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Quarterly Progress Report No. 3

Task 8--Strontium-90 Fueled

Thermoelectric Generator Development

May 1, 1961 through July 31, 1961

MND-P-2483-3

NUCLEAR MARTIN DIVISION

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FOREWORD

This quarterly report covers the period from May 1 through July 31, 1961. It has been prepared by The Martin Company according to the requirements of Contract AT(30-3)-217, Task 8, with the U. S. Atomic Energy Commission.

CONTENTS

SUMMARY

The SNAP 7 program is being conducted by The Martin Company for the purpose of developing four radioisotope-fueled thermoelectric power generation systems. An important phase of this program is the processing of Strontium-90 into heat sources for these systems.

I. INTRODUCTION

The SNAP 7 program covers:

(1) Design, fabrication, test and delivery of four radioisotope-fueled thermoelectric generator systems to meet the rigorous environmental requirements of field use by the United States Coast Guard and the United States Navy.

(2) Fabrication of the Strontium-90 fuel for two of the aforementioned generators.

The four deliverable generator systems under the contract are as follows:

(1) SNAP 7A: 5-watt electric generation system for U. S. Coast Guard light buoy, Subtask 8.1.

(2) SNAP 7B: 30-watt electric generation system for U. S. Coast Guard fixed light station, Subtask 8.2.

(3) SNAP 7C: 5-watt electric generation system for U. S. Navy weather station, Subtask 8.3.

(4) SNAP 7D: 30-watt electric generation system for U. S. Navy boat-type weather station, Subtask 8.4.

Fuel processing was isolated as a separate subtask, Subtask 8.5, after initiation of the program, to permit a more detailed surveillance of this aspect of the program. This report has been divided into three major sections: one covering Subtasks 8.1 and 8.3, one for Subtasks 8.2 and 8.4 and one for Subtask 8.5; however, it should not be overlooked that this is a highly interrelated program where variations in any subtask may produce significant effects in one or more of the others.

II. SNAP 7A AND 7C FIVE-WATT ELECTRIC GENERATION SYSTEMS--SUBTASKS 8.1 AND 8.3

A. INTRODUCTION AND SUMMARY OF SIGNIFICANT TECHNICAL ACHIEVEMENTS

The SNAP 7A and 7C generators were analyzed and designed during the first quarterly report period (Ref. 1); and during the second report period, many components and subassemblies were manufactured (Ref. 2). The highlights of the current report period were:

(1) Assembling the thermoelectric reliability breadboard model and initiating performance tests.

(2) Assembling the operating model of the 10-watt thermoelectric generator and preparing it for electrical tests.

(3) Finalizing installation details for both the SNAP 7A and SNAP 7C generation systems.

B. ENGINEERING--EQUIPMENT DESCRIPTION, DESIGN TECHNIQUES AND PROCEDURES, AND TEST FOR SNAP 7A AND 7C

To analyze, design, assemble and test SNAP 7A and 7C generators and their electrical systems--including design and modifications required for field installations--are the objectives of Subtasks 8.1 and 8.3.

The SNAP 7A system, made up of a 10-watt thermoelectric generator, a dc-to-dc converter and voltage regulator, and a nickel-cadmium battery pack capable of delivering five watts at 12 volts, is to be installed in a Coast Guard buoy (8X26E) to operate a flashing light. The system is designed to be maintenance-free for two years. Initially, the buoy will be located in Arundel Cove at the Curtis Bay Coast Guard Station. The SNAP 7C system, comprising a 10-watt thermoelectric generator, a dc-to-dc converter and regulator and a 28-volt nickel-cadmium battery package, will deliver four different voltages as required by the Naval Research Laboratory's automatic weather station. It will be located on the Antarctic continent at Little America Station No. 5 (Fig. 1). Special provisions will ensure proper operation of the generator and weather station in the extreme Antarctic climate.

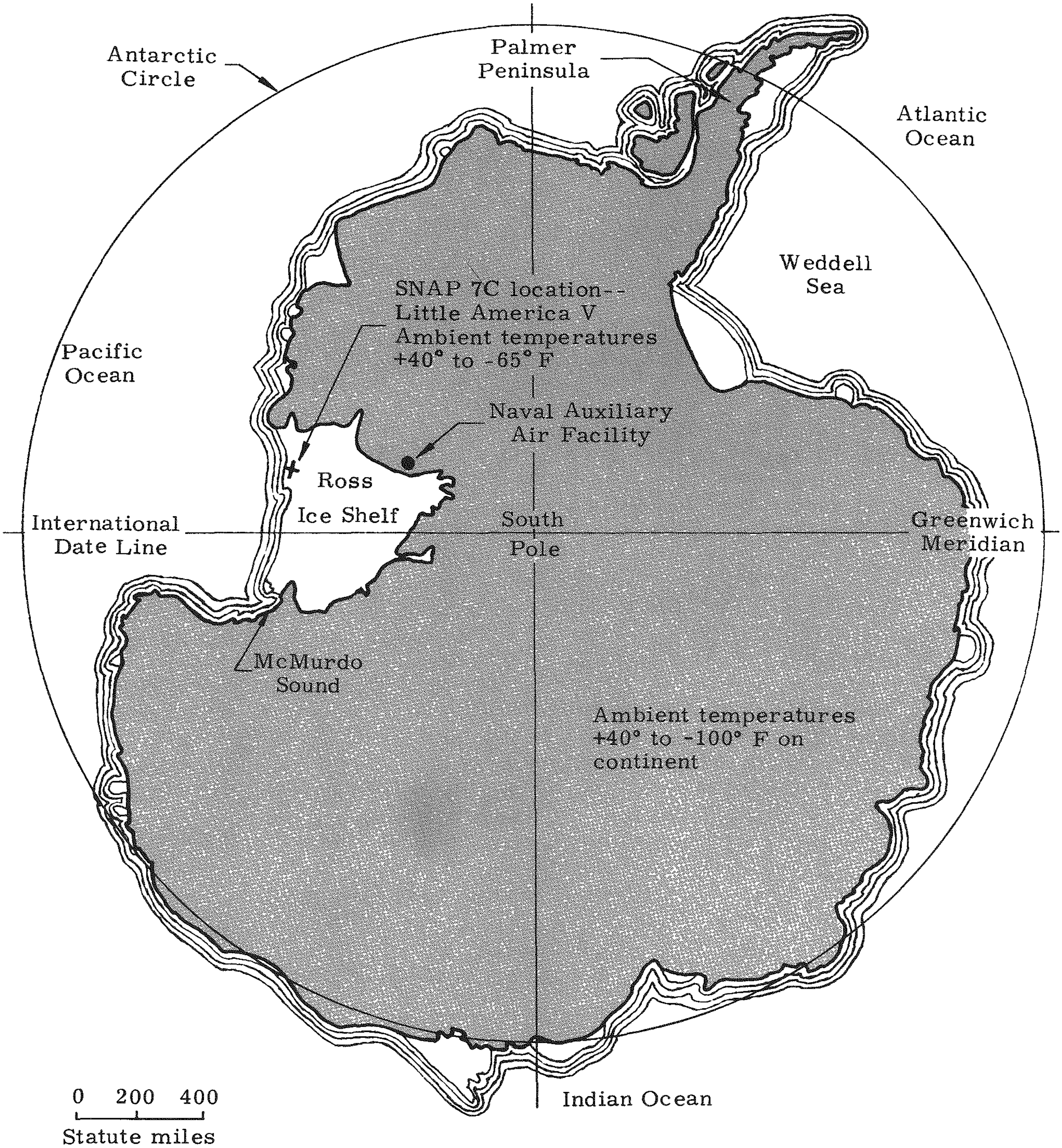

Fig. 1. Map of Antarctica

1. Design

During this report period, the installation design of the SNAP 7A light buoy system was completed and approved by the Coast Guard. The position of the generator, the location of the battery-converter enclosure and the arrangement of electrical connections were reviewed. Of the several possible installation designs, the Coast Guard approved the one displayed in Fig. 2, which shows the 10-watt generator located 11 feet under water in the counterweight tube. The tube will be sealed and filled with fresh water to facilitate the transfer of heat to its walls. The 12-volt battery package and the dc-to-dc converter are to be located in a waterproof enclosure in the battery pocket above the generator. In previous data obtained by the Coast Guard, the upper portion of the battery pocket usually indicated a temperature about 20° F higher than the surrounding air temperature. The position of the battery-converter enclosure--in the battery pocket about 18 inches above the bottom--was chosen, first, to keep it out of any water splashed into the pocket, and second, to keep it below the buoy water line for a more uniform temperature.

Design engineers, during this report period, considered the problems of connecting the electrical output of the generator to the dc-to-dc converter and connecting the battery to the lantern leads. Since the generator is to be surrounded by water, the type of electrical connection is critical. Two were investigated: waterproof cable and connections, and conduit with cables. After a study of several designs, one employing conduit was chosen. In this design a 3/4-inch Hastelloy C pipe extends from the top of the generator up through the body of the buoy into a junction box at topside. The cap on the two-inch pipe which surrounds the 3/4-inch pipe seals off the tube and thus prevents water from splashing up from the counterweight tube into the junction box. Also, the cap provides lateral support for the conduit (Fig. 3). The junction box contains terminal blocks for the power leads and the thermocouples from the generator, as well as power and test leads to both the lantern and the battery-converter enclosure. From this point, temperatures, voltages and currents, can be checked to evaluate the operation of the system.

The shipping pallets, including shock mounts and restraining cables for the 10-watt generators, and the fuel shipping casks are shown in Fig. 4. These pallets were designed to meet ICC safety criteria for shipping by truck, rail, ship or aircraft.

During this period there were two minor generator design changes:

(1) The compression spring was redesigned to increase the bearing pressure on the thermoelectric elements.

Fig. 2. SNAP 7A--Light Buoy

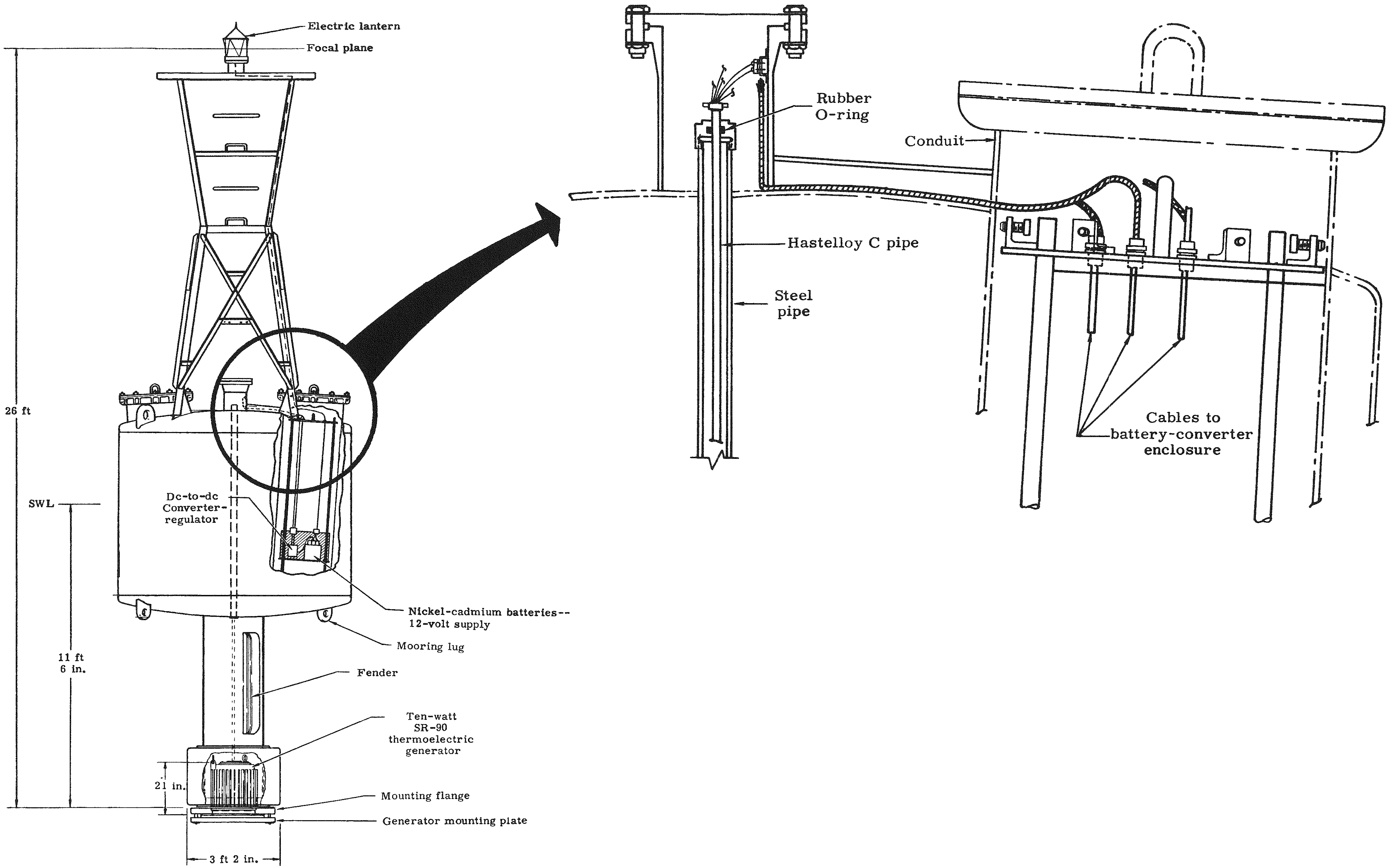

Fig. 3. Electrical Corrections--SNAP 7A Coast Guard Light Buoy

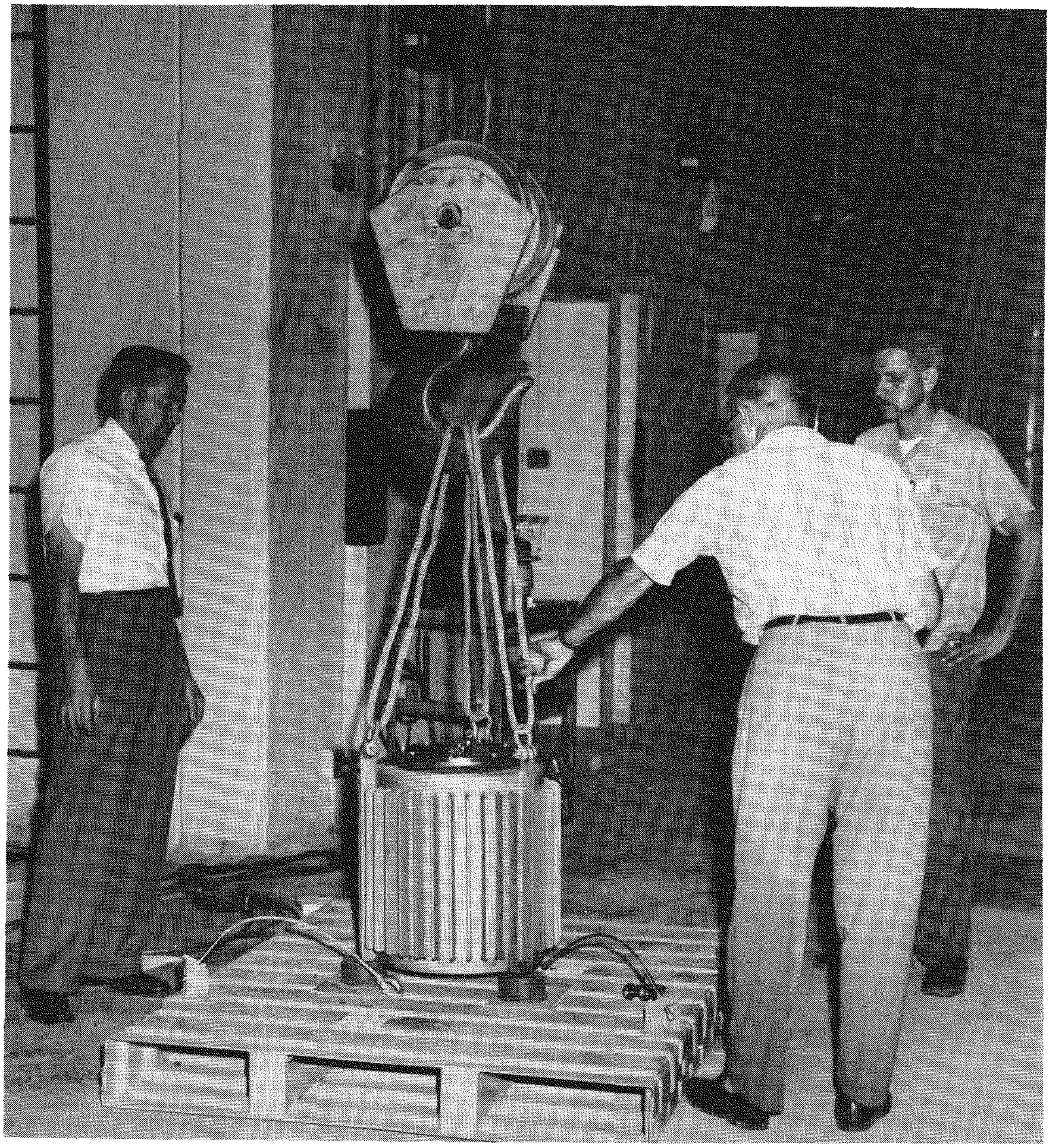

Fig. 4. Fuel Shipping Cask and Pallet

(2) The use of bent connecting wires appeared to be a handicap in the assembly of the thermoelectric modules; therefore, shorter, straight wires, with connecting bars between the pairs, were used to facilitate assembly without affecting the operation of the unit.

The weather station container for the SNAP 7C field installation, as designed, consisted of a plain cylinder 36 inches in outside diameter and 96 inches in height with a watertight cover held in place with a V-band clamp. Originally, a C-130 aircraft was to be used for transportation of the weather station to the field site in Antarctica; however, because of higher priority missions, a C-130 will not be available. The choice of replacement aircraft, an R4D, made it necessary to revise the SNAP 7C container. Because of size and weight limitations, a two-section cylindrical container, rather than one eight-foot tube, is required. The O-ring seal used for the cover in the single tube is not suited for the two-section tube design. Therefore, the flange was redesigned to take a flat silicone gasket that can be held watertight with a V-band clamp. The top cover will use a similar flange and clamp. An artist's concept of the current design appears in Fig. 5.

The battery and converter enclosure in the current design is watertight and measures 20 by 16 by 6 inches. It will be installed in the system container as Fig. 5 indicates. To simplify the assembly, the battery and converter will be mounted on a panel that can be removed from the enclosure. A vent valve in the enclosure will prevent incidental pressure buildup from battery gases.

The cylindrical container, when installed, will rest upon a Navy-supplied platform of 2- by 8-foot planks buried in the ice. The Navy will also supply 2- by 8-foot planks for the four outriggers that support the container and prevent sinking in the ice. At the installation, there will be a thin layer of ice and snow over the container cover, with the mast and bushing exposed.

2. Dc-to-Dc Converter

The preliminary design of the dc-to-dc converter was started with the extrapolated data from the reliability model. Although these data were not suitable for a final design, they were sufficiently close to provide information for a first review of the converter design, to point out problem areas and to provide checks on material and transistor ranges, component sizes and tolerances.

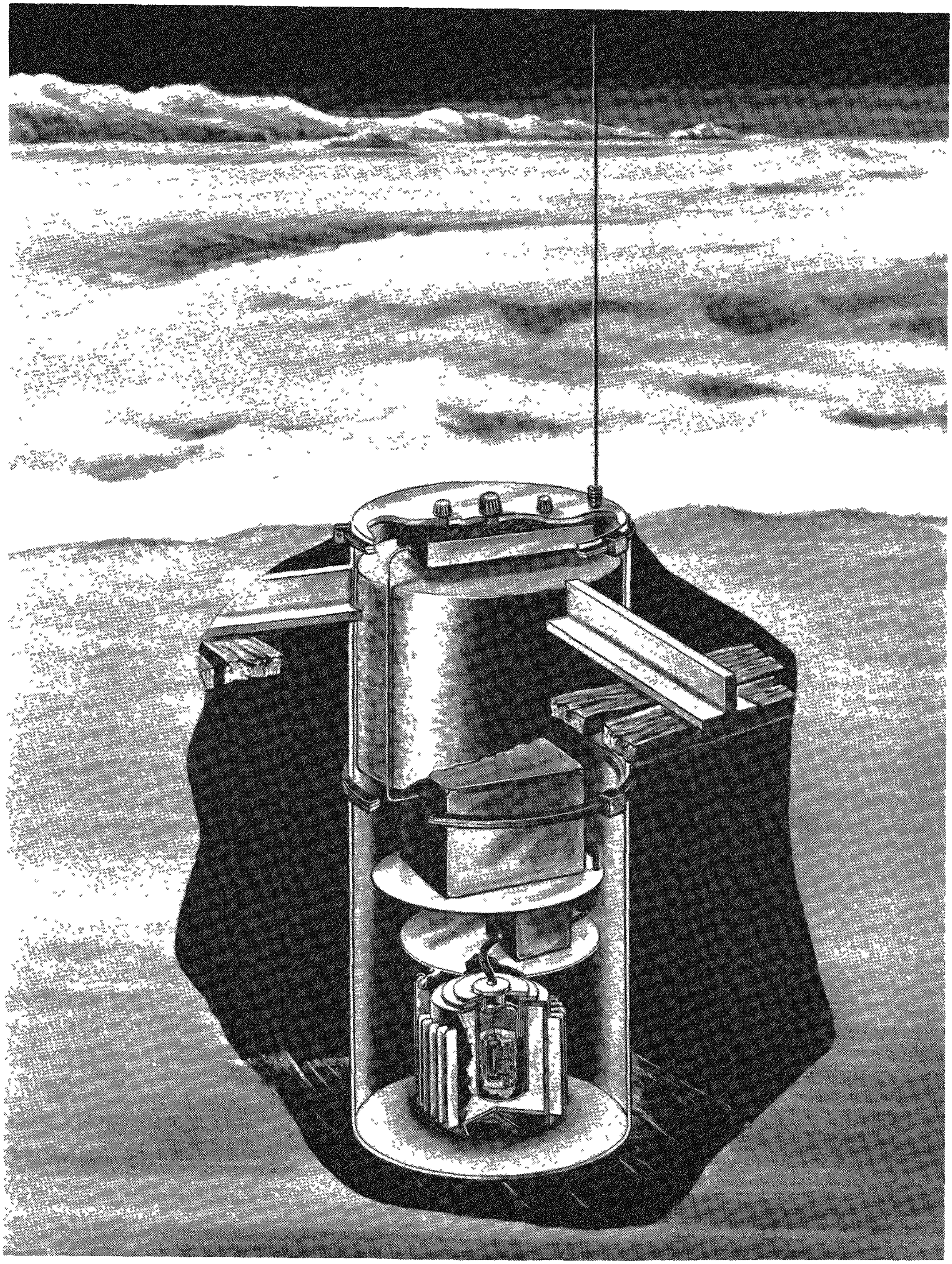

Fig. 5. SNAP 7C--Simplified Concept of Weather Station

3. Reliability and Life Tests*

At the beginning of this report period, three thermoelectric couples of the 10-watt generator size were installed in the laboratory test fixtures. Two of the couples were coated around the hot junctions with Sauereisen Cement, and the third couple was left bare. The results of this test indicate conclusively that Sauereisen Cement appreciably retards sublimation of the elements at the hot junction.

Assembling of the reliability model was completed and testing was started on May 3, 1961. Preliminary test results indicated excessively high temperatures at the cold junctions. As a result of these data, the unit was disassembled and inspected. There were signs of oxidation on the element assemblies, and three couples showed high resistance. When the elements were reassembled, an aluminum grease was added between the cold sink bars and the outer cover. The cold junction temperatures were thus lowered to 145° F, still 30° F above the design conditions. With a hot junction temperature of 930° F, the power output of the initial 15-couple unit was 2.5 watts, equivalent to 10 watts for a full generator. The extrapolated maximum generator output, with a hot-to-cold junction temperature difference of 705° F, was 8.7 watts.

The reliability model was reassembled on July 12, 1961, and tests were initiated. The output power was approximately 20% below the design objectives. The lower power output was attributed to the thermoelectric elements which were tentatively accepted with a merit factor lower than design limits. These elements were used to determine whether it was possible to reduce the element design criteria and produce a satisfactory generator. The results indicate that the original acceptance criteria for the elements must be maintained.

It is intended to replace the test elements with elements conforming to original design specifications. The reliability model will then be tested for 10-1/2 months, with the hot junction temperature maintained at 900° F. At the end of this period, final data will be taken at anticipated operating conditions, and parametric curves will be drawn and extrapolated to 60 couples.

4. Operating Model Assembly

All materials and components for the 10-watt operating model of the generator were received and the generator was assembled.

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*D. Toler and H. Miller

The instrumentation for reading temperatures in the generator was completed by installing thermocouples as follows: one thermocouple, plus a spare, on the fuel block; one each on the hot and cold junctions of a P- or N-type element on Couples 1 and 5 in Module 5, and similarly, on Couples 1 and 5 in Module 6, as a spare; one thermocouple on the heat sink; and one thermocouple on the heat sink bars. The thermocouples were terminated at the hermetically sealed connector. Electrical performance tests of the 10-watt generator were scheduled for early August 1961.

Checkout facilities for the operating models of the 10-watt generator are complete. A dry box was constructed to provide detailed testing, and facilities were made available for high temperature tests and verification of the operational capability of the generator.

III. SNAP 7B AND 7D 30-WATT ELECTRIC GENERATION SYSTEMS--SUBTASKS 8.2 AND 8.4

A. INTRODUCTION AND SUMMARY OF SIGNIFICANT TECHNICAL ACHIEVEMENTS

The SNAP 7B and 7D 60-watt thermoelectric generators were designed, and the associated engineering drawings were released for fabrication and procurement during the second quarterly report period (Ref. 2).

During the period of this report (May 1 through July 31), materials were received and fabrication of components was initiated. Toward the end of this report period, preliminary design investigations were started on SNAP 7B and 7D installations.

B. ENGINEERING--EQUIPMENT DESCRIPTION, DESIGN TECHNIQUES AND PROCEDURES, AND TEST FOR SNAP 7B AND 7D*

The objectives of Subtasks 8.2 and 8.4 are to analyze, design, assemble and test SNAP 7B and 7D generators and their electrical systems. The SNAP 7B system, composed of a 60-watt thermoelectric generator and a dc-to-dc converter and voltage regulator, is to be used for charging a nickel-cadmium battery system in a Coast Guard fixed-light station. The SNAP 7D system, similar to the SNAP 7B, will be utilized to provide power to a boat-type weather station which, anchored 100 miles or more off shore, will broadcast weather data on a regular, programmed schedule. This is a joint U.S. Navy and National Bureau of Standards effort (Fig. 6).

In the preceding quarterly report (Ref. 2), the design and the results of analysis for heat transfer, radiation shielding and electrical output of the 60-watt generator were presented. The effort during the period of the current report has been limited to the following:

(1) Starting the heat transfer analysis for the SNAP 7B installation.

(2) Observing the Naval weather station boat to determine the installation problems of the SNAP 7D system.

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*H. Morton

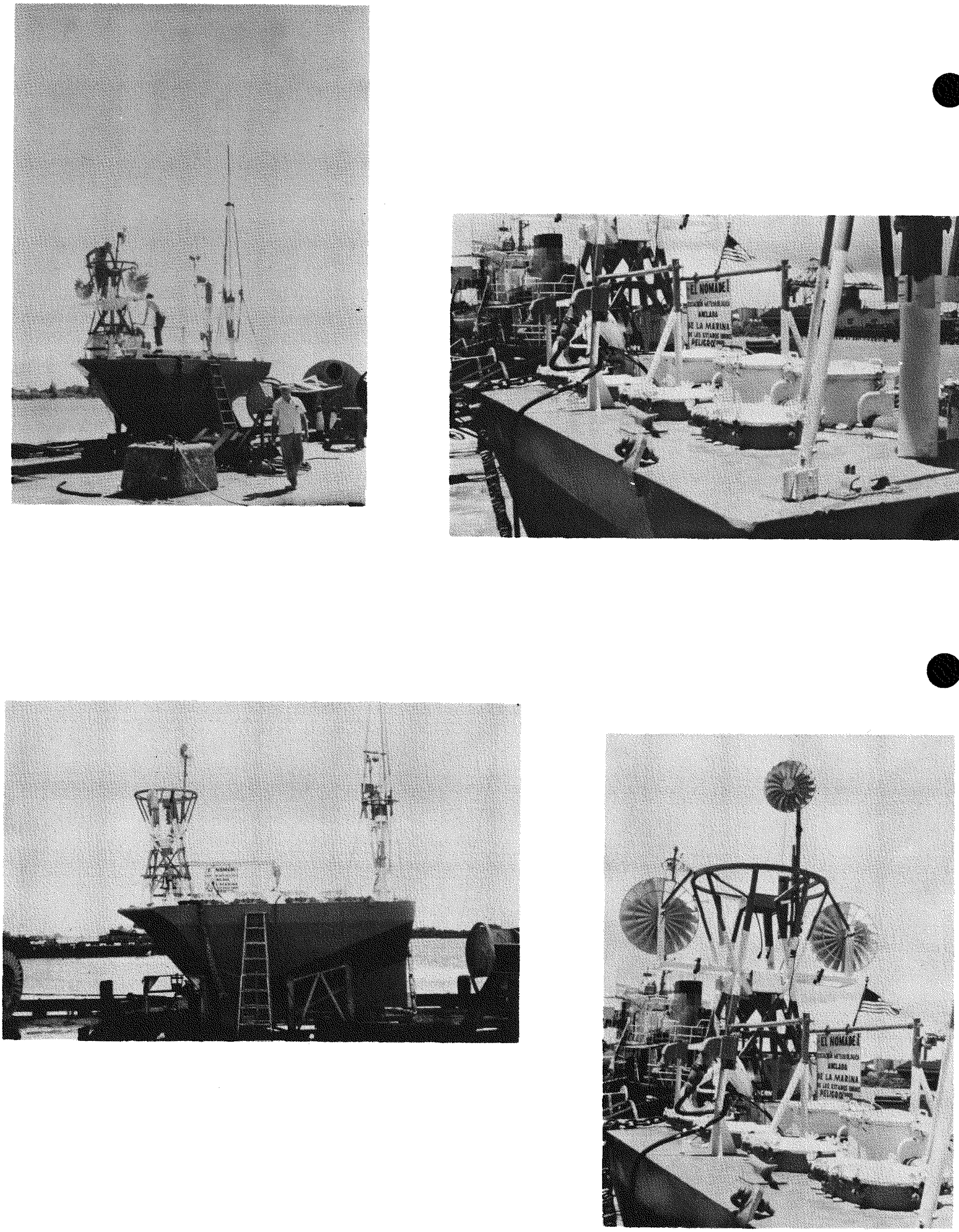

Fig. 6. Views of U. S. Navy Boat-Type Weather Station

(3) Starting the tests of the thermoelectric elements for the 60-watt generators.

1. Design

Since the installation design for the SNAP 7B system was initiated near the end of the period, complete design data are not yet available. A cylindrical one-section container similar to the one used initially for the SNAP 7C system is being considered for housing SNAP 7B.

The weather station boat in which the SNAP 7D generator will be installed is in dry dock for overhaul at Choptaw Point Coast Guard Station, Mobile, Alabama. Since this boat is to be returned to its location in the Gulf of Mexico for approximately a year, it was examined during the quarter to determine the best method of installing the generator and its electrical system. An arrangement which appears to meet the requirements of the generator, as well as those of the Navy and weather station personnel, was conceived.

Several possible installations were considered. Because of the present utilization of the weather boat, the second compartment (port or starboard) from the bow was chosen for the generator. The generator will be lowered, through a new hatch, to a shelf near the keel, then clamped to the center bulkhead to prevent shifting due to the motion of the boat. The section will be filled with oil, covering the generator, to improve heat transfer through the hull to the sea. To prevent surging, a cover will be used over the oil. The electrical and thermocouple wires will be run through stainless steel tubing, with A-N fittings, through the bulkhead, by means of a standard bulkhead tube fitting. The battery-converter enclosure will be in the adjoining compartment. A terminal block will be used for the thermocouple leads to facilitate testing. Figure 7 is an artist's concept of the installation.

2. Reliability and Life Tests

Sixty-watt couples of both Martin and Minnesota Mining and Manufacturing Company (3M) materials were life tested for continued performance of the N and P elements. After 2112 hours of test, the 3M P and Martin N elements showed only very slight changes in properties.

Tests will continue with typical couples for the 60-watt generator design.

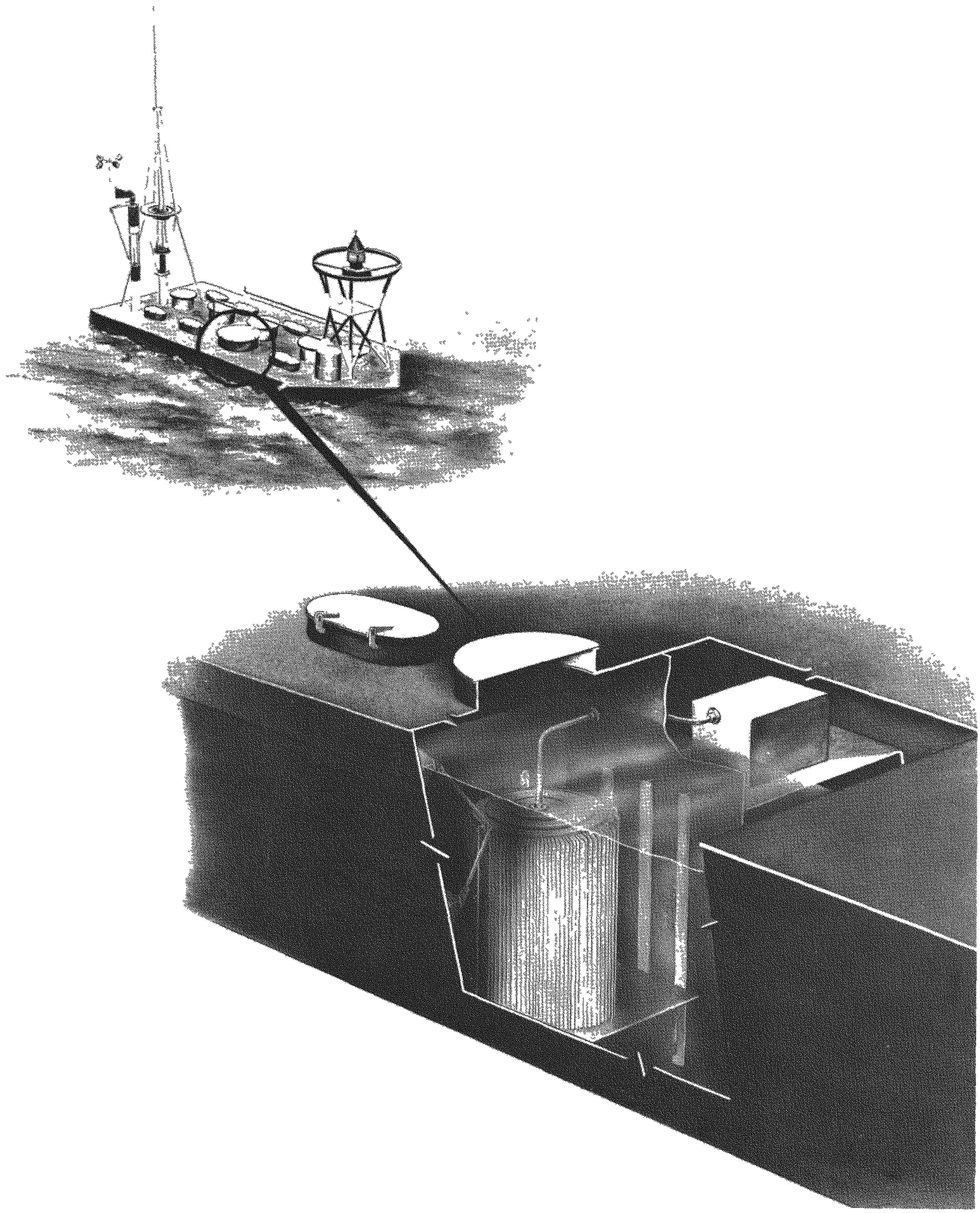

Fig. 7. Artist's Conception of Boat-Type SNAP 7D

IV. FUEL PROCESSING FOR SNAP 7B AND 7D GENERATORS--SUBTASK 8.5

A. INTRODUCTION AND SUMMARY OF SIGNIFICANT TECHNICAL ACHIEVEMENTS

During this quarter, effort has been expended primarily in three areas, namely:

(1) Fuel process engineering.

(2) Nuclear chemistry audit.

(3) Manufacturing fuel processing equipment.

The significant achievements in each area are discussed in the following paragraphs.

1. Fuel Process Engineering

The objectives of fuel process engineering for the SNAP 7 program are to design isotope fuel processing equipment for the Quehanna facility which will be used to convert strontium carbonate (as received) to strontium titanate pellets, and to encapsulate the fuel pellets in Hastelloy C containers. These capsules will be the isotope fuel heat sources for the 60-watt thermoelectric generators.

During this report period the major effort was directed toward the design of the fuel processing system and components, and the establishing of shielding requirements. A safety analysis was also conducted to evaluate the possibility and consequences of accidents.

2. Nuclear Chemistry Audit

A comprehensive evaluation, or audit, of the entire chemical process was performed during this report period. The results of the study will be published subsequently; however, significant aspects are covered in Section C of this chapter.

3. Manufacture of Fuel Processing Equipment

Fabrication of many of the process equipment items, such as those listed, was initiated during this quarter.

(1) Containment box for Cell No. 2.

(2) Stainless steel storage tanks.

(3) Electric furnace for calcining and sintering.

(4) Jacketed precipitation vessel.

(5) Hydraulic pelleting press.

(6) Automatic heliarc welder.

(7) Electrically driven ball mill.

(8) Stainless steel valves.

(9) Stainless steel tubing and fittings.

(10) Miscellaneous instrumentation and accessory hardware.

Additional hot cell items and components are near design completion and will be manufactured during the next period.

B. FUEL PROCESS ENGINEERING*

The main effort during this quarter has been directed toward the design of the fuel processing equipment. Since the general process was not changed from that defined in the preceding quarterly report, this report will point out only the minor changes and corrections and, in some areas, amplify the previous data. Refer to Fig. 8.

The most obvious change in the system was the relocation of the containment box in Cell 2. Using three-cell operation, as indicated in the last quarterly report, permitted relocating equipment from the Cell 2 isolation room into Cell 1 proper. This change made it possible to use the transfer port in the right rear corner of Cell 2 for the transfer of items to and from the containment box. So that these transfers could be made with maximum safety, a transfer can system was designed similar to the "alpha can" system used at Los Alamos Scientific Laboratory. It consists of an adapter in the containment box designed to receive and load a sealed can in such a manner that the exterior of the can is not exposed to contamination. After the can is loaded, it may be withdrawn from the adapter, through the rear cell wall, into a shielded housing. (The shielding is sufficient to allow personnel to handle the housing.) The housing is then moved to another cell or other designated zones where the can may be removed by a reverse procedure. Air cooling at 10 standard cubic feet per minute (scfm) has been provided to keep the can temperature below 200° F during the transfer of the hottest item, a fuel capsule. It is planned to make all normal transfers into and out of Cell 2 by this method.

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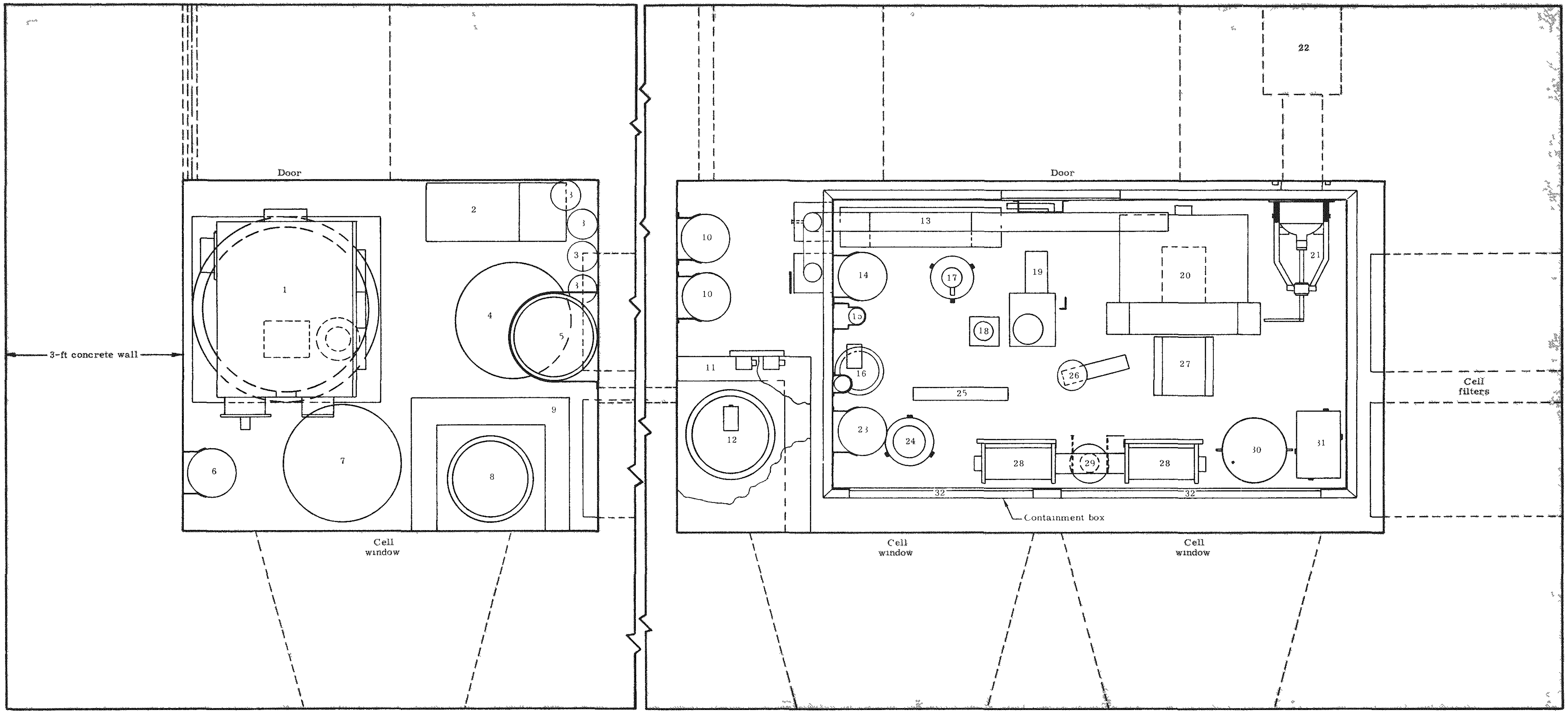
*C. Young



LEGEND

1	Shipping cask	17	Blender
2	Vacuum pump	18	Die charger
3	Air filters	19	Remote welding assembly
4	Waste filter	20	Furnace
5	Vacuum tank	21	Transfer mechanism
6	Transfer tank	22	Transfer port
7	Waste drum	23	Overflow tank
8	Storage cask	24	Filter housing
9	Lead brick shielding	25	Pellet transfer tray
10	Transfer tanks	26	Balance
11	Lead shielding	27	Furnace loading table
12	Waste tank	28	Absolute filters
13	Air inlet duct	29	Exhaust duct
14	Filtrate tank	30	Shielded capsule holder
15	Metering vessel	31	Cleaning tank
16	Precipitation vessel	32	Containment box windows

Fig. 8. Floor Plan - Cells 1 and 2.

1. Transfer Box

In addition to the transfer can, a large transfer box* will be provided for emergency use. Its function is to permit removal of the large pieces of equipment from the containment box for maintenance. The transfer box is large enough so that the largest piece of equipment in the cell may be lifted by an air hoist from the containment box into the transfer box for removal. During a transfer, the boxes are sealed to each other and the doors are mated in such a way that no external surfaces are exposed to contamination. Air cylinders provide the required locks and door actuation. The box control system may be operated from a distance (up to 30 feet); and since the system is to handle relatively low radiation levels, no other shielding is necessary.

2. Containment Box Entry

In addition to the transfer can and the transfer box, another access into the containment box will be through a two-foot (diameter) door in the rear of the box. This door is used primarily during the initial equipment installation. It is equipped with a flange so that a special suit arrangement can be used for entry, provided the radiation levels are low enough. Such entry may be feasible for the final decontamination of the containment box.

3. Valves

Manually operated valves have been selected for all fuel process equipment to give maximum reliability. The 3/8-inch and 1/2-inch valves located in the radiation areas are all stainless steel Hoke valves with bellows seals plus backup packing on the stem. They are provided with stub tube ends and will be equipped with Swagelok fittings for connection into the system. Ball valves will be used for the 3/4-inch lines.

4. Sampling System

The sampling system selected utilizes a plug cock with a calibrated metering hole to remove a sample from the process line or tank concerned. The sample, which is varied in size according to the anticipated activity levels, is then washed through the lines by a dilution and rinse water aliquot and received in a special glove box located in the isolation room behind Cell 2. Since all volumes are known, the volume received in the glove box can be checked to assure that there is no plugging nor hangup in the system.

*This box is patterned after one employed in the alpha-tight Transuranium Development Facility at ORNL.

5. Cell 2 Lighting

The containment box will be lighted by four 21,000-lumen, mercury vapor, wide angle lights. To further diffuse the light for shadow reduction and glare prevention, a sandwich-type window is used under the lights. It consists of a sheet of thin matted T-80 Plexiglas; the main containment window, which is 1/2-inch Plexiglas G; and, on the underside of the window, a thin sheet of pigmented 2447 Plexiglas.

6. Air Filters

The air filters for the Cell 2 containment box will consist of two Flanders Airpure absolute filters in parallel for inlet and exhaust. Each filter is rated at 68 cfm at one inch of water, and may be used singularly or jointly by controlling the flow with butterfly-type valves. The inlet filters are external to the box; the exhaust filters, internal. A large filter of the same type, rated at 170 cfm at one inch of water, is located between the exhaust filters and the duct that leads to the exhaust fan. These filters will resist temperatures to 1000° F and will withstand moderate steam cleaning.

7. Metering Tank

A liquid level indicator has been added to the metering tank. This gage will be calibrated at installation (within 0.5%) and will permit controlled transfers of intermediate volumes controlled by standpipes. A check valve is to be used in the vacuum line to prevent flooding in the event of accidental overfill of the tank.

8. Electric Furnace

The selected glowbar furnace requires a one-gallon per minute (gpm) water flow to maintain the 125° F maximum exterior surface temperature. Original plans required no water, but no furnace manufacturer contacted was willing to attempt to supply such a furnace without water cooling to operate within the maximum allowable temperature envelope. The low exterior temperature was selected to keep the containment box as cool as possible and, also, to reduce the possibility of damage to the manipulator booting which will be of a plastic material. Analysis shows that if the water cooling were to fail completely, the furnace exterior surface temperature would stabilize at 298° F, causing an ambient temperature increase in the containment box of approximately 19° F within acceptable limits. The furnace is equipped with a program-type controller and an automatic recorder. A spare thermocouple control has been built into the furnace for increased system reliability.

9. Ball Mill

The ball mill design was reviewed and the decision was made to add cooling fins to the ball mill can to permit the processing of isotope quantities greater than 5000 curies. The equipment now permits the handling of 10,000 curies of Strontium-90 with a Strontium-89/Strontium-90 ratio of three to one. Originally, the expected power output per gram necessitated rather bulky batches and the 5000 curies appeared to be the optimum mill batch. However, based on the fuel received by Oak Ridge from Hanford, higher power outputs are being achieved and it may become desirable to mill batches greater than 5000 curies at one time.

10. Shielding Analysis*

During the past quarter the following items were given consideration:

(1) The amount of shielding required during fuel processing to allow brief entry into the hot cell.

(2) The amount of shielding required for storing and shipping fuel process wastes.

(3) A review of the shielding analysis of the Quehanna hot cells as related to Strontium-90 processing.

To allow brief entry into the Strontium-90 processing cells, shielding will be required for the water-jacketed capsule chill blocks and the slurry (or digestion) tank. Further, the dose rate from the radioactive dust which will collect in the processing box must be estimated.

The maximum loading of the water-jacketed copper chill block is 20,000 curies of Strontium-90. Under this maximum loading, 4-1/2 inches of lead are required to reduce the dose rate at the surface of the lead shield to 200 milliroentgens per hour (mr/hr). The digestion tank has a capacity of 25 gallons. The maximum loading here will be 168,000 curies of Strontium-90. To reduce the dose rate at the tank's surface to 200 mr/hr, five inches of lead are required.

Shielding of the thicknesses for the capsule storage cask and the slurry tank will allow entry into the cells for varying periods of time, depending upon the accumulated dosage of the individual and the amount of radioactivity present.

*A. Spamer

An attempt was made to determine the amount of Strontium-90 which could collect as particulate matter in the processing box and give a dose rate of 500 mr/hr at a distance of six feet. Two configurations were used for analysis: one, a point source; the other, an infinite plane source. The point source gave a value of 134 curies; the plane source, a value of 2.5 millicuries per square centimeter (mc/cm^2), which gives a total of 140 curies when spread over a 6- by 10-foot area. Converting these figures to weight gives a calculated total amount of Strontium-90 of about one gram. The corresponding amount of strontium titanate is about two grams.

While 2.5 mc/cm^2 appears to be a high value for Strontium-90 collected over the whole box area, it should not be too high for specific areas. Severe dusting has been experienced at ORNL during weighing and die loading operations (Ref. 3). Because of this accumulation of dust, partial decontamination of the box between batches may be required to prevent excessive dose rates.

Figure 9 is a curve showing the thickness of lead shielding required to reduce the surface dose rate from a given curie-quantity of Strontium-90 (100 to 100,000 curies) to 10 mr/hr. The curve was prepared for use in estimating the shielding required for various pieces of processing equipment. It should give conservative shielding estimates since the self-absorption of the source and absorption by structural materials were not accounted for in the calculations.

The processing waste liquids will be disposed of in 55-gallon drums lined with three inches of concrete. The remainder of the drum volume is to be filled with vermiculite, which will absorb the liquid waste. An analysis of the drum showed that it could contain a maximum of 24 curies of Strontium-90 for a dose rate equal to or less than 10 mr/hr at one meter from the center of the drum.

The effectiveness of the shielding around the equipment depends upon the accuracy of the estimates of curie quantities to be shielded. If the estimate is low, the calculated amount of material which may be put into a shielded vessel will be higher than that actually allowable. More storage or shipping containers than the calculations indicate would be required. If the estimated activity is high, the shielding will be conservative for the real contents; additional activity could be added to the vessel.

The types and amounts of shielding in the Quehanna Laboratory hot cells used for processing Strontium-90 are discussed in Ref. 4, the Quehanna Facility Safety Report. This work was published prior to the existence of measurements made at ORNL of the dose rates from a 1000-curie source of Strontium-90. Although the ORNL experimental data indicate that the dose rates reported for the Quehanna equipment

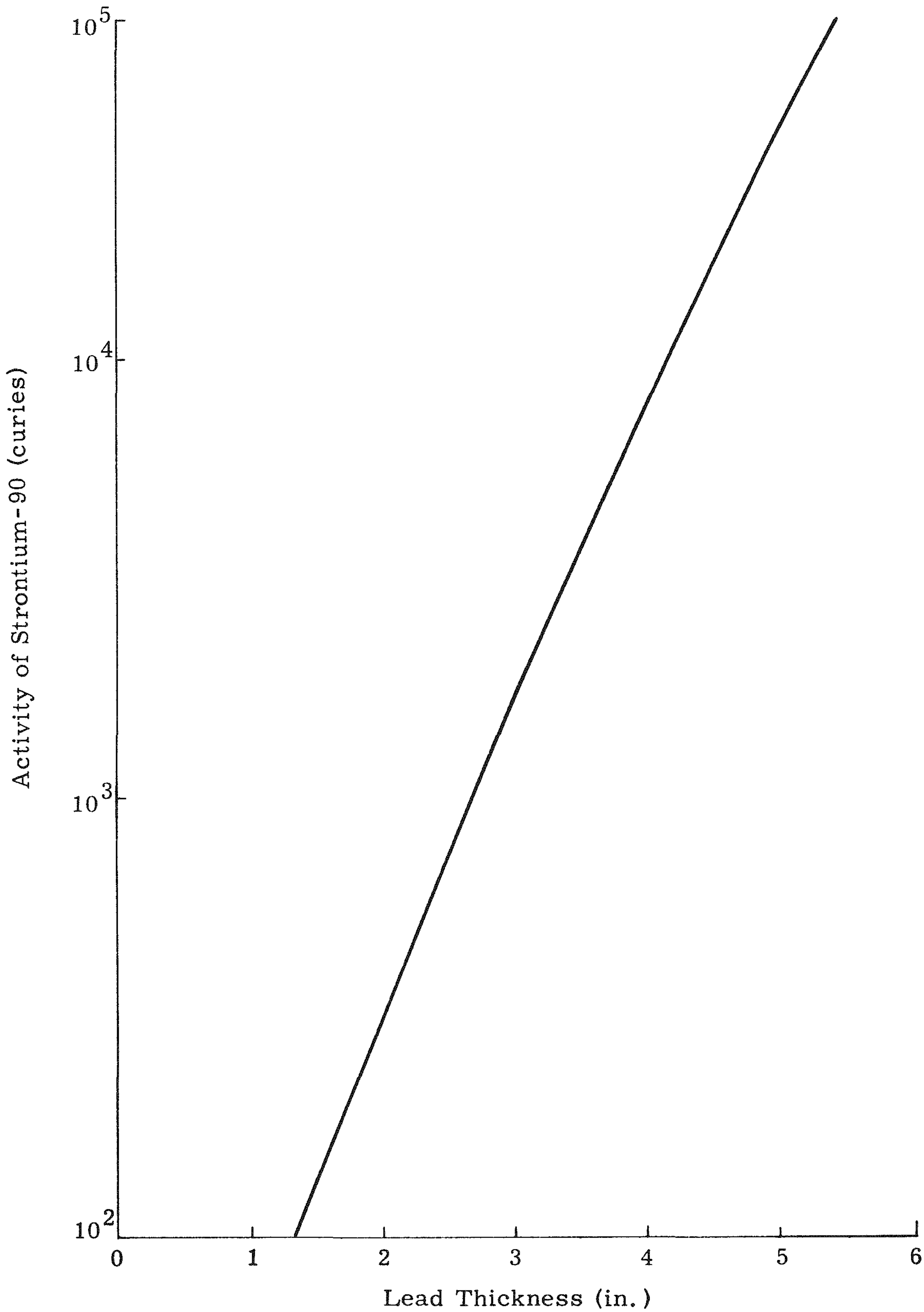


Fig. 9. Shielding Required to Limit Dose Rate to 10 mr/hr at One Meter from Center of Source of Strontium-90

are conservative, the shielding analysis was repeated on the basis of the ORNL results. The cell configuration used in the present analysis is shown in Fig. 10 and the results are given in Table 1. Dose rates at the front of the cell through the ferrophosphorous concrete walls and through the windows were calculated. Dose rates at the top of the cell, through the two-foot (thickness) ordinary concrete roof slabs, were also calculated. Two curie strengths (20,000 and 100,000) and two Strontium-90 forms (liquid and titanate) were used in the calculations.

The shielding provided in the hot cell walls and windows reduces dose rates to extremely low values. The cells with two-foot walls are adequate for processing 20,000-curie batches of Strontium-90. The highest dose rates shown in Table 1 occur at the roof of the cell; however, they are still low enough to allow an individual to remain on the roof for a reasonable length of time.

11. Safety Analysis*

A preliminary safety-failure analysis was initiated during this period to evaluate the possibility and radiobiological consequences of accidents during processing. Each individual step, as defined previously (Ref. 2), was investigated, and credible accidents were postulated for each step.

Typical of the fuel-processing accidents postulated, and their consequences, are:

(1) Dropping of the shipping cask by overhead crane.

The cask is designed to withstand a fall of 30 feet, a height greater than it could possibly be subjected to in the Facility.

(2) Trapping in the system of CO_2 gas formed during processing.

The vessels and tubing will contain any pressure buildup due to trapped gases.

(3) Leakage during the transfer of liquids from one vessel to another.

During all transfer procedures at least two barriers will exist between personnel and radioactive fluid. In preprocessing, the second barrier is the hot cells; therefore, if leakage from components occurs, remote cleanup procedures can be applied.

*V. Kelly

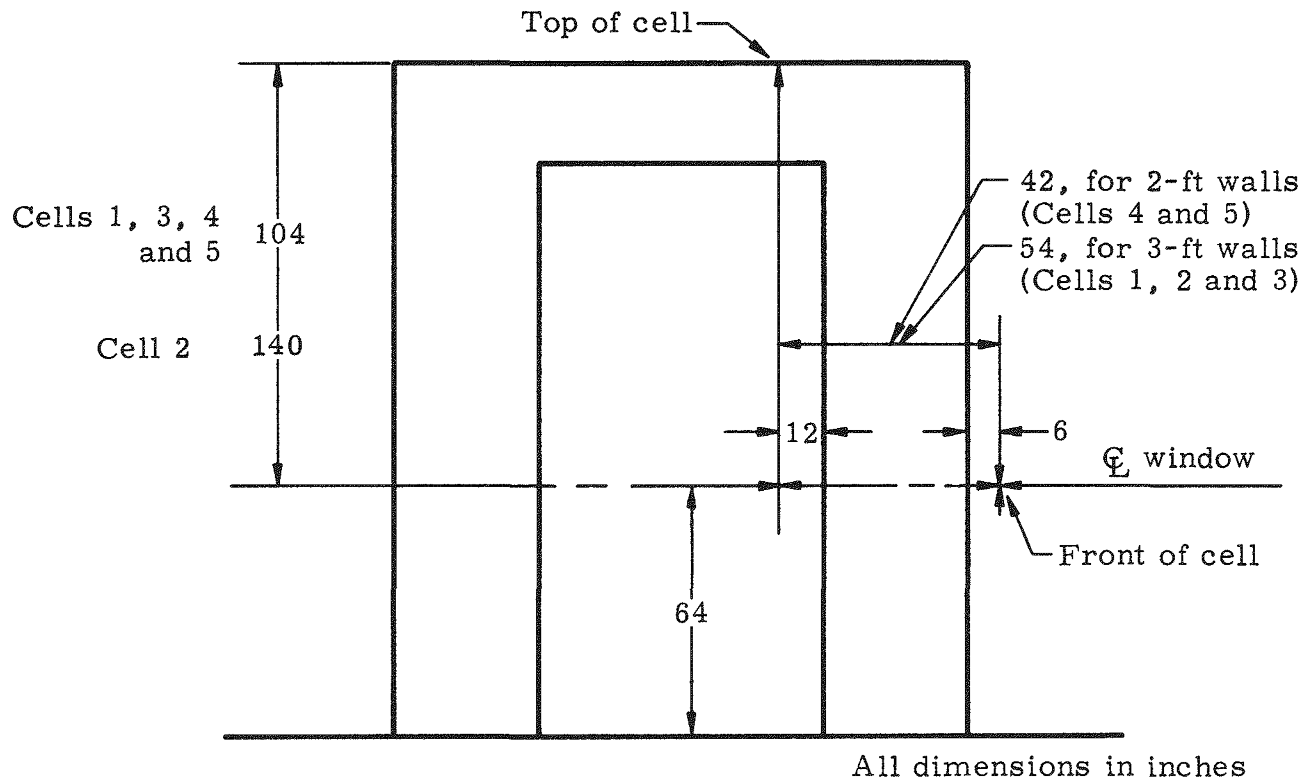


Fig. 10 Configuration Used for Shielding Analysis of Strontium-90 Processing Cell

TABLE 1

Calculated Dose Rates from Strontium-90 Processing Cell

Location	Form	Amount of Sr-90 (curies)	Cell 2 2-foot walls (mr/hr)	Cells 1, 3, 4 and 5 2-foot walls (mr/hr)
Front of cell	Liquid	20,000	1.37	2.5
		100,000	6.85	12.4
	Titanate	20,000	3.55	6.4
		100,000	17.8	32.2

Location	Form	Amount of Sr-90 (curies)	Cells 1, 2 and 3 3-foot walls (mr/hr)	Cells 4 and 5 2-foot walls (mr/hr)
Top of cell	Liquid	20,000	0.23×10^{-5}	4.6×10^{-3}
		100,000	1.1×10^{-5}	2.3×10^{-2}
	Titanate	20,000	0.6×10^{-5}	1.2×10^{-2}
		100,000	2.9×10^{-5}	5.9×10^{-2}

Location	Form	Amount of Sr-90 (curies)	Cells 1, 2 and 3 3-foot windows (mr/hr)	Cells 4 and 5 2-foot windows (mr/hr)
Top of cell	Liquid	20,000	0.0032	0.66
		100,000	0.016	3.4
	Titanate	20,000	0.0082	1.7
		100,000	0.042	8.9

NOTE:

Roof of cell is ordinary concrete; walls for ferrophosphorous concrete.

(4) Loss of coolant to the vessels, causing the temperature of the solution to rise. The solution decomposes, releasing gas and contaminated particles to the ventilation system. This is assumed to pass through the filters and stack to the environment outside the building.

An individual inhaling the contaminated air will receive an internal dose that is below the maximum emergency dose allowed for occupational workers.

A majority of the released activity will be contained in the filters within the cell. These filters can be changed remotely and are treated as high level waste.

If the system is accidentally closed, the trapped gas will be contained under the maximum pressure buildup.

(5) Rupture of the cooling lines within the containment box.

The force of the escaping water is not sufficient to penetrate the Plexiglas windows.

The release of water at elevated temperatures will not increase the internal pressure significantly. However, loss of differential cell pressure will occur.

(6) Release of radioactive powder due to a spill or gas release. The powder particles are assumed to settle out in the containment box and/or are released to the ventilation system.

If the particles settle out, remote vacuum cleaner procedures will be used to clean the box.

If a release to the ventilation system occurs, the dose received by an individual outside the facility will not exceed the maximum emergency dose allowed for occupational workers.

The majority of the activity will be contained within the filters within the cell. They can be removed remotely and treated as high level waste.

(7) Mechanical failure of equipment.

Any piece of equipment can be moved to a storage area by means of a transfer box.

(8) Excess contamination is released to the waste storage system.

The waste storage system is protected by a filter arrangement. However, provisions have been made to shield the storage tanks with concrete. High level waste may be removed for disposal.

(9) Accidental release of contamination to vacuum systems.

The vacuum system is enclosed in a containment box with an absolute filter in the vacuum pump inlet. It is likely the contaminated material will be caught in the filter, which can be changed remotely in Cell 1.

These accidents, with other slight variations, have been applied to the various steps in the fuel processing system. Under no conceivable condition was the maximum emergency dose to occupational workers exceeded. In all cases, it was proven that the components will maintain their structural integrity if gas becomes accidentally trapped within the system.

The analysis will be reviewed and changed accordingly as the step-by-step procedures of the system are finalized. A final failure analysis report, based on these procedures, will be prepared.

12. Fabrication of Strontium Titanate Pellets*

An investigation has been conducted to optimize the variables in fabricating strontium titanate pellets. The variables investigated were the calcining schedule and ball milling time. The effect of controlling these variables is shown in Tables 2 and 3. The illustrations shown in Fig. 11 are sintered pellets of the material described in Tables 2 and 3.

Although the material used in the investigation was precipitated in two 1300-gram batches, it was later divided into four equal batches and processed as shown in Tables 2 and 3. Calcining was accomplished in aluminum oxide crucibles. The times at temperatures and rate of increase were found to be very important with calcining as well as in sintering. This rate should not exceed 150° C per hour.

As indicated in Tables 2 and 3, the higher calcining temperature combined with increased time and longer ball milling yielded the highest density pellets. A calcining temperature of ~1350° C for two hours with a four-hour ball milling will yield pellets of ~95% theoretical density. As shown later, this schedule is for processing a batch of 315 grams of strontium carbonate-titanium oxide precipitate blend and

*W. Precht

TABLE 2

Effect of Calcine and Ball Mill Variations on Sintered Density

Material and Impurity	Time: Calcine* (hr)	Time: Ball Mill (hr)	Sample Number	Pressed: Weight (gm)	Pressed: Height (in.)	Pressed: Diameter (in.)	Sintered: Weight (gm)	Sintered: Height (in.)	Sintered: Diameter (in.)	Density (%)
$SrTiO_3$ + 11 wt % Ca^{++}			1A	73.90	0.740	1.766	73.60	0.621	1.460	88.16
		2	1B	73.90	0.740	1.766	73.60	0.615	1.460	88.98
			1C	73.92	0.740	1.766	73.65	0.615	1.460	88.98
	1									
			2A	73.85	0.740	1.766	73.60	0.610	1.450	91.02
		4	2B	73.87	0.740	1.766	73.45	0.610	1.450	90.82
			2C	73.90	0.740	1.766	73.70	0.609	1.450	91.43
$SrTiO_3$ + 11 wt % Ca^{++}			3A	73.30	0.666	1.766	73.10	0.573	1.505	88.57
		2	3B	73.45	0.666	1.766	73.15	0.576	1.505	88.16
			3C	73.35	0.666	1.766	73.05	0.577	1.505	87.76
	2									
			4A	73.45	0.714	1.766	73.05	0.601	1.477	87.96
		4	4B	72.90	0.707	1.766	72.50	0.596	1.477	87.96
			4C	72.90	0.707	1.766	72.50	0.597	1.477	87.96

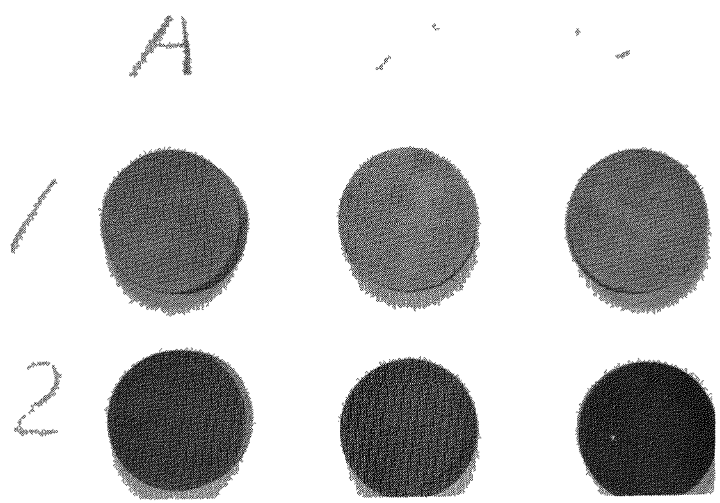
*Calcining temperature--1250° C

TABLE 3

Effect of Calcine and Ball Mill Variation on Sintered Density

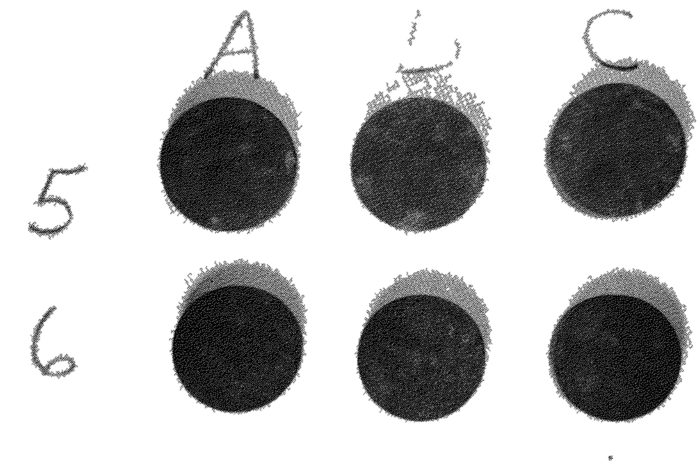
Material and Impurity	Time: Calcine* (hr)	Time: Ball Mill (hr)	Sample Number	Pressed: Weight (gm)	Pressed: Height (in.)	Pressed: Diameter (in.)	Sintered: Weight (gm)	Sintered: Height (in.)	Sintered: Diameter (in.)	Density (%)
$SrTiO_3$ + 11 wt % Ca^{++}	1	2	5A	73.40	0.638	1.766	72.45	0.573	1.593	79.2
			5B	73.40	0.637	1.766	72.50	0.573	1.590	79.2
			5C	73.40	0.637	1.766	72.60	0.573	1.600	77.9
		4	6A	73.40	0.635	1.766	72.90	0.554	1.514	91.43
			6B	73.40	0.637	1.766	72.90	0.554	1.516	90.20
			6C	73.40	0.636	1.766	72.90	0.558	1.514	90.82
$SrTiO_3$ + 11 wt % Ca^{++}	2	2	7A	73.40	0.678	1.766	73.15	0.569	1.477	93.06
			7B	73.40	0.678	1.766	73.15	0.569	1.477	93.06
			7C	73.40	0.678	1.766	73.10	0.569	1.477	92.86
		4	8A	73.40	0.703	1.766	73.05	0.578	1.453	95.3
			8B	73.40	0.701	1.766	73.05	0.576	1.453	95.5
			8C	73.40	0.700	1.766	72.95	0.576	1.453	95.5

*Calcining temperature--1350° C



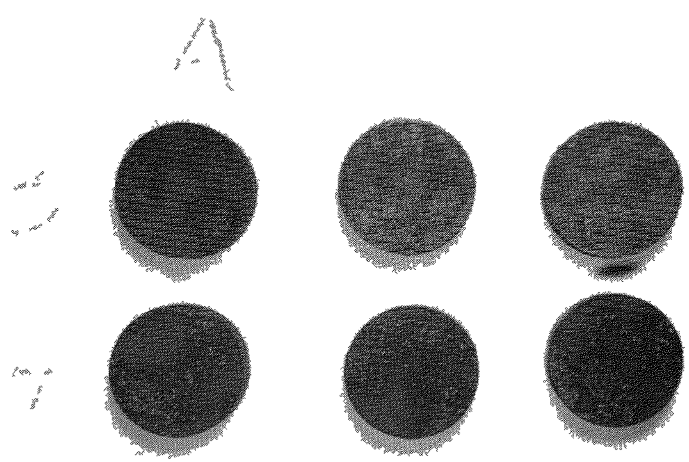
a. $SrTiO_3$ Pellets

1250° C calcine, 1 hr
Row 1, 2-hr milling
Row 2, 4-hr milling



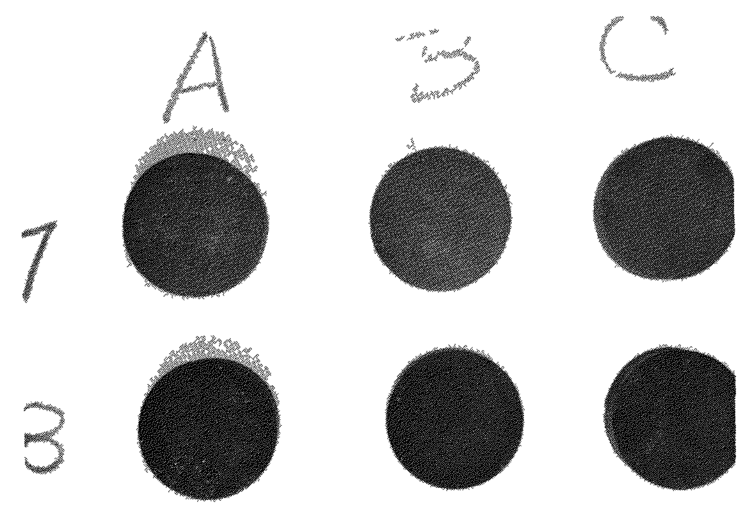
c. $SrTiO_3$ Pellets

1350° C calcine, 1 hr
Row 5, 2-hr milling
Row 6, 4-hr milling



b. $SrTiO_3$ Pellets

1250° C calcine, 2 hr
Row 3, 2-hr milling
Row 4, 4-hr milling



d. $SrTiO_3$ Pellets

1350° C calcine, 2 hr
Row 7, 2-hr milling
Row 8, 4-hr milling

Fig. 11 Strontium Pellets

is subject to change when larger batches are used.

The inconsistency in trying to increase density by increased calcining time indicated the importance of controlling the temperature rate increase during calcining (Tables 2 and 3). The rate of temperature increase (300° C per hour), for the material calcinated at 1350° C for one hour and ball milled for two hours, was twice that of the other batches shown in Tables 2 and 3. As a result, this material shows a significantly lower final density. It is recommended that the rate of temperature increase for calcining as well as sintering be controlled so it does not exceed 150° C per hour.

After wet ball milling, each batch was filtered through a stainless steel filter that had a pore diameter of 20 microns. Early analysis of the filtrate using this filter showed a strontium content of 8 ppm. During the runs shown in Tables 2 and 3, however, the filter was mechanically and chemically cleaned. After cleaning the filter, the filtrate showed a strontium content of 200 ppm, indicating that the clogged filter resulted in a finer pore size. It was therefore recommended that a filter with a pore size of ~5 or 10 microns be investigated so as to maintain a lower strontium content in the filtrate.

13. Limit of Scale Up

The pelletizing process described previously was established to process 315 grams of carbonate-titanium oxide blend. Thermal decomposition and reaction of such a blend yields 250 grams of strontium titanate which is calculated to be equivalent to ~5000 curies. An effort was made to determine the amount to which the batch size could be scaled, using the same equipment as for the established process.

A single fuel capsule will contain 950 grams of strontium titanate (17,500 curies). The first attempt to scale up was to process a batch equal to 950 grams of strontium titanate. The results of this attempt are shown in Table 4. The low final density of these samples is attributed to lack of proper ball milling and the fact that approximately 50 grams of material were hand crushed and added to the blend after the ball milling. The other samples with densities of ~90% theoretical and shown in Table 4 were equivalent to 610 grams of $Sr\text{-}Co_3 + TiO_2$ or ~9500 curies. Samples 8A, 8B and 8C of Table 4 are representative of final pellet density of ~95% theoretical.

As indicated in Table 4, the batch size capacity for the equipment now in use is approximately 250 grams of strontium titanate (~5000 curies when using $Sr\text{-}90TiO_3$). The data in Table 4 show that when larger batches are processed with this equipment, the ball milling time increases. A 250-gram batch can yield pellets of ~95% theoretical

TABLE 4

Effect of Quantity Scale Up on Final Density and Appearance

Equivalent Curies per Lot	Carbonate-TiO_2 Blend Weight (gm)	Weight After Calcining (gm)	Ball Mill Time (hr)	No. Pellets per Lot	Average Final Density (%)	Remarks
4,500	317	250	4	3	95	Good appearance. All pellets sound
9,500	610	479	8	6	89	Good appearance. Two pellets. slightly laminated
78,000	1220	960	4	12	80	Poor samples. All pellets laminated

density with a four-hour ball mill. When a 479-gram batch was ball milled for twice this length of time, or eight hours, the average density was 89% theoretical. It is conceivable that the increased ball milling time required for the larger batch would yield a product similar to the smaller one. However, the time required would not make this worth while.

14. Al_2O_3 Filter Crucible

An aluminum oxide (Norton No. 23209) crucible was evaluated for use as both a filter and calcining container. The initial precipitate was first filtered through the crucible with the aid of a partial vacuum. The crucible and material were then dried in an oven and placed in a furnace for calcining. The rate of temperature increase and time at calcining temperature were controlled so they equalled all previous runs. The result, however, showed excessive reaction between the calcined material and the crucible. The reaction product was a white powder that was found on the surface where the calcined powders were in contact with the crucible. The crucible also showed excessive swelling around the bottom portion where most of the precipitated material had settled. This section of the crucible was also severely cracked. After standing in air overnight, the white powder appeared yellow in color and was hygroscopic. No effort was made to identify this reaction product. The calcined titanate also showed a higher degree of sintering than was expected. It may have been caused by the weight of the powder at the beginning of the run or possibly by furnace override. The calcining run following this test also showed a slight indication of a similar reaction even though the normal calcining crucibles had been used. The reaction at the interface of the crucibles and $SrTiO_3$ was considerably less and the increased sintering effect was not as severe as in the filter crucible.

Because of the results obtained when an aluminum oxide filter crucible was used, and because 2.3% strontium was found in the filtrate, it was decided that in the future a stainless steel filter would be employed. Calcining was to be accomplished in zirconium oxide crucibles.

C. NUCLEAR CHEMISTRY AUDIT--SUMMARY OF RESULTS*

In general, the following major changes or simplifications have been recommended in the Strontium-90 fuel process:

(1) Modify the shipping cask unloading procedure to ensure dissolution of the $SrCO_3$ and decontamination of the cask

*J. L. Bloom

with a minimum volume of acid and water. (Changes in this area are based on operating experience obtained recently at Hanford and ORNL on the HAPO-2 cask.)

(2) Eliminate the "slurry tank," filter aid filter and attendant piping. (This change has been made possible by developments at Hanford and ORNL which permit the $SrCO_3$ to be loaded into the shipping cask without the use of a filter aid.)

(3) Eliminate the caustic scrubbers in vent and vacuum manifolds. (Investigation of anticipated HNO_3 concentrations in off-gas streams indicated that the gases may be discharged to the air handling system without scrubbing. Elimination of the scrubbers markedly simplifies the venting of the various tanks, precludes any possibility of inadvertent mixing of the caustic solutions with process streams and reduces the waste disposal problem.)

(4) Possible replacement of the ball mill for attrition of the calcination product by a heavy-duty Waring Blendor. (Work at ORNL has demonstrated the efficacy of the Blendor in preparing the calcined material for cold pressing. Its use eliminated the tedious ball mill operation and a subsequent filtration and drying step. Experiments are being conducted to determine the optimum Blendor operation.)

(5) Use of an alternate filtration step after precipitation whereby either a stainless steel filter or a porous ceramic filter can be employed in the same apparatus. (Since the ceramic filter can be used as a crucible for the calcining step and thus eliminates a powder transfer operation, it is preferred from an operational point of view. However, because of the danger of possible chemical reaction with the ceramic filter, it is desirable to be able to substitute the stainless steel filter, if required, during actual operation.)

(6) Reduction of the calcination temperature from 1350° to 1150° C. (This will extend the life of the furnace and reduce the possibility of crucible deterioration or breakage at the expense of a slight possible reduction in ultimate pellet density. Since the specific activity of the Sr-90 produced at Hanford far exceeds the value anticipated at the inception of this program, nominal reduction in the pellet density does not jeopardize attainment of the design power density. In fact, it will be necessary to employ inert spacers to fill the void volume of each fuel capsule.)

D. FUEL PROCESS EQUIPMENT

Manufacturing was initiated for many of the equipment items required for the Strontium-90 fuel process in Cells 1 and 2 of the Quehanna Hot Cell Facility. Some of the items follow.

1. Containment Box for Cell No. 2

The latest engineering design for the containment box which included provisions for the "alpha can" transfer mechanism and the top transfer box were approved for fabrication. The interior walls of the box are constructed of stainless steel, while the exterior structural members are made of carbon steel. An internal traversing bridge crane is provided to position and remove heavy equipment remotely.

2. Stainless Steel Tanks

Vendor drawings were approved for fabrication of the stainless steel storage tanks handling nitric acid solutions. These included slurry, metering, fuel storage and waste storage tanks.

3. Electric Furnace

A globar electric furnace for calcining and sintering was selected from several different makes of furnaces. Special features such as replaceable globars and water cooling were included in the small furnace to reduce operating temperatures in Cell 2 and increase furnace operating life.

4. Jacketed Precipitation Vessel

Vendor drawings were approved and fabrication was initiated for the jacketed precipitation vessel. Special accessories for this vessel included internal heating, water cooling, electric mixing and numerous special inlets.

5. Hydraulic Pelleting Press

Engineering drawings to be used in fabricating the hydraulic press required to compact strontium titanate pellets were released. Hydraulic equipment and raw materials were procured to manufacture the special frame and housing. This press is designed for hot cell operation with remote handling of powder, pellets and die components.

6. Automatic Heliarc Welder

Manufacturing of the heliarc welder for the Strontium-90 fuel capsules was initiated during this quarter. This unit is similar to the previous SNAP capsule welders and also the Strontium-90 ORNL welder. The basic difference is the size of the capsules that can be welded. Like the previous welders, the welding chamber is flooded with inert gas and may be pressurized to several atmospheres for capsule leak test inspection after welding.

7. Electrically Driven Ball Mill

Vendor drawings of a modified standard ball mill were approved for fabrication. Modifications were suggested concerning the release of heat from the unit and several fins were added to the body to increase thermal convection. The complete assembly is operated by an electrically driven motor.

8. Miscellaneous Hardware

Stainless steel tubing and fittings were ordered for the chemical processes required in the Quehanna hot cells. Manually operated for stainless steel valves were procured to provide proper control for the process flow streams. Both the ball-type and throttling valves were ordered for use with precipitate slurry streams and clear filtrate solutions. Installation plumbing will be performed in Quehanna.

V. REFERENCES

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3. Crompton, C. E., Trip Report, March 22, 1961.

4. "Radioactive Materials Laboratory Safety Report--Martin Nuclear Facility, Quehanna Site," The Martin Company, MND-2410, September 1960.