Research in Progress

Space Available

For $10,000 you can rent five cubic feet on the Space Shuttle for a few days. That may seem like pretty cramped and expensive travel accommodations, but it's a bargain at the price. NASA has made the small self-contained payload canisters, called Getaway Specials, available to anyone with a legitimate research purpose, and a group of Caltech students has been quick to seize the opportunity. They are packing two experiments into a Getaway Special that will investigate growth of seeds and crystals away from the effects of the earth's gravity.

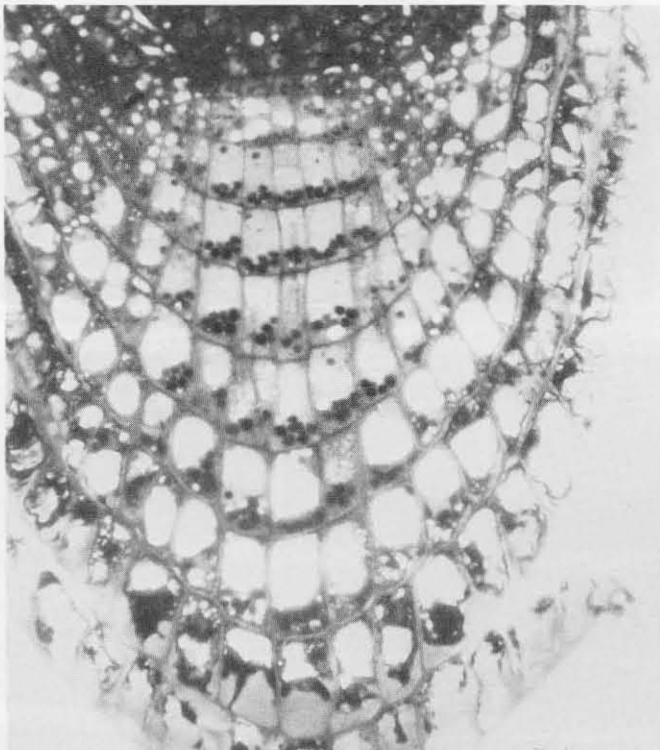
These two experiments emerged from 10 proposals developed over the summer of 1980 by the Student Space Organization (SSO). The organization had been formed the previous winter by Ralph Weeks (BS 1981) and John Whitehead, now a senior in engineering, to manage the space research project from start to finish. It has involved a group of about 15 students, whose ranks shift somewhat every June. Kirk Haselton, a sophomore in applied physics, is currently the project manager.

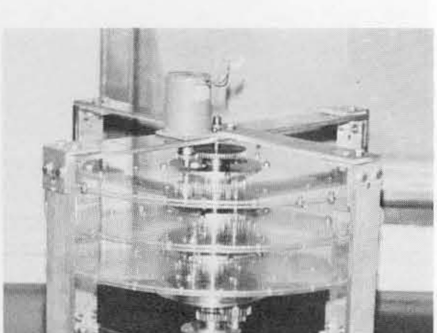
Whitehead originated the biology experiment to explore the mechanisms by which plants perceive and respond to the direction of gravity. One hypothesis holds that dense organelles, called amyloplasts, settle to the bottom of root tip cells and lead to downward growth. To test this hypothesis and to determine the threshold of geotropism, the SSO students are sending radish seeds (because they grow very fast) on a journey into zero gravity, where they can germinate under very low artificial gravitation conditions.

The seedlings will be mounted in small depressions drilled into five Lexan disks, which will rotate at different speeds. Since acceleration is greater at the rim than at the hub of a disk, the seeds will be mounted at four radii across each disk, thus subjecting them to acceleration forces representing 20 data points between 10^{-4} g near the hub of the slowest disk to $10^{-1.5}$ g at the rim of the fastest one. When the Shuttle reaches orbit, a motor will start the disks rotating, and water will be injected to soak the seeds and germinate them. After 30 and 60 hours, a fixative solution will halt seed growth until recovery of the canister.

Then begins the laboratory work, using electron microscopy to determine the position of the amyloplasts within the cells and to observe whether they started to settle at the same stimulus level at which the roots began to grow downward. The student research team is expecting to find that these events do happen at the same time — at about 10^{-3} g.

The crystal in the second experiment will have a four- to five-day growing period. Darrell Schlom and Connie Bennit, sophomores in engineering and chemical engineering respectively, are conducting this experiment to study the





An electron micrograph (right) shows that amyloplasts (the small, round, dark spots) have settled to the bottom of cells near the middle of this radish root tip grown in normal gravity. The student space experiment will test the hypothesis that this settling determines downward growth. The radish seeds will journey into space mounted on rotating disks similar to those in the prototype apparatus shown at far right.

During a visit to Caltech, John L. Robbins (left) and Roger Vernon (right) of Great Western Inorganics (which is sponsoring the Getaway Special) discuss materials to be used in the seed growth experiment with students John Whitehead and Darrell Schlom.

mechanisms of crystallization; Paul Shlichta of the JPL technical staff is principal adviser.

In the presence of gravity a growing crystal creates a convection current in the surrounding fluid, which affects the way it grows. Other crystallization research already performed on Skylab and in simulation experiments indicates that the space-grown crystals develop more slowly than earth-grown ones but possibly are of more uniform composition with more regular defect structure. The student experiment will take photographs of the crystal as it grows and will collect more quantitative data than have previous experiments. This will allow testing of various theories of how crystals grow by molecular diffusion in the absence of gravity.

Candidates for the most appropriate crystal material are currently under consideration; so far, potassium dihydrogen phosphate (KDP) appears the most likely. Warm, saturated solution will be introduced into the chamber containing the crystal, and it will then be cooled, causing the crystal to grow.

Thermal control is going to be one of the more challenging engineering problems of the project, since temperature is crucial, but different, for each experiment, and each chamber must be insulated and controlled separately. Teams of two or three students are attacking specific problems that are common to both experiments and must be coordinated, since the size and weight constraints of the canister (less than 200 lbs.) dictate a minimum of equipment. For example, the battery power source and digitally controlled data storage system are being designed as single units for both experiments. Also, the experimental apparatus must be strong enough (although very light) to survive the vibrations of the Shuttle's launch, so the students must devise some high-technology solutions to the various mechanical design questions.

Like any other research project, this one can't run on enthusiasm alone, and the students of the SSO have been exposed to all the realities of program management, including funding. Great Western Inorganics of Golden, Colorado, has provided the $10,000 for the Getaway Special ticket and has offered to supply any necessary chemicals. Other firms, including Hughes Aircraft Company, TRW Inc., Xerox Electro-Optical Systems, and Northrop Corporation have supplied some funds, and still others have donated equipment, from bubble memory systems (Intel Corporation) and computer chips (National Semiconductor Company) to temperature sensors (Omega Engineering). The biggest funding problem is student support over the summer, when the major portion of the work is performed, since it is impossible to devote full time to it during the academic year.

Most of the remaining work will have to be done this coming summer. If all goes well, the radish seeds and the seed crystal, with all the carefully designed controlling apparatus of the experiments, will be launched into microgravity on the Space Shuttle's seventh flight, now planned for April 1983. □ *–J.D.*

Middle Stages

ALTHOUGH catalysts perform the important function of speeding up chemical reactions, many of them commercially significant, most catalysts have been discovered by chance. As long as they worked and managed to generate appropriate quantities of the desired product, understanding the catalytic process (which promised to be an obstinate problem) didn't seem necessary. But recently, the urgency of developing new sources of energy and increasing industrial production has aroused a growing interest in catalysis. Chemists are trying to look into the transitory steps of the catalytic mechanism and determine what actually happens on a molecular level — how the bonds between atoms are broken and the fragments reconnected in another way. Caltech's Catalysis Group is approaching the problem from a number of different angles.

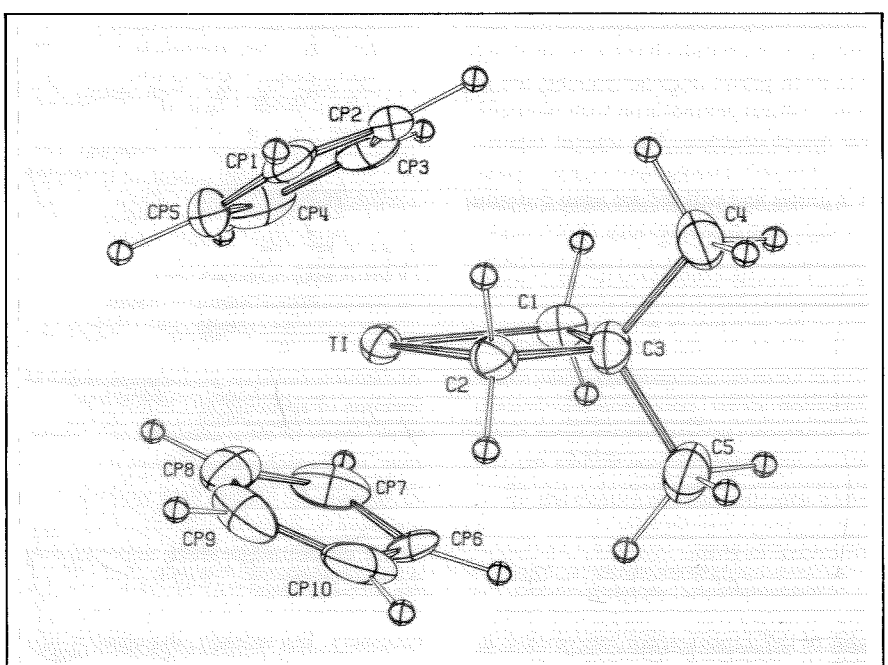
One of the group, Robert Grubbs, professor of chemistry, is studying the intermediate products of catalysis — the compounds that are formed very briefly by the catalyst and the reactants before the end product of the reaction is reached. One requirement of a catalyst, however, is that it remain chemically unchanged; that is, it cannot be consumed in the overall process. Therefore any reactions of the catalyst itself must be reversible, so that it can return to its original form. By their very nature, then, these intermediate products are extremely unstable; they must be stable enough to be produced but unstable enough to react further. In fact, according to Grubbs, if you can actually isolate a product and see it, then in most cases it's probably too stable to be part of the catalytic scheme. How can you go about identifying something that exists too briefly to be observed?

Grubbs starts out by establishing, as models, all the potential mechanisms for the molecular transformation. Then his task is to determine which one of all the possibilities actually takes place — a frustrating problem because the true mechanism cannot actually be proved. Rather, all the others must be disproved. And before you can begin to do that, you have to devise experiments to tell the different models apart.

For example, in their current work on the polymerization reaction (in which small units are linked together in long chains), Grubbs and his group, including graduate students Jorge Soto and Michael Steigerwald, have generated a large family of possibilities. They have been able to eliminate one sizable section of these and are now concentrating on narrowing the field still further. One way of doing this is to exploit the differences between reactions. For instance, in the remaining group, one set of possible polymerization mechanisms involves hydrogen migration in the intermediate steps, and a small but significant set does not. Current experiments (measuring isotope effects in the polymerization of ethylene) to distinguish whether hydrogen migration actually does take place in the true catalytic reaction seem to indicate that it does not, allowing Grubbs to zero in another step closer to his target.

One of his goals has already been reached — isolation of an intermediate in the olefin metathesis reaction, one of the most important processes in organometallic chemistry, which, with the assistance of a transition metal as a catalyst, interconverts olefins. This process is now a key step in the production of detergent and perfume intermediates as well as a new class of plastics. Olefins are hydrocarbons with one carbon-carbon double bond, which is cleaved and reformed in the catalytic reaction. For example, propylene, a C_3 olefin, is converted to ethylene, a C_2 olefin, and 2-butene, a C_4 olefin. Going through all the steps (over several years) of narrowing down the modeled possibilities of all the mechanisms, Grubbs last year isolated and defined the structure of one of the proposed intermediates in a carefully selected (not a model) catalytic system. He found that the olefin metathesis reaction has two intermediates that interconvert. He has demonstrated that his intermediate, in which titanium links up three carbon fragments in an unusual arrangement of atoms, does proceed through the steps of bond breaking and reformation necessary to the catalytic reaction. Working with Grubbs on this project were graduate students Kevin Ott, John BOSCO Lee, Dan Straus, and Suzzy Ho.

Another current project concerns developing a catalytic scheme to convert carbon monoxide from coal into useful organic materials. Graduate students Ken Doxsee and Tom Coolbaugh are involved in this project. And, as it has always been with catalysis, Grubbs finds that balancing all the intermediate equilibria still involves a considerable amount of luck. □
–J.D.



This computer drawing illustrates the structure of an intermediate, isolated by Robert Grubbs, in a titanium catalyzed olefin metathesis reaction. The key structural feature is the four-membered ring at right defined by the titanium atom (TI) and the carbon atoms, C1, C3, and C2.