

Consort 3 Flight Experiments

Francis C. Wessling*

University of Alabama in Huntsville, Huntsville, Alabama 35899

and

George W. Maybee†

McDonnell Douglas Space Systems Company, Huntsville, Alabama 35806

The third sounding rocket payload in the Consort program was launched from the White Sands Missile Range on May 16, 1990. It carried 12 experiments designed to investigate materials processes in low gravity. All of the experiments were reflights from the Consort 2 payload that was launched in November 1989 but failed to achieve microgravity because of a malfunction in the launch vehicle. Four national Centers for the Commercial Development of Space participated in the mission. The payload included five experiments and two accelerometer systems that flew on Consort 1 and seven new experiments, designed and developed since Consort 1. Experiments from Consort 1 incorporated hardware modifications and changes in experimental parameters based on mission results. The new experiments covered a variety of polymeric and biological investigations. A new power distribution and control system designed to provide discrete, computer-supervised, experiment power monitoring and control was flight qualified on Consort 3. Consort 3 featured very late access (3–5 h before launch vs 27–30 h for Consort 1) for installation of sensitive biological specimens. The integrated payload and mission sequence of events are described. Changes in the Consort 1 experiments are defined and the objectives, methods, and expectations for new experiments are discussed.

Introduction

CONSORT 3 is a continuation of low gravity materials processing flights launched from White Sands Missile Range (WSMR) in New Mexico on May 16, 1990. The first flight, Consort 1,¹ launched on March 29, 1989, carried a variety of low gravity experiments and provided valuable results.² A payload designated Consort 2 was developed following the flight of Consort 1. It was launched on November 15, 1989. However, only 17 s after launch, while the second stage booster was firing, the payload was prematurely separated and the mission was terminated. This resulted from a failure in the rocket's guidance system, which has since been corrected. Although the mission was aborted, the experiment payload was recovered intact after a nominal parachute landing. The payload experiments were thoroughly reviewed and underwent requalification testing for reflight on Consort 3.

Consort flights provide 6–8 min of low gravity (10^{-4} to 10^{-5} g) time for materials experiments. The objectives of the Consort series are to 1) promote the commercial development of a sounding rocket industry in the United States, 2) develop a cadre of scientists and engineers with experience in low gravity materials investigations, and 3) encourage commercial and industrial involvement in low gravity experiments.

Consort launches and experiments are funded by a cooperative arrangement between the NASA Office of Commercial Programs (OCP), industries, universities, and other government agencies. The Consort program is managed by the Consortium for Materials Development in Space (CMDS) at the University of Alabama in Huntsville. The Consortium is one of 16 Centers for the Commercial Development of Space (CCDS) established by the NASA-OCP to promote commercial uses of space.

The Consort rockets are launched at WSMR with the support of the Naval Ordnance Missile Test Station. Space Services Incorporated (SSI) provides launch support and integration of the rocket to the payload module. McDonnell Douglas Space Systems Company integrates the experiments into the payload module. Experiments are provided by the various CCDSs working with their university, government, and industrial investigators.

In parallel with the Consort program, the CMDS manages Joust, which will provide 13–15 min of suborbital low gravity flight time for materials processing payloads, beginning in early 1991. Joust payloads will be launched from the Eastern Test Range in Florida. Experiment interfaces and accommodations for Joust are being designed for compatibility with Consort payloads. This provides the opportunity for reflight of Consort experiments in Joust payloads with minimum hardware modification and integration cost. Further information on the Joust and Consort programs is provided elsewhere.³

Consort 3 experiments were provided by four CCDS: 1) the CMDS at the University of Alabama in Huntsville, 2) the Center for Advanced Materials at Battelle, 3) the Center for Cell Research at Pennsylvania State University, and 4) Bioserve Space Technologies at the University of Colorado. The experiments and investigators are listed in Table 1.

Payload Module and Flight Plan

Experiments were integrated into a payload module as shown in Fig. 1. The payload was approximately 3.6 m in length, 0.44 m in diameter, and weighed approximately 280 kg. The lower two segments contained five experiments and two accelerometer systems that had previously flown on Consort 1. Seven new experiments were contained in the upper segment. Thus, a total of 14 different experimental apparatus were flown on Consort 3. The intent was to phase in experiments from flight to flight so that each experimenter had an opportunity for two flights. This provided opportunity to improve hardware and repeat earlier results.

The entire payload module was sealed to maintain a pressure of 1 atm throughout the flight. The experiments were mounted onto longerons that were attached to a bulkhead on one end and pinned on the other. This facilitated the integra-

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*Professor, Department of Mechanical Engineering; Associate Director, Consortium for Materials Development in Space. Member AIAA.

†Manager, Space Technology Projects. Senior Member AIAA.

Table 1 Consort 3 experiments

Experiment	Principal investigator	Institution or company affiliation
Demixing of immiscible polymers	J. M. Harris	Univ. of Ala., Huntsville (UAH)
Electrodeposition cells	P. Concus	Univ. of Cal., Berkeley
	C. Riley	UAH
	H. Abi-Akar	
	G. W. Maybee	McDonnell Douglas Space Systems Company (MDSSC)
Elastomer-modified epoxy resins	J. M. Harris	UAH
	F. C. Wessling	UAH
	J. Geibel	Consultant, Phillips Petroleum
Foam formation	S. P. McManus	UAH
	F. C. Wessling	UAH
	C. Zisette	Thiokol Corporation
Flight experiment computer	C. C. Rupp	Marshall Amateur Radio Club
Accelerometers	J. A. Bijvoet	CMDS
Power distribution and control unit	C. Schwarz	MDSSC
Materials dispersion apparatus	J. Cassanto	Instrumentation Technology Associates (ITA)
Automated generic bioprocessing apparatus	L. Stodieck	University of Colorado
	B. Spooner	Kansas State University
	J. Guelkema	
	P. Wong	
	M. Luttges	Bioserve Space Technologies
Biomodule	R. Hammerstedt	Penn. State Univ. CCDS Center for Cell Research
Polymer thin film	C. Zisette	Thiokol Corporation
	D. DeGeorge	U.S. Air Force Astronautics Lab
Polymer membrane processing, multiphase polymer curing, and plasma polymerization	V. McGinniss	Battelle CCDS Principal Investigator
	F. Jelinek	
	L. McCauley	

tion of the experiments onto the payload module and permitted an experiment to be assembled and tested on its own mounting plate. For launch operations, the payload was thermally conditioned to maintain an internal module temperature of $20 \pm 5^\circ\text{C}$. A summary of physical parameters for each experiment is given in Table 2.

The payload module provided power to most of the experiments through a power distribution and control unit (PDCU). The PDCU was developed specifically to accommodate Consort payload experiments. During flight, the PDCU received power from either of two nickel-cadmium rechargeable battery packages and distributed it to payload experiments and support systems. Each battery package had a capacity of 174 Wh at 28 V. Power from a third battery package, reserved for the elastomer-modified epoxy resins experiment, was controlled by an experiment computer. During ground operations, the PDCU also received and distributed power from a ground power source via a power/signal umbilical connection. The PDCU provided for discrete experiment on/off control, current limiting, and power usage monitoring as well as battery monitoring and charging.

The Consort rocket, called Starfire 1 by SSI, has two stages. The first stage is a Thiokol TX664 booster in a Terrier casing.

Table 2 Experiment physical parameters

Experiment	Dimensions, cm			Weight, kg
	Length	Width	Height	
UAH demixing	45.7	36.8	18.4	6.5
UAH electrodeposition	40.9	36.8	30.5	6.8
UAH epoxy resins	16.5	36.8	11.4	2.3
UAH foam formation	62.2	36.8	24.1	16.1
ITA MDA #1	26.7	11.4	8.9	2.3
ITA MDA #2	26.7	8.9	7.6	4.0
Bioserve AGBA	61.0	34.3	13.5	16.5
Penn. State biomodule	28.7	36.8	8.9	6.2
Thiokol thin film	38.7	36.8	4.3	9.2
Battelle IPMP & resins	47.2	36.8	15.2	21.9
Battelle plasma	25.4	36.8	16.5	14.3

The second stage is a Black Brant provided by Bristol Aerospace. Telemetry, boost guidance, rate control, and recovery systems are also provided. The rocket is launched from a rail that is raised to a vertical position shortly before launch. This provides for late access to the payload, allowing experimenters to load sensitive biological specimens within a few hours of launch.

The planned sequence of events for the Consort 3 flight is given in Table 3. Approximately 7.2 min of low gravity time was attained. The low gravity period for Consort 1 was slightly less, approximately 7.0 min. The payload was recovered from the desert floor, approximately 80 km from the launch complex, by Army helicopter.

Experiments

The experiments flown on Consort 3 are described below. Experiments incorporated in Consort 3 that flew on Consort 1 are described only briefly. Full descriptions can be found else-

Table 3 Flight sequence of events

Event	Time, s	Altitude, km, MSL	Range, km	Velocity, m/s
Max Q	6.0	3.2	0.2	627.3
BBVC ignition	12.0	6.6	0.3	523.6
S-19 end of guidance	18.0	10.3	0.6	726.3
Max Q-BBVC	19.0	11.1	0.6	766.0
BBVC burnout	44.5	49.1	3.2	2242.4
Yo-Yo despin	51.0	63.2	4.3	2177.5
Nose tip eject	54.0	69.7	4.8	2148.5
P/L motor separation	57.0	76.1	5.3	2119.6
Body rates nulled	67.0	95.7	6.9	2024.5
Begin micro-g	72.0	105.8	7.9	1977.2
Apogee	290.0	318.6	43.1	157.6
End micro-g	502.0	116.7	74.2	1934.6
RCS spin-up	514.0	93.0	75.8	2048.0
S-19 power off	520.0	80.6	76.6	2104.9
Max Q re-entry	553.0	21.4	79.2	1127.8
Main chute deployed	619.0	4.8	79.6	93.6
Impact	875.0	0.0	79.6	9.1

where.^{1,2} The new experiments are described herein more fully.

Demixing of Immiscible Polymers

The demixing of immiscible polymer (DIP) experiment on Consort 1 consisted of a set of 12 quartz cuvettes filled with immiscible mixtures like polyethylene glycol and water and dextran and water. The cuvettes (1.5 ml cubes) were arranged in two rows of six cuvettes inside an adiabatic chamber. External to each cuvette was a stirring motor with a magnet. A small stirring bar was inside each cuvette. The cuvettes were intended to be stirred for 29.2 s and then photographed every 15 s for 35 exposures. The immiscible liquids were expected to segregate during the low gravity time of the flight. Photographs taken on Consort 1 indicate that three of the stirring bars never coupled with the magnets when stirring commenced on Consort 1. The stirring started on Consort 1 only 0.2 s after the second stage rocket motor separated from the payload and the rate control system was enabled. These events caused noticeable accelerations in the payload and may have pushed these stirring bars away from the magnets. Stirring was initiated well after the second stage separated from the payload and the rate control system had nulled body rates on Consort 3. In addition, the motor speed was decreased to prevent the stirring bars from being thrown away from the magnetic coupling.

Based on results from previous Shuttle experiments^{4,5} mixtures were chosen that would demix within the 7 min of low

gravity. Consort 1 photographs showed that the remaining nine cuvettes' mixtures appeared to be more highly homogenized than in earlier experiments. Thus, the mixing on Consort 1 was more thorough than in earlier experiments. As a result, the immiscible liquids did not demix within the low gravity period. Formulations of mixtures that have a quicker demixing rate were used on Consort 3.

Elastomer-Modified Epoxy Resins

This experiment investigated the morphology and strength of elastomer-modified epoxy resins and can lead to better glues for manufacturing. The results of the experiment may lead to a better understanding of the demixing process and to better epoxy products. The addition of rubber to epoxy lowers the tensile strength approximately 28% and the modulus of elasticity approximately 21% but increases the fracture energy by a factor of 20.⁶

The system selected for this experiment consisted of three elastomer-modified epoxy resins. The elastomers are commercial products and are of the general class of materials called carboxy-terminated copolymers of butadiene and acrylonitrile (CTBN elastomers). These elastomers can be reacted with epoxy resins (typically a low molecular weight epoxy resin) to yield an epoxy-capped elastomer. Because the elastomer is capped by the epoxy resin, it can be cured just like a normal epoxy resin. However, because epoxy resins and the CTBN elastomers have different solubility parameters as the curing progresses, the driving forces for solubility decrease and the

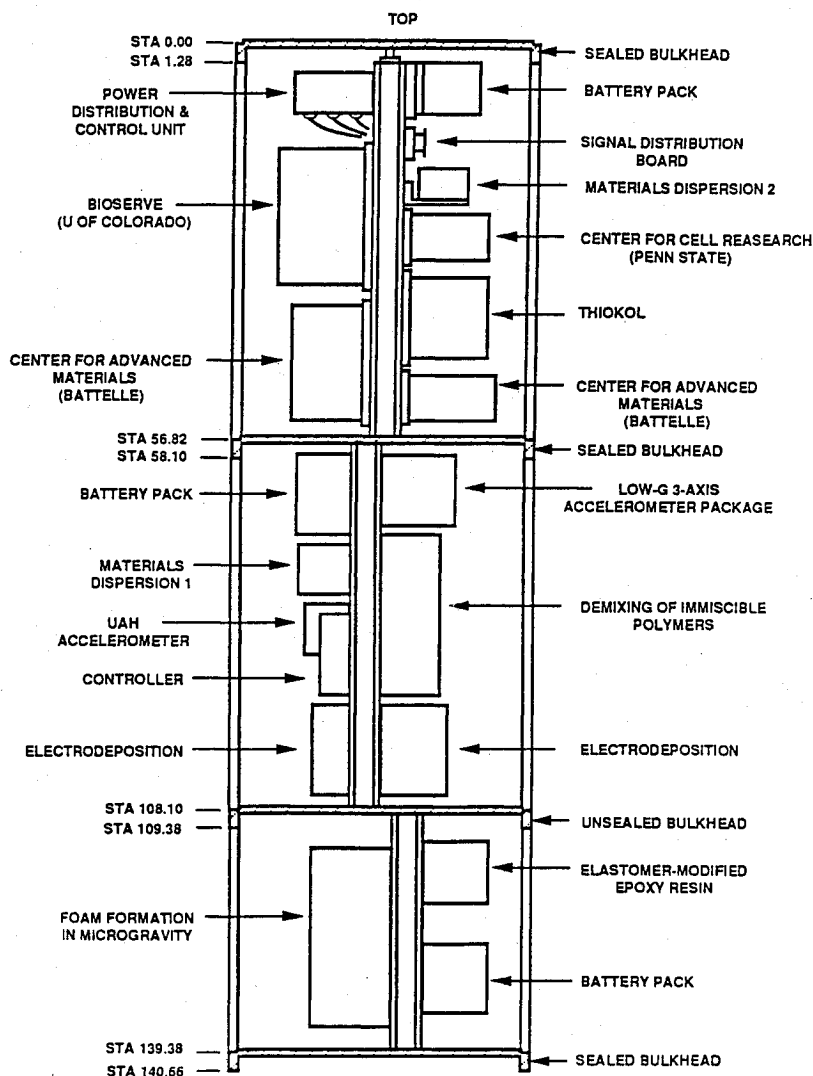


Fig. 1 Consort 3 payload module experiment layout.

elastomer phase separates.⁷ The curing process is slightly exothermic and the separated phases have different densities; thus, convective mixing may be influenced in the demixing process. The resultant deposition of rubber in epoxy was determined by electron microscopy.⁸

The elastomer-modified epoxy resins experiment was mechanically unchanged on Consort 3. The device is essentially a flat plate heater that heats a sample from both sides. The heater had a square cross section 114 mm on a side. The thickness of the heated space was variable. Consequently, this experiment had a dedicated battery. The heater raised the sample temperature from 20 to 200°C in 94 s seconds on Consort 1. Crosslinking of the epoxy proceeded so rapidly on Consort 1 that the system became rigid before phase segregation could occur. A system in which phase segregation proceeds more rapidly was used on Consort 3.

Electrodeposition

Whereas the electrodeposition experiment on Consort 1⁹ was dedicated to determining if amorphous nickel deposition was possible in low gravity, Consort 3 was directed toward reproducing results of Consort 1 as well as answering some scientific questions and studying codeposition in more detail. The nickel deposited on Consort 1 became increasingly x-ray transparent as the rate of deposition was increased. The codeposition experiment showed that particles suspended for 15 s in low gravity did not remain dispersed. Weak attractive forces that were not significant in 1 g resulted in clumping or coagulation. The electrodeposition apparatus flown on Consort 1 was also used for Consort 3, with modifications to accommodate new experimental objectives. The apparatus allowed installation of cells with variable voltages, different electrode surface areas, and various spacings between the electrodes. Thus, a variety of voltages and current densities were studied as well as various chemical compositions. Three cells (1, 2, and 3) contained cobalt sulfate. These were operated at 3, 4, and 5 V to determine if the rate of deposition influences the cobalt deposit morphology as it does with nickel. Cell 4 contained nickel sulfamate and buffers. It was operated at 7.5 V and stirred to determine if convection affects the deposition form. It was expected that stirring at 7.5 V would lead to an extremely high deposition rate. Cell 5 attempted to reproduce the x-ray transparent form of nickel made on Consort 1. This was again set at 9 V, which led to a high deposition rate. One cell (6) contained nickel sulfate as opposed to nickel sulfamate. This cell was operated at 7.5 V to determine if the deposit was x-ray transparent. The remaining four cells (7-10) contained particulates. Two contained cobalt sulfate with chromium carbide particles and two contained nickel sulfamate with diamond dust. One chromium carbide cell and one diamond dust cell included a surfactant. This demonstrated whether more vigorous stirring alone is sufficient to overcome the attractive forces or if a surfactant is also necessary. These latter four cells were photographed during low gravity to record the stirring process.

Foam Formation Experiment

The foam formation experiment aboard the Consort 1^{10,11} payload consisted of two separate piston and cylinder assemblies that contained the chemical ingredients (polyol mixture and isocyanate) that were mixed to generate the rigid polyurethane foam. The isocyanate piston was actuated by nitrogen gas pressure to cause the isocyanate to flow into the cylinder containing polyol. The two liquids were then mixed for 20 s by a motor-driven propeller. The mixed ingredients were then driven out of the chamber by the second nitrogen gas driven piston through a small orifice and a wire mesh funnel. The reason for using an orifice was to decrease the flow rate of the foam coming out of the chamber, allowing the formation of a foam sphere. The final foam remained attached to the funnel in the form of a ball.

Consort 3 flew an aluminum particle and polyurethane foam mixture. The aluminized foam hardware was not significantly different from that used for the Consort 1 mission. The changes were related to incorporating the solids (aluminum powder or flakes) into the apparatus. The principal changes were in the hardware related to the nitrogen gas driving the mixture out of the apparatus and the flow restriction caused by the exit orifice. The apparatus still had the two separate piston and cylinder assemblies that contained the chemical ingredients (polyol mixture, aluminum powder or flakes, and isocyanate) to be mixed.

High pressure (approximately 10 atm) of nitrogen gas was used to drive the isocyanate into the polyol. However, a gas pressure regulator stepped down the pressure to approximately 5 atm for pushing the mixture through the exit funnel. Thus, the flow restricting orifice was not needed. It was removed because of possible clogging by the aluminum particle laden foam.

Materials Dispersion Apparati

There were two materials dispersion apparati on Consort 3. The first one was flown on Consort 1 and successfully processed samples in low gravity. Its description is similar to that given here for the Materials Dispersion Apparatus-2 (MDA-2).

The MDA-2 is a private-sector-developed compact automated device used to mix two to three fluids in the low gravity of space. The MDA weighs 1.8 kg and occupies a volume of less than 2.8 liters. The minilab represents a cost effective technique to obtain multiple data points on a sounding rocket flight. MDA-2 is an upgrade of the MDA payload successfully flown on the first Consort sounding rocket flight in March of 1989. Seventeen experiments in biomedical and materials processing in space (MPS) areas were flown. This version of the MDA was also flown on the Space Shuttle in June of 1990 aboard STS-37.

The MDA operates on the following principle: two blocks of inert material, each with an equal number of sample test wells in the upper and lower half, are held together under pressure with a sealing mechanism in an aerospace housing. The test wells are misaligned at launch, thus separating the fluids to be mixed. After microgravity has been achieved, the blocks are moved into alignment by means of a motor-cam mechanism allowing the fluids to contact. An option exists to mix a third or fourth fluid to fix the process while in the microgravity environment or prior to/during re-entry. Mixing occurs through the liquid-to-liquid diffusion process. Each MDA can have up to several dozen test wells and each test well can accommodate samples in the 50-1000 μ l range.

The MDA was designed for biotechnology experiments, specifically to grow and mass produce high quality crystals in space such as peptides, hormones, enzymes, antibodies, and other biological macromolecules. Additional classes of experiments that can be accommodated are fluid sciences and thin film membrane casting.

Automated Generic Bioprocessing Apparatus

The Automated Generic Bioprocessing Apparatus (AGBA) was developed as a valuable tool to study a variety of biomaterials processes in reduced gravity. In the configuration flown on Consort 3 (see Fig. 2), this payload weighed approximately 18 kg and occupied 0.04 m³. Principal investigators from the University of Colorado, Kansas State University, NASA, and industry conducted several studies involving up to 150 individual samples. The AGBA permitted active mechanical mixing, passive diffusive mixing, temperature control by heating, and real-time data acquisition for the study of reaction kinematics.

The AGBA experiments for Consort 3 were designed to process a large number of biological samples during microgravity. Fluid samples were stored in misaligned wells machined into

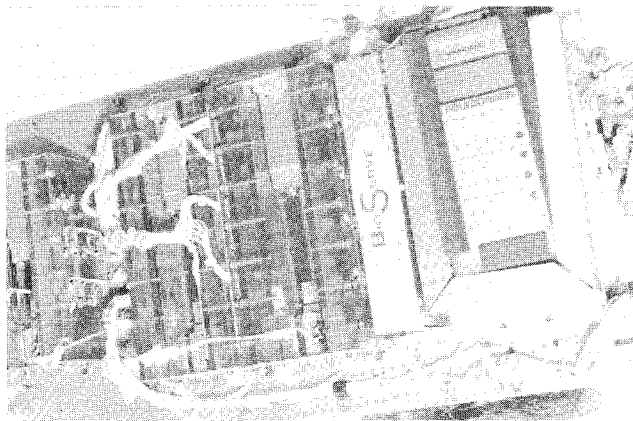


Fig. 2 Automated generic bioprocessing apparatus.

six sets of Lexan blocks. Each set of blocks consisted of one fixed and one sliding block and housed from 12–40 sample wells, depending on block-set configuration. The sample wells of the fixed block in each set contained process precursor materials. The wells in the sliding block contained process initiation and termination materials. In some cases, the wells in the moving block contained miniature mixing devices to produce active mixing. The remaining wells permitted passive diffusive mixing.

Upon entry into microgravity, all of the sliding blocks were driven to allow the wells in the mixed block to align with the first well in the moving block. This action would also actuate mixing devices in the wells that were so equipped. The fluids that were thereby brought into contact with each other were then allowed to mix for the full duration of the microgravity period. Just prior to exit from reduced gravity, a signal was generated that caused the sliding block to move again, which brought the second well in the moving block (process termination materials) into contact with the well in the fixed block, thereby terminating and preserving the reaction products.

Two of the six sets of blocks offered temperature control. They were raised to an elevated temperature of 40°C 1 h prior to launch. The other four sets of blocks were operated at ambient temperature conditions.

For 32 samples, real-time data were obtained on reaction kinematics to compare with ground controls. These samples were instrumented with light emitting diodes (LEDs) and photoresistors for recording of optical density data during flight. Data were multiplexed into a Polycorder data logger and telemetered to the ground. In addition to optical density, ambient temperature in the AGBA and the temperature of the temperature controlled block were recorded. Following recovery, samples were removed from the AGBA and stored for return to Bioserve's laboratories for analysis.

During the Consort 3 flight, experiments were conducted in a variety of topic areas, including self-assembly processes,^{12–14} microorganism nutrient uptake, liposome formation,^{15,16} and binding of nitrogen-fixing bacteria to plant roots. All of these processes may be affected in reduced gravity because of the profound effects on the fluids in which the reactions occur. In reduced gravity, convective mixing and resulting shear forces are greatly reduced. In addition, sedimentation and buoyancy forces are nearly eliminated. Diffusion, surface tension, and electrostatic interactions become much more important. As such, bioprocesses studied in reduced gravity will provide new insight into the effects of the fluid environment and may lead to novel processes or materials of significance to the biomedical industry.

Biomodule

The life science experiments on the Spacelab 3 mission provided "the greatest amount of information ever obtained from a biological payload in space and may well be the most signifi-

cant contribution on biological systems in space ever gained from a single mission."¹⁷ Profound perturbations in muscle function and structure, decreases in bone mass and elasticity, diminished pituitary function, and alteration of the hematopoietic system were documented in rats. These changes can be traced to problems related to cellular secretion. It is quite possible that the physical (and psychological) dysfunctions suffered by man from long exposure to the space environment also are related to similar physiological imbalances.

Secretion is the basis of the cellular communication essential for the integrated function of the various tissues of the body. A roll call of diseases of concern on Earth—dysfunction of muscle, bone, cardiovascular, and immunological systems—reveals a strong dependence on orderly cellular secretion, and increased awareness of the details of the regulatory pattern of secretion can serve as the basis for successful pharmacological intervention and correction. The problem is one of dissecting the process into its individual steps to allow a detailed characterization of each obligatory component. The only methods currently available are pharmacological treatments to grossly perturb the secretory processes or fortuitous isolation of genetic variants that display recognizable phenotypic features. Space flight offers a third alternative—not surprising, since life developed on Earth during 3.5 billion years under relatively constant gravitational force. Use of microgravity as a noninvasive mode of perturbing the secretory process will allow detection of heretofore unrecognized "factors" essential to the regulation of secretion.

A central theme of the Pennsylvania State University (PSU) CCDS, the Center for Cell Research (CCR), is the study and commercial development of processes related to cell physiology, biochemistry, and endocrinology. Within that context, an important objective of the PSU-CCR is to develop the equipment (see Fig. 3) needed to study the effects of microgravity on biological systems at the cellular level. Of paramount importance is the capacity to analyze, in detail, cellular response(s) to extracellular stimulation. This requires design of generic (and reusable) equipment for containing cells and adding exogenous solutions; detection of perturbations of specific aspects of the secretory process; and stabilization of cells (or storage of data) after the observation period is completed.

The Consort missions provide an excellent opportunity to begin the test of this approach. Low-cost access to short periods of microgravity will enable future customers to define their biological system and determine points of gross dysfunction, thereby making later orbital missions much more cost effective. Provision of late access (3 h before launch) allows removal of cells from any home laboratory in the U.S., transport to the launch site, and test within 18 h. PSU is building the necessary hardware and then testing the use of a simple biological system that reflects many of the general features sought by potential customers.

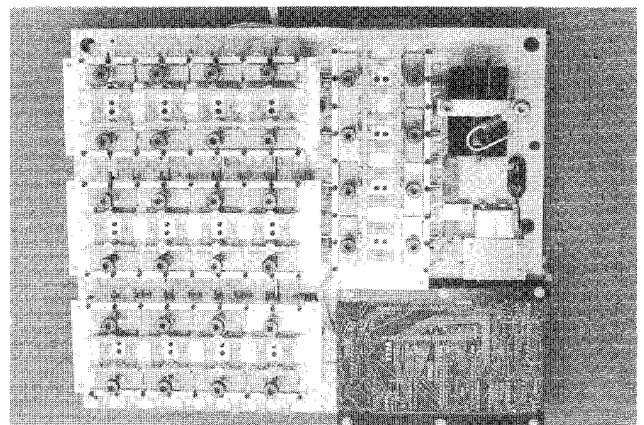


Fig. 3 Biomodule apparatus.

This equipment has the following features: the main Biomodule unit occupies a volume of less than 3 liters, weighs less than 9 kg, and draws only power and liftoff signal from the payload systems. Up to 64 silicone "T" devices to hold cells (main body) and solutions (side bladders) are provided (see Fig. 3). The main body is isolated from the side bladders by mechanical pressure on the connecting tubes and upon receipt of a preprogrammed signal from a dedicated local computer, the pressure is released and a solution is displaced from the side bladder into the main body. This format allows addition of either of two solutions (e.g., stimulant and fixative) to the cells at any time during the mission. Later supplementation of the main body with any of a variety of optical detectors would allow monitoring of parameters of the cell suspension.

The biological system used to test this device is chameleon skin, where a microtubule dependent color change stimulated by melanocyte stimulating hormone (MSH) occurs. The cells are hardy enough to withstand the rigors of space travel; the system is well characterized; the visible reaction occurs within 3 min; and the system remains hormonally responsive for greater than 12 h. The hypothesis [microgravity alters microtubule organization and results in the loss of cell function (MSH stimulated color change)] was tested by monitoring morphological features of cells, with and without MSH addition, at various times during the Consort mission.

Successful completion of these experiments should allow both refinement of the Biomodule and modification of the experimental protocol to allow evaluation of more complex medically relevant cells and tissues.

Thin Film Zero "G" Experiment

Specific liquids can be formed into thin film structures with a shape that is determined by surface tension forces, as in soap bubbles.¹⁸ Thin film structures have been proposed for many applications in the space environment. Examples include communication reflectors, solar collectors, solar sails, and precursor structural members.¹⁹ An ability to tailor the optical and electric properties of these films is fundamental to many applications. The optical and electrical properties can be altered by the addition of solid particulate to the film. Of particular interest is the incorporation of metal powders to increase the conductivity and reflective character of the film. However, in the Earth's gravitational field, the metal particulate drains from the liquid film before it can be used or cured.²⁰ This phenomenon results from the difference in densities between the metal powders and the liquid polymers. The densities of most film-forming polymers approximate 0.8-1.2 g/cm³, whereas the densities of metal powders range from 2.69 g/cm³ for aluminum to as great as 19.32 g/cm³ for gold.²¹ Forming the film in a microgravity environment will eliminate this effect. The scope of this investigation was limited to the characterization of flake aluminum dispersed in a film matrix formed and cured in a microgravity environment.

The experimental apparatus shown in Fig. 4 was specifically designed and fabricated to be flown on the Consort 3 sounding rocket. During the 7-min period of microgravity, the liquid polymer containing flake aluminum was formed into a thin film and cured.

The operation of the apparatus was controlled by two timers. The timers were actuated during liftoff by a greater than 5-g vertical impulse. Times were set based on the historical record of Consort 1 and ballistic predictions for Consort 3. The experiment was entirely self-contained and required no power or communication services from the rocket or payload systems.

The apparatus consisted of a sealed reservoir that contained the film-forming polymer and flake aluminum. A mechanical stirring blade and perforated film-drawing plate were housed in the reservoir. As microgravity was achieved, the mechanical stirrer was activated to remix the polymer and aluminum par-

ticles that may have separated during the spin stabilized ascent phase of the flight. The film-drawing plate was then pulled from the reservoir to form the thin films. Once the films were formed, two ultraviolet (uv) lamps were activated to cure the films. The uv lamps had a spectral band with the greatest intensity centered around 250 nm. The electric power to operate this apparatus was contained in four 12-V, sealed lead acid batteries located below the reservoir assembly. They provided power to the stirring motor, film-drawing motor, and ultraviolet lamps. The apparatus was designed, fabricated, and tested to withstand the anticipated flight load and vibration environment.²²

Selection of a suitable photopolymerizable liquid mixture was made. Union Carbide's Cyacure System was chosen for its ability to be rapidly cured in an ambient atmosphere, and its compatibility with Fluorad FC-430, a surfactant. Cyacure UVR6110 is a medium viscosity (350-450 cP at 25°C) cycloaliphatic epoxide. This material undergoes rapid polymerization in the presence of strong onium salt photoinitiators. The cationic initiator selected was Union Carbide's Cyacure UVI 6974; 3M's Fluorad FC-430 was selected as the surfactant to aid in the formation and stabilization of the thin film structure. The aluminum flake selected had a particle size distribution centered around 15 μ . This material was optimized for its film forming ability and rapid curing characteristics.

A postflight analysis of the telemetry data from Consort 3 was made to determine the period and extent of microgravity experienced. The recovered films from the experiment were characterized for aluminum distribution, uniformity, density per unit surface area, and particle size distribution as located on the surface or within the film matrix. The electrical conductivity and capacitance of the thin film were measured under static and high frequency fields. Optical spectra were made of the thin films from 220 nm through 22 μ . In addition, the mechanical strength of the thin films were determined. All microgravity films characterized were compared to equivalent films formed under unit gravity.

Three Polymer Experiments

The Battelle Advanced Materials Center for the Commercial Development of Space developed a sounding-rocket experiment package to investigate the effect of microgravity on polymer processes. The Battelle experiment package consisted of three experiments: 1) investigations into polymer membrane processing (IPMP), 2) multiphase polymer curing, and 3) plasma polymerization in microgravity. The experiments are shown in Figs. 5 and 6. Experiment systems and hardware are discussed individually below.

Investigations into Polymer Membrane Processing

Polymer membranes have been used for over 25 years in the separations industry for such applications as desalination, fil-

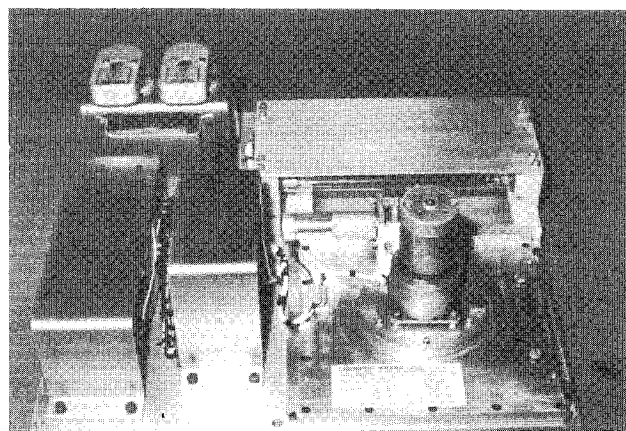


Fig. 4 Thin films zero "g" apparatus.

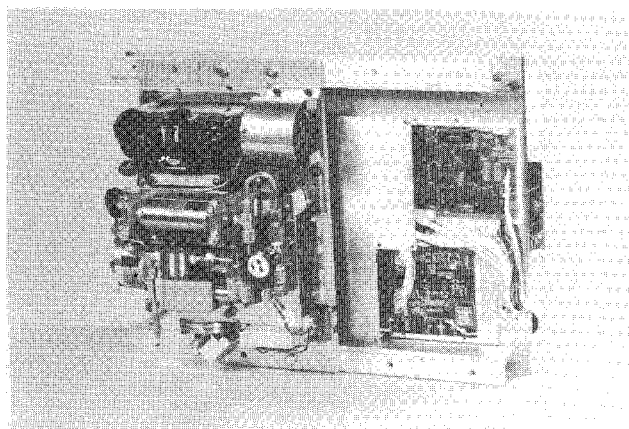


Fig. 5 Plasma polymerization in microgravity apparatus.

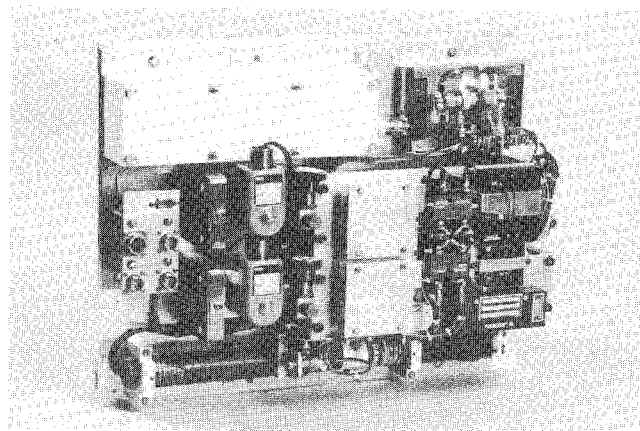


Fig. 6 Polymembrane processing and multiphase polymer curing apparatus.

tration, atmospheric purification, electrolysis, and dialysis. Polymer membranes are commercially processed by evaporation casting. In this process, a polymer membrane is prepared by casting a mixed solution of polymer and solvent into a thin layer. The cast solution is then evaporated to dryness.

The importance of the evaporation step in the formation of thin-film membranes is not yet fully understood. However, recent experimental work demonstrates that the convective mass transfer owing to densification cannot be ignored and, in fact, influences the structure of the membrane.

The overall objective of the investigations into polymer membrane processing (IPMP) experiment was to gain a fundamental understanding of the role of convection-driven currents in the transport processes that occur during the evaporation casting of polymer membranes and, in particular, to investigate how these transport processes influence membrane morphology. In this flight experiment, mixed solvent systems were flash evaporated in the absence of convection to control the porosity of the polymer membrane. Ground-based control experiments were also performed. It was expected that the resulting pores in the space-processed membrane would be of a more uniform configuration than that processed on Earth.

This experiment began to investigate the effects of convection on membrane morphology. Experimental hardware consisted of a small membrane chamber and a larger vacuum chamber, connected with a normally closed solenoid valve. A thin-film polymer material was swollen in a solvent solution and inserted into the small chamber at ambient pressure. When the payload reached microgravity, the valve was opened and the solvent evaporated into the vacuum chamber. Prior to leaving the microgravity regime, timing circuits deactivated the relay so that the solenoid valves would reclose. Two IPMP units were flown on the *Consort 3* mission.

No data acquisition or feedback was planned for the sounding rocket flight. Standard scanning electron microscopy (SEM) techniques were performed after retrieval to determine pore size and distribution. Ground-based control experiments were run and compared to the microgravity experiments.

The microgravity of space will permit researchers to study polymer membrane casting in a convection-free environment. Results of the experiment will improve the knowledge base regarding the effects of convection in the evaporation casting process. Longer duration IPMP experiments are scheduled on the Space Shuttle (STS-31) in early 1990. It is anticipated that the increased understanding of the membrane casting process gained from these flight experiments will translate into improved terrestrial commercial processing techniques with the ultimate goal of optimizing membrane properties.

Multiphase Polymer Curing Experiment

A major focus of the Battelle Polymer Program relates to multiphase polymer component systems. Current industrial processing involves the interaction of polymers with other materials such as fibers, metal oxides, or glass or carbon fibers. The products of such interactions are nonuniform because of gravity-driven settling or dispersion of the different-density materials. To compensate for dispersion effects, special additives have been developed to promote uniform interaction among the different phases on Earth.

This experiment studied phase dispersion phenomena under microgravity conditions with the objective of acquiring a better understanding of the fundamental properties of thermoplastic and thermosetting composites. This payload was a follow-on experiment to similar successful drop tower and Learjet low gravity experiments performed earlier. It is anticipated that the knowledge gained from this series of experiments can be transferred to enhance commercial processes resulting in new or improved multicomponent polymer systems with tailored mechanical and/or electrical properties by providing uniform dispersion of phases and controlled particle size distributions.

The experiment involved the curing by heating and catalysis of liquid samples into polymers. The experiment consisted of 15 vials containing polymer resin/catalyst systems that cure at approximately 120°C. An electrical heating tape and a temperature sensor/switch were enclosed in a Teflon container that provided physical, electrical, and thermal insulation. The heating tape was wrapped around the vials. The vials were heated to curing temperature during the microgravity period.

Plasma Polymerization in Microgravity

Plasmas have been used to generate polymer films and coatings since the early 1970s. Both polymeric and inorganic particles have been observed when organic vapors are fed into plasma discharges. The mechanisms by which these films and particles nucleate and grow in plasmas are not well understood. The objective of the plasma polymerization experiment was to further the understanding of plasma particle/film formation.

Nucleation and growth of particles and associated films was observed in a microgravity environment, and the structure and chemical composition of the final particles were compared with particles generated under identical plasma conditions in a ground-based laboratory. It was anticipated that the particles would be suspended in the plasma discharge throughout their growth period, permitting the growth of larger particles and facilitating study of the growth processes.

The experimental system consisted of a discharge chamber with axial dual cylindrical electrodes and window ports, organic gas stored at low pressure, and several systems to measure and record the plasma process (laser, camera, photodiode array, temperature and pressure sensors, and microprocessor). The experiment involved generation of carbon particles or films from acetylene in a 10-kHz AC plasma. An HeNe laser beam was expanded, transmitted through the

chamber, and passed through special imaging optics to a camera. The camera recorded light scattering patterns. Other sensors measured the intensity of the Rayleigh scattering perpendicular and along the polarization of the laser beam and the temperature and pressure in the chamber. Postflight analysis included analysis of film and microprocessor data, and the generated particles were recovered and analyzed using SEM/EDX, x-ray diffraction, Raman, and infrared techniques.

Summary and Conclusions

The 7 min of microgravity time available for Consort 3 experiments was sufficient to gain extensive data on materials processing. Five experiments that flew on Consort 1 flew again on Consort 3. These were modified to improve performance and to make changes in materials and protocols dictated by Consort 1 results. The demixing of immiscible polymers experiment was able to photograph the initial stages of demixing and was able to see some systems completely separated after their mixing in low gravity. In addition, the effect of container shape and liquid contact angle on demixing was photographed. Ultimately, this information can be used for biological cell separation. The elastomer-modified epoxy resins experiment yielded samples processed in low gravity that were adequate for determining the morphology of these resins, as well as providing specimens for tensile testing. New forms of epoxy may ultimately emerge. The electrodeposition experiment was able to obtain samples of nickel and cobalt deposited over a range of current densities and also obtained codeposited particles and metals for later analysis. These results may yield new forms of catalysts. A sample of polyurethane foam with aluminum particles was produced in low gravity. This sample will be analyzed for cell morphology and also for thermal conductivity and compressive strength. The foaming process was photographed during microgravity to assist in understanding the formation process. A better understanding of the foam formation process should result. The materials dispersion apparatus will allow several researchers the opportunity to study a variety of phenomena including bone marrow studies, organic cell growth, and diffusion phenomena. The small test chambers allow many researchers to perform experiments in a small volume.

There were seven new experiments on Consort 3, affording flight opportunities for several organizations that have never flown sounding rocket experiments. These experiments investigated the potential for a number of future commercial applications. Three of the new experiments were directly related to the study of biological functions. The automated generic bioprocessing apparatus will provide an opportunity for studying biological reaction kinematics. This includes tubulin self-assembly into microtubules, fibrin polymerization and collagen polymerization, microorganism nutrient uptake, liposome formation, and binding of rhizobia to plant root hairs. The biomodule unit will test cellular secretion, which is the basis of cellular communication. This affects the integrated function of the various tissues of the body. An improved materials dispersion apparatus will also be flown with more biological experiments.

Four of the new experiments dealt with polymers in low gravity. The thin film zero "G" experiment used ultraviolet light to cure a polymer liquid containing suspended aluminum flake. The resultant thin film was evaluated for the distribution of aluminum flakes and for optical and electrical properties. The investigation into polymer membrane processing sought to gain an understanding of the effect of convection-driven currents in the transport processes that occur during the evaporation casting of polymer membranes. The multiphase polymer curing experiment studied phase dispersion in order to acquire a better understanding of the fundamental properties of thermoplastic and thermosetting composites. The plasma polymerization in microgravity experiment generated carbon particles or films from acetylene in a 10-kHz alter-

nating current plasma. A photographic record was made of the process.

The 12 experiments packaged in the Consort 3 payload promise to return a large amount of new data and significantly increase our understanding of many microgravity materials processes and possible commercial applications.

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