Analysis of Microgravity Experiments Conducted on the Apollo Spacecraft

R.J. Sharpe
The University of Alabama, Tuscaloosa, Alabama

M.D. Wright
Marshall Space Flight Center, Marshall Space Flight Center, Alabama
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R.J. Sharpe
The University of Alabama, Tuscaloosa, Alabama

M.D. Wright
Marshall Space Flight Center, Marshall Space Flight Center, Alabama

National Aeronautics and Space Administration

Marshall Space Flight Center • MSFC, Alabama 35812

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<tr>
<td>Be</td>
<td>beryllium</td>
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<tr>
<td>Bi</td>
<td>bismuth</td>
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<td>CO$_2$</td>
<td>carbon dioxide</td>
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<td>Cu</td>
<td>copper</td>
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<td>In</td>
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<td>MSFC</td>
<td>Marshall Space Flight Center</td>
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<td>PFTBA</td>
<td>perfluorotributylamine</td>
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NOMENCLATURE

\( a \)  \hspace{1cm} \text{particle radius}

\( D \)  \hspace{1cm} \text{dielectric constant of the medium}

\( E \)  \hspace{1cm} \text{electric field strength}

\( U \)  \hspace{1cm} \text{velocity}

\( \delta \)  \hspace{1cm} \text{particle thickness}

\( \zeta \)  \hspace{1cm} \text{particle potential}

\( \eta \)  \hspace{1cm} \text{viscosity of the medium}
1. INTRODUCTION

NASA’s Apollo program set out to complete the challenge given it by President John F. Kennedy in 1961 to land humans on the Moon and safely bring them back to Earth. By its completion, the program sent a total of 12 men onto the lunar surface and back safely. This ushered in a new era of exploring worlds beyond the Earth combined with a broader understanding of the Earth’s place in the solar system.

Traveling to the Moon and back in the Apollo capsule required a 3-day journey in each direction. Astronauts were given a strict list of operations and maintenance from Johnson Space Center, Houston, TX, to be performed as they made their journey. However, one operation that was often ignored was the scientific opportunity that the Apollo capsule provided. Taking advantage of some of the longest presences in space to date, the spacecraft offered a laboratory in which some of the first human-tended microgravity experiments could take place. These experiments, while few in number and subordinate to the primary goal of lunar science, laid the foundation for the programs of Skylab, Spacelab, and several focal points of the International Space Station.

Throughout the duration of the Apollo program, a total of five experiments (not including the three biomedical experiments which also took place) were conducted in the microgravity environment of the command, service, and lunar modules. One of them—a light flashes experiment in which methods were tested to explain flashes of light the astronauts would see while in space—was the only one considered life science. The four remaining experiments (two of which span different missions within the program) were all considered microgravity/materials experiments.

This Technical Memorandum (TM) outlines those four microgravity experiments that laid the foundation for later eras of human-tended space science. The experiments fell within three major categories: composite materials, organic chemistry/proteins, and fluid dynamics. The descriptions, results, and significance of these experiments are discussed in this TM.
2. COMPOSITE CASTING EXPERIMENT AND DEMONSTRATION

Microgravity experiments in materials processing, a foreign concept prior to the Apollo missions, became reality as NASA scientists began attempts at developing metals and alloys that had previously been extremely difficult, if not impossible, to form. I.C. Yates, Jr., NASA scientist in the Marshall Space Flight Center (MSFC) Process Engineering Laboratory, noted that using a microgravity environment to synthesize unique materials had been a topic of discussion for several years. He hypothesized that by removing the buoyant force of gravity along with thermal convection, such immiscible materials could be developed in homogeneous mixtures. His experiment, conducted on Apollo 14 (launched January 31, 1971) by astronauts Alan Shepard, Stuart Roosa, and Edgar Mitchell, is shown in this section.

The composite casting experiment consisted of a small heating apparatus that could accept various samples, heat them, and then cool them back to the solid state for examination and comparison with similar materials developed on Earth. The apparatus featured an electrical heater, a section for storage in the heater that could also be used to cool, and 18 samples in sealed vials. An image of the instrumentation is given in figure 1.

![Image of the composite casting experiment](image)

Figure 1. Workup of the composite casting experiment.
The size of the capsule inside the heater allowed for it to be effectively shaken while heating in order to loosen any particles sticking to the sides of the capsule. Each sample was designed to be heated for 10 min to temperatures of up to 235 °F (112 °C) and then cooled for 30 min, returning to an average temperature of 100 °F (37.7 °C).

The samples tested varied from organic compounds to metallic mixtures and were chosen based on several guidelines that ensure the collection of useful data from the experiment. For organic composites, the samples included a melting point no higher than 170 °F (76.7 °C), a low flammability and vapor pressure, and availability, amongst others. Most importantly, though, was the requirement to have a significant difference in specific gravity (or density), which would result in immiscibility. For metallic composites, the requirements were more situational. For instance, candidates considered were metal wires, glass filaments, metallic particles, and others. Still, significant differences in density were required to show a possible mixing of previously immiscible materials. The selected 18 samples were split into three categories: powder compacts (used to observe redistribution of particles after heating), metallic and gas mixtures, and immiscible materials (mostly organic compounds). The duration of the flight allowed for only 11 of the 18 samples to be tested. These samples were returned to MSFC for examination and comparison.

The powder composites, comprised of varying amounts of indium (In), bismuth (Bi), and tungsten, showed a more uniform mixture in zero gravity than similar materials developed on Earth. This suggested that powder metal composites could be developed more homogeneously in zero gravity than on Earth.

The metallic and gas mixtures were examined and showed results similar to the powder composites. Each sample completed showed a relatively uniform distribution of metal particles and/or gas particles upon heating and cooling. These mixtures were composed of In-Bi fibers, copper (Cu)-beryllium (Be) fibers, as well as argon gas and other materials. Sample 11, which contained 68% paraffin (general linear alkane groups having the formula \( C_nH_{2n+2} \)), 7% Be-Cu fibers, and 25% argon gas, displayed a strong homogeneous gas distribution as shown in figure 2.

The organic/immiscible materials continued the positive results. Samples of paraffin and sodium acetate were mixed in varying concentrations. When the results were examined, it was found that although the distribution of material between the two chemicals was not uniform, several traces of each chemical were found within each region of the samples. Shown in figure 3 is an image of sample 6, a 1:1 mixture of paraffin and sodium acetate.

In conclusion, it was determined that composites previously thought to be immiscible on Earth could be more homogeneously mixed in the absence of gravity. Although the range of materials tested was limited, this experiment became the foundation for numerous experiments on later missions. As an example, a test very similar to this one took place on Space Shuttle Endeavor in STS-47: Spacelab J, in which immiscible alloys of Cu-lead and aluminum-In were mixed for the purpose of superconductor development.
Figure 2. Sample 11 of 68% paraffin, 7% BeCu fibers, and 25% argon after heating and cooling.

Figure 3. Sample 6 of the composite experiment, showing sodium acetate (light colored) dispersed in paraffin (dark colored).
3. STUDY OF ELECTROPHORESIS IN SPACE

Another popular area of study demonstrated on Apollo was electrophoresis, a phenomenon in which charged particles or fluids are put in motion when an electric field is applied. The study was chosen by NASA for experimentation on Apollo because it was comprised of relatively simple experiments with the potential to yield a significant amount of useful data for study on Earth. The experiments, which took place over both Apollo 14 and Apollo 16, are discussed in sections 3.1 and 3.2.

In electrophoresis, charged particles in a fluid move toward the oppositely charged electrode in an experiment. They will move with a velocity \( U \), according to the following equation:

\[
U = \frac{\zeta ED}{4\pi \eta},
\]

where \( \zeta \) is the particle potential, \( E \) is the electric field strength, \( D \) is the dielectric constant of the medium, and \( \eta \) is the viscosity of the medium. The equation itself is commonly known as the Helmholtz-Smoluchowski relation. This equation, however, has several dependencies. The shape and size of each particle plays a major factor in the actual speed, although it is not represented in the equation. In addition, in round particles, the ratio of the particle’s radius \( a \) to the thickness of the ionic atmosphere \( \delta \) plays a factor. When the ratio is \( >100 \), equation (1) applies, but when it is as low as 0.1, a different relationship can be expressed:

\[
U = \frac{\zeta ED}{6\pi \eta}.
\]

The equation potentially becomes more complex when the ratio is between those two values. This separating effect, while visible on Earth, was thought to be slightly distorted due to the Earth’s effects of sedimentation and convection currents. During the workup of the Apollo 14 and 16 experiments, the 0.1 ratio equation was applicable.

3.1 Apollo 14 Experiment

Dr. James Bredt suggested the development of an electrophoresis experiment that would take place during the return from the Moon. The instrumentation would consist of a metal frame casing with a small, rectangular window for viewing. A pump was attached to circulate the fluid, and a light was placed in the apparatus to better photograph particles during the experiment. Another instrument was added to remove gaseous bubbles, and the Apollo Hasselblad camera was used to take photographs. The entire apparatus is shown in figure 4.

The experiment was comprised of two different colored dyes (blue and red), chosen for their distinguishable color and mobility at the desired pH value. In addition, these dyes showed
an ability to separate, within a short distance, a requirement for the small Apollo workup. Two secondary experiments were also suggested using high molar mass biological materials (hemoglobin for one and a salmon deoxyribonucleic acid (DNA) sample for the other), but this experiment bore the highest chance of success. When the samples were completed, they were brought to the Lunar Receiving Laboratory for quarantine and then returned for study.

As was feared, the biological experiments failed, showing no significant electrophoresis. It was later found that this was due to contaminant bacteria that destroyed the biological material before the experiment was ever conducted.4

The red and blue dyes, however, showed a distinct electrophoretic effect.3 Their separation, while not perfectly sharp, showed a significant sharpness increase in comparison to similar experiments conducted on Earth. This gave NASA scientists significant evidence that the absence of sedimentation and convection in microgravity had improved the separation of the two dyes as compared to Earth-bound experiments.

3.2 Apollo 16 Experiment

The Apollo 16 (launched on April 16, 1972) electrophoresis experiment learned from the failures and complications of Apollo 14 and developed a more effective electrophoresis experi-
ment with a much stronger chance of success with the high molar mass material. Scientists decided that the instrumentation of the test should be very similar to that used in Apollo 14, albeit several upgrades could be made. A diagram of the Apollo 16 workup is shown in figure 5.

![Diagram of the Apollo 16 electrophoresis apparatus.](image)

Using the supports shown in the diagram, the new apparatus was designed so that a camera could be attached to the device and take pictures automatically (one every 20 s). The sample cells would be made of 10-cm-long polycarbonate tubes.

Seeking again to find a successful organic material for study, NASA scientists decided on the use of polystyrene latexes for the experiment. Due to their availability, high molar masses, and scientists’ familiarity with them, two samples of the latex with varying charges were chosen. The experiment itself proceeded much in the same manner as did the Apollo 14 electrophoresis experiment with the results being returned to Earth for investigation.

Scientists found that the polystyrene did show a migration in the samples, although it was not as pronounced as they had hoped. A small amount of swirling of the two materials was noted near the interface, making a sharp separation more difficult to interpret. However, none of the experimental/instrumental errors of the Apollo 14 workup affected the Apollo 16 experiment. Furthermore, it was noted that a small amount of material in the sample casing escaped through a loose seal, accounting for the “blob of stuff” that astronaut Ken Mattingly noted during the experiment. These results served as further confirmation to scientists that the lessening of sedimentation and convection in microgravity enhanced the electrophoretic mobility of the samples.
This research served as a major motivation for research in the increased purification of biological materials, such as medicines and vitamins. It was hoped that by finding more efficient methods to carry out electrophoresis, the purity of these materials could be enhanced. The research conducted here laid groundwork for future microgravity experiments such as the work of separating proteins on Spacelab J.⁶
4. HEAT FLOW AND CONVECTION EXPERIMENTS

In another experiment that took place over two separate Apollo missions, NASA scientists designed a set of experiments to explore the forces in microgravity that can cause convection in fluids. The Apollo 14 experiment was carried out by astronaut Stuart Roosa, and the Apollo 17 experiment was conducted by Ron Evans.

Convection, the movement of particles within a fluid, or the movement of two fluids of different densities or at different temperatures, is a common event on Earth due to gravitational forces. This property of fluids is crucial in consideration of the separation of liquids, precipitation, etc. However, in a microgravity environment, this effect on fluids becomes significantly less pronounced. As a result, the smaller convection force can be viewed in contrast to their minute effect which is often masked on Earth due to gravity. NASA scientists, in one of the first fluids experiments in space, sought to understand how different fluids would perform in this environment. This understanding would benefit scientists in their consideration of future materials research. The experiments are discussed in sections 4.1 and 4.2.

4.1 Apollo 14

The Apollo 14 convection experiment consisted of a square box apparatus designed to carry out three different types of fluids experiments in different areas: the radial cell, the flow pattern cell, and the zone cell. The radial cell was comprised of a sealed circular dish and was designed to observe radial heat flow in a gas. The flow pattern cell, designed to test if surface tension alone could produce a strong enough effect for convection, consisted of an aluminum dish with a uniformly distributed heater. The zone cell was made up of two see-through cylinders of material with a central heater in each cylinder. It was designed to observe convection based on temperature color maps in two substances with different viscosities. An image of the apparatus is given in figure 6.

The radial cell would be filled with carbon dioxide (CO$_2$) gas. The flow pattern cell was filled with a heavy oil called Krytox® (Dupont™) which was topped with aluminum flakes so that the flow could be observed. The zone cell contained water in one cylinder and a sugar mixture in the other so that liquids with different viscosities could be tested. These tests made use of liquid crystal strips which could be used to make temperature measurements based on a calibration scale. The experiments were run and the results delivered back to Earth for investigation.

The radial cell was found to exhibit convection based on the observation of sharp shifts in temperature that could not be caused by simple heating. The reason for the convection was also difficult to interpret. Scientists decided that the temperature changes were due to oscillatory convection. In addition, they initially suspected that the convection itself had taken place due to small amounts of gravity, but no concrete evidence was able to prove it. This led them to believe that some previously unexpected force might have caused the convection.
The flow pattern cell showed much more conclusive results. Convection was observed, allowing scientists to conclude that surface tension alone could produce the effect in the absence of gravity. However, the exact pattern of the convection was only partially interpreted.

The zone cell results showed similar temperature oscillations as in the radial cell experiment, albeit not as large. However, due to the cylindrical construction of the zone cells, no correlations were able to be drawn from the data.

These results gave scientists both new answers and additional questions. Surface tension-driven convection was proven, but the force that had caused convection in both the radial and zone cells was still unaccounted for. Researchers postulated that fluid volume expansion might be a factor as well as a possible interfacial tension that might have caused convection. In addition, other hypotheses were developed to account for the observed temperature oscillations. The experiment as a whole yielded several useful results and was considered a success, but the unanswered questions prompted NASA scientists to conduct a second convection experiment during the Apollo 17 mission.

### 4.2 Apollo 17

The second convection experiment aboard Apollo 17 (launched December 17, 1972) employed a similar design with slight modifications in both the apparatus and sample selection.
Scientists decided that both the radial and flow pattern tests should be conducted again in similar fashion. However, the zone test was replaced in favor of the lineal test, which was designed to observe heat flow (and possible convection) in a confined container in microgravity. It would be filled with the Krytox oil used in the flow pattern test on Apollo 14. The updated radial test contained argon gas instead of CO\textsubscript{2}. The flow pattern test consisted of two experiments with varying fluid depth—2 mm and 4 mm. The fluid used was Krytox, which was topped with an aluminum powder so that flowing could be observed. Both the radial and lineal tests contained the liquid crystals for temperature measurements.

The flow pattern test showed results similar to the Apollo 14 experiment, although an unexpected event took place when it was carried out. Astronaut Ron Evans reported that large bubbles began to form in each test while the oil was injected into the cell. He attempted to eliminate them by stirring but was only able to remove small amounts. Images of the two cells are given in figure 7.

Researchers speculated that these bubbles had an affect on the results of the experiment, but ground tests concluded that they did not adversely affect the outcome. Convection driven by surface tension was observed, further confirming the results and conclusions of the Apollo 14 flow pattern experiment. Furthermore, researchers concluded that convection can occur at lower temperature gradients in microgravity than on Earth.

The radial and lineal tests, unfortunately, showed no significant convection. These data suggested that the outcome of the Apollo 14 experiment could have been caused by simple vibrations of both the apparatus and spacecraft.

Based on data from both experiments, researchers were able to conclude that, in space, convection of fluids could be abated, eliminated, or utilized for practical means. These conclusions carried several implications. For example, although it is safe to assume that convection in space is much weaker than in gravity, assuming that none exists is erroneous. Also, vibrations can fairly easily induce convection patterns in microgravity.
These experiments provided invaluable data to scientists seeking to reconcile effects on fluids caused by gravity from those caused by other factors. A significant step in understanding the properties of fluids in microgravity, the tests laid the groundwork for further experiments on space-based platforms (Skylab, Spacelab, etc.).
5. LIQUID TRANSFER DEMONSTRATION

As NASA scientists began planning the future of space travel, they considered the possibility of extended interaction between two spacecraft in orbit. As a result, this introduced the possibility of transferring materials, payloads, and fuel from one craft to the other. However, researchers had no prior experience regarding how the transfer of fluids in space might behave. In preparation for these situations, the liquid transfer experiment of Apollo 14 analyzed the behavior of a fluid as it was forced through a tube from one container to another. Through the experiment’s results, scientists hoped to prove that fluid transfer in microgravity behaved similarly to how it did on Earth. The experiment is discussed in this section.

In a simple workup, the liquid transfer demonstration consisted of a series of tests in which a fluid would be hand-pumped through clear tubing. The experiment would be recorded as it took place, and the results would be reported. The apparatus itself was comprised of a tank with two independent containers, two clear tubes, and a piston pump that could be used to transfer the fluid. Researchers decided that two experiments would be conducted. The first would use regular tanks while the second would experiment with three configurations of baffles in the tanks. These baffles, one being a curved web design and the second using a standpipe liner design, would be used to prevent fluid surging and improve flow patterns. The apparatus is shown in figure 8.

Scientists decided to use perfluorotributylamine (PFTBA) as the fluid for the experiment, which met the necessary safety requirements for the spacecraft. Furthermore, the PFTBA (as depicted in fig. 9) showed almost no static contact with the walls of the tank, a trait characterized by most common fuels. A small dye was added to the PFTBA for color, and it was shown to have no major effects on the physical properties of the fluid.

Scientists proposed that the unbaffled tanks would not be able to effectively carry out successful fluid transfer, and their hypothesis was mostly confirmed. Near the beginning of the experiment, a large bubble formed over the tank vent, causing problems in the transfer. Furthermore, it was found that very low amounts of the overall supply volume were successfully transferred to the receiver tank. The rest either remained in the supply tank or escaped through the tank’s vent.

The baffled tanks showed much more promising results. Three separate tests were conducted using varying baffle designs. The first test, which transmitted the fluid from the curved web to the standpipe liner tank at 0.83 cm$^3$/s, performed exceptionally well, transmitting all of the material with little loss. The second experiment transferred the PFTBA in the opposite direction at 0.67 cm$^3$/s, and it proved successful as well. The third test was another attempt at the first design but at a speed of 3.5 cm$^3$/s. Transfer still took place, but not as efficiently as it had at lower speeds. Scientists speculated that more efficient baffle designs could be developed to better handle the fluid transfer at higher rates.
Figure 8. Workup of the liquid transfer demonstration—standpipe liner baffle (left tank) and curved web baffle (right tank).

Figure 9. Chemical structure of PFTBA.
In light of these results, NASA researchers considered the experiment a resounding success, proving that fluids could effectively be transferred from one tank to the other in the presence of microgravity. Much like the other experiments in the Apollo program, these tests became foundational for future experiments.\textsuperscript{10} For example, Space Shuttle mission STS-53 featured the Fluid Acquisition and Resupply Experiment, which performed tasks similar to Apollo 14 in low-Earth orbit.
6. CONCLUSIONS

Although an often overlooked aspect of the landmark Apollo program, these experiments marked the birth of NASA’s microgravity science. Each demonstration provided crucial information to scientists seeking to develop more reliable materials on Earth for use in space. Furthermore, the experiments laid the groundwork for future experiments conducted in an attempt to further clarify the results and anomalies presented during Apollo. Experiments performed on Skylab, Spacelab, and the International Space Station all had their inspiration from these results.
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This Technical Memorandum (TM) discusses the microgravity experiments carried out during the later missions of the Apollo program. Microgravity experiments took place during the Apollo 14, 16, and 17 missions and consisted of four experiments in various materials processing concentrations with two of the four experiments taking place over the course of two missions. Experiments consist of composite casting, electrophoresis, heat flow and convection, and liquid transfer. This TM discusses the background, the workup, execution, and results of each experiment. In addition, the historical significance of each experiment to future applications/NASA programs is discussed.

materials processing, microgravity, Apollo 14, Apollo 16, Apollo 17, composite casting, electrophoresis, heat flow and convection, liquid transfer
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