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Monodisperse Latex Reactor (MLR)

A Materials Processing Space Shuttle Mid-Deck Payload

By Dale M. Kornfeld
Space Science Laboratory

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16. ABSTRACT

The Monodisperse Latex Reactor experiment has flown five times on the space shuttle, with three more flights currently planned. The objective of this project is to manufacture, in the microgravity environment of space, large particle-size monodisperse polystyrene latexes in particle sizes larger and more uniform than can be manufactured on Earth. Historically it has been extremely difficult, if not impossible, to manufacture in quantity very high quality monodisperse latexes on Earth in particle sizes much above several micrometers in diameter due to buoyancy and sedimentation problems during the polymerization reaction. However the MLR project has succeeded in manufacturing in microgravity monodisperse latex particles as large as 30 micrometers in diameter with a standard deviation of 1.4 percent. It is expected that 100 micrometer particles will have been produced by the completion of the the three remaining flights.

These tiny, highly uniform latex microspheres have become the "FIRST SPACE PRODUCT," that is, the first material ever to be commercially marketed that was manufactured in space. The U.S. National Bureau of Standards has certified the first batch of "space latex," which was transferred to NBS by NASA in July 1984, and they will begin marketing this material in mid-1985 as the U.S. national 10-micrometer Standard Reference Material.

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TECHNICAL MEMORANDUM

MONODISPERSE LATEX REACTOR (MLR)

A Materials Processing Space Shuttle
Mid-Deck Payload

The Monodisperse Latex Reactor (MLR) is a Microgravity Science and Applications experiment, the purpose of which is to produce large particle-size monodisperse polystyrene latexes in microgravity in sizes larger than can be manufactured on Earth.

A latex is a suspension of very tiny (micrometer-size) plastic spheres in water, stabilized by emulsifiers. The objective of this experiment is to grow "billions" of these microspheres to sizes larger than can be grown on Earth, while keeping all the spheres within the batch the same size and "perfectly" spherical. The word "monodisperse" means exactly the same size, and it is defined in this experiment as maintaining a standard deviation of diameter of less than 2 percent for all microspheres in the batch. Thus far, latex batches have been returned to Earth after several shuttle flights with standard deviations better than 1.4 percent.

The reason that high-quality monodisperse latex is so desirable is because a researcher need only examine or measure one particle at random from any batch and yet have confidence that all the other particles in the batch will be exactly the same size and will not need to be measured. This would be very important when the latex particles are used as size standards against which other objects in the same general size range are compared, or when they are used as membrane probes or as carriers for drugs or biological species that require extremely accurate knowledge of the volume or surface area of the particles.

A typical MLR latex reactor batch, such as that carried aboard its most recent shuttle flight (STS-11), produces 1.7 billion microspheres of 30 micrometers diameter, or 45 billion microspheres of 10 micrometers diameter; the difference in numbers is due to each latex recipe being held constant at about 25 percent total solids content by weight. The microspheres comprising these latexes can only be grown in quantity on Earth up to about 5 micrometers in diameter while remaining monodisperse due to buoyancy and sedimentation effects. They cannot be stirred sufficiently to maintain the suspension during polymerization because stirring causes shear-induced coagulation which destroys the latex's monodisperse qualities and produces mostly coagulum. However, in microgravity the absence of buoyancy effects has thus far allowed growth of the spheres to 30 micrometers in diameter, and hopefully to much larger sizes in later flights. The MLR has now flown five times on the space shuttle (STS-3, -4, -6, -7, and -11), and three more flights are presently scheduled to be completed by mid-1986 (Table 1).

This experiment has now produced the first commercial space product; i.e., the first commercial material ever to be marketed that has been manufactured in space. The 10 micrometer particle-size latex manufactured during the STS-6 shuttle mission was officially accepted by the U.S. National Bureau of Standards (NBS) on July 17, 1984. After NBS completes certification of this latex in early 1985, they plan to market it to research workers worldwide as the United States national 10 micrometer Standard Reference Material. NASA transferred 15 grams of this 10 micrometer latex to NBS where it was diluted and packaged by NBS into 600 vials of 4 ml each.

Each of these vials was priced by NBS at $384, thus giving the batch of 15 grams a total market value of $230,000. This computes to about $15,300 per gram of solid polymer, or some $434,000 per ounce. If the chemistry and all four reactors operate properly, about $2 to $3 million worth of latex can be manufactured during each space shuttle flight. NBS has also officially requested that NASA produce for them 30 grams of 30 micrometer latex and 80 grams of 100 micrometer latex which they also plan to market. This is expected to be accomplished by the end of the next three flights.

The MLR flight hardware is mounted in the shuttle mid-deck in place of three storage lockers as shown in Figure 1. The rectangular apparatus shown in Figure 2 is the Support Electronics Package (SEP), and the round drum is called the Experiment Apparatus Container (EAC). Figure 3 shows the EAC with its protective dome removed to display how the four separate independent chemical reactors are carried during each MLR flight. Each of these four reactors can produce a batch of 100 ml of latex by a process called seeded emulsion polymerization. The reactors are loaded 3 days before launch with 100 ml each of the latex recipe, that is, the chemicals calculated to grow the desired large-size latex particles when the hardware is activated in space. A reactor consists of a small hollow steel cylinder with a stirrer at the bottom and a piston at the top. The piston moves to allow expansion and contraction of the latex as a function of temperature and conversion, and an LVDT monitors this movement. Heating tape is wrapped around the cylinder, and four thermocouples are mounted in it. Pre-programmed microprocessors within each reactor control its time and temperature profile. Once in space, the crew turns on the reactors and everything else is automatic. The reactors heat up and then stabilize at the desired hold temperature of 70°C, the chemicals inside slowly polymerize, and the temperature and volume-change data is read out as a function of time to a tape recorder inside the SEP. After 20 hours of operation the reactors turn themselves off. However, the crew must manually turn off the power supply and tape recorder.

THE CHEMICAL PROCESS

The MLR space latexes are manufactured by using as starting material the largest particle-size monodisperse latex that can be made on Earth, and then taking this "seed" latex into the microgravity environment of space and doubling the diameter of each existing microsphere in the latex without allowing any new microspheres to form. This product latex is then returned to Earth and examined microscopically to verify that the chemicals reacted as expected to generate the desired new larger-size monodisperse latex. If this new latex is determined to be of high quality — meaning all the existing microspheres grew exactly the same amount — then this new product latex is taken back into space again and each microsphere is doubled in diameter again. Thus, the microspheres are doubled in diameter repeatedly during each succeeding flight until the final desired size is reached. See Figure 4 for transmission electron photomicrographs of the seed and product latexes from the STS-3 and -6 missions, and Figure 5 for scanning electron photomicrographs of the seed and product latexes from the STS-7 and -11 missions.

It has not been possible so far to grow the seed spheres larger on each flight than a doubling of their diameter, because of current hardware and timeline limitations, without also producing large amounts of coagulum. However, it is expected that later improved hardware would be able to overcome this problem and allow increased seed growth in fewer steps.

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The original small-size seed particles are made on Earth by mixing together chemicals known as the monomer, the initiator, the emulsifier, and the inhibitors. The monomer is the repeating unit of the chemical molecule styrene that links together end-to-end to polymerize into the long chain polystyrene polymer. The initiator is the chemical species that breaks down upon heating and causes the styrene monomer units to link together. The emulsifier is a soap-like material that stabilizes the particles by preventing them from sticking together, and the inhibitors help to prevent the reaction from starting prematurely or new particles from forming in the water phase. When this mixture is heated, with gentle stirring, up to 70°C, the reaction starts and the latex particles begin to grow. Growth ceases when all the monomer in the system is converted to polymer. At this point each microsphere is usually about 0.4 micrometers in diameter.

To obtain larger particles, a carefully calculated amount of additional monomer is added at room temperature to the seed latex which has been prepared as described above. The added monomer is absorbed equally by each of the existing seed particles, as if they were tiny sponges, causing them to swell to over twice their original diameter and become soft and sticky. Additional initiators, inhibitors, and emulsifiers are then added to this monomer-swollen latex, and it is heated again with gentle stirring to 70°C. Once hot, the monomer inside each swollen seed particle begins to polymerize, causing each latex particle to actually shrink slightly as low-density monomer is being converted into high-density polymer, but resulting in each particle achieving a final net size increase to approximately twice its original diameter. Several of these sequential seeding steps can be performed on Earth with the product latexes remaining very monodisperse, until the particles grow to sizes above about 2 or 3 micrometers.

The process becomes very difficult, however, when larger sizes are attempted. High-quality monodisperse polystyrene latexes with particle sizes up to several micrometers in diameter are relatively easy to prepare on Earth; however, when the particles exceed this size range they tend to become polydisperse (all different sizes), or to coagulate, or both. This is due to various physical and chemical processes inherent in seeded emulsion polymerization systems. These include the sensitivity of the latexes to emulsifier concentration and mechanical shear (stirring). If the added emulsifier is insufficient to stabilize the growing latex particles, they will flocculate to form coagulum. If too much emulsifier is added, a new crop of tiny particles will be formed, along with the main crop of large ones, and the latex particle-size distribution will be bimodal (two different sizes) rather than monodisperse.

At particle sizes well below 1 micrometer, the emulsifier concentration range is rather forgiving, but at larger sizes this operable range becomes smaller and smaller, until at sizes above 1 micrometer it becomes "knife-edge," meaning the reaction can go either way — make a good-quality monodisperse latex one time, and make a poor-quality polydisperse latex usually with lots of coagulum the next time it is performed. As even larger sizes are attempted, the reaction always fails and gives only poor-quality polydisperse latexes. As one attempts to manufacture particles in sizes significantly larger than ∿2 micrometers, the monomer-swollen particles have an increasing tendency to cream (rise to the top of the reaction pot) at the beginning of the reaction, and the partially-polymerized particles to settle out during the later stages of the reaction. This is due to the density difference between the particles and the water in which they are suspended, and because Brownian motion can no longer hold them in suspension due to their large size. According to Stokes law, the terminal velocity of a sphere falling (or rising) through a fluid is directly proportional to the square of its radius and the difference in density between it and the fluid. As the
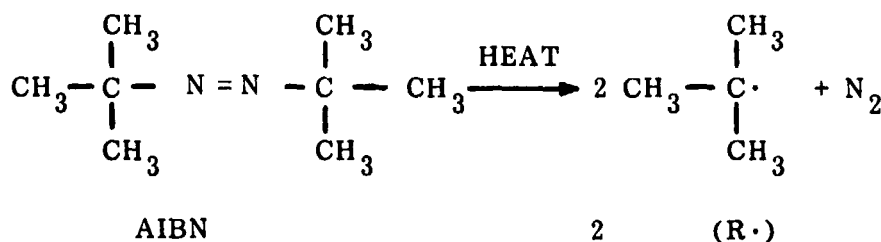
polymerization proceeds, low-density styrene monomer (density = 0.905 g/cm^3) with which each seed sphere is saturated is converted into high density polystyrene polymer (density = 1.05 g/cm^3). Since the polystyrene seed particles are swollen with at least six times their weight with additional monomer and thus their average density (ranging from about 0.95 to 0.92 g/cm^3) is now less than that of water, the swollen particles tend to cream before and during the early stages of the reaction (while they are full of monomer and thus lighter than water) and to settle during the later stages (when most of the monomer has been converted to polymer and they are heavier than water). Of course, stirring can be increased to offset this creaming and settling, but stirring at a rate sufficient to keep the particles in suspension usually results in coagulation because the large particles are very sensitive to mechanical shear. This means that if the particles are stirred too rapidly while they are polymerizing, they will hit each other and stick to form bunches or even huge lumps of coagulum.

However, if the average density of the monomer-swollen particles were the same as that of the fluid they were suspended in, then creaming or settling would not occur. It would be possible to match the densities of the particles and the water phase at either end of the reaction by changing the monomer composition, such as by substituting a heavier monomer like p-chlorostyrene or vinyltoluene-t-butylstyrene mixtures for pure styrene, or by adding electrolytes or non-electrolytes to the water phase, but particle and fluid densities cannot be matched at the beginning, the end, and continuously throughout the reaction due to the continuous change in density of the particles as they polymerize. If a heavier monomer is used for swelling, the particles could be made more closely neutrally buoyant at the beginning of the reaction, but being heavier, would settle more rapidly during the middle and later stages of the polymerization.
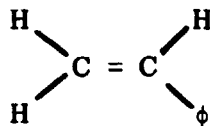
All the particles must also be held at a constant and uniform temperature throughout the entire course of the reaction or, again, a poor-quality polydisperse product will be produced. If one portion of the latex reaction vessel varies more than 1 or 2°C, the particles in the hotter portion will grow slightly faster and therefore slightly larger than the particles in the cooler portion. The hotter, faster-growing particles will actually rob monomer from the cooler, slower-growing particles.

Therefore, due to these and other difficulties inherent in producing these large-particle monodisperse latexes on Earth, it was decided to perform the polymerization in microgravity where stirring could be held to the absolute minimum required for good heat transfer within the reactor.
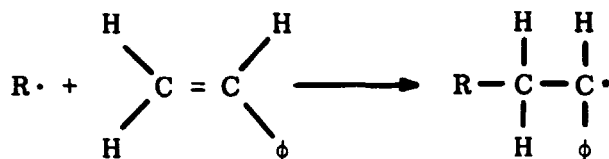
The series of chemical reactions by which liquid styrene monomer is converted into hard colorless polystyrene polymer can be shown as follows: When the initiator, Azobisisobutyronitrile (AIBN) in this case, is heated to 70°C it decomposes into two radicals (symbolized by R·) plus a molecule of nitrogen gas (N$_2$) according to the equation:
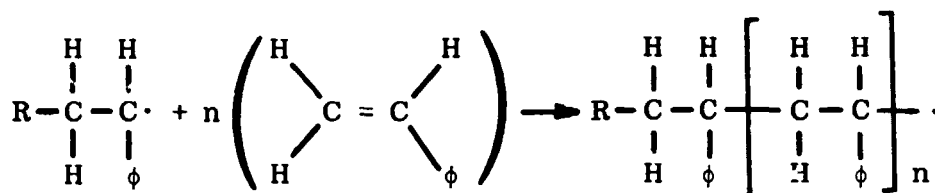
$$CH_3-\underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{C}}-N=N-\underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{C}}-CH_3 \xrightarrow{\text{HEAT}} 2\ CH_3-\underset{\underset{CH_3}{|}}{\overset{\overset{CH_3}{|}}{C}}\cdot\ +\ N_2$$

AIBN   2   (R·)

One of the radicals (R·) then initiates the chain-building process, adding to a styrene monomer molecule

$$H \diagdown \diagup H$$
$$C = C$$
$$H \diagup \diagdown \phi$$

by attacking its double bond.

$$R\cdot + \quad \begin{matrix} H & & H \\ \diagdown & & \diagup \\ & C = C & \\ \diagup & & \diagdown \\ H & & \phi \end{matrix} \longrightarrow R-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle H}{|}}{C}}-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle \phi}{|}}{C}}\cdot$$
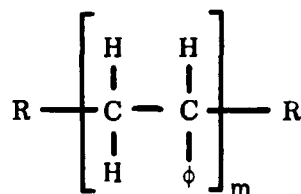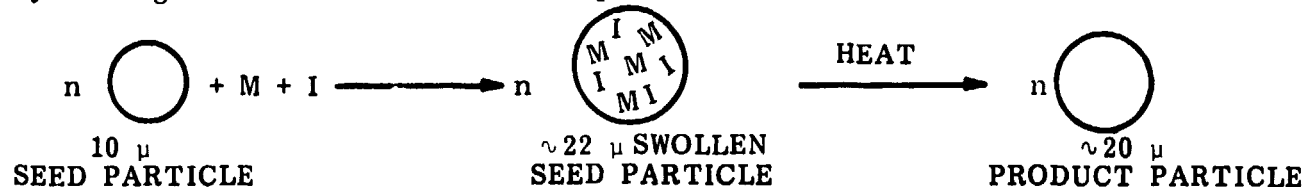
The secondary carbon atom $-\overset{|}{\underset{|}{C}}\cdot$ is now highly reactive due to this attack and adds another styrene molecule; and then this one adds another, and so on, building a long linear chain of styrene units

$$R-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle H}{|}}{C}}-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle \phi}{|}}{C}}\cdot + n\left(\begin{matrix} H & & H \\ \diagdown & & \diagup \\ & C = C & \\ \diagup & & \diagdown \\ H & & \phi \end{matrix}\right) \longrightarrow R-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle H}{|}}{C}}-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle \phi}{|}}{C}}\left[\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle H}{|}}{C}}-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle \phi}{|}}{C}}\right]_n\cdot$$

until this growing chain meets another growing chain, or another (R·) and they join, thus stopping the growth and forming a molecule of polystyrene.

$$R\left[\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle H}{|}}{C}}-\overset{\overset{\textstyle H}{|}}{\underset{\underset{\textstyle \phi}{|}}{C}}\right]_m R$$

Billions of these long chains of polystyrene are formed inside each existing latex seed particle, increasing its diameter to the desired new larger size. This can be represented in the following drawing in which additional styrene monomer (M) and initiator (I) is dissolved inside the existing polystyrene seed particles and then polymerized by heating to increase the size of the particles:

$$n \bigcirc + M + I \longrightarrow n \bigcirc \overset{\text{HEAT}}{\longrightarrow} n \bigcirc$$

| 10 μ | ∿22 μ SWOLLEN | ∿20 μ |
| SEED PARTICLE | SEED PARTICLE | PRODUCT PARTICLE |

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Note that each final product latex particle shrinks slightly from its monomer-swollen diameter, after all the monomer has been converted to polymer, but still exhibits a net increase of doubling its seed diameter if a ratio of about 6/1 monomer to polymer is used.

Figure 6 graphically illustrates the difference in quality between the space-made latex (top photo) which was transferred to NBS, and the remaining portion of the same seed latex from the same bottle which was polymerized on Earth under identical conditions except for microgravity as a ground control (bottom photo).

Once it is demonstrated that these large-size monodisperse latexes can be routinely produced in quantity and high quality, they can be marketed for many types of scientific applications. Biomedical research applications include such things as drug carriers and tracers in the body, human and animal blood flow studies, membrane and pore sizing in the body, and medical diagnostic tests. Other applications include use as calibration standards for optical and electron microscopes, Coulter counters, laser light-scattering equipment, and for many other types of laboratory equipment.

This project was directed by Dr. John W. Vanderhoff, Principal Investigator (Lehigh University, Bethlehem, PA) along with Co-Investigators, Drs. Fortunato J. Micale and Mohamed S. El-Aasser (also of Lehigh University) and Dale M. Kornfeld (NASA/Marshall Space Flight Center). Two former graduate student research assistants, Drs. E. David Sudol and Chi-Ming Tseng, received their Ph.D. degrees at Lehigh University during the course of this program; and a third student, Mr. Anthony Silwanowicz, received his M.S. degree as part of this program. Their theses work represents all the chemical latex recipe research carried out during this program. This effort was sponsored by the Microgravity Sciences and Applications Division of the Office of Space Science and Applications, NASA Headquarters. The Principal Investigator team research program was managed by NASA/MSFC, Huntsville, AL, under contract NAS8-32951, initiated on February 22, 1978. Flight hardware development was also managed by NASA/MSFC. The MLR reactors and ground support equipment were designed and manufactured by General Electric Company Space Division, Philadelphia, PA, and the SEP and EAC cannister were designed and manufactured by Rockwell International Space Operations and Satellite Systems Division, Downey, CA.

PUBLICATIONS

U. S. Patent No. 4,247,434; issued January 27, 1981, "Process for Preparation of Large-Particle-Size Monodisperse Latexes." Inventors: J. W. Vanderhoff, F. J. Micale, M. S. El-Aasser, and D. M. Kornfeld.

J. W. Vanderhoff, M. S. El-Aasser, F. J. Micale, E. D. Sudol, C. M. Tseng, A. Silwanowicz, and D. M. Kornfeld: Preparation of Large-Particle-Size Monodisperse Latexes in Space: The STS-6 and STS-7 Mission Results. Presented at AICHE 1984 Summer National Meeting, Philadelphia, Pennsylvania, August 19-22, 1984.

J. W. Vanderhoff, M. S. El-Aasser, F. J. Micale, E. D. Sudol, C. M. Tseng, A. Silwanowicz, and D. M. Kornfeld: Preparation of Large-Particle-Size Monodisperse Latexes in Space: The STS-3, STS-4, STS-6, and STS-7 Mission Results. In Proceedings of the Second Symposium on Space Industrialization, NASA CP-2313, ed., Camille M. Jernigan (Huntsville, Alabama, February 13-15, 1984), October 1984.

J. W. Vanderhoff, M. S. El-Aasser, F. J. Micale, E. D. Sudol, C. M. Tseng, A. Silwanowicz, D. M. Kornfeld, and F. A. Vicente: Preparation of Large-Particle-Size Monodisperse Latexes in Space: Polymerization Kinetics and Process Development. Journal of Dispersion Science and Technology, 5(3&4), 231-246 (1984).
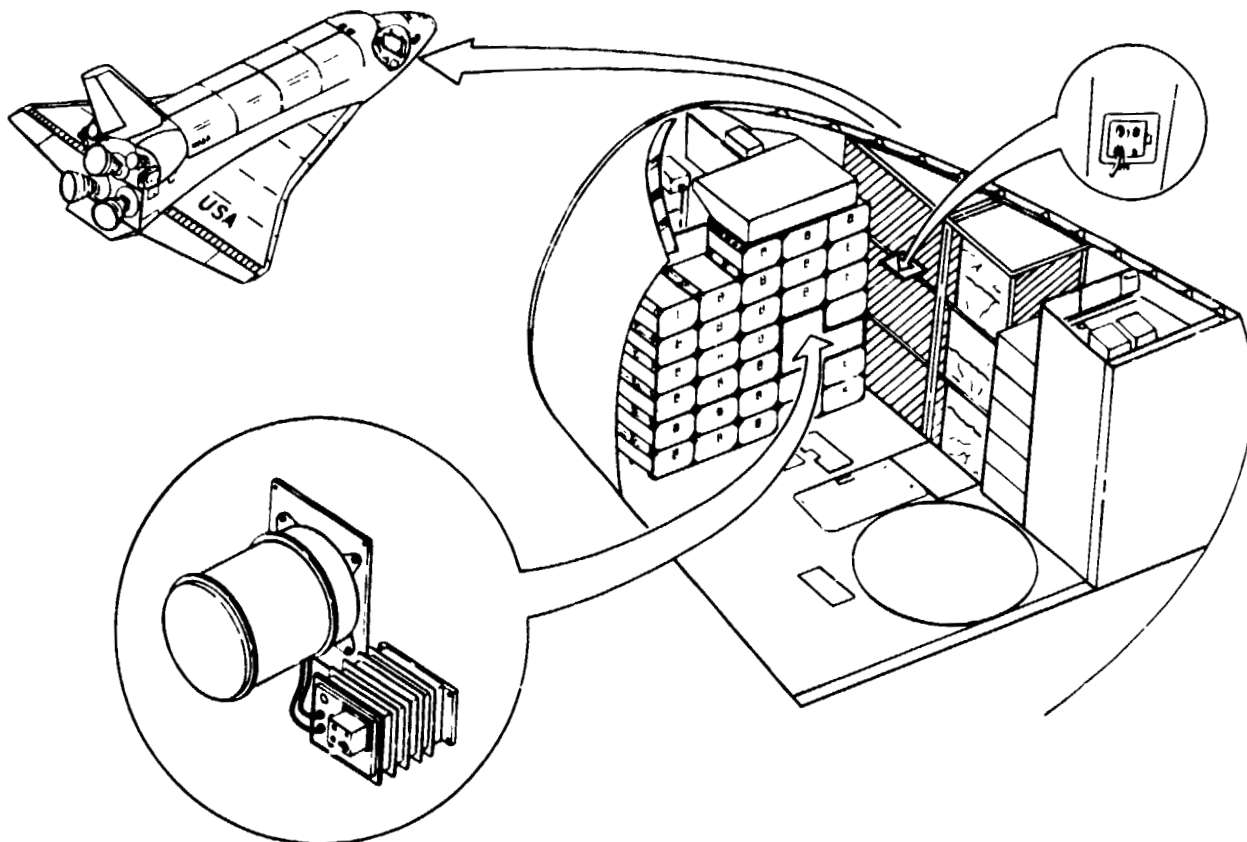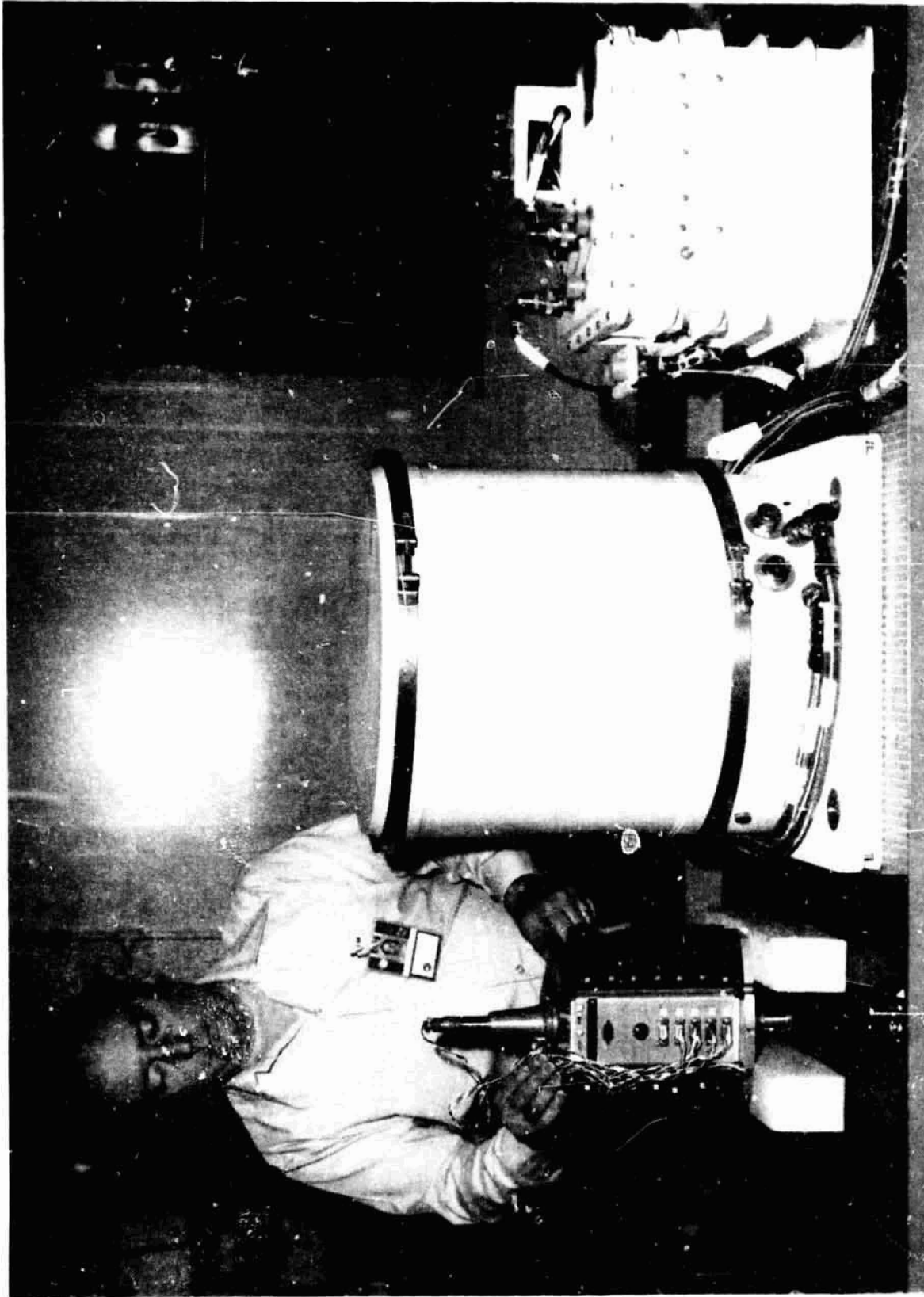
Figure 1. STS accommodations for MLR.

Figure 2. NASA MLR Co-Investigator Dale M. Kornfeld exhbits one of the latex reactors. Four of these reactors are mounted inside the Experiment Apparatus Container (center). The Support Electronics Package is shown at right.
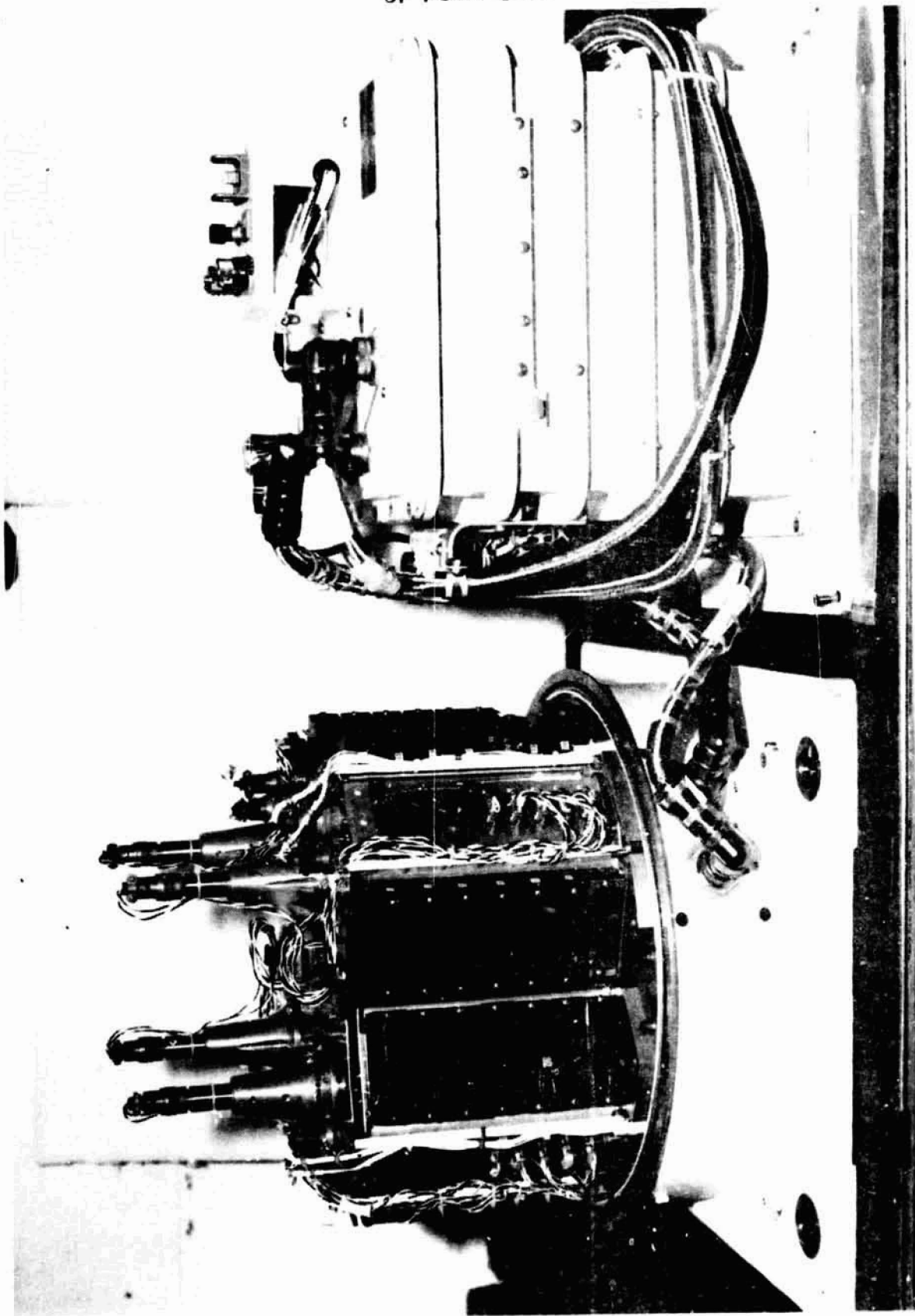
Figure 3. The EAC (left) is shown with its cover removed to expose the four latex reactors as they are mounted for flight. The SEP (right) contains a data tape recorder and malfunction detection circuitry, and provides power to the reactors.

2.5 μ SEED

STS-3

3.4 μ PRODUCT
Recipe # 1

4.1 μ PRODUCT
Recipe # 2

5.0 μ PRODUCT
Recipe # 3
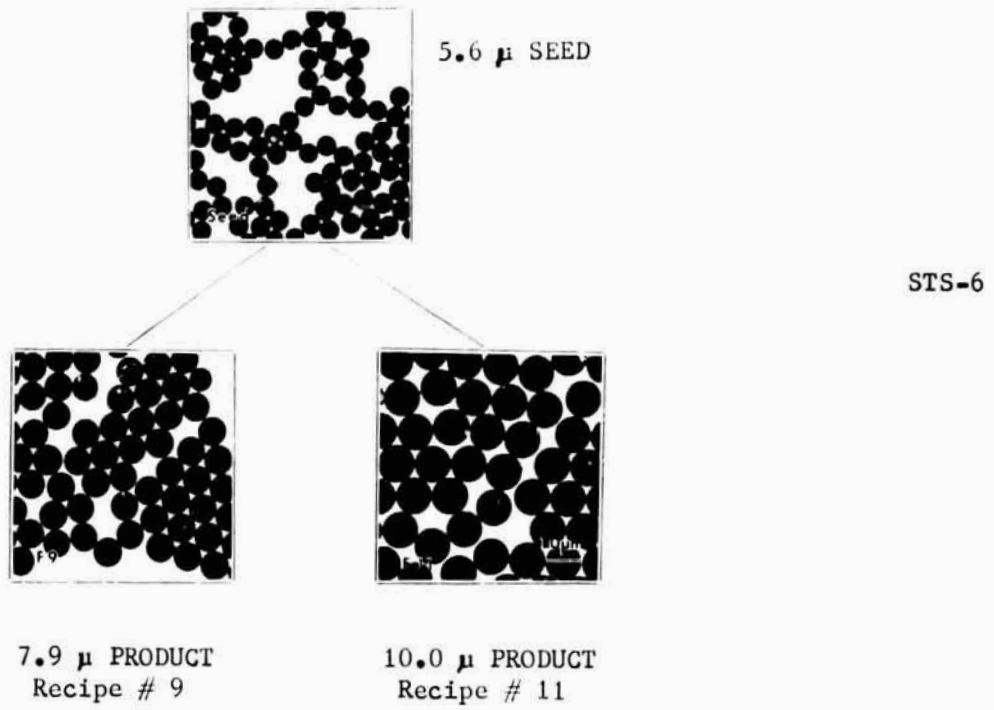
5.6 μ SEED

STS-6

7.9 μ PRODUCT
Recipe # 9

10.0 μ PRODUCT
Recipe # 11
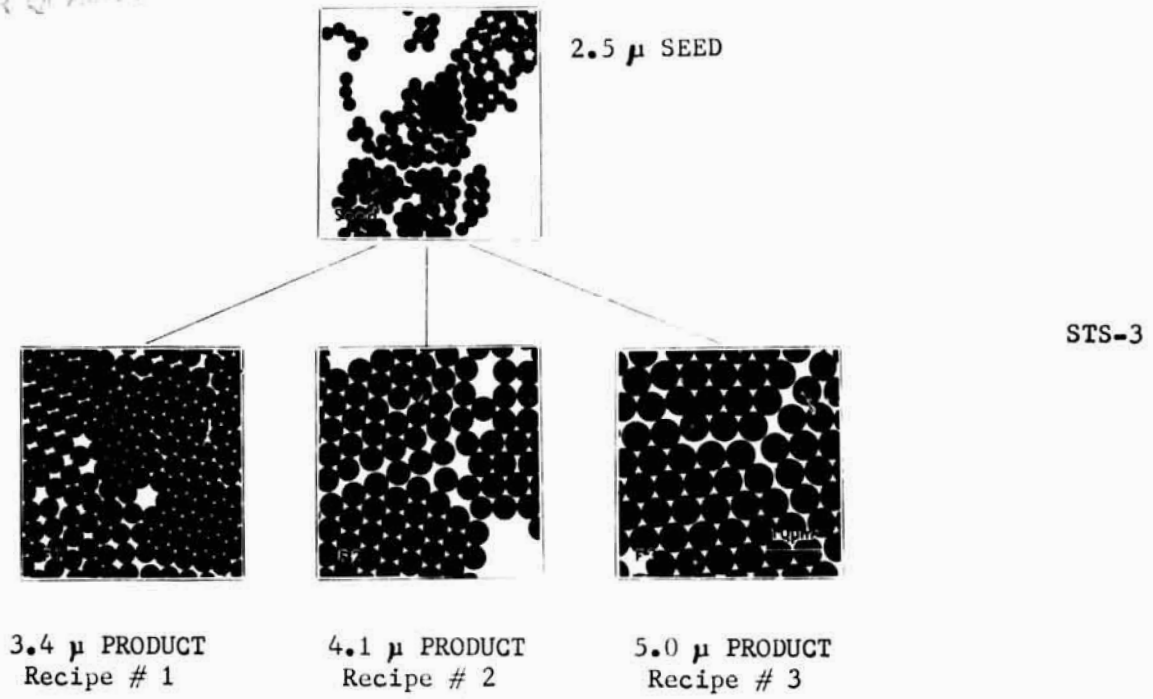
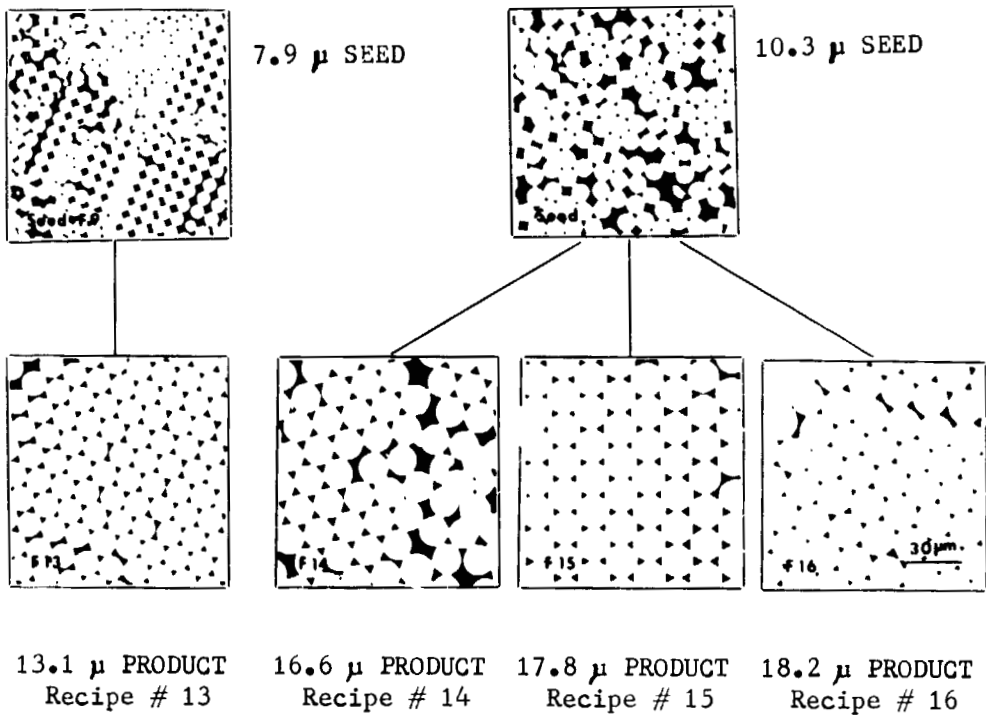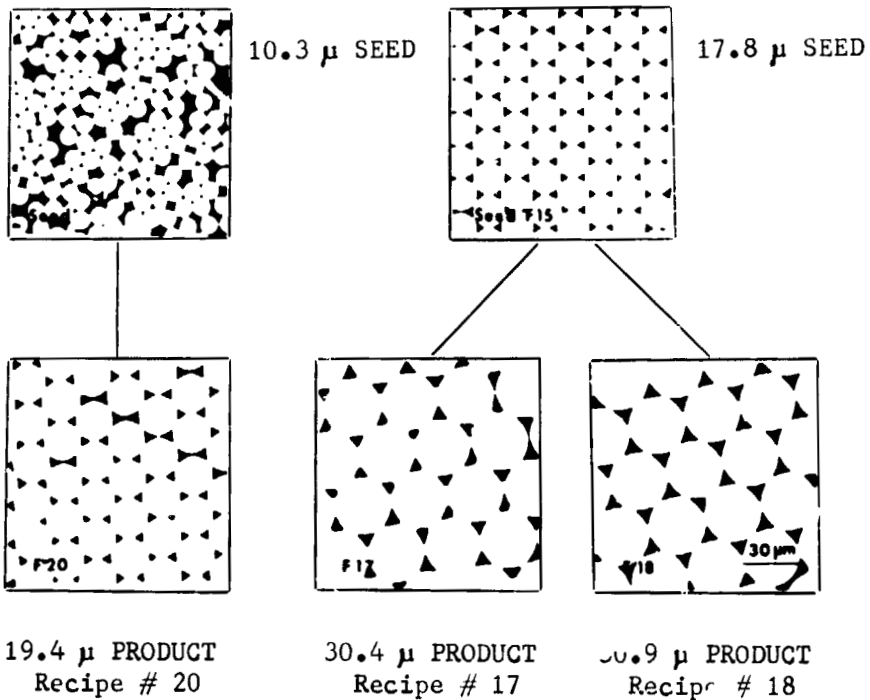Figure 4.   Transmission electron photomicrographs of the MLR seed and product
latexes from the STS-3 and STS-6 shuttle missions.

7.9 μ SEED

10.3 μ SEED

STS-7

13.1 μ PRODUCT
Recipe # 13

16.6 μ PRODUCT
Recipe # 14

17.8 μ PRODUCT
Recipe # 15

18.2 μ PRODUCT
Recipe # 16

10.3 μ SEED

17.8 μ SEED

STS-11

19.4 μ PRODUCT
Recipe # 20

30.4 μ PRODUCT
Recipe # 17

ᴊ0.9 μ PRODUCT
Recipe # 18
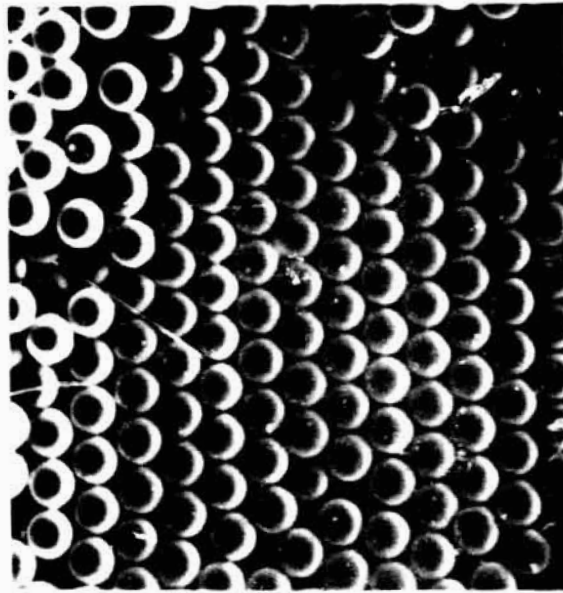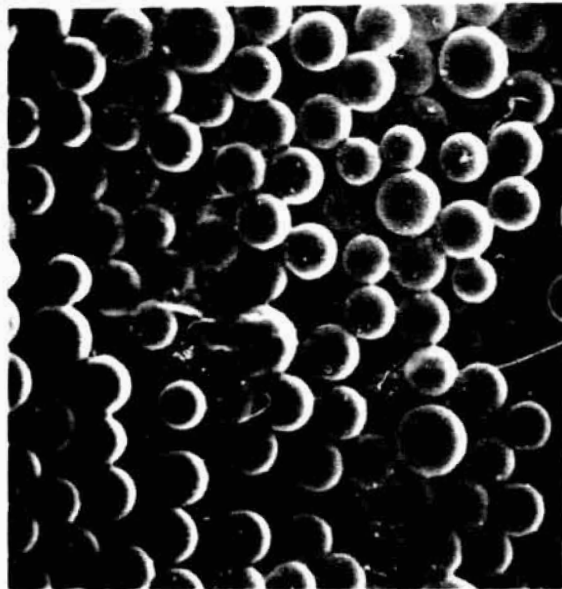
Figure 5.   Scanning electron photomicrographs of the MLR seed and product
latexes from the STS-7 and STS-11 shuttle missions.

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Space-manufactured latex

Ground-control latex

Figure 6. Scanning electron photomicrographs (800X) of the 10 micrometer
high-quality latex manufactured in space (top) and the corresponding
ground-control latex (bottom) manufactured on Earth under
identical conditions from the same seed, except
for microgravity.

TABLE 1. MLR FLIGHT SUMMARY

Flight No. (Date)	Recipe No.	Seed Size (μ)	Product Size (μ)	Monomer/Polymer Swell Ratio	Pre/Process Stir Speed (rpm)	Remarks
STS-3 March 22, 1982	1	2.5	3.4	2/1	13/13	Very good product latex
	2	2.5	4.1	4/1	13/13	Very good product latex
	3	2.5	5.0	10/1	13/13	Very good product latex
	4	0.19	0.26	2/1	13/13	Latex polymerized prematurely; no data recorded
STS-4 June 27, 1982	5	5.5	7.5	2/1	13/13	An electrical failure in the SEP prevented all four reactors from heating properly and all four latexes were only partially polymerized; therefore, no usable product was obtained.
	6	5.5	9.0	4/1	13/13	
	7	5.5	9.3	5.7/1	13/13	
	8	5.5	10.8	6.2/1	13/13	
STS-6 April 4, 1983	9	5.6	7.9	2/1	13/13	Excellent product latex used as seed for Recipe #13.
	10	5.6	--	4/1	13/13	This reactor failed to heat, thus no product.
	11*	5.6	10.0	6/1	13/13	Excellent product latex; FIRST SPACE PRODUCT.
	12	0.19	0.26	2/1	13/13	Repeat of Recipe #4; product not fully converted.
STS-7 June 18, 1983	13	7.9	13.1	6/1	13/13	Excellent product latex
	14	10.3	16.6	4/1	13/13	Excellent product latex
	15	10.3	17.8	6/1	13/6	Excellent product latex used as seed for Recipes #17/18.
	16	10.3	18.2	6/1	6/3	Excellent product latex; new slower stir speeds
STS-11 February 3, 1984	17	17.8	30.4	5/1	13/6	Excellent product latex
	18	17.8	30.9	5/1	6/3	Excellent product latex
	19	10.3	18	6/1	13/6	Reactor apparently contaminated; mostly coagulum.
	20	10.3	19.4	6/1	6/3	Excellent product latex.

*The 10 micrometer product latex from Recipe #11 was transferred to the National Bureau of Standards on July 17, 1984, and certified for sale in April 1985, thus becoming the first "product" to be manufactured in space and marketed on Earth. NBS determined this latex to have 1.1% std. dev. of diameter.

APPROVAL

MONODISPERSE LATEX REACTOR (MLR)

A Materials Processing Space Shuttle
Mid-Deck Payload

By Dale M. Kornfeld

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

A. J. DESSLER
Director, Space Science Laboratory