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PLUTONIUM-238 RADIOISOTOPE FUELED
LIFE SUPPORT SYSTEM TEST

E. W. Johnson, J. R. Wysong, and R. E. Zielinski

AEC Research and Development REPORT

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MIAMISBURG, OHIO

OPERATED FOR

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R. E. Zielinski

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SUMMARY

The purpose of this investigation was to determine the feasibility of radioisotopes in Life Support Systems. In order to perform this evaluation, Mound Laboratory designed and fabricated radioisotope capsules for operation in the Vacuum Distillation Water Recovery Unit designed and fabricated by the General Electric Company, Missile and Space Division, for the Aerospace Medical Research Laboratory, United States Air Force. This report discusses the design and fabrication of the radioisotope capsules, the experimental tests conducted at Mound Laboratory, the analysis of the reclaimed water, and the post test examination and evaluation of the radioisotope capsules.

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INTRODUCTION

Weight is a major consideration for any space voyage as every pound of payload requires about 1500 pounds of thrust to attain orbit. A man on an extended space voyage requires from 9 - 12 pounds of water per day for food and drink and for wash water. A water recovery unit that could reprocess urine and used wash water would be a distinct advantage from a minimal weight viewpoint. An ideal unit would operate with a minimum of maintenance, have a high water-recovery efficiency, require only small amounts of electrical power, and have a minimum weight.

Water recovery units in manned space vehicles are judged by 1) chemical purity of recovered water, 2) bacteria content of recovered water, 3) amount of expendables required, 4) amount of electrical power required, 5) weight and volume requirements, 6) maintenance required, and 7) reliability. The radioisotope heat source would provide a constant, reliable heat source for distillation and pyrolysis with a great reduction in size and weight over electrical heaters. Electrical power would be used only for process controls and phase separation.

A thirty-day demonstration test of such a device was made. The systems developer and fabricator was the General Electric Company, Missile and Space Division, Valley Forge, Pennsylvania, under Contract Number AF33(615)-3308 with Aerospace Medical Research Laboratory, Wright Patterson Air Force Base, Ohio. Mound Laboratory, Miamisburg, Ohio (operated for the U. S. Atomic Energy Commission by Monsanto Research Corporation, Contract Number AT-33-1-GEN-53), was responsible for the design and fabrication of the radioisotope fueled capsules and the demonstration test.

The water recovery unit tested at Mound Laboratory and developed by General Electric Company, Life Support Engineering Operation, MOL Department had the following design characteristics:

- Water input rate: 15 pounds per day
- Waste liquid: Urine and/or wash water
- Possible Crew size: 2 men
- Recovery process: Vacuum distillation and vapor pyrolysis
- Liquid waste storage: 5 pounds
- Solid waste storage: 5 pounds
- Hot, potable-water storage: 5 pounds
- Cold, potable-water storage: 5 pounds
- Electrical power available: 28 VDC and 115 VAC 60 cycle
- Life: 5 Missions
- Water efficiency: 90% recovery (minimum).

The recovery unit was designed to operate in the following environment:

- Gravity: Normal earth gravity and design for zero "G"
- Temperature: 15.6° to 35°C
- Pressure: 380 to 760 mm Hg
- Humidity: 30 to 80% (relative)
- Atmosphere: Normal earth and design for 100% oxygen
- Acceleration: Design for 15 cps at 8 G in any plane.

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SYSTEM USED IN TEST

The system* used for this test consisted of a storage bottle for the collection of urine, an evaporator, a pyrolysis unit, a condenser, a condenser water pump, and an output collector. The urine was metered into the evaporator by a solenoid valve controlled by a liquid level sensor in the evaporator. The urine was heated to 50°C by 210 thermal watts of radioisotopic heat in the form of three 70-watt sources. The liquid boiled and the vapor passed through a phase separator. The phase separator assured that liquid was not carried with the vapor flow. The water vapor then proceeded through the pyrolysis unit where it was heated to 650°C in the presence of a ruthenium catalyst, the heat being supplied by a 70-watt radioisotope heat source. The pyrolysis unit catalytically reduced the impurities in the water vapor and they were removed through a vacuum vent in the condenser as the water vapor was condensed.

The vapor was condensed at approximately 15°C. A time-actuated pump drew the water from the condenser and stored it in a collection bottle. The solid waste from the urine was continually conveyed to the storage reservoir. Figure 1 is a diagram of the system.

Of the three main subsystems, collection, processing, and storage, only the processing section is described in detail.

COLLECTION

The collection subsystem collects and transports the urine to an intermediate storage tank. The waste liquid is then metered into the processing subsystem.

PROCESSING

Evaporator Fill Control

The evaporator is filled to maintain a certain liquid level by three conductivity probes in the evaporator and a voting control circuit. If for some reason the evaporator level falls below a safe level, an emergency mode is automatically initiated and water is metered into the evaporator from the potable water reservoirs.

Evaporator

The urine evaporator is a vessel containing ports for the admission and discharge, respectively, of urine and steam. Heat is provided by either three electrical heaters (Figure 2a) or three radioisotope heaters (2b) with each heat source integral with its own heat exchanger. A bladed rotor driven at 60 rpm by the evaporator motor and gear box through a Quad-Ring shaft seal provides the artificial gravitational field necessary to nucleate convective boiling in a zero gravity environment. The evaporator is constructed of Tufam - coated corrosion resistant 6061-T6 aluminum.

*System description furnished by General Electric Corporation.

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Figure 1. Flow Diagram of System Used In The Test.

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a

b

Figure 2. *Evaporator With Electrical Heaters (a) and Simulated Heat Source Being Positioned In Heat Exchanger (b).*

Evaporator Phase Separator

The evaporator phase separator consists of a closed impeller (similar to the impeller of a conventional centrifugal pump) driven at high speed. Liquid is separated from the water vapor by an impeller and returned to the evaporator.

Pyrolysis Unit

The pyrolysis unit consists of a counter-flow heat exchanger, a heat source (either electrical or radio-isotope), a catalyst, and a vacuum-insulated, radiation-shielded jacket (Figure 3). Steam entering the pyrolysis unit flows initially through the tubes of the counter-flow exchanger and is heated by the steam leaving the exchanger. Exiting from the exchanger, the steam flows through a tube containing the centrally-located heater surrounded by catalyst, the latter performing the additional function of assisting the heat transfer to the steam. Upon leaving the heater, the steam enters the catalyst bed completely surrounding the heater tube, turns and flows back through the catalyst bed to the counter-flow exchanger and out, giving up some of its heat to the incoming steam.

Early in the design phase of the program, it was believed that a steam temperature of as high as 1800°F would be required to decompose the ammonia with a platinum catalyst. Accordingly, Hastelloy X was chosen for the fabrication material, based upon its strength at that temperature and its resistance to corrosion by the common metallurgical test environments. Since no materials performance data were available for Hastelloy X in an ammoniacal environment, tests were run upon TIG welded lap joints (with and without Hastelloy X filler wire) and electron-beam butt-welded joints and tube-to-bulkhead joints. General Electric tests confirmed the selection of Hastelloy X for this application.

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Figure 3. *Installation of Heat Source Into Pyrolysis Unit. a) Inserting Heat Source Into Pyrolysis Unit. b) Securing The Catalyst and Heat Source. c) Inserting Pyrolysis Unit Into Vacuum Insulation Jacket.*

Condenser

The conically-shaped condenser is constructed of aluminum with an external cooling coil and foam silicone rubber insulation. The condenser is Tufram coated for corrosion resistance and non-wetting surfaces; however, a section of the internal surface near the apex is anodized to provide a wetting surface.

Condenser Level Control

The condenser level control consists of two capacitance probes and an electronics package. The capacitance between the probes varies significantly between the steam environment and when covered with water so that an electrical circuit is activated which operates the water pump.

Potable Water Pump

The pump draws the potable water from the apex of the condenser when the liquid water covers the level control. The pneumatically-powered, positive-displacement, piston pump draws the water through a check valve and into the cylinder of the pump. Gaseous oxygen then drives the piston forward forcing the water through a check valve and into the water storage reservoir. The oxygen is then vented to the cabin for breathing.

STORAGE

The storage subsystem stores, heats or cools, and dispenses the potable water. Storage is in three reservoirs with a capacity of five pounds of water each.

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The reservoirs contain elastic expulsion bladders for zero gravity operation. The water is dispensed from the reservoirs by the timed actuation of solenoid valves so that a known amount of water exits for a set time of valve opening.

RADIOISOTOPE CAPSULES

The heat for the system during the test was supplied by four similar radioisotope heat sources. Specifications are as follows:

Diameter:	1.227 in.
Length:	5.450 in.
Configuration:	Cylindrical with concave ends dished inward 0.125 in.
Thermal Output:	-70 watts
Fuel:	Plutonium-238 dioxide
Pressure Vessel Material:	Tantalum-10 wt % tungsten
Liner Material:	Haynes alloy No. 25
Outer Clad:	Platinum

CAPSULE DESIGN

The capsule was made of three concentric cylinders fabricated to fit at close interface tolerances to enhance the thermal conductivity (see Figures 4 and 5). The capsule was designed for a life of five years, even though the length of the test was 30 days, so that only minimal redesign would be necessary should the system be accepted as flight-qualified hardware. Four capsules of this design were used in the system.

The inner liner, made of Haynes alloy No. 25, is a 20-mil thick cylinder, 0.750-in. in diameter (O.D.) and was used to chemically contain the fuel (Figure 6). Thirty-mil caps were used to seal the liner.

Figure 4. Life Support Capsule Assembly.

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Figure 5. Photo of Capsule Showing Laminar Cylindrical Structure.

Figure 6. Life Support Capsule Subassemblies.

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The intermediate cylinder or strength member, made of Ta-10 wt % W, was designed to withstand the maximum pressure developed after five years. Since the wall thickness was greater than 0.1 times the outer diameter, the cylinder was considered a thick-walled cylinder. Thus, the tangential wall stress, being greater than the longitudinal or radial stresses, was the principal design factor. The tangential wall stress for a thick cylinder is found by the equation:

$$T_t = \frac{(r_o/r_i)^2 + 1}{(r_o/r_i)^2 - 1} P \quad (1)$$

where, T_t is the tangential wall stress,

r_o is the outside radius,

r_i is the inside radius, and

P is the internal pressure.

The stress on the caps was calculated by assuming they were circular flat plates with fixed edges and under a uniformly distributed load. The stress is maximum at the edge and is given by.

$$T_c = \frac{3}{4} \frac{r_c^2}{t_c^2} P \quad (2)$$

where: T_c is the cap stress,

r_c is the radius of the cap,

t_c is the thickness of the cap, and

P is the internal pressure

Equation 1 when rearranged is.

$$T_t = P \frac{\frac{t_u}{r_o} + \frac{1}{2} \frac{t_u^2}{r_o^2}}{\frac{t_w}{r_o} - \frac{1}{2} \frac{t_w^2}{r_o^2}} \quad (3)$$

where T_t , r_o and P are the same as above, and t_u is the wall thickness of the cylinder.

It can be shown that the wall stress (T_t) is a minimum when t_u/r_o is approximately 0.36 at any pressure. Thus, for optimum design,

$$T_t = 2.1 P \quad (4)$$

when the minimizing ratio $\frac{t_u}{r_o} = 0.36$ is used.

The creep of the capsule was the design criterion used for the cylinder. Design of capsules has been based on a maximum of two percent creep over the life of the capsule; that is, the capsule was designed so that the creep would not exceed two percent in the expected life of the capsule. This same criterion was used for the Life Support Capsule.

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The creep properties of Ta-10 wt % W are shown in Figure 7. The maximum creep corresponding to two percent creep at 1000°C for five years was determined to be 7.61×10^{-9} in./in./min using:

$$\text{Creep rate} = \text{elongation}/\text{time}$$

The wall stress corresponding to this creep rate is 43,000 psi for Ta-10 wt % W. Thus, the maximum allowable wall stress in order not to exceed this creep rate is 43,000 psi.

The pressure, due to alpha decay of plutonium-238, after five years, will be 3000 psi. This value was calculated from the expression

$$P = \frac{R}{V_v} N_o [1 - \exp(-\lambda t)] [(T_o - T_a) \exp(-\lambda t) + T_a] \quad (5)$$

where R is the universal gas constant

V_v is the void volume in cm^3

N_o is the number of moles of plutonium-238 at encapsulation

t is the time in years

$[1 - \exp(-\lambda t)]$ is the fraction of decayed plutonium-238 for t years,

T_o is the operating temperature, and

T_a is the ambient or atmospheric temperature.

Figure 7. Material Stress as a Function of Creep Rate for Ta-10 wt % W at 1000°C.

With these values for creep rate and pressure build-up and a safety factor of four, the wall stress was calculated as 7200 psi, the wall thickness 0.225 in., and the cap thickness 0.220 in. (Figure 6).

Dished-out end caps were used as they had withstood impact tests. The platinum outer clad (Figure 6) served only to protect the capsules from the hot, corrosive vapors in the pyrolysis unit. They were fabricated from 0.010-in. sheet which was rolled and seam welded to form a cylinder. The end caps for the clad were also of 0.010-in. platinum and were made to fit the profile of the dished-out endcaps of the strength member. These were electron beam welded to the platinum cylinder.

FABRICATION AND FUELING OF CAPSULES

The capsules were fueled with sintered plutonium-238 dioxide pellets, which were less than 0.710 in. in diameter and weighed about 28 grams per pellet. These pellets were calorimetered prior to encapsulation and microspheres (also pre-calorimetered) were added so that each capsule emitted just over 70 watts (i.e., 70.71, 71.49, 72.34, and 70.71 watts). The fueling and closure welds were done in an alpha glove box.

CAPSULE LINERS

The Haynes alloy No. 25 bottoms and caps were TIG welded at 20 amp, 15 sec/rev, with three seconds for beam fade-out. An argon gas flow of 15 cfm was used for the beam, and the arc gap was set at 35 mils. The bottoms were welded in a helium atmosphere and the caps were welded in an argon atmosphere. In both cases an aluminum cooling block was used to hold the liner and to provide a heat sink. The liners protruded 78 mils from the cylindrical cooling block.

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Photographs of sample liner bottom welds are shown in Figures 8 and 9. The same parameters were used for both the sample welds and the bottoms. Pieces of the same material and same geometrical shape and size as the actual piece were used for the sample welds.

Sample welds were also made before and after the closure welds of the fueled liners, but since these were made in a glove box, photographs could not be made. These welds were examined visually to insure good closure.

The liners, after the closure welds were completed, were decontaminated by scrubbing. The wipes on the liners were less than 500 cts/min which is acceptable by the Health Physics Section of Mound Laboratory.

Each of the liners were helium leak checked on a Veeco model MS9AB leak detector after the bottoms were welded on. The standard maximum allowable leak was assumed to be 1×10^{-7} cc/sec. The calibrated leak, which was used to standardize the instrument, was 4.5×10^{-8} cc/sec. All of the capsule components checked less than the standard leak.

Leak checks were made on the closure welds (after decontamination) for two of the liners; two could not be checked because of problems with the leak detector. However, decontamination of the liners could not be accomplished to less than 500 cts/min if the weld were leaking. The liners that were checked fell below the maximum allowable rate of 1×10^{-7} cc/sec.

Figure 8. TIG Weld Sample Before Welding Liner Bottom.

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Figure 9. TIG Weld Sample After Welding Liner Bottom.

STRENGTH MEMBERS

The bottom caps of the strength members were electron beam welded before the liners were inserted for final closure. The parameters used were 145 kV, 13.5 ma, and 21.5 sec/rev. Figure 10 shows a photograph of a weld sample of Ta-10 wt % W made on a solid bar 2 in. in diameter as no sample piece similar in geometrical shape and size was available. Penetration of the welds was approximately 290 mils, sufficient to penetrate the cap and 25% deeper into the wall (the amount necessary to make a good weld).

One weld was sufficient to encapsulate the fuel for the life of the capsules from a strength standpoint. These welds (the bottoms of the strength members) leak checked to better than 4.5×10^{-8} cc/sec.

The liners were rechecked for alpha contamination after leak checking and before insertion into the strength members (They were less than 500 cts/min).

The closure welds for the strength members were electron beam welded using the same parameters as for the bottoms. The four capsules were again leak checked and the rate fell within the allowable helium leak rate.

Figure 10. Weld Sample of Ta-10 wt % W.

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PLATINUM CLAD

The platinum cladding was welded in a similar manner as the other two components. The bottoms were electron beam welded first and then leak checked. The capsules were inserted into the liners and the closure welds made. Figure 11 shows a photograph of the seam weld. Since the cladding was only for a leak tight container, the 50% penetration was more than enough. Figure 12 shows the weld of the cap to the tube. After the bottom welds and the closure welds were made the completed capsules were leak checked and all the capsules proved to be acceptable.

Figure 11. Platinum Seam Weld.

Figure 12. Weld of Cap to the Tube.

ELECTRICAL TESTS

The Vacuum Distillation Water Recovery Unit was tested for five days to determine whether the unit was operating correctly before the radioisotopes were installed. The unit operated as expected with a few minor problems that were soon corrected. Electrical heaters were used in place of the radioisotopes in order to check recording equipment and general performance of the unit.

Distilled water was used at the initiation of the test in order for the unit to heat before urine was entered. After the temperatures of the unit stabilized (1.5 hr), the first four liters of urine were added to the unit. During the remainder of the test approximately eight liters of urine were added to the unit in a 24-hr period with an output of approximately eight liters.

The unit ran exceptionally well with temperatures and pressures remaining fairly constant until the vacuum pump filled with water causing a rise in condenser pressure. This resulted in a drop of temperature in the steam coming out of the pyrolysis unit and a strong ammonia odor in the output water collected. Whenever the situation was corrected, it happened again twelve hours later. This was occurring with such frequency that a cold trap was installed into the system. After the installation of the cold trap, the system became stabilized.

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Two factors were noted concerning the water that was collected. Whenever the temperature of the steam out of the pyrolysis unit dropped below 150°C and whenever the pyrolysis temperature dropped below 600°C, the collected water had a pH reading greater than 6.8 and an ammonia odor was evident.

The test was successful in checking out the monitoring system and the test unit itself. Problems that were encountered were ones that had to do with mechanical aides to the unit and were not attributed to the unit. These problems once corrected resulted in a stabilization of the data which remained fairly constant during the test

RADIOISOTOPE FUELED TEST

TEST RUN

The radioisotopes were loaded into the unit on November 15, 1966. During the warm-up period with distilled water input, there was a noticeable change in the evaporator temperature after 6 1/2 hr of operation. Crossed leads into the level sensor were causing the liquid level in the evaporator to remain low. This was corrected and the unit again functioned properly. After the pyrolysis temperature had reached 635°C and the steam out of the pyrolysis unit had reached 148°C, the first four liters of urine were introduced into the unit.

For the next three days, the unit ran consistently with the pyrolysis temperature varying from 635°C to 670°C and the steam out of the pyrolysis unit varying between 150°C to 170°C. These temperatures were higher than during the electrical test but this was due, in effect, to the greater efficiency and more even heat of the radioisotopes. The water output during this time was approximately eight liters which was above the anticipated output. The pH of the output varied between 4 and 5, which was considered excellent.

However, on November 19, the unit became very erratic, the condenser pressure reached a high of 65 torr, no water was pumped out, and output water backed up into the vacuum line. The pH reading at this time was very high, and from the odor, it was clearly evident that the output was contaminated with organic materials. A preliminary analysis indicated a malfunction in the pumping mechanism of the unit. The piston of the pump was repaired. Operation was resumed but the pH reading still remained high. This high pH indicated that the catalyst in the condenser was saturated and poisoned during the output back-up. When the unit was dismantled, it was found that the catalyst was saturated with solid waste and also that the level sensors of the evaporator were blocked.

On November 20, the system was restarted after all repairs were completed. Distilled water was again run through the unit to flush it. The next day the unit again performed erratically with the condenser pressure oscillating until it reached a high of 38 torr. At this time it was found that (1) the pump oil contained water, (2) the inlet solenoid valves were clogged, and (3) the solenoid valve downstream of the phase separator failed to seat allowing an air blast to enter the evaporator whenever the level sensor required water. All problems were corrected while the unit was running. Furthermore, it was decided that the condensate pump would be manually operated because of electronic failures in the pumping controls. Again the unit resumed constant running with both the vapor temperature in the pyrolysis unit and the pyrolysis temperature remaining fairly constant. The pH reading varied from 4.8 to 5.8 (still a safe reading).

The pH reading on November 23, was 8. A system check showed everything operating normally. The pH reading varied from 8 to 9 and an attempt to lower it by increasing the pyrolysis temperature was unsuccessful. During this period, the temperature of the pyrolysis unit never rose above 480°C. The temperature of the steam out of the pyrolysis unit varied from 130° to 393°C.

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On November 29, the condenser's pumping mechanism failed to put out any water. The failure of the water vapor to condense resulted in a high pyrolysis temperature. This was caused by a low level in the evaporator effecting a temperature rise. The unit was cooled by placing dry ice atop the evaporator. This cooled it sufficiently.

On November 30, the unit was shut down and dismantled. Inspection of the component parts indicated a blockage of the inlet to the pyrolysis unit by solids. This was probably caused by a large amount of water flowing into the evaporator and boiling with such force that it carried solids into the inlet of the pyrolysis unit. The catalyst was slightly contaminated with materials from the evaporator. The pyrolysis unit heat source was buckled along the seam weld of the platinum and there was a slight buckle of one of the end caps.

The unit was reassembled and started on November 31. A mixture of water and alcohol was fed into the unit to sterilize the lines. On December 1, urine was again fed into the unit after it had reached operating temperatures. The unit was stabilized and the pH varied between 4 and 5. Within four hours a back pressure developed caused by a blocked check valve in the cold water storage tanks of the unit. The valve was removed and operations returned to normal. The unit ran consistently with a pH varying between 4 and 5 and pressure varying from 4 to 11 torr.

On December 2, a leak developed in the rotary shaft seal which resulted in a pressure rise to 19 torr. The leak was sealed by first applying a thin layer of grease on the seal and then a coat of silicone rubber sealant to act as a face seal. This effected an immediate pressure drop to 9 torr.

The unit again functioned properly. Slight variations in pressure were noticed due to condensation buildup in the the cold trap. The pH varied between 4 and 5 until December 3, when it suddenly jumped to 8. On December 4, the pH decreased to 5. No cause was determined for the rise in pH value

On December 4, the pressure rose to 11 torr, increased to 14 torr, then dropped to 7 torr. Cause of this was attributed to unregulated manual feed (too much urine suddenly introduced into the evaporator). This caused a large amount of concentrated urine to be put through the pyrolysis unit. This condition finally cleared itself with a concurrent pressure reduction to 5 or 6 torr.

On December 5, the pH reading was between 7 and 8. Again this can be attributed to unregulated manual feed in filling the evaporator unit. After the urine was forced out of the pyrolysis unit, normal operation resulted and the pH was 5.2.

However after 11 hr, the pH was up to 6.8. Again manual feed was the cause of the malfunction of the unit. The pH rose to above 8. The evaporator was flushed with water to reduce the pH of the output. During the next several days of operation, the pH rose and fell several times with an accompanying rise and fall in temperature. On one occasion there was a rise in condenser pressure from 4-11 torr to 62 torr. This was a direct result of unregulated manual feed in which the level sensor of the evaporator unit was not shut off, thus causing urine to pour into the evaporator and flood through the pyrolysis unit and contaminate the catalyst.

On December 8, the coolant pump failed and the coolant lines were connected directly to a water faucet. This innovation was reliable and was used until the pump was overhauled.

The unit was shut down for repairs on December 9, because the coolant pump was not functioning, the pH was above 10, the evaporator was flooded, and the catalyst was contaminated. The pyrolysis unit was dismantled, the contaminated catalyst replaced, the evaporator was flushed clean, and the coolant pump overhauled. The capsule that was positioned in the pyrolysis unit had a slight crack in the end cap. This was judged minor and the capsule was reinstalled. The unit was placed in operation with a mixture of alcohol

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and water cycled through it to flush it out. During this flushing operation, the coolant pump again failed and it was decided to run the coolant from the water faucet as this would not disrupt the system's operation and would maintain the coolant flow. On December 10, the pyrolysis vacuum was turned off for repair work. The input alcohol-water mixture was stopped and the unit allowed to stabilize. The inlet solenoid valves and lines were cleaned. A face seal was installed on the stirrer shaft and this was put back into operation. The repairs were completed and the system was allowed to stabilize with water cycling through it before urine was introduced into the system.

The temperatures and pressure remained constant and the pH varied from 7 to 8 until December 13, when the pH suddenly increased to 10. This was attributed to unregulated manual feed.

An attempt to stabilize and optimize the system by putting a fan under the evaporator to cool it did not have the desired effect, so the system was shut down on December 13, for repairs. At this time a clad failure was noted on the end cap of the pyrolysis capsule.

On December 14, the system was cleaned. The evaporator was disassembled and cleaned and a phase separator of stainless steel mesh was installed. The pyrolysis unit was contaminated with solids at the inlet and in the heat exchanger. This unit was disassembled and cleaned and new catalyst installed. The pyrolysis capsule with the crack in the clad was inserted into the evaporator and an evaporator capsule placed in the pyrolysis unit. The unit was re-assembled with distilled water cycling through the system. During the warm-up period, a cracked gage on the high vacuum line was discovered. Repairs could not be made and the high vacuum gage was removed from the system. Also, the inlet solenoid valves and lines were apparently blocked, so a close check had to be kept on the system and the level in the evaporator had to be controlled manually to maintain a proper level.

On December 15, urine was added to the system after the pyrolysis temperature exceeded 600°C. The unit again resumed normal operation. The pH was 6 and remained fairly constant.

On December 15, the stirring motor in the evaporator froze; the unit was now running without a stirrer. Manual control of the liquid level in the evaporator proved difficult and on numerous occasions the level became quite low and the evaporator was filled too quickly. The pH rose during this time and hit a peak value of 9.3. The system appeared to slowly correct itself as the pH began decreasing. An air bleed was added in the evaporator in an attempt to speed up the corrective measures.

The air bleed was cut down repeatedly and the values of temperature and pH (at 5) remained steady. On December 16, the pH rose to 7 and the bleed valve was again opened. This produced no change and the pH varied from 5 to 7 until test termination.

On December 17, the system was shut down and dismantled.

COMMENTS

Probably the most acute problem in the present design of the system and the one which did the most to hamper a successful continuous run of the test was the unreliability of the evaporator fill control. The evaporator should be filled to maintain a certain level. The three conductivity probes in the evaporator did not function properly. They tended to malfunction on a time cycle of about eight days of running. Since they did malfunction, it was necessary to resort to manual filling of the evaporator. When the evaporator was filled too quickly, a carry-over of liquid waste into the pyrolysis unit contaminated the ruthenium catalyst. Whenever this occurred, the output would be of very poor quality. Thus, more accurate level controls in the evaporator are advisable.

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Also, the phase separator in the evaporator can be improved. It could not successfully combat the mechanical malfunction of the level sensors if the evaporator overfilled. Stainless steel mesh was added to the system too late in the test run to determine whether or not it contributed favorably to the phase separation step.

Apparent in the analysis reports of the minerals present in the output water was a relatively high amount of aluminum as compared with the other minerals present. This was present from the initiation of the test until the time the aluminum condenser pump was replaced in the system by a stainless steel pump.

Abrasion of the interior was visibly evident in the dismantled pump, indicating that urine attacked the aluminum pump to an appreciable extent. Therefore, the stainless steel pump should be incorporated as a permanent part of the system.

The potable water pump controls failed during the test and a timing activator was installed into the unit. The activator is of a poor design because the pump now operates on one set cycle no matter how much water is in the condenser. A control influenced by the level of the condenser would be much better. The original circuit design could be improved and would be better than the timing activator now attached to the unit.

The ruthenium catalyst did not appear to minimize metal "boil-off" sufficiently, although it was modified for the test run to provide significantly more surface area and vapor contact time. Perhaps another modification is in order.

INTEGRITY OF CAPSULES

Three of the capsules were positioned in the evaporator in aluminum holders. They provided a total of about 210 thermal watts. The other capsule was positioned in the pyrolysis unit.

The system was shut down four times during the test.

- (1) On November 19, the system was shut down for repair and disassembled. The appearance of the four capsules had not changed.
- (2) On November 31, the system was again shut down and disassembled. This time only the pyrolysis unit was disassembled completely. There was a noticeable buckle along the seam weld of the platinum clad. The capsule end cap located at the top of the pyrolysis unit had also buckled. Upon closer examination no breaks in the cap were noted and the capsule was put back into the pyrolysis unit in the same position.
- (3) On December 9, another shutdown occurred when the pyrolysis unit was completely disassembled. Again the capsule was examined and the only change was a black line visible along the buckle on the end cap. There was no change in the size of the buckle along the seam or end cap. The end cap was examined under a microscope and it was ascertained that the spot was either a piece of Hastelloy X imbedded in the cap or a slight crack with a very minute depth. Therefore, it was judged to be safe and was reinstalled into the pyrolysis unit.
- (4) On December 13, the system was disassembled for repairs and modification. At this time a cladding failure was discovered on the end cap of the capsule positioned in the pyrolysis unit. The failure seemed to originate at the beginning of the buckle. Microscopic examination showed that the failure began at the slight crack that was discovered on the previous

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shutdown. This capsule was replaced with one from the evaporator and the unit reassembled. The capsules in the evaporator showed no external changes whatever.

- (5) On December 17, the unit was disassembled and capsules examined. The replacement capsule in the pyrolysis unit had a buckle in the end cap positioned at the inlet-exit end of the pyrolysis unit. The capsules in the evaporator showed no physical changes. The capsule that failed and was placed in the evaporator showed no more changes.

POST TEST CAPSULE EVALUATION

On December 13, the Life Support Vacuum Distillation Water Recovery Unit was disassembled. Upon disassembly of the pyrolysis unit, the capsule enclosed was observed to have a split along the platinum liner. The capsule was examined and photographed to determine the cause of the liner failure.

Figure 13 shows the top edge or end of the capsule as it was positioned in the pyrolysis unit. This photograph shows several indentations but none of these penetrate the clad material completely. The pyrolysis unit had a maximum temperature of approximately 800°-850°C. The surface temperature of the capsule was estimated to be 100°-150°C more than that of the thermocouple reading (670°C). (The temperature of the capsule is not definite because the pyrolysis thermocouple was not at the surface of the capsule but extended only to within 3/4 in of the capsule.) This high temperature caused the platinum to anneal and become susceptible to slight external abrasions and scratches.

Figure 14 shows the opposite end of the capsule and shows the split quite clearly. Figure 15 shows a scale in order to make a size comparison of the previous two photographs.

Figure 13. Capsule End at Exit of Pyrolysis Unit. Figure 14. Capsule End at Entrance of Pyrolysis Unit Showing Split in Platinum.

Figure 15. Photograph of Scale for Comparison Purposes (inches).

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Figures 16 through 21 show photographs of the pyrolysis capsule container. Figure 16 is a photograph of the top supporting member and Figure 17 is for a size comparison. No defects are apparent on this member. Figure 18 shows the bottom of the container where the split capsule was supported. A portion of the support was melted away because of a short during the electrical testing and the void can be clearly seen in these photographs. Figure 19 shows the same surface from a different view and the void is not as apparent in the photographs. Apparently the melted material created a ridge which resulted in an uneven distribution of pressure on the radioisotope capsule.

Figure 16. Top Support of Pyrolysis Unit.

Figure 17. Scale for Size Comparisons.

Figure 18. Bottom of Pyrolysis Capsule Container.

Figure 19. Bottom of Pyrolysis Capsule Container.

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Figure 20. Capsule 1-2.

Figure 21. End View of Capsule 1-2.

During the course of the experiment, the system (including the pyrolysis unit) was disassembled several times. It is felt that repeated disassembly caused the platinum to wear thin where it met the support member. The improper mating of the support and capsule was caused by a dimensional difference or a ridge in the support member. Evidence shows that the platinum in the area of the split was worn very thin from rubbing and/or stretching, the latter being more probable as the capsule was clamped in place at the ends and the mating surfaces of the support and capsule were compressed. Subsequent disassemblies and assemblies finally caused the platinum to fail.

The capsule was placed in the evaporator unit and one of the evaporator capsules was placed in the pyrolysis unit for the balance of the test.

After completion of the test, the platinum clad was removed from all of the capsules and the inner capsule surfaces were examined. Capsules 1-2 and 5-6 remained in the evaporator (low temperatures) for the duration of the test. Examination of the surfaces showed no apparent attack or corrosion (Figures 20 to 25).

Capsule 7-8 (Figure 26) is the capsule in which the platinum split. Examination of the bottom edge of the capsule showed where the hot vapors corroded the capsule. Figures 27 and 28 show this end more clearly. Figure 29 shows the opposite end of this capsule (the end opposite the split) and no corrosion is apparent on this end of the capsule. The water vapors at high temperatures are believed to be the cause of the corrosion at the one end.

Figure 30 shows the capsule which replaced the split capsule. Figures 31 and 32 show the ends of this capsule. The entire surface seemed to be oxidized. After inserting this capsule an air flow was admitted to the pyrolysis chamber. This had not been done before. Platinum is somewhat porous to oxygen and it is assumed that the oxygen and the high temperature caused the oxidation, since the capsule with the split showed corrosion only at the split end and not over its entirety.

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Figure 22. Opposite End of Capsule 1-2.

Figure 23. Capsule 5-6.

Figure 24. End View of Capsule 5-6.

Figure 25. Opposite End of Capsule 5-6.

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Figure 26. Capsule 3-4.

Figure 27. Corroded End of Capsule 3-4 Where Platinum Clad Had Split.

Figure 28. Corroded End of Capsule 3-4 Where Platinum Clad Had Split.

Figure 29. Opposite End of Capsule 3-4.

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Figure 30 Capsule 7-8.

Figure 31. End View of Capsule 7-8.

STRENGTH MEMBER

The four radioisotope capsules employed in the test were placed in an alpha glove box prior to extraction of the fuel from the capsules. The Ta-10 wt % W strength members were examined visually during the fuel removal process.

Capsule 1-2 which was situated in the evaporator during the entire test period showed no discoloration or any other physical change on the exterior of the strength member. Upon removal of the inner liner, the interior of the strength member was also visually examined and no changes were evident.

Capsule 3-4 was located in the evaporator until the last four days of the test run during which time it was positioned in the pyrolysis unit. At the completion of the test, the capsule was removed from the pyrolysis unit and a buckle was noticed in the platinum clad. The entire exterior surface of the strength member was discolored as if it were oxidized. When the inner liner was removed, the interior surface was visually examined and no changes were noted. The oxidation appeared to be only a surface layer on the exterior of the strength member, with no penetration at all. X-ray diffraction analysis of scrapings of the exterior surface indicated the presence of α and β Ta_2O_5 and tantalum metal.

Figure 32. Opposite End of Capsule 7-8.

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As this capsule was in the evaporator for the initial 29 days of the test and the capsules that were in the evaporator during the entire test period showed no change, it is assumed that discoloration occurred during the four days the capsule was located in the pyrolysis unit. The capsule originally in the pyrolysis unit was not affected by total surface discoloration even though it had a crack in the platinum clad for some time. The only variable present when capsule 3-4 was in the pyrolysis unit was an air bleed that was attached to the system for 15 hr on the last day of the test.

The air bleed was used to set up an air flow through the pyrolysis unit. The air flow varied from 2 to 5 torr-liters/sec at various times during this period.

At the completion of the test, capsule 3-4 was removed from the unit; the platinum clad was not cracked. Therefore, it seems plausible that oxygen permeated the platinum and formed the coating of oxide on the surface of the strength member. The oxide was an evenly distributed coating and this lends credibility to the fact that the platinum clad did not fail. If that clad had failed, the oxide would tend to be distributed in a greater amount around the point of failure.

Capsule 5-6 was positioned in the evaporator during the entire test. The strength members showed no discoloration or any other physical changes on the exterior. The interior was also unaffected.

Capsule 7-8 was located in the pyrolysis unit initially. On the 29th day of the test, it was transferred to the evaporator. When the test was completed, the platinum clad was removed and it was noted that corrosion had affected the number 8 end cap of the strength member, the end cap nearest the failure of the platinum clad. There were no other physical changes of the external surface of the strength member. The interior was unchanged. The corroded end cap was sectioned for metallographic studies. These were localized attacks on the outer perimeter but not on the interior surface. The corrosion penetrated to approximately 0.06 in. at its deepest point. This is only 28.1% of the thickness of the end cap and, therefore, had no effect on the integrity of the capsule. The area of attack seemed to be circular and it appeared to be spread out from the point where the crack had developed in the platinum end cap. A photomicrograph (Figure 33) shows the deepest point of attack and the irregular type of attack.

Scrapings of the corrosion were also taken for x-ray diffraction analysis. The results of the analysis of the corrosion of the end cap indicate the presence of α and β tantalum oxide (Ta_2O_5) and tantalum metal. The corrosion centered around the crack in the end cap. This crack was large enough (approximately 0.25 in.) to admit the vapor and liquid that was present in the pyrolysis unit at a nominal rate. The oxygen present in the environment then attacked the surface of the strength-member end cap nearest the crack in the platinum.

The rate at which the oxidation occurred when there existed a known crack lends credence to the fact that there were no cracks in the platinum clad of the number 3-4 capsule.

The electron beam welds of all the end caps of the strength members were pitted. This was a result of the welding procedure and obviously did not compromise the integrity of the strength members because wipes taken on the surface of the capsule strength members showed no radiation count. No corrosion of the welds was observed.

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INNER LINER

The inner liner of capsule 1-2 showed no physical change on either its interior or exterior surface. The inner liners of capsules 3-4, 5-6 and 7-8 appeared no different. The Haynes alloy No. 25 inner liner came out of the Ta-10 wt % W strength member very easily, indicating that little or no creep occurred to the inner liner.

A section of the inner liner end cap number 5 of capsule 5-6 was taken for metallographic study along with a center section of the same inner liner. The outside of the Haynes alloy No. 25, which was in contact with the Ta-10 wt % W strength members, showed no physical changes (Figures 34 and 35). There was, however, a minute degree of localized attack at the fuel-metal interface. The degree of attack did not appear to affect the integrity of the alloy (Figures 36 and 37). The weld metal was visible after etching and seemed to resist attack. The depth of corrosion was approximately 0.40% of the thickness of the wall. This corrosion is the result of plutonium-238 oxide powder reacting with the Haynes alloy No. 25 surface.

Figure 33. Exterior Surface of No. 8 End Cap Showing Depth of Corrosion. 250X.

Figure 34. Exterior Surface of Mid-Section of Inner Liner 5-6. 250X.

Figure 35. Exterior Surface of Inner Liner 5-6. 500X.

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Figure 36. Interior Surface of Mid-Section of Inner Liner 5-6. 250X.

Figure 37. Interior Surface of Mid-Section of Inner Liner 5-6. 500X.

The photomicrographs of the interior surface of the number 5 end cap showed a small degree of corrosion (Figures 38 and 39) where the plutonium-238 oxide powder had attacked the Haynes alloy No. 25 end cap. The exterior surface was unaffected.

Photomicrographs of the Haynes alloy No. 25 number 8 end cap were also taken. Again, the interior surface was affected by a small degree of corrosion (Figures 40 and 41). The exterior surface (Figure 42) was unchanged even though it was next to a corroded Ta-10 wt % W end cap.

Figure 38. Interior Surface of Inner Liner End Cap No. 5. 250X.

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Figure 39. Interior Surface of Inner Liner End Cap No. 5, 500X.

Figure 40. Interior Surface of Inner Liner End Cap No. 8 (Unetched Surface), 250X.

Figure 41. Interior Surface of Inner Liner End Cap No. 8 (Etched Surface), 250X.

Figure 42. Exterior Surface of Inner Liner End Cap No. 8, 250X.

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FUEL

The caps of the inner liners were removed and the fuel taken out of the inner liner. There was no physical change of the fuel. The sintered plutonium-238 oxide pellets held together and remained unchanged from their appearance when the capsules were fueled.

ANALYSIS OF WATER OUTPUT

RADIOLOGICALLY

Radiologically the output water of the system had no contamination whatsoever, proving the design integrity of the alpha-emitting heat source. Records of test results and the procedure for analysis are included in the appendix.

CHEMICALLY

Seven water samples taken during the radioisotope test run showed mentionable concentrations of aluminum, copper, and zinc. Aluminum was the major constituent in all the samples. Originally, it was thought that the aluminum concentration was a carry-over from the aluminum cylinder in the condenser pump. However, when this was replaced by a stainless steel cylinder, the aluminum concentration was still rather high (10^3 ppb). The only other reason for the high aluminum concentration would be corrosion of the interior of the evaporator. When the evaporator unit was disassembled, the interior coated with Tufram showed corrosion with the bladed rotor shaft having a large corroded area.

After the completion of the test, the condenser was disassembled and a deposit found on the bottom was analyzed. Chemical analysis, x-ray diffraction analysis, and emission spectroscopy showed silicon present in the deposit. Since silicon is a constituent of aluminum 60-61, it was concluded that the Tufram coating on the interior of the evaporator did not resist corrosion as well as anticipated and that the urine penetrated through this coating and attacked the aluminum, resulting in high aluminum concentration in the output.

The copper and zinc concentrations were relatively high (approximately 2×10^2 ppb) during the initial days of the test. However, when a copper line and brass valve leading from the condenser to the collection bottle were removed, the zinc concentration dropped to approximately 35 ppb and the copper dropped below 10 ppb. Therefore, it can be concluded that there was carry-over of copper and zinc from this line. Data are presented in the appendix.

BACTERIOLOGICALLY

Two samples were collected during the test run specifically for bacteria analysis. The first of these showed a relatively high bacteria count (4700/ml total bacteria) while the second sample showed no count. However, the first sample was not analyzed until approximately three hours after it was delivered. Thus, the first analysis could not be considered very accurate because there would have been enough time for bacteria to multiply before the analysis was made. All of the collected samples, however, were analyzed by the agar pour plate technique and showed a minute count per milliliter.

Tested for organic carry-over, the samples indicated that whenever a flooding of the evaporator occurred, the organic materials built up in the system and were released during a later phase of recovery. The sample tested immediately after a flooding condition would show small amounts of ammonia, urea and total carbon. The very next sample would show a rise in all three indicating an organic buildup being released. Data are presented in the appendix.

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EXIT GAS ANALYSIS

Intermittently, a sample of the exit gas from the condenser was withdrawn. An evacuated 10-cc bulb was attached to the low vacuum line by a 10/30 joint. By opening the stopcock, a representative exit gas specimen was introduced into the glass bulb. The total pressure of this gas was about 10 torr. The specimen was analyzed on a CEC Model 21-620 mass spectrometer. Data obtained on the 10 samples are presented in the Appendix.

No ammonia was observed in the analyses. It was thought that all ammonia was absorbed on the stainless steel inlet system since the apparatus is not equipped with a heated inlet system.

The $\text{CO}_2/\text{C}_3\text{H}_8$ differentiation was done by comparison of peak area of mass 44 (CO_2^+ and C_3H_8^+) to mass 43 (C_3H_7^+ , a fragment formed from parent C_3H_8 by electron impact).

There are wide variations in the concentration of components. Most interesting are the hydrocarbon, hydrogen, and carbon dioxide. High values for hydrocarbons could mean ineffectual catalysis action. Conversely, the high carbon dioxide/hydrogen ratios could mean oxidation of the hydrocarbon by water vapor. From potability aspects, the second effect produces the desired sort of harmless reaction products.

CONCLUSIONS

All of the considerations described in this report indicate that radioisotope capsules are well qualified for use in Life Support Systems.

Flight qualification of the capsules would demand only slight design modifications, one of which would be a better clad material for high temperature use. However, the problems encountered with the clad were caused by the capsule support member and not inherent in the clad material. A better method of containing the capsule in the unit would alleviate this problem.

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APPENDIX I

SAMPLES ANALYZED FOR LIFE SUPPORT PROGRAM

Procedure.

The samples were analyzed for alpha radiation by liquid scintillation¹ counting. One milliliter of urine or distillate was added to vials of a scintillation solution consisting of

- 1 PPO , 2,5-Diphenyloxazole, seven grams per liter
- 2 POPOP - 1,4-bis-2 (5 phenyloxazoly) benzene
- 3 Naphthalene - 100 grams per liter
- 4 P-Dioxane - one liter

A background count was taken for each vial prior to the addition of the sample. The value listed under sample is the deviation from this value. Because of the normal variation of single background count determinations the average value of 26 cts/min is considered a much more realistic number. The range of values of background counts was 21 to 32 cts/min. Only two samples out of 89 determinations exceeded the upper limit and then only by one ct/min. In summary, no detectable amount of alpha activity was observed in any of the samples.

¹The efficiency for counting alpha radiation by liquid scintillation method is 100%

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Table I

ALPHA RADIATION IN URINE SAMPLES

Date of Analysis	Sample No.	Background (cts/min)	Total (cts/min)	Sample (cts/min)	Date of Use
Electrical Test					
11/3/66	1	27	29	+2	11/3/66
	2	27	26	-1	11/3/66
	3	27	23	-4	11/4/66
	4	27	25	-2	11/4/66
11/4/66	5	26	29	+3	11/5/66
	6	25	28	+3	11/5/66
11/5/66	7	24	28	+4	11/6/66
	8	23	26	+3	11/6/66
11/8/66	9	25	27	+2	11/7/66
	10	25	25	0	11/7/66
Radioisotope Test					
11/16/66	11	25	26	+1	Not used
	12	22	23	+1	11/16/66
	13	27	23	-4	11/17/66
	14	25	28	+3	11/17/66
11/17/66	15	27	27	0	11/18/66
	16	28	25	-3	11/18/66
11/18/66	17	27	27	0	11/21/66
	18	28	29	+1	11/21/66
	19	28	30	+2	11/22/66
	20	28	25	-3	11/23/66
	21	28	27	-1	
11/22/66	22	25	26	+1	11/23/66
	23	26	27	+1	11/24/66
11/28/66	24	28	28	0	11/25/66
	25	25	25	0	11/25/66
	26	24	26	+2	11/26/66
	27	26	30	+4	11/26/66
	28	25	28	+3	11/27/66
	29	23	26	+3	11/28/66
	30	23	28	+5	11/29/66
11/29/66	31	25	25	0	12/1/66
12/6/66	32	26	26	0	12/2/66
	33	27	28	+1	12/2/66
	34	27	24	-3	12/2/66
	35	27	22	-5	12/3/66
	36	24	21	+3	12/3/66

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Table I cont.

ALPHA RADIATION IN URINE SAMPLES

Date of Analysis	Sample No.	Background (cts/min)	Total (cts/min)	Sample (cts/min)	Date of Use
12 6 66	37	24	25	+1	12 4 66
	38	24	24	0	12 5 66
	39	26	24	+2	12 6 66
	40	26	26	0	12 6 66
	41	24	24	0	12 7 66
	42	24	25	+1	12 7 66
12 8 66	43	27	28	+1	12 8 66
	44	25	33	+8	12 10 66
	45	25	28	+3	12 10 66
12 9 66	46	28	28	0	12 11 66
12 12 66	47	28	28	0	12 11 66
	48	29	31	+2	12 12 66
	49	27	29	-2	12 12 66
	50	32	33	+1	
	51	30	30	0	12 13 66
	52	29	28	+1	12 13 66
12 12 66	53	28	30	+2	12 14 66
	54	29	31	+2	12 14 66
Total Average		26.2	26.9	+0.7	

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Table 2

ALPHA RADIATION IN DISTILLATE SAMPLES

Date Drawn	Date Analyzed	Sample No.	Background (cts/min)	Total (cts/min)	Sample (cts/min)
Electrical Test					
11/4/66	11 8 66	1	24	23	-1
11/6 66		3	25	28	+3
11/7 66	11 9 66	4	25	28	+3
11/8 66		5	25	28	+3
Radioisotope Test					
11/16 66	11 16 66	6	24	24	0
11/17/66	11 17 66	7	27	26	-1
		8	27	24	-3
11 18 66		9	26	24	-2
11/21 66	11 21 66	10	24	23	-1
11/22/66	11 23 66	11	25	26	+1
11/23 66		12	25	23	2
		12a	25	24	-1
11 24 66	11 28 66	13	21	30	+9
11/25/66		14	23	26	+3
		15	26	28	+2
11/26 66		16	21	26	+5
11/27/66		17	24	25	+1
11/28/66	11/29/66	18	26	25	-1
11/30/66	12 1/66	16a	27	27	0
12/3/66	12/6/66	19	27	27	0
12/4/66		20	25	27	+2
12/5/66		21	26	26	0
12/6/66		22	27	26	-1
12/6/66	12/9/66	23	28	28	0
12/7/66		24	29	32	+3
12/8/66		25	25	27	+2
12/11/66		26	28	25	-3
12/12 66		27	30	30	0
12/13/66	12/15/66	28	30	31	+1
12/15/66		29	26	24	-2
12/17/66	12/17/66	30	27	28	+1
12/15 66		30a	26	26	0
12/15 66		31	27	29	+2
12/15/66		32	29	31	+2
12-15-66		33	28	29	+1
Total Average			25.9	26.7	+0.8

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APPENDIX II

SPECTROGRAPHIC ANALYSIS OF WATER SAMPLES WITH ELECTRICAL HEATER AS HEAT SOURCE

	Spectrographic Analysis (ppb)		
	Sample 1	Sample 2	Sample 3
ZINC	36	130	26
CADMIUM	<5	<5	<5
BORON	18	30	8
PHOSPHORUS	20	105	20
IRON	5	73	7
MOLYBDENUM	*	*	*
MANGANESE	3	9	<3
ALUMINUM	1375	1500	1500
BERYLLIUM	.03	.03	<.03
COPPER	500	500	350
SILVER	5	5	<.5
NICKEL	5	20	5
COBALT	<5	<5	<5
LEAD	10	10	<10
CHROMIUM	3	17	3
VANADIUM	10	<10	<10
BARIUM	4	12	4
STRONTIUM	<1	<1	<1
MERCURY	--	--	--
ARSENIC	25	<25	<25

Date: 11 18-66

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SPECTROGRAPHIC ANALYSIS OF WATER SAMPLES WITH RADIOISOTOPE AS HEAT SOURCE

Date:	Spectrographic Analysis (ppb)						
	12-6-66	12-6-66	12-4-66	12-6-66	12-13-66	12-15-66	12-15-66
ZINC	200	250	14	38	35	63	<5
CADMIUM	<5	<5	<5	<5	<5	<5	<5
BORON	8	<3	14	<5	7	7	<5
PHOSPHORUS	90	60	<25	<25	55	55	30
IRON	75	75	26	20	40	25	9
MOLYBDENUM	*	*	*	<10	<10	<10	<10
MANGANESE	8.5	<2.5	<2.5	3.5	<3	<3	<3
ALUMINUM	2250	1500	1000	465	1000	525	70
BERYLLIUM	<.03	<.03	<.03	<.03	<.03	<.03	<.03
COPPER	225	175	6	7	25	<3	<3
SILVER	<.5	<.5	<.5	<.5	4	<.5	3
NICKEL	16	<5	<5	<5	<5	<5	<5
COBALT	<5	<5	<5	<5	<5	<5	<5
LEAD	45	<10	15	15	40	<10	30
CHROMIUM	21	10	<5	<5	<3	<3	<3
VANADIUM	<10	<10	<10	<10	<10	<10	<10
BARIUM	5	14	1	4	80	<1	<1
STRONTIUM	<1	<1	<1	<1	<1	<1	<1
MERCURY	—	—	—	—	—	—	—
ARSENIC	<25	<25	<50	<50	<25	<25	<25

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APPENDIX III

BACTERIOLOGICAL ANALYSIS OF WATER SAMPLES WITH
ELECTRICAL HEATER AS HEAT SOURCE

	Sample Number		
	1	2	3
Ammonia (mg l)	87	250	75
Chloride (mg l)	9	250	7.5
pH	9.2	9.2	9.1
Conductivity (μ mhos/cm)	270	1570	260
Total Hardness (CaCO_3)	<1	<1	<1
Total Alkalinity (CaCO_3)	298	1300	356
Urea	88	<.1	<.1
Total Plate Count	—	—	—
Total Carbon	710	25	2800

Date: 11-18-66

BACTERIOLOGICAL ANALYSIS OF WATER SAMPLES WITH
RADIOISOTOPE AS HEAT SOURCE

	Sample Number						
	1	2	3	4	5	6	7
Date:	12-6-66	12-6-66	12-4-66	12-6-66	12-13-66	12-15-66	12-15-66
Ammonia (mg l)	10	6.3	17	8	1500	4.2	3.0
Chloride (mg l)	48	36	6.5	6.5	18	11	4.0
pH	4.1	4.4	8.9	8.2	9.7	4.0	8.3
Conductivity (μ mhos/cm)	205	165	175	170	2900	77	116
Total Hardness (CaCO_3)	14	5	6	6	<1	<1	1
Total Alkalinity (CaCO_3)	0	0	226	86	6200	38	52
Urea	10	10	<.1	<.1	80	<.1	1
Total Plate Count	—	TCN**	—	o/ml**	—	—	<1 ml**
Total Carbon	135	2000*	5.2	4	45*	12	5

— No Data

* AI interference

** Agar Pour Plate Technique

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APPENDIX IV

SUMMARY OF MASS SPECTROMETRIC ANALYSES OF EXIT GAS

Date Taken	Time Taken	Exit Gas Components, Mole %									
		H ₂	CH ₄	H ₂ O	N ₂	C ₂ H ₆	O ₂	Ar	CO ₂	C ₃ N ₈	C ₃ N ₈ +CO ₂ ^a
11-17	0900	24.4	0.8	4.3	27.4	1.0	5.7	0.7	35.5	0.0	35.5
11-22	0930	0.0	9.2	0.7	27.8	5.2	0.7	0.9	5.6	49.8	55.4
11-29	1000	31.3	0.7	0.4	24.9	0.9	6.6	0.2	33.1	1.6	34.7
12-2	1000	0.0	0.6	0.1	67.8	3.7	14.5	4.3	6.2	2.8	9.0
12-5	1300	0.0	0.5	1.1	54.0	25.4	2.8	5.0	10.4	0.7	11.1
12-8	1000	20.3	0.1	0.0	51.4	0.0	2.1	1.4	14.2	0.0	14.2
12-15	1032	35.8	0.0	0.2	38.9	0.0	3.4	0.4	21.1	0.0	21.1
12-15	1500	42.4	1.1	0.1	29.8	1.1	3.9	0.4	19.4	1.7	21.1
12-16	1455	58.5	0.2	0.0	22.0	0.0	1.2	0.1	17.6	0.0	17.6
12-16	1525	5.0	0.1	0.0	76.5	0.0	1.3	0.8	15.5	0.0	15.5

^aBoth mass 44; Differentiation is made by the operator's judgement as to fragments observed at mass 43 (C₃H₇⁺)

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