The 10-way valve selects the original reaction cylinder.
- 18) Right before the start of a zero-g phase, the injection piston pushes formic acid into the reaction cylinder initiating the formation of a gel. A laser shining through the piston will be obscured when the cylinder reaches its critical point, detected by a cadmium sulfide cell.
- 19) After 20 seconds, a gel will have formed.
- 20) The process is repeated 9 times during flight.

19.6 Post-Flight

A manual release mechanism on the adjustable check valves attached to each cylinder is activated, slowly depressurizing the reaction cylinders. The liquid carbon dioxide siphon tank is turned off and all delivery lines are depressurized.

19.7 Offloading

The equipment is disconnected from the liquid carbon dioxide siphon tank, off-board vents, and is unstrapped from the floor. The containment unit is then taken off the plane by forklift. Equipment will be brought back to Wisconsin by car.

III. OUTREACH

20. OUTREACH PLANS

20.1 OUTREACH OBJECTIVES

The goals of our outreach are to:

- Encourage and inspire elementary, middle, and high school students to attend college and get involved with engineering and science
- Show the exciting opportunities provided by the University of Wisconsin and NASA
- Get students and the public interested and excited by science happening in their state
- Inform students and the public about aerogel and its applications
- Spread outreach to elementary, middle, and high school students from underrepresented backgrounds (ethnicity, language, gender, and economic status)

To accomplish these goals, we propose developing and organizing the following activities.

20.2 PLANNED OUTREACH ACTIVITIES

20.2.1 TechTV

Brandon Mercer, producer at TechTV, has enthusiastically agreed to have TechTV accompany our team to Houston upon acceptance to the Reduced Gravity Student Flight Opportunities Program. TechTV is a young cable network with a rapidly growing audience featuring shows about technology, computers, and science. TechTV is available in many areas on standard cable and is available everywhere on digital cable packages.

TechTV's own description: "TechTV is the only 24-hour cable television network dedicated to showcasing the impact technology has on our everyday lives and the world at large. By creating and delivering entertaining and insightful programming regarding today's and tomorrow's technology news, events, products, and people, TechTV enables viewers to stay current and connected with all things related to technology. TechTV is currently available in more than 33 million households and distributes content to 70 countries. With an average of 1.6 million unique visitors in Q1, techtv.com enhances the TV viewing experience with compelling companion content and interactivity."

TechTV will be producing a news segment about our experiment and RGSFOP for one of their nightly television programs. Topics including the KC-135A, aerogel, and engineering will be discussed. The segment will also be distributed to

their partners ABCNews and CNN Headline News, who incorporate TechTV segments into their nightly news broadcasts. Additionally, local ABCNews affiliates around the country will have the option of picking the story up.

20.2.2 Expand Your Horizons Program

Expand Your Horizons is a program sponsored by the University of Wisconsin to encourage young women to enroll in engineering. Emily Prewett on our team is involved with this program. We will be giving a presentation at this program about our research with emphasis on showing the exciting opportunities the University of Wisconsin and NASA have for young women.

20.2.3 Big Brothers/Big Sisters of Madison Field Trip

Overview:

This initiative is a half-day workshop and tour reach-out program to all children involved in the Madison-area Big Brother Big Sisters Program.

Big Brothers Big Sisters Program Background:

The Big Brothers and Big Sisters (BBBS) organization is a nation-wide program with a locally-run branch in Madison, Wisconsin affiliated with the United Way of Dane County.

Most or all young participants or “Littles” are from single parent households, usually living with their mothers. They include both males and females, ranging in age from 5 years old to 14 years old at time of placement and are mostly minorities. Almost all live in the economically deprived areas of the city below or near poverty level and many have experienced physical, emotional or drug/alcohol abuse either directly or indirectly. They often have academic and social problems in school with little available help at home. These children and their parent(s) choose to be in the program because they want to enrich the children’s lives with new social and academic experiences, and develop a bond with a personal positive role model.

Volunteers or “Bigs” in the program also have diverse backgrounds. All wish to improve and enrich the lives of at-risk youths by providing positive and stimulating activities in a supportive atmosphere. Ben Longmier on our team is such a volunteer.

Goals of the Initiative:

- Allow participants to experience the wonder and importance of college first hand, and impress upon them that they can go to college and shouldn’t give up on their educations, no matter the barriers or outside influences.
- Excite participants about science in the classroom and in our world, and inspire a love of science, space exploration, technology and learning itself that will hopefully propel them to succeed in school and more fully enjoy their lives.

Logistics of the Initiative:

On a specific Saturday in the spring, as many as ten Bigs will come to the University of Wisconsin College of Engineering with their Littles. They will be greeted by the Zero-G Aerogel Team and brought to the Engineering Centers Building, a 50-million dollar building constructed in 2002 dedicated to the engineering campus. After a tour of the building of what it's used for and how college students use it for class, study and research, the Littles, Bigs and Zero-G Aerogel Team members will go to a small auditorium for a short presentation about our project. The talk will cover the objective of research in zero-gravity, what zero-gravity is and how it is achieved, and fun recognizable photos and videos to communicate ideas. We will then show our equipment and talk about aerogel, followed by encouragement to the children on how they can get involved in zero-g and other research in the future. The talk will be kept short to keep the children's attention, and will aim to be both informative and inspiring.

The Zero-G Aerogel Team members will then split up to take small groups of Littles and Bigs, based on the Littles grade and age. The groups will then participate in hands-on interactive activities to help the children understand zero-g and aerogel. Activities will include:

- Discussions of what a gel is (accompanied by edible gelatin to play with and eat)
- Trying out dehydrated "space-food"
- Airplane making and flying contests that attempt to replicate the KC-135A flight path
- Bucket swinging demonstrations, object dropping demonstrations, and mathematical problem solving workshop to help understand gravity and its effects

Each small group will then tour the engineering campus with their Bigs and Zero-G Aerogel Team member. The tour will show the buildings and facilities on campus, but focus on the importance of college, what to do in order to get into college, and how fun and interesting it is to study at a post-high school level. After the tours everyone will meet again in a different engineering building for a nutritious group lunch. During lunch a team member will sit at each table to facilitate discussion and to answer questions.

20.2.4 www.zerogaerogel.com

For the past three years we have operated a website at www.zerogaerogel.com. In a Google search for "aerogel" our site is currently number 11. We are linked to by Lawrence Berkeley National Laboratory, the National Geographic Channel, and Los Alamos National Laboratory. The site explains our projects in plain and technical English along with fun and scientifically accurate descriptions of what aerogel is and how it is made. The site also has video segments of our experiments produced by the

National Geographic Channel and ourselves and features the most comprehensive collection of aerogel photos on the web.

This year although our experiment is new, it is still related to aerogel production in zero-g. We are going to revamp our entire site and develop a new Flash-enabled site with more depth into aerogel, interactive web activities, and video segments produced by TechTV. We will also construct a new “Outreach” page with posters we have produced about aerogel and the KC-135A to download and print.

We also plan on working with the University of Wisconsin Institute for Chemical Education, a world-renowned innovator of educational materials, in developing some high-school lab experiments with gels.

Additionally, we will continue entertaining questions about aerogel and zero-g through our email links on the site, which we currently receive on the order of 7 to 10 per week. We respond to questions in English, Spanish, and Italian and will be adding a Russian questions link this year, as the number of Russian immigrants in our state is increasing rapidly.

20.2.5 espanol.zerogaerogel.com

Although for the past three years our website www.zerogaerogel.com has entertained and responded to questions in Spanish, we do not currently have content written in Spanish on the site. We receive a tremendous amount of traffic from Spanish speakers from both the United States and other countries. Our goal is to translate our site into Spanish with the aim of making it readable by Spanish speakers ranging from elementary school students to university students in Spanish-speaking countries. We will also compile a list of links linking to aerogel- and space-related sites written in Spanish.

20.2.6 All-Day Presentations to Science Classes at Nicolet High School

As part of our on-going outreach efforts with Nicolet High School in Glendale, Wisconsin, we will be presenting to 5 science classes either in spring or fall of 2004. The presentations will focus mainly on the physics of weightlessness and the chemistry of aerogel. The presentations will include a PowerPoint presentation along with fun videos of us in weightlessness and new video produced by TechTV. We will also bring in samples of gels and aerogels to show to the classes. We currently have made arrangements with Joy Brandstrom, Earl Feltyberger, and Karyl Rosenberg at Nicolet High School to give presentations to their science classes. We are working with these teachers to conduct high-school lab experiments involving aerogels in their classes as well.

20.2.7 Presentations to Science Classes at Madison Memorial High School

We have been invited by Ben Senson, teacher at Madison Memorial High School, to give a presentation to his aerospace students. The class is open to junior and senior high school students. We will give a PowerPoint presentation and show

some video from our flight, focusing on the KC-135A flight and talking about aerogel as an aerospace material. We've included Ben Senson's email below inviting us to present in his classes.

From "Benjamin J. Senson" <bsenson@madison.k12.wi.us>
Sent Wednesday, October 15, 2003 4:36 pm
To BEN WESLEY LONGMIER <bwlongmier@students.wisc.edu>
Cc
Bcc
Subject Re: zero-g talk

Dear Ben and Zero-G Team,

The Memorial High School Aerospace course would love to have you back for another presentation regarding the use of the KC-135a "Vomit Comet" for research. The talk went very well last year and was greatly appreciated by the students. Please feel free to use this reply as a letter of invitation to have us participate in you educational outreach component of the project.

I look forward to working with you in the spring.

Most Sincerely,

Benjamin J. Senson
Aerospace Instructor
Memorial High School

20.2.8 Engineering Saturday for Tomorrow's Engineers At Madison Program

The University of Wisconsin College of Engineering runs ESTEAM to bring prospective students to the University of Wisconsin engineering campus and show them the unique and exciting opportunities the University of Wisconsin has. High school students are brought to campus on a given Saturday and tour the engineering departments they are interested in. Student organizations involved in engineering projects can volunteer to participate in the program and setup a display to show the students what kinds of projects they can get involved with.

We will be participating in this program and will be giving a brief talk to students on their tours through the engineering campus about the Reduced Gravity Student Flight Opportunities Program and how they can get involved as freshmen.

20.2.9 ASPIRE

ASPIRE (Achieving Success by Promoting Interest in Higher Education) is a student-run mentoring program at the University of Wisconsin that brings middle school students in the Madison, Wisconsin area to campus for activities with a University of Wisconsin student mentor. The program presents college as a realizable goal for students from backgrounds underrepresented in institutions of higher education (ethnicity, first-generation college students, economic background).

Margaret Fink, leader of the organization, has invited us to participate in this program. The organization is striving to not only inspire middle school students but also to teach them something new on their visits to campus this next year. We will be setting up a workshop for visitors with hands-on activities, photos, and videos related to NASA, aerogel, and our zero-gravity research. Visits will take place on 5 Saturday mornings February-March 2004.

20.2.10 Wisconsin Space Conference Presentation

As grant recipients from the Wisconsin Space Grant Consortium, we are invited to present our research at the next annual Wisconsin Space Conference, which will be held in August of 2004. We will present the technical findings from our experiment and detail the method of implementation and engineering developed for the experiment to an audience of scientists, engineers, and businessmen from around the state.

20.2.11 American Institute for Aeronautics and Astronautics Presentation

As five of the members on our team are also members of the AIAA, we will be presenting the results of our research to our AIAA chapter. The talk will be mostly technical and will focus on the challenges of engineering for weightlessness and the applications of our experiment to weightless materials processing.

20.2.12 Press Plan

In addition to coverage from TechTV, we intend to contact the following news organizations about our experiment.

The Daily Cardinal

The Daily Cardinal is one of the University of Wisconsin's primary student newspapers and is widely read on campus. Maya Ziv-el, writer for *The Daily Cardinal*, has expressed interest in writing an article about our experiment and our organization in a future issue of their publication.

The Wisconsin State Journal

The official state newspaper *The Wisconsin State Journal* is frequently interested in University of Wisconsin activities. We plan on contacting them to see if they would be interested in writing an article about our zero-gravity research.

The Milwaukee Journal-Sentinel

The Milwaukee Journal-Sentinel is Wisconsin's most widely-read newspaper. The publication has published an article about our research in the past. We plan on

contacting the newspaper to see if they would be interested in doing an update on our research.

The Glendale Herald

The local newspaper of Glendale, Wisconsin, *The Herald* has expressed interest in doing a story on our project and our outreach at Nicolet High School.

WISN-TV Milwaukee and WKOW-TV Madison

With TechTV distributing to local ABC affiliates, we will contact Milwaukee's local ABC affiliate WISN-TV and Madison's local ABC affiliate WKOW-TV to see if they would carry the segment on their nightly news.

IV. ADMINISTRATIVE REQUIREMENTS

21. NASA/JSC HUMAN RESEARCH CONSENT FORM

This experiment does not involve human research.

22. IUCAC

This experiment does not involve any animal research.

23. FUNDING/BUDGET STATEMENT

23.1 Itemized Budget

Item	Price	Quantity	Total
304 Stainless Steel Components			
Nipple	\$2.50/ea	58	\$145
Bushing	\$5.00/ea	18	\$90
Quick Connect	\$12.00/ea	36	\$432
Tee	\$3.00/ea	36	\$108
Ninety (elbow)	\$1.50/ea	10	\$15
Pressure Gauge	\$40.00/ea	9	\$360
Digital Thermometer	\$30.00/ea	9	\$270
8"-Pipe Segment	\$9.00/ea	10	\$90
Cross	\$4.50/ea	2	\$9
Valves and Actuation			
Actuated 10-Way 11-Port Valve	\$1,500.00	1	\$1,500
Actuated 4-Way Valve	\$250.00	1	\$250
1-Way Check Valve	\$90.00/ea	4	\$360
Relief Valve	\$90.00/ea	2	\$180
1-Way Adjustable Check Valve	\$150.00/ea	10	\$1,500
Electrical System			
Laser Diodes	\$10.00	9	\$90
CdS cells	\$5.00/pack	1	\$5
Electromechanical Actuator	\$850	2	\$1,700
High Pressure Piston	\$50	2	\$100
Heating Coil	\$25	9	\$225
Variac	\$25	2	\$50
Control Electronics	\$100		\$100
Materials			
High Pressure Hose	\$100	1	\$100

IV Bag	\$15	2		\$30
Miscellaneous Materials	\$200			\$200
Teflon Jackets	\$15.00/ea	14		\$210
Structural Supports	\$200			\$200
Chemical Reagents	\$100			\$100
CO ₂ tank	\$9/mo.	4 months		\$36
Machining, Welding, and Testing				
Machining and Welding	\$300			\$300
Testing	\$50			\$50
Travel and Accommodations				
State Fleet Rental Car	\$28/day	12 days		\$336
Hotel in Houston	\$75/day/room	10 days	2 rooms	\$1,500
Hotels on the Way to Houston	\$60/day/room	2 days	2 rooms	\$240
Total				\$10,881

23.2 Current Sources of Funding

Associated Students of Madison student organization operations grant	\$500
Earthtech corporate sponsor	\$250
University of Wisconsin Chemistry Department discretionary funds	\$750
University of Wisconsin Electrical Engineering funding	\$500
University of Wisconsin Engineering Physics funding	\$500
University of Wisconsin Mechanical Engineering funding	\$500
University of Wisconsin Polygon Chapter	\$500
University of Wisconsin Space Science Engineering Center funding	\$3,500
Wisconsin Space Grant Consortium Travel Grant	\$1,500
Wisconsin Space Grant Consortium Summer Research Grant	\$3,500
Total Funding Currently Available	\$12,500

24. PARENTAL CONSENT FORMS

All team members are over 18.