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Argonne National Laboratory

ENGINEERING DEVELOPMENT OF FLUID-BED FLUORIDE VOLATILITY PROCESSES

Part 5. Description of a Pilot-scale Facility for Uranium Dioxide-Plutonium Dioxide Processing Studies

by

G. J. Vogel, E. L. Carls, and W. J. Mecham

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Part 1: Bench-scale Investigation of a Process for Zirconium-Uranium Alloy Fuel, by D. Ramaswami, N. M. Levitz, J. T. Holmes, and A. A. Jonke (ANL-6829)

Part 2: Bench-scale Investigation of a Process for Aluminum-Uranium Alloy and Stainless Steel-Cermet Fuels, by D. Ramaswami, N. M. Levitz, and A. A. Jonke (ANL-6830)

Part 3: Fluid-bed Fluorination of Uranium Dioxide Fuel Pellets, by L. J. Anastasia, J. D. Gabor, and W. J. Mecham (ANL-6898)

Part 4: Fluidized-packed Beds: Studies of Heat Transfer, Solids-Gas Mixing, and Elutriation, by J. D. Gabor and W. J. Mecham (ANL-6859)

Part 6: Preparation of Dense Uranium Dioxide Particles from Uranium Hexafluoride in a Fluidized Bed, by I. E. Knudsen, N. M. Levitz, and A. A. Jonke (ANL-6902)

Part 7: The Corrosion of Nickel in Process Environments, by A. A. Chilenskas and G. E. Gunderson (ANL-6979)

Part 8: Pilot Plant Development of a Process for Uranium Alloy Fuels, by J. T. Holmes, H. L. Stethers, and J. J. Barghusen (ANL-6973)

Part 9: Computer Programs for Alloy-fuel Process Calculations, by J. T. Holmes and D. Ramaswami (ANL-6992)

Part 10: Bench-scale Studies on Irradiated Highly-enriched Uranium Alloy Fuels, by A. A. Chilenskas and K. S. Turner (ANL-6994)

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES

ENGINEERING DEVELOPMENT OF FLUID-BED FLUORIDE VOLATILITY PROCESSES

Part 5. Description of a Pilot-scale Facility for Uranium Dioxide-Plutonium Dioxide Processing Studies

SUMMARY

Fluid-bed, fluoride volatility processes are being developed for the recovery of uranium and plutonium from spent nuclear fuels of power reactors. In these processes, chemical separations are achieved by formation of the volatile hexafluoride compounds of uranium and plutonium.

A pilot plant has been constructed at Argonne National Laboratory for studying the following two major process steps, as applied to uranium dioxide-plutonium dioxide fuel:

(1) The fluorination of uranium and plutonium oxides to hexafluorides.
(2) The conversion of uranium and plutonium hexafluorides to dense dioxide mixtures suitable for refabrication of fuel.

A major objective is the demonstration of optimum process conditions for these two steps for synthetic reactor fuel compositions, including those containing mixtures of inactive fission products. The conversion unit can also be used for separating uranium and plutonium by selective reduction of plutonium hexafluoride from hexafluoride mixtures. The nominal throughput of the pilot plant is 10 kg/day. Current experimental work involves the testing of equipment with uranium only. After successful demonstration runs, work with plutonium will commence.

UO_2-PuO_2 pellets, immersed in a fluidized bed of alumina, will constitute the charge to the fluorinator. The hexafluorides can be formed by either simultaneous oxidation (which disintegrates the pellets) and fluorination, or by batch oxidation followed by fluorination. The reactor walls at the fluorination zone are kept at a constant temperature of about 500°C by externally-attached cooling coils through which a two-phase, air-water mixture is passed. Process gas from the fluorination zone passes through a disengaging section and a cooled porous-metal filter at the top of the fluorinator unit. The product hexafluorides are condensed out of the gas stream in a condenser cooled to -70°C by a circulating heat-transfer liquid. The fluorinator is made of nickel. Monel is also used for equipment and components of the fluorinator system that operate at lower temperatures. Waste streams in this step are the solid nonvolatile residues in the fluorinator and the noncondensible gases leaving the condenser. The fluoride content

of the waste gas is removed from the gas stream by a bed of activated alumina before the waste gas is sent to the exhaust-air treatment.

Hexafluorides are converted to dioxides by reaction of the hexafluoride vapor with steam and hydrogen at about 600°C in a fluid bed of the product dioxide. The product is recovered as fluidizable particles and drained from the Inconel converter. Effluent gaseous wastes are filtered and water-scrubbed before the gas is sent to the exhaust-air treatment.

The major process equipment is mounted in a large glovebox for containment of the toxic, alpha-emitting plutonium. Auxiliary equipment is housed in a smaller glovebox. An air purge maintains the alpha box under pressure negative to that in the personnel area. The chief hazard will be the possibility of volatile plutonium hexafluoride being released from the process equipment. To remove such material from the air exhausted from the alpha boxes, this air is passed through water scrubbers to hydrolyze any hexafluoride to nonvolatile material; multiple, high-efficiency filters then remove solids from the exhaust air before final venting to the atmosphere.

Special features of the installation concern the adaptation of equipment, gas supplies, utilities, material handling, and instrumentation and control to alpha-box operation. A remote panelboard is provided for instruments measuring temperature, pressure, gas flow and composition, and product collection. Control during runs, including valve operation, is also carried on from the panelboard. Material movement and equipment maintenance are done manually by special glovebox techniques.

I. INTRODUCTION

In the United States government-operated reprocessing plants and in the first United States commercial plant, now being constructed by Nuclear Fuel Services, Inc., uranium and plutonium (if present) are recovered by an aqueous, solvent-extraction process. Nonaqueous processing methods - volatility, pyrometallurgical, and pyrochemical - are being studied to develop a more economical process. The status of these processes is reviewed in the Reactor Fuel Processing quarterly magazine.[1]

In volatility processes, separation of the uranium from plutonium and also the separation of uranium and plutonium from fission products and matrix metals (if present) are accomplished by means of differences in volatilities of the halides. A high decontamination from fission products comparable to that obtained by solvent-extraction processing is desired. Fluoride systems have been studied mainly, although some processes have been based on the chlorides and some data have been obtained on iodides and bromides. A general review of the fluoride volatility processes is available.[2] One example of a fluoride process under investigation involves the dissolution of uranium and the metal matrix in fused salt with the addition of hydrogen fluoride, followed by the volatilization of the uranium as uranium hexafluoride with the addition of fluorine.[3] In another variation of the fluoride process, fuel elements of uranium in either a zirconium or aluminum metal matrix are reacted with gaseous hydrogen chloride in a fluidized bed to form the matrix metal chloride, which is volatile, after which the uranium remaining in the fluidized bed is volatilized by reaction with fluorine.[4,5,6] Considerable experimental data, including distribution of fission products, are available on processing irradiated metallic uranium fuel by liquid bromine trifluoride.[7] Volatile chlorides of uranium serve as the basis for a Belgian,[8,9] a German,[10] and a Japanese[11] separation process under development.

In the fluid-bed, fluoride volatility process discussed here, fluorides are formed by reaction with elemental fluorine, the reaction taking place in a fluidized bed of inert material. Because of their extensive use in current and planned reactors, oxide fuels are receiving emphasis in the present developments at Argonne National Laboratory (ANL). However, metal and carbide fuels can be processed by fluoride volatility methods after suitable head-end steps. Recent ANL reports have summarized laboratory experimental investigations and engineering-scale development of the fluid-bed, fluoride volatility techniques directed toward the processing of uranium oxide fuels.[12,13,14]

Laboratory studies pertinent to fluid-bed, fluoride volatility processing of uranium dioxide-plutonium dioxide have been published as part of the program.[15-20] The emphasis in these studies on plutonium recovery

arises from an interest to develop a more economical processing method applicable to the commercial, thermal power reactors fueled with low-enrichment uranium, and to the fast breeder power reactors, which utilize plutonium in uranium cores (up to about 20%) and also uranium blankets in which plutonium is bred. This plutonium must be recovered to provide an economical fuel cycle.

Figure 1 illustrates several conceptual fuel cycles involving volatility reprocessing for a thermal reactor. Partial or complete decontamination may be employed, and the products may be in either oxide or fluoride form. Figure 2 shows a conceptual flowsheet for the principal steps of a fluid-bed, fluoride volatility process in which the plutonium is not separated and a mixed plutonium oxide-uranium oxide fuel is produced for recycle to the reactor. The process steps shown are: (1) removal of Zircaloy cladding by volatilization of zirconium tetrachloride; (2) fluorination, to produce the volatile hexafluorides of uranium and plutonium; (3) fractional distillation, to separate the hexafluorides and remove fission products; (4) conversion of hexafluorides back to dioxides, for convenient storage or for recycle directly to the reactor. The plutonium recycle flowsheet can be applied to fast-reactor fuels and to thermal-reactor fuels in which plutonium recycle is employed. Figures 1 and 2 are illustrative only and do not summarize the continuing work in flowsheet development at ANL.

Figure 3 is a perspective view of the major chemical process units of the ANL pilot plant (the fluorinator and the converter) as they are installed in a large glovebox (alpha box), which provides the alpha containment of the plutonium-bearing fuel. The fluorinator and the condensers for collecting the volatile uranium and plutonium hexafluorides are shown at the right in the figure. The converter unit, in which hexafluorides are converted to dioxide, is shown at the left. The separation of plutonium from uranium by the selective conversion of plutonium hexafluoride (vapor) to plutonium tetrafluoride (solid) by thermal decomposition will also be studied in the converter unit. The purpose of the present installation is to study the pilot-scale behavior of the uranium and plutonium reactions in the absence, and in the presence, of synthetic mixtures of inactive fission products. (Tracer levels of radioactive fission products may be used also.) The fission-product separation step (including distillation) and irradiated fuel processing are to be studied in other facilities.

The present report describes the design of a pilot-scale (~10 kg/day) uranium dioxide-plutonium oxide processing facility for those interested in evaluating the equipment aspects of potential new reactor-fuel processing methods in general, and fluid-bed, fluoride volatility processes in particular. The present pilot-scale facility, the contained equipment, and the process steps are described in the other sections of this report. Because of the experimental nature of this facility, certain safety features incorporated into the design might not be required in a future full-scale facility.

Figure 1

CONCEPTUAL FUEL CYCLE FOR OXIDE-FUELED REACTORS

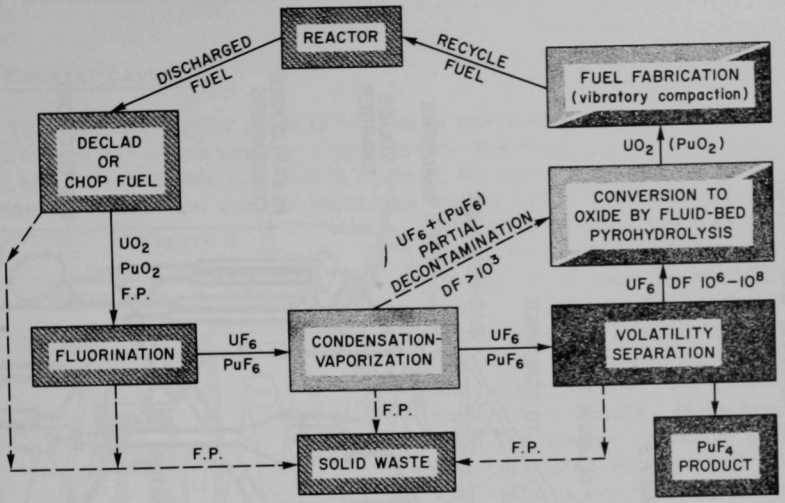


Figure 2

CONCEPTUAL FLOWSHEET FOR A FLUID-BED, FLUORIDE VOLATILITY PROCESS

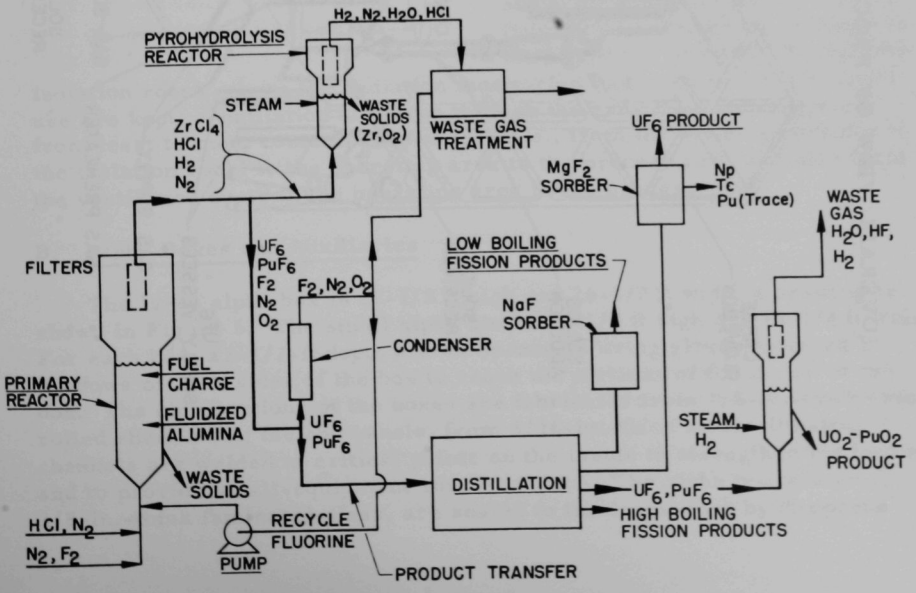
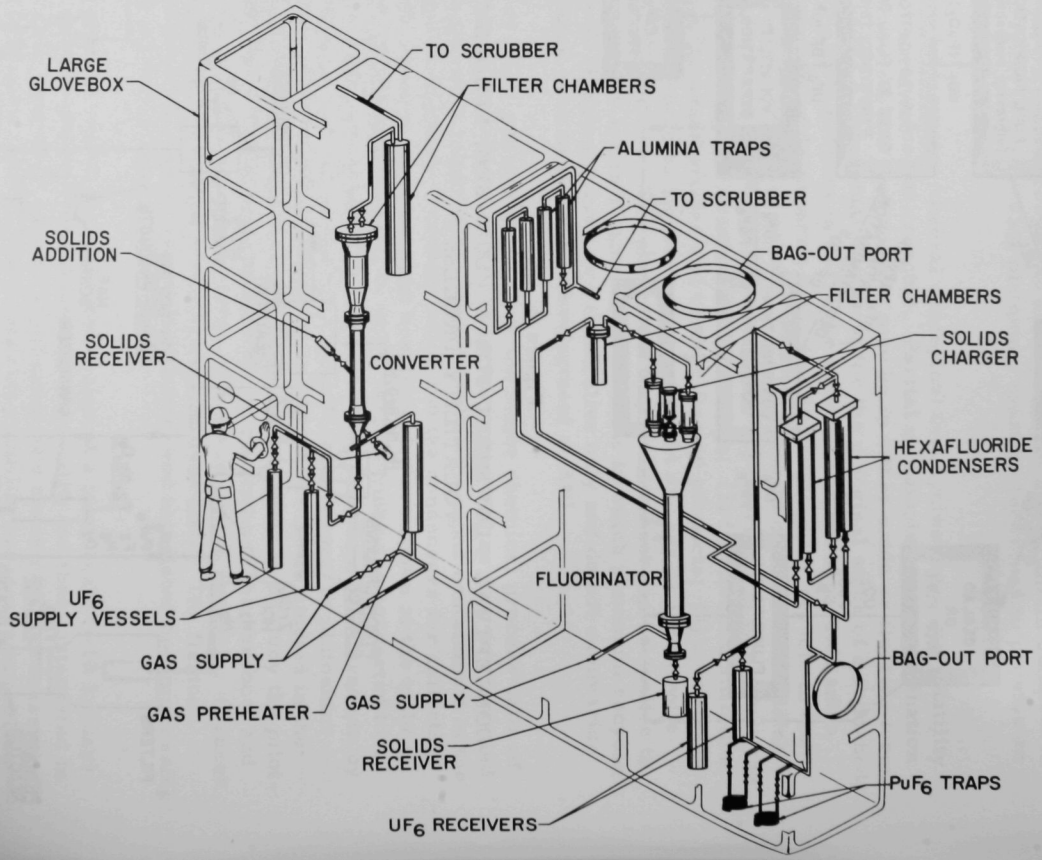


Figure 3

OVERALL VIEW OF PROCESS EQUIPMENT IN A LARGE GLOVEBOX



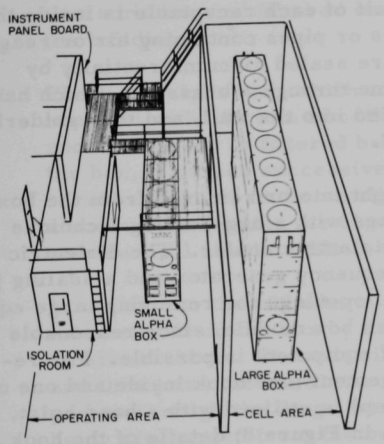
II. GENERAL FACILITIES AND ALPHA-CONTAINMENT FEATURES

A. General Layout

The plutonium pilot plant is housed in one room, 28 ft x 38 ft x 33 ft high, except for some auxiliary equipment--the final scrubber, pump, hold tank, and filters of the ventilation system, the compressor unit of the refrigeration system, and the inert-gas supply cylinders. The room layout in Figure 4 shows the location of the alpha boxes and the panelboard. Plutonium processing equipment is housed inside the large alpha box, which is inside a concrete cell in the room. Auxiliary equipment that might contain plutonium is housed in a smaller alpha box in the room. Fluorine cylinders are stored in a ventilated enclosure in the room. The room is serviced by an overhead crane having a rail elevation of 25 ft.

Figure 4

ROOM LAYOUT OF PILOT-SCALE FACILITY



The panelboard, containing the remote process control and recording instruments, overlooks the operating area from its position above the isolation room. Access from the main building corridor to the facility is provided through the isolation room, where the radiation monitoring instruments for personnel use are kept. Ventilation air flows through the area once (not recycled) from least to most contaminated areas: i.e., from the building corridor to the isolation room to the operating area to the process cell, and also from the ventilation ducts in the operating area to the process cell.

B. Alpha Boxes and Auxiliaries

The large alpha box is 17-1/3 ft high and 26-1/2 ft wide; a drawing is shown in Figure 5. The small alpha box is 10-1/2 ft high and 13-1/4 ft wide. For each box, a 3-1/2-ft depth allows operators using gloves mounted in windows of both sides of the box to reach all portions of the inside of the box. The side sections of the boxes are fabricated from 1/8-in.-thick cold-rolled sheet steel; the end panels, from 3/16-in.-thick steel. Unistrut channels are welded at critical points on the inside to strengthen the boxes and to provide small-equipment support points. The alpha box windows, 3/8-in.-thick laminated glass, are sealed to the framework by Neoprene

channel stripping and a room-temperature-setting plastic sealant. Usually, two pairs of rubber gloves are mounted in each window. Inside and outside metal surfaces of the boxes are painted with chemical resistant finishes. The modular construction of these gloveboxes is part of the original ANL design.

Airtight seals are required for the electrical cable, thermocouple leads, and air and reagent gas lines that pass through the box walls. The electrical lines through the box walls are sealed by inserting a special (Mineral Insulated) electrical cable through a hole in a cap screw and soldering the outer sheath of the cable to the screw. Before the soldering, the screw is placed in a hole in the wall, gasketed, and held by a jam nut as shown in Figure 6. Thermocouple feedthroughs are 48-point, pressure-tight, wall-mounted receptacles; one half of each receptacle is inside the box, the other half outside. Metal tubes or pipes containing air or reagent gases and passing through a box wall are sealed circumferentially by welding to the wall or by passing the line through a brass nut, which has been screwed into a mating fitting welded into the wall, and then soldering the line to the nut as shown in Figure 7.

Equipment and materials are brought into or removed from the boxes through 8-, 22-, or 30-in.-diam. openings with a bag-sealing technique utilizing polyvinylchloride bags and a dielectric sealer. The dielectric sealing unit comprises a 4-kW, high-frequency generator and a sealing press with a 37-in. dielectric bar. The 30-in. openings for removing large equipment items are located at the top of each box module, since reasonable vertical, but little lateral, movement of equipment is possible. For removing heavy equipment, a hook arrangement (one hook inside and one outside the bag and held together by a clamp) is utilized with a hand hoist. Key steps of the bagout operation are shown in Figure 8; details of the hook are shown in Figure 9. In addition to the above openings, 4-in.-diam. sphincter-type openings are available for adding items of small size. These items are placed in a standard cylindrical cardboard or metal carton and pushed through the sphincter opening into the alpha box by another carton, which is then left in place to maintain box closure.

Hydraulically operated lifts elevate personnel to working positions on the face of the larger glovebox. Two men can stand on the lift platform, which is 7 ft long and 2 ft wide. After release of a spring-loaded pin, which mechanically locks the lift in position, the vertical movement is started by pushing a constant-pressure push button mounted on the movable platform. The rate of vertical movement is approximately 20 ft/min. For lateral movement, foot-actuated brakes on the wheels must be released before the lifts can be pushed manually. Each lift has guard rails on the front, guard chains on the back, and kick plates on the platform. The occupant of the lift is separated from the roller chains that move the platform by a 6-ft-high partition. The design features of the lift are shown in Figure 10.

C. Ventilation System

The room-ventilation scheme provides that ventilation air flows toward, and is exhausted from, the area with the highest possibility of contamination. Air enters the room from two inlets on the room wall and from the building corridor through louvers in the entrance doors. The ventilation air then follows one of the following four paths through the room and boxes, as shown in Figure 11:

1. The small alpha box, which is not expected to be highly contaminated, is ventilated with 150 cfm of air from the room. This air is passed through absolute filters (at the box) before entering, and on leaving, the box. The exit air is humidified in the ventilation scrubber and filtered once again before being discharged from the building.

2. The large alpha box, which is treated as highly contaminated, is supplied with 600 cfm of cell air that has been pulled from the room. The air is filtered before entering this box. On leaving the box, it passes successively through the small exhaust-air scrubber (No. 1) in the small box, three absolute filters in series, a booster ventilation air blower, the large ventilation-air scrubber, and the final absolute filter.

3. In addition to the 600 cfm of ventilation air, 450 cfm of air can be exhausted from the box by an alternate route, which bypasses the small scrubber. The exit air passes through a filter at the box, and then joins the ventilation air exhausted from the room, which passes through the large ventilation scrubber and filter before being discharged from the building.

4. Approximately 100 cfm of air is exhausted from the fluorine cylinder storage cabinet. The air is joined by the exhaust air from the small box and is scrubbed once and filtered.

All air that has entered the room is water-scrubbed (humidified) and filtered at least once before being exhausted from the building; this treatment is performed twice (in sequence) for air exhausted from the box having a higher activity level. Thus any plutonium hexafluoride or uranium hexafluoride that might have contaminated the box atmosphere can be converted to a solid form that can be filtered. A small scrubber, located in the small box, scrubs exhaust ventilation air from the large box; a large scrubber, located outside the room, scrubs all ventilation air exhausted from the room area, including that from the small scrubber. All air exhausted to the building stack is monitored for radioactivity, and the amount of activity is recorded.

Process off-gas from the fluorinator and converter units is exhausted with alpha-box air after being given special treatment to remove radioactive and fluoride components. Fluorinator off-gas (after leaving the hexafluoride condenser and passing through the chemical traps to remove uranium, plutonium, and fluorine) consists of oxygen, nitrogen, or inert gases, with a total flow rate of 1 cfm or less. This process gas is mixed with large-box ventilation air before passing to the scrubber (No. 1 in the small box); the final treatment of this air is as described in (2) on the previous page. Converter off-gas, containing water, hydrogen fluoride, hydrogen, and nitrogen, in a total flow stream of about 1/2 cfm, passes through the converter off-gas process scrubber (No. 2 in the small box) to remove hydrogen fluoride. This effluent gas is sent to the scrubber (No. 1) handling the exhaust air (600 cfm) from the large box.

The large scrubber, in which is scrubbed all ventilation air exhausted from the alpha facility, is housed in the fan loft. Details of the unit are shown in Figure 12. The scrubber (made of stainless steel) is horizontal, approximately 13 ft long x 3-1/2 ft square, with two banks of four spray nozzles each, a demister section, and a heating section. The rated design capacity is 4000 cfm.

The small scrubber (No. 1 in the small box), which treats the exhaust ventilation air from the large box air and the process off-gas, is a vertical, spray-nozzle type. The scrubber system includes a demister section for removing entrained water droplets, and a steam-heated coil for ensuring that no droplets (which could cause filter plugging) remain in the scrubbed stream. Air and any particulate solids from the heating section pass through three absolute filters in series. These filters are standard units, 24 in. square and 12 in. deep. Layouts of the scrubber unit and filter stacks are shown in Figure 13. The scrubber is designed to handle a normal air flow of 600 cfm. Water for scrubber spray is pumped by a vertically-coupled pump with a 3/4-in. discharge and a 1-in. suction. The liquid discharging from the pump passes through a filter and a flowmeter before it reaches the spray nozzles. A tank is available for holding steam condensate from the air heater. This condensate is added to the water hold tank to compensate for evaporation losses. Liquid level can be determined visually in the scrubber and in the hold tank. The size of the largest bag-out ring (30 in.) and the height of the alpha box (10.5 ft) determine the maximum size of the scrubber components. The tower is 18 in. in diameter and 6 ft high; the pump tank is 26 in. in diameter and 2 ft high. Mild steel is the material of construction.

The converter process scrubber (No. 2) is described in Section IV-B of this report.

Pressure in the alpha boxes is maintained at approximately minus 0.5 in. (water gauge) by adjusting manually the damper in the ventilation ducts.

The ventilation flow rates are monitored by an orifice-manometer system. The box pressures are recorded at the panelboard, and a high pressure in either alpha box will activate an alarm circuit. In the case of the large box, the alarm circuit can also automatically activate an air-controlled valve, which opens a damper to provide an additional route for exhaust. In the case of the small box, a damper must be opened manually for increased flow of purge air.

D. Services and Process Gas Supplies

Process gas supplies and services provided to the alpha boxes include fluorine, nitrogen, oxygen or air, hydrogen, vacuum, instrument air, electricity, and a circulating liquid for cooling and heating the condensers. Also provided at different room locations are steam, air, water supply and drains, electricity, and the gas mixture for the alpha-counter probes.

1. Gas Supplies

Eight lines carry process gas to the equipment in the large box. These lines, one each for fluorine, oxygen, and hydrogen, and five for nitrogen are shown in the diagram of the gas supply system in Figure 14. These lines originate at gas cylinders in the room or basement and pass through the small alpha box where the control valves and metering orifices are installed. With the exception of the fluorine line, fiberglass filters (Mine Safety Appliance-DZ-78001) capable of 99.7% removal of 0.3-micron particulate are mounted in each line at the entry to the large box. Also, pressure check valves are installed in all lines to minimize the effects of reverse gas surges.

a. Fluorine. Three fluorine supply cylinders are housed in a sheet-metal enclosure, together with surge vessels, a pressure-reducing valve, and a packed bed of sodium fluoride to remove any hydrogen fluoride from the fluorine. The enclosure is held at a negative pressure by an exhaust air duct connected to the ventilation scrubber. Slits on bottom of the doors of the enclosure provide for a ventilating air flow of approximately 100 cfm, which is approximately four times that suggested for fluorine storage facilities. Each cylinder, when connected for use, is separated from the others by a metal partition. Empty or spare cylinders are stored in the back part of the enclosure and are transferred manually to the operating position. Cylinder valves are manipulated from outside the enclosure by extension handles attached to a ratchet wrench.

The fluorine gas cylinders (6-lb capacity each, at 400 psi max) are connected via pressure-reducing and shut-off valves to two surge tanks (2.2-cu ft capacity each). In process runs, these tanks are filled intermittently to 100 psig and serve as the process-connected fluorine supply. This supply of fluorine is throttled to approximately 35 to 40 psia by a pressure-

reducing valve controlled from the panelboard. The hydrogen fluoride impurity is removed at this point by sorption on a bed of heated sodium fluoride. The supply line leaves the fluorine tank enclosure and passes into the small alpha box. Between the enclosure and the alpha box, the line is enclosed in a secondary pipe so that any gas release will be to the enclosure rather than the room. The fluorine flow-control valve, which is actuated manually by an operator or automatically by the temperature of the fluidized bed in the fluorinator, and a metering orifice are housed in the small alpha box. Also located in the small alpha box is an air-operated, shutoff valve controlled remotely from the panelboard. Besides the instantaneous measurement of flow by the orifice meter, the cumulative fluorine usage is obtained from the recorded pressure drop in the calibrated surge tanks. To remove fluorine in the lines, a connection is provided to a vacuum source and to a purge nitrogen supply through double-valving arrangements. The fluorine-containing line between the small and large alpha boxes is enclosed in a secondary pipe.

b. Nitrogen. The five nitrogen lines entering the large alpha box consist of two controlled and metered supplies to the fluorination and converter reactors; one supply for purging pressure transmitter lines; one for providing quench-cooling gas to the fluorinator filters; one supply of "pulse" nitrogen, which may be superimposed on the main stream of fluidizing gas; and one high-pressure supply of 100-psig nitrogen, for blowback of filters. The source for the last is a bank of two nitrogen cylinders with pressure regulators; for the other four, the source is a bank of 12 cylinders. In the small alpha box are the control and shutoff valves and the metering orifices. Solenoid and manual shutoff valves are provided in each line.

c. Oxygen. The oxygen supply system consists of a bank of two cylinders with associated pressure regulators. The path of the lines and the controls are similar to those in the nitrogen supply lines.

d. Hydrogen. The hydrogen supply system is also similar to the nitrogen system. A bank of four cylinders is provided.

2. Vacuum System

Gas evacuated from the fluorination process equipment is passed successively through a sodium fluoride trap and two chemical traps in series in the large alpha box. These traps, filled with activated alumina, remove fluorine and other fluorides. Another trap is used at the vacuum-pump inlet. Gas is discharged from the 38-cfm-capacity vacuum pump to the scrubber (No. 1). The vacuum system is shown as part of the ventilation system in Figure 11.

3. Process Instrumentation Air

Reduced-pressure, dry instrument air from the building header is

supplied to the panelboard for operation of the controllers and the control valves, and to each alpha box for operation of the transmitters and the air-operated, shutoff valves. A high-efficiency filter for solids removal is placed in each air supply line and in each instrument air signal line between the box and the panelboard. This precaution is observed since radioactive particles could contaminate a line opened in a maintenance operation.

4. Electricity

Electricity is supplied through a 100-kVA, three-phase, 60-cycle transformer from the building supply system. This transformer feeds two 200-Amp distribution boxes for the panelboard circuits. Miscellaneous room circuits, including room and cell lighting and convenience outlets, are fed from a 37-1/2 kVA, three-phase transformer.

5. Breathing Air

A special air-supply system is provided in connection with supply-air breathing masks for emergency use by operating personnel. Hose connections for four masks are made at the supply manifold in the corridor outside the operating room. Connection is made to the building air system, but two reserve supply tanks are employed for stand-by use. Each tank has a capacity of 400 cu ft of humidified breathing air.

6. Gas Supply for Alpha Radiation Monitors

A 90% argon-methane mixture (designated P-10) is supplied from a two-tank manifold in the basement. This mixture flows continuously through the alpha radiation detector probes of the proportional counters.

7. Air and Water Cooling Systems

Process equipment operating at temperatures below 100°C is cooled by air or distilled water. A two-phase air-water system is used for control of temperatures above 100°C, and is described in the fluorinator cooling Section III-B-1-d of this report. The air and water are discharged into the small exhaust-air scrubber (No. 1).

8. Process Refrigeration

Hexafluoride condensers and cold traps require low temperatures for efficiently collecting the hexafluorides, and in the present system, a -70°C condenser operation has been specified. In addition, the condensers have a heating cycle in which their temperature is raised to about +70° C to transfer the hexafluoride product. A circulating liquid system (see Figure 15) is provided for the cooling and heating of these condensers. A heat-transfer liquid, tetrachloroethylene, is circulated between the heating

and cooling sources in the room and the condensers and cold traps in the large alpha box. Manifold connections are made to separate heating and cooling sources for independent operation of the condensers and cold traps on either a heating or cooling cycle. Cooling is provided by a mechanical, two-stage, 10-hp refrigerator unit with a design capacity of 1500 BTU/hr at -70°C. Heating is by means of a thermostated electric heater. Total flow of the heat-transfer liquid is measured and recorded. The liquid lines that pass through the alpha-box wall are insulated and sealed by use of a urethane foam which is cast in place between the pipe lines and an outer support box. The support box is attached to the wall of the alpha box and is sealed with Thiokol cement.

III. FLUORINATOR SYSTEM

A. Introduction

A fluidized-bed reactor and associated equipment are installed in the large alpha box of the pilot-scale facility to convert uranium oxide-plutonium oxide fuel material into the volatile hexafluorides of uranium and plutonium. This fluorination step effects virtually complete separation of uranium and plutonium from those fission-product and other metals that form nonvolatile fluorides at the reaction temperature. Fluorine gas, diluted with an inert gas (e.g., nitrogen), serves as the fluorinating agent and the fluidizing gas. The fluorination reaction at about 500°C is rapid and readily controlled; fluidization aids in the removal of the heat of reaction and serves to maintain nearly uniform temperatures in the fluorination reaction zone.

A chemically inert granular solid, e.g., ceramic alumina, is the main fraction of the fluidized bed. In the fluorination of pellets of uranium dioxide, which themselves are too massive to be fluidized, the granular solid is fluidized in the voids between the pellets to aid in the heat removal and to dilute the finely-divided solids. The alumina will dilute any fission-product fluorides remaining after the fluorination of the uranium and plutonium and will serve as a vehicle for removing these fission products from the reactor.

Cylindrical-oxide pellets (1/2 in. x 1/2 in.) have been fluorinated by two operating methods. (An alternative procedure to the fluorination of pellets is to produce by chemical pulverization a powder which is then fluorinated batchwise or continuously.) In one method, called direct fluorination, the diluted fluorine is passed through the pellet bed in the lower portion of a bed of fluidized alumina.

The reaction of uranium dioxide (UO_2) proceeds in a two-step manner:

(1) $UO_2 + F_2 \longrightarrow UO_2F_2$;

(2) $UO_2F_2 + 2F_2 \longrightarrow UF_6 + O_2$.

The reaction of plutonium dioxide (PuO_2) proceeds mainly by the steps:

(3) $PuO_2 + 2F_2 \longrightarrow PuF_4 + O_2$;

(4) $PuF_4 + F_2 \rightleftharpoons PuF_6$.

In a second method, called two-zone fluorination, the pellets are again charged into the lower portion of a fluidized bed. The fluorine, instead of passing through the pellet bed, as in direct fluorination, enters the fluid

bed at a point above the pellet bed. Oxygen, diluted with nitrogen, passes through the pellet bed zone from below and forms triuranium octaoxide (U_3O_8) powder, which has a lower density than uranium dioxide and spalls off the pellet surfaces. The U_3O_8 powder moves to the fluidized-bed zone and reacts with the fluorine. If the oxidation is conducted separately from the fluorination in the lower zone, improved reaction control is achieved. The reactions involved are:

$$(5)\quad 3UO_2 + O_2 \longrightarrow U_3O_8;$$

$$(6)\quad U_3O_8 + 3F_2 \longrightarrow 3UO_2F_2 + O_2;$$

$$(7)\quad UO_2F_2 + 2F_2 \longrightarrow UF_6 + O_2.$$

Laboratory and engineering studies of the above reactions have been reviewed in previous ANL reports.[12-18] Details of the transport behavior of solids in two-zone operation are described in two reports in the series.[12,13]

The head-end process step of separating the oxide fuel from the cladding is being studied in other facilities and will be either a chemical, or a combination of mechanical and chemical, means. For elements clad with Zircaloy, a fluid-bed method of chemical decladding with gaseous hydrogen chloride is applicable using the methods described in other reports on zirconium-uranium alloy fuel processing.[4] For oxide elements clad with stainless steel, a mixture of HF and O_2 may be used to remove the metal and pulverize the oxide. Alternatively, mechanical chopping may be used to expose the oxide fuel for application of the oxidative method producing U_3O_8 powder (described above). The U_3O_8 powder is physically separable from the unreacted stainless steel in the oxidative method, which can be performed in a separate oxidation reactor or in the fluorination reactor itself if the two-zone operation is followed. Preliminary investigations of these cladding-separations schemes applied to simulated oxide fuel elements are described in another report in this series.[12]

A process material flow diagram for the fluorination of uranium dioxide pellet fuel is shown in Figure 16. The conceptual flowsheet of the two-zone fluorination shown in this figure is based on experimental studies with uranium (no plutonium) and is intended to serve only as an indication of the reference design throughput of the equipment and as an illustration of operations that may be performed in subsequent experimental studies.

B. Equipment and Instrumentation

The fluorination system of the pilot-scale process development facility includes gas supplies, a fluid-bed fluorinator with connected off-gas filters,

a secondary off-gas filter, hexafluoride condensers, a pump for circulating part or all of the fluorine-containing process off-gas, a gas analysis system, and chemical traps for removing fluorine and hexafluorides. In addition, receivers for process materials are included: UF_6-PuF_6 product receivers, PuF_6 cold traps for cleanup operations, and solid-waste receivers. All process components of the fluorinator are located in the large alpha box, except the pump for recirculating fluorine and the control valves for the gas supplies. To support the head of the fluorine pump mechanically, the head is mounted on a shielding wall and enclosed in its own alpha box. The control valves for the gas supplies are located in the small alpha box, as described in Section II-D of this report.

An illustration of process-stream flows for the fluorinator system is shown in Figure 16; the equipment and instrumentation schematic diagram is shown in Figure 17; the layout of the major equipment items and process piping is shown in Figure 18 (for clarity, several valves are shown with connecting lines omitted). The layout of the fluorine circulation pump system is shown in Figure 19. Construction details of the fluid-bed fluorinator, the secondary filter, and the large UF_6-PuF_6 condenser are shown in Figures 20, 21, and 22, respectively. Specifications for welding fabrication and for inspection of nickel process vessels are contained in the appendix.

1. Fluorinator

Input and exit process-stream flows for the fluorinator are indicated in Figure 16. Fluidizing gas enters a gas distributor at the bottom of the fluorinator. A side entry for gas is also provided. Process gas leaves through the disengaging section and filters at the top of the fluorinator assembly.

a. Construction of the Fluorinator Unit

The fluorinator is of welded A-nickel construction except for the disengaging section, which is L-nickel for added strength. The The fluidization section of the fluorinator consists of a 4-ft section of 3-in., schedule 40 pipe. The conical disengaging section, fabricated from 3/16-in. plate, is welded to the top of the fluidization section. The disengaging section is in the form of an inverted, oblique, truncated cone, 20 in. long, with a top 14 in. in diameter. All internal angles are greater than 60° with the horizontal in order to minimize powder holdup.

A detail of the baffled-cone gas distributor for the fluid-bed fluorinator is shown in section A-A of Figure 20. Fluidizing gas enters the side of the cone, and the baffle prevents solid back-up in the gas line, even if the gas supply is cut off. The gas distributor unit is flanged to the bottom of the fluid-bed section of the fluorinator. The seal gasket is a solid,

annealed-copper ring with a circular cross section of 0.10-in. diameter. Similar gasketed flanges are located at the top of the fluorinator unit.

Dimensional details of the fluorinator and its attached heating and cooling components are shown in Figure 20. Heat is supplied from tubular, electric-resistance heaters wrapped around the circumference of the unit. To aid conductive heat transfer, the heating elements are bonded to the walls by copper applied by flame spraying. (See appendix for specification details.) A thin coating of stainless steel is applied similarly over the copper to prevent oxidation. Separately controlled heating zones are provided, each zone having spare heaters in the event of a heater burn-out.

Internal reactor temperatures are measured by chromel-alumel thermocouples. The thermocouple assembly, which consists of the thermocouple wires imbedded in a compacted powder insulation inside a closed nickel sheath, is slipped through a Swagelok connector and a tube extending from the reactor wall. The tip of the thermowell can be positioned at different radial distances inside the reactor by loosening the nut on the Swagelok fitting and moving the thermocouple assembly. The thermocouples are arranged in a common vertical line with the electrical leads of the resistance heaters so that two half-circular sections of preformed thermal insulation can be clamped easily to the walls of the fluorinator. The tubes that hold the thermocouple assemblies can be used for pressure-tap connections or sampling ports if the assemblies are not in place.

b. Fluorinator Off-gas Filters

Two filter chambers are mounted atop the disengaging section of the fluorinator unit. These chambers (3-1/2-in. diam., schedule 40 pipe, 40 in. long) house sintered-Monel, bayonet-type filters, which remove entrained dust from the fluorinator off-gas. The lower 18-in. portions of the filter chambers are provided with water-cooling coils (Figure 20). The cooling portion of the filter chambers have internal fins to aid in cooling the off-gas to below 150°C before the gas reaches the filter elements. A provision is also made for introducing a stream of cold, inert gas as a "quench" to aid further in keeping the filters cool, if necessary.

The porous, sintered Monel filter elements are 9 in. long, bayonet-type, and have a filtering area of 0.18 sq ft per element and a nominal porosity rating of 20 microns.

The filter units operate in parallel. To prevent continual buildup of filter cake, each filter is equipped with an automatic blowback system which supplies a 1/2-sec pulse of 80-psig nitrogen gas to the filter every 15 minutes. To minimize the amount of nitrogen added to the process gas, the process gas itself supplies part of the blowback gas.

This is accomplished by passing the nitrogen through the high-pressure inlet of a Venturi ejector (on the exit line of the filter) through which the process gas normally flows. On blowback, the process gas on the low-pressure side of the ejector is pulled into the ejector system and becomes part of the blowback gas.

A secondary filter chamber containing an 18-in.-long, bayonet filter is located downstream of these primary units to trap any entrained solids in event of a failure of the primary filter elements. The secondary filter chamber details are shown in Figure 21.

c. Solids Charging and Discharging

Solids are charged to the fluorinator through an air-lock charger mounted, like the blowback filter units, on a flange at the top of the disengaging section. (The charger is shown in Figure 20, but not in the other figures.) The charger is constructed of a flanged nickel pipe, 3-in. diam., schedule 40, 18 in. long, with a metal plug valve at the bottom (connecting with the fluorinator) and a blind flange for a top seal.

Solids are discharged from the fluorinator through the small (1-1/2-in.) flanged nipple at the bottom of the gas distributor (Figure 20). A metal plug valve, connected to this flange, controls solids removal. The solids discharge through a flexible metal hose connected to the cover of a solids receiver vessel. The latter is a metal can (paint-can type) with a diameter of about 8 in. and a height of about 14 in. When the solids receiver is to be removed, the cover and connecting line are detached, and another cover is placed on the can.

d. Fluorinator Cooling System

For cooling the reaction zone of the fluorinator (i.e., the lower 24 in.), a two-phase mixture of air and water is passed through an external coil (Figure 20) attached to the wall of the fluorinator. Air is passed continuously through the coil, and water is injected at three points along the coil, when called for by temperature controllers at each of three cooling zones. A schematic flow diagram of the air-water system is shown in Figure 23. The zone temperature control provides for flexibility when it is desirable to create or overcome longitudinal temperature gradients within the reactor. The water hold tanks (Figure 23) are equipped with sightglasses and are located at the panelboard to permit measurement of the volume of coolant used, and to provide a positively controlled supply of water to the alpha box.

The advantages of an air-water cooling system over air alone include higher heat-transfer coefficients and higher enthalpy change for water. Smaller piping is allowed for the water-air mixture than for air alone, and

fins on the reactor cooling surface are not required. The use of water alone for cooling at the process temperature of 500°C is not practicable because localized overcooling would occur and control would be difficult. This is minimized in the air-water system, the heat being removed chiefly by the vaporization of the water carried by the air stream.

In addition to the reaction-zone coolant system described above, three additional cooling zones are provided: (1) the fluorinator walls above the reaction zone, (2) the disengaging section, and (3) the filter chambers. Water is passed through these stainless-steel coils, since the aim is to cool the off-gas rapidly from 500°C to about 150°C to minimize the thermal decomposition of plutonium hexafluoride. Enough heat-transfer surface is available in the internally-finned filter section to cool the gas to 150°C before it reaches the filters. The source of water supply for these coils is a measuring tank near the panelboard.

2. Hexafluoride Condensers

Uranium and plutonium hexafluoride from the fluorinator off-gas are collected in two series-connected condensers. The condensers are shown in the material flow diagram of Figure 17 and the equipment instrumentation schematic diagram of Figure 18. Construction details are shown in Figure 22. Nickel and Monel are the materials of construction. The U-shape of the condenser reduces its overall length. In each leg of the condenser, the coolant flows through an inner central tube to which longitudinal fins are welded. The solid hexafluoride products are condensed in the annular space between the fins and the outer 4-in. diam. shell. The U-shape also eliminates the need for expansion joints when the condenser temperature is raised in order to transfer products or to fluorinate the condenser to recover any deposited plutonium tetrafluoride. The central coolant tubes are double-walled pipes, consisting of an outer 3/4-in. diam. pipe to which the fins are welded, and an inner tube (1-1/16-in. thick wall) swaged in place. The double wall reduces the possibility of contaminating the coolant if a leak should develop in the finned tube. The heat-exchange liquid flowing through the central tubes is cooled by a 6000-BTU/hr mechanical refrigeration unit designed for -70°C operation and described in Section II-D-8 of this report. Transfer of hexafluorides from the condenser by vaporization will be done by heating the heat-exchange fluid to approximately 80°C.

Results of mock-up tests [described in a previous report[21]] with one of these condensers disclosed the desirability of heating the outer walls of the condensers during the condensing operation in order to increase the capacity of the units. Heat for this purpose is supplied by resistance heaters wound on the outer shells. These heaters also provide for heating the units to a temperature of 350°C for the purpose of refluorinating plutonium tetrafluoride formed by the decomposition of plutonium hexafluoride.

A remote-reading scale measures the weight of the condensed product. The basic design features have been described previously.[22] The system consists of a platform-type beam scale, in which the movement of the beam from the null balance point is detected by a photocell. The electrical signal from the photocell operates a reversible electric motor which moves a sprocket wheel and adds or subtracts chain to rebalance the beam. Position of the sprocket is remotely recorded on a millivolt recorder by using a 10-turn helipot electrical system.

All process and refrigerant lines are attached to the condensers so as to avoid interference with the measurement of small changes in weight. This is accomplished by using lever and torsion-arm arrangements at least 6 ft long. These lines are covered with a flexible insulation having a vapor barrier.

3. Process-gas Recycle Pump

The off-gas from the condensers may contain substantial amounts of fluorine during certain periods of fluorination. To aid in the overall efficiency of fluorine utilization, this off-gas can be recycled to the fluorination reactor by a remote-head diaphragm compressor. This recirculation pump has a rated capacity of 2.0 scfm at 15-psia inlet and 30-psia discharge pressure. A layout of the prime mover and the remote head is shown in Figure 19. A 3-in. diam. copper line connects the two heads and contains about 2 gallons of a fluorinated oil required for the fluid coupling between the remote head and the primary head of the pump unit. The remote head contains a nickel diaphragm sealed between heavy nickel flanges, 18 in. in diameter. Because of the weight and the applied impulses of the reciprocating hydraulic fluid, the unit is securely mounted on a concrete shielding wall. An alpha box encloses the remote head, and a 4-in. vent duct connects this enclosure to the large alpha box. A pulley hoist is mounted inside the enclosure to facilitate removal or repair of the remote head. Process-gas lines from the pump to the large alpha box are enclosed in conduit for extra protection against leaks. Pneumatic control of the variable-speed electric motor of the pump unit allows variation of pumping flow by remote operation from the panelboard.

4. Hexafluoride Product Receivers

Uranium hexafluoride and plutonium hexafluoride collected in the condensers are transferred to nickel receivers for storage within the alpha box and for transfer to the converter unit (see Figures 16 and 17). During the transfer by vaporization from the condensers, the product receivers are cooled by immersion in a bath maintained at a low temperature by the refrigeration system used to cool the condensers. The product receivers are fabricated from a 2.5-ft length of 4-in.-diam., schedule 40, nickel pipe. Two connecting lines and a pressure gauge are attached to the top of each

receiver, and Monel valves are attached to these lines. The receiver is connected to another process vessel by means of flare connectors.

5. Plutonium Hexafluoride Cold Traps

Two cold traps, of a coil design and fabricated from 5-ft lengths of 3/4-in.-diam. copper tubing, are used to collect the plutonium hexafluoride formed during the fluorination of plutonium tetrafluoride collected in lines and equipment. These traps are connected in series and immersed in a cooling bath. Flare connections permit the traps to be removed for weighing.

6. Chemical Traps

Sodium fluoride and activated alumina chemical traps in the off-gas line downstream of the condensers remove any hexafluorides and fluorine in the process off-gas before the gas is sent to the process scrubber. The traps, fabricated from 4-in. diam. brass tubing, are each 5 ft long. The bed of active material is supported by a perforated plate inside the trap, and nickel wool prevents solids from entering the connecting lines at either end. Except for the top cover, the trap is assembled before the bed material is charged. Attachment of the cover, as well as all other connections, is done by silver alloy brazing. The spent trap units are disposable without the necessity of disassembly. Thermocouples are mounted on the outside wall to indicate bed temperatures. The location of these traps in the alpha box is shown in Figure 18.

7. Gas Analyzer

A schematic diagram of the continuous-process gas analyzer for measuring fluorine and hexafluoride concentrations is shown in Figure 24. Analysis is based on the thermal conductivity of the gas which flows through the analyzer continuously at flows of 100 to 500 cc/min. In the first stage of the analyzer, the change in gas conductivity is measured before and after UF_6 is removed from the gas stream by sodium fluoride at 100°C. In the second stage, the gas conductivity is measured before and after the following conversion of the fluorine to chlorine by reaction with sodium chlorides:

$$2NaCl(s) + F_2(g) \longrightarrow 2NaF(s) + Cl_2(g).$$

The analyzer is installed in the large alpha box; electrical controls and recorded outputs of the thermal conductivity cells are located at the panelboard. Although not incorporated in the present facility, other process-gas components can be measured by thermal conductivity cells. Plutonium hexafluoride may be removed selectively by thermal decomposition at temperatures of 200 to 300°C, and oxygen may be determined by a special magnetic thermal conductivity cell.

8. Process Valves

Two types of process valves are used. The first is a manually operated, 1/4-in., Monel diaphragm valve which is used for low gas flow rate applications and where temperature or pressure requirements are not stringent.

The second and major type of process valve is a 1/2-in., Monel, bellows-seal type. This valve is either manual or air-operated and has either a metal-to-metal seat for 350°C service, or a Teflon-to-metal seat for low-temperature service. The construction of the valve is shown in Figure 25. The valve bonnet is bolted to the valve body and can be removed for cleaning and repairing the plug or seat. The valve bellows will withstand a static load of 200 psi. The process shut-off valves had a leakage rate of less than 0.1 micron cu ft/hr (1×10^{-6} standard cc/sec) across the seat when installed.

9. Photographs of Facility Components During Installation

More detailed and realistic information on components of the facility are furnished by photographs taken during installation. Figure 26 shows the prime mover unit of the fluorine circulation pump, which is shown schematically in Figure 19. A view of the fluorinator being lowered into the large alpha box through a top bag-out port is shown in Figure 27. The panelboard of the fluorinator system and its surrounding operating area, including the top of the smaller alpha box, are shown in Figure 28. The personnel lift located in the process cell and serving the large alpha box, is shown in Figure 29.

C. Procedure for Fluorination

A typical run, based on selected flowsheet conditions, will start with the charging of weighed amounts of alumina grain and uranium dioxide-plutonium dioxide pellets to the fluorinator. The alumina will be fluidized with nitrogen gas while the reactor is being heated to the operating temperature.

The high-rate period of fluorination will be carried out at a controlled temperature while fluorine and oxygen are being admitted to the fluorinator and hexafluoride is being collected in the product condensers. During this period in which high fluorine-utilization efficiency is maintained, the process gas will not be recirculated, but will pass out through the activated alumina traps and the scrubber systems.

In the low-rate period of fluorination, final volatilization of uranium and plutonium hexafluorides will be conducted with recirculation of process gases containing fluorine. In this step, the plutonium hexafluoride may be

condensed in the small cold traps rather than in the larger product condensers.

During fluorination, temperatures, flow rates, gas compositions, heat-generation rate, and the amount of hexafluoride product collected will be measured. The inert fluid bed will be sampled for analysis of uranium and plutonium contents during and after a run.

Hexafluorides will be vaporized from the condensers into a product receiver after the run. This material will be used as feed for subsequent steps in which the contained PuF_6 will be thermally decomposed to PuF_4 or in which the PuF_6-UF_6 mixture will be converted to PuO_2-UO_2 in the converter system discussed in Section IV.

D. Fluorination Program

Rates and efficiencies of fluorination will be measured as a function of the process variables: temperature, gas flow rate and composition, and quantities of feed and bed materials. The rate of collection of PuF_6 product, the amount of residual uranium and plutonium and the inert fluid bed, and the material balances for uranium and plutonium are of major interest. Efficiencies of operation of the condensers and chemical traps will be determined.

Other matters to be investigated concern the effect of alternative charging and feeding arrangements, the study of decladding and head-end treatments, and the chemical effects of added fission-product compounds.

IV. CONVERTER SYSTEM

A. Introduction

A fluidized-bed reactor and associated equipment are being installed in the alpha containment box to convert the gaseous uranium and plutonium hexafluoride products of the fluorination process to an oxide powder that can be recycled to the fluorination process or removed from the alpha box and stored with greater safety and economy than is possible for the volatile hexafluorides. The effects of process variables in the production of a high-density oxide powder suitable for fabrication into compacted fuel elements can also be studied with this equipment. Experimental data have been obtained previously on the conversion of uranium hexafluoride to dioxide in a fluidized-bed reactor.[23] The present unit will be used to obtain data with plutonium hexafluoride alone, or with uranium hexafluoride-plutonium hexafluoride mixtures. In addition, the equipment can be used for the study of the separation of plutonium from uranium in UF_6-PuF_6 mixtures by selective thermal decomposition of the PuF_6 to nonvolatile PuF_4.

Operating conditions for the conversion of the mixed hexafluorides to oxides are expected to be similar to those developed in the uranium hexafluoride conversion experiments. The processing cycle is divided into two periods: in the first period the uranium and plutonium hexafluorides, steam, and hydrogen are reacted at 650°C; in the second, or cleanup period, the steam and hydrogen alone are fed to complete the conversion of the intermediate solid compounds. The mixed oxide product is removed after this cleanup period.

The reactions for the conversion of plutonium hexafluoride to the oxide are yet to be established; those for uranium hexafluoride are:

First Period: $UF_6(g) + H_2(g) \rightarrow UF_4(s) + 2HF(g)$; $\Delta H_{298°K} = -69$ kcal/mole U;

$3UF_6(g) + 8H_2O(g) + H_2 \rightarrow U_3O_8(s) + 18HF(g)$; $\Delta H_{298°K} = -8$ kcal/mole.

Second Period: $UF_4(s) + 2H_2O(g) \rightarrow UO_2(s) + 4HF(g)$; $\Delta H_{298°K} = +47$ kcal/mole;

$U_3O_8(s) + 2H(g) \rightarrow 3UO_2(s) + 2H_2O(g)$; $\Delta H_{298°K} = -13$ kcal/mole.

A greater than 15 times stoichiometric excess of hydrogen and slightly more than the stoichiometric amount of water are required to produce a high-density oxide. Quantities of reagents and products used as a basis in the design of the present equipment are shown in the material-balance flowsheet (Figure 30). A step not shown in the flowsheet is the method proposed for controlling particle size in the conversion step. Oxygen is introduced to provide a partial break-up of UO_2 particles by formation of U_3O_8, which is

reduced again to UO_2 with hydrogen. Particle-size control may also be provided by physically adding seed particles to the bed.

B. Equipment and Instrumentation of the Converter Unit

The pilot plant converter equipment includes gas supply sources, a gas heater, a fluidized-bed converter (including filters), a secondary filter, a gas-analysis system, and an exit-gas scrubber, and chemical traps. An equipment-instrumentation flowsheet is shown in Figure 31; the alpha box equipment layout plan is shown in Figure 32. The converter is fabricated of Inconel; other equipment and lines are nickel.

Process-gas supplies include steam, air, nitrogen, and hydrogen. The process feed is a uranium hexafluoride-plutonium hexafluoride mixture. Steam is supplied by an automatically controlled, constant-pressure steam generator, electrically heated. Water entrained in the steam is vaporized in an electrically heated superheater, made from a 1-1/2-in. diam. pipe, 16 in. long. The hexafluoride gas mixture is vaporized from 4-in. diam., cylindrical vessels, which have removable heating coils for supplying heat of vaporization. Water-cooling coils are provided for emergency cooling. Air, nitrogen, and hydrogen are supplied from high-pressure cylinders and are dried before being used. All gas flowrates are metered and automatically controlled by an orifice-differential pressure system.

The gas entering the converter (steam, air, nitrogen, or oxygen) is preheated in a 33-in. long, 1-1/2-in. diam. pipe, packed with 1/2-in. nickel Rashig rings; heat is supplied by electrical resistance elements. Input to these clamshell heaters is controlled automatically with respect to the wall temperature of the preheater at the gas exit.

The converter is composed of three sections, connected by flanges: a conical bottom section, a fluid-bed section, and a cooling-filtering section. The assembled reactor is shown in Figure 33, and drawings of the individual sections are shown in Figures 34, 35, and 36. The gaseous hexafluoride mixture is fed into the bed through a small-diameter tube extending into the conical opening; the steam and hydrogen mixture is fed through the annulus between this tube and the surrounding pipe. Solid samples and product are withdrawn through the other opening in the conical bottom into a 1-1/4-in. diam., water-cooled container. Adequate mixing of the bed is attained without a gas-distributor plate.

The fluid-bed section of the converter is fabricated from a 2-in. diam., 24-in. long pipe. Three 1500-watt and two 750-watt, stainless alloy sheathed, tubular, electric resistance heaters are bonded to the outside wall by a coating of copper applied by a metallizing technique. Thermocouples are inserted through side inlets, which also serve as pressure taps. One side inlet, similar to that for removing product, is available for adding seed par-

ticles to the fluid bed. Other openings include a solids-overflow pipe and a port, normally plugged, through which a boroscope can be inserted for examining the interior. Two surface-temperature thermowells are located on the lower half of the wall of the fluid-bed section.

The cooling and filtering section of the converter is fabricated from a 4-in.-diam. pipe and is 24 in. long. The lower half is wrapped with two cooling coils of six turns each, which are copper-bonded to the wall. A two-phase, air-water mixture is the coolant. A port is provided at the middle of the section for viewing the interior. The upper flange has five openings, four for the filters and one for a thermowell. The dust cake is removed from the filters by blowback with a pulse of high-pressure nitrogen.

A secondary filter vessel for the containment of solids in the event of primary filter failure is fabricated from 3-1/2-in. diam. pipe, 21 in. long. A single bayonet filter, 18 in. long, made of porous nickel is contained in the vessel. A 1500-watt heater is attached to the outer wall.

The hydrogen content of the process gas effluent from the converter is analyzed by a thermal conductivity cell. Another hydrogen detector is provided to monitor the hydrogen content of the air environment around process components of the converter system as a safety precaution. This detector monitors the air in the large alpha box, the air in the operating area of the cell, and the air in the vicinity of the hydrogen supply cylinders. Normally no hydrogen is present in these locations, but if a leak appears, the instrument sounds an alarm and indicates the area of the leak.

Effluent process gas from the converter and filters contains hydrogen fluoride, which is removed in a scrubber (Figure 30). The countercurrent, packed-bed scrubber is constructed from Monel alloy 400. Details are shown in Figure 37. The packed-bed section, 4 ft long and constructed from 4-in.-diam. pipe, has sixty-four, 1-1/2-in.-high, cooling fins attached to its outer surface. The gas being scrubbed enters the bottom and passes through the packed section to the cyclone separation, where excess liquid is removed. Caustic solution is pumped from the hold tank to the top of the packed section. The design of the hold tanks was dictated by criticality considerations.

Control, indicating, and recording instruments are mounted on a panelboard outside the cell area and adjacent to the fluorinator panelboard. Most process variables (flowrates, temperatures, and pressures) are recorded.

Gas evacuated from process lines is scrubbed by passage successively through sodium fluoride and alumina-filled traps. These are 4 in. in diameter, and 5 and 5-1/2 ft long, respectively. The sodium fluoride trap can be heated.

C. Procedure for Converter Operation

A typical run in the initial experiments will start with the loading into the converter of a weighed amount of either uranium dioxide or a mixture of uranium-plutonium oxides of known particle-size distribution. The gas purges and the filter blowback system will be activated and the bed fluidized with nitrogen until the operating temperature of 650°C is reached. Then the steam-hydrogen mixture will be substituted for nitrogen and, when equilibrium is established, the uranium-plutonium hexafluorides will be fed for a predetermined feeding period (a half-hour in initial tests). In the cleanup period that follows this feeding period, the hexafluoride feeding is stopped and the steam flow is increased automatically to compensate for the loss of gas volume. Cleanup is continued for a predetermined period (a half-hour in initial tests), after which part of the product oxide is removed from the bed and the cycle is restarted by feeding the hexafluorides. Bed samples and product are withdrawn by opening a valve connecting the reactor with a previously evacuated container.

Product oxide will be analyzed for fluoride content and density and also for particle-size distribution. Seed particles may be physically charged to the bed by opening a valve connecting the reactor to a previously pressurized container holding the seed particles. The seed particles are produced either by grinding, or by reoxidizing and reducing the oxide product. An alternate method for producing seed particles by *in situ* oxidation was described in Section IV. A above.

D. Program for Conversion of Hexafluorides to Dioxides

If the unit is to convert hexafluorides to oxide solids for storage, the converter will be operated with an 8-in. deep bed at 650°C. Feed rates and quantities will be those shown on the material-balance flowsheet (Figure 30). For determining optimum conditions for producing a high-density oxide, the variables to be studied may include bed height, temperature, ratios of reactants, feed rates, number and position of hexafluoride feed nozzles, and duration of the process periods.

E. Program for Decomposition of Plutonium Hexafluoride to Plutonium Tetrafluoride

The decomposition of plutonium hexafluoride to the tetrafluoride is also to be studied in this experimental facility. Because plutonium hexafluoride is more easily decomposed than uranium hexafluoride, this method can be used to effect a separation between uranium and plutonium. The thermal decomposition proceeds according to the reaction: $PuF_6(g) \rightleftarrows PuF_4(s) + F_2(g)$. Initial experiments have been carried out in the laboratory scale.[7,8] Chemical agents that reduce the hexafluoride may also be used to produce PuF_4.

The process variables to be studied are temperature, flow rates, surface area of decomposition zone, and the rate and efficiency of the decomposition. Density and purity of the plutonium tetrafluoride product are of interest.

APPENDIX

Recommended Specifications for Fabrication and Inspection of Nickel Process Vessels

The following is a partial list of specifications used by the ANL Chemical Engineering Division. The specification items cited below are those especially important for nickel process vessels to be put into service in fluoride volatility processes such as the one described in this report. Acknowledgment for preparing the specifications is made to the Design Group of this Division, especially J. H. Schraidt and H. O. Smith.

Specification Sections

B.2.1 Procedure for inert-gas arc welding of nickel by the consumable-electrode process or by the tungsten-arc process using nickel 200 filler metal, or by the tungsten-arc process for single fusion.

C.1 Degreasing
G.1 Radiographic examination
H.1 Leak testing for vacuum service
H.2 Leak testing for internal-pressure service
H.3 Leak testing by liquid-penetrant examination
K.1 Copper flame spray metal application

B.2.1.0 PROCEDURE FOR INERT-GAS ARC WELDING OF NICKEL BY THE CONSUMABLE-ELECTRODE PROCESS OR BY THE TUNGSTEN-ARC PROCESS USING NICKEL 200 FILLER METAL, OR BY THE TUNGSTEN-ARC PROCESS FOR SIMPLE FUSION

Huntington Alloy Products Division of INCO technical bulletin T-2, "Fusion Welding of Nickel and High Nickel Alloys," may be used for more detailed information on welding procedures and other pertinent data.

2.1.1 Base-metal Preparation
Before welding, the mating surfaces and adjacent areas shall be thoroughly cleaned of all dirt, oil, grease, metal fillings, or other foreign matter.

2.1.2 Filler Metal
Filler metal, where required, shall be INCO, Huntington Alloy, Nickel 200.

2.1.3 Weld Porosity

Weld porosity can be controlled by proper care and attention to the factors (gas, current, arc length, speed of welding) that affect weld quality.

2.1.4 Defects

Any cracks or blowholes on the surface of any weld bead shall be removed by chipping or grinding before depositing the next successive bead.

2.1.5 Gas

High-purity, welding-grade gas is to be used. To prevent contamination of the protective inert-gas atmosphere surrounding the arc during the welding process, it is essential that all welding be done in a still air environment. In most cases, the work area must be shielded from air movement due to fans, welding generators, or other air currents. An abundance of gas should be used at all times. Gas cups should be sufficiently large to permit delivery of required gas flow to the welding area at low velocity.

2.1.5.1 Argon gas is to be used in the consumable electrode process.

2.1.5.2 Helium gas is to be used in the tungsten-arc process where simple fusion of light-gauge materials is required.

2.1.5.3 Argon gas is to be used in the tungsten-arc process where filler metal is added as required when welding heavy sections.

C.1.0 DEGREASING

All traces of oil and grease are to be removed by immersion in any standard vapor degreaser. Large parts may be cleaned by washing with trichlorethylene or equivalent. Water-soluble degreasing compounds may not be used.

G.1.0 RADIOGRAPHIC EXAMINATION

All welds designated to be inspected in accordance with this specification shall be examined throughout their entire length by the X-ray or gamma-ray method of radiography, except that the gamma-ray method shall not be used with aluminum alloys.

1.1 Preparation

All butt-welded joints to be radiographed shall be repaired as follows: The weld ripples or weld surface irregularities, on both the inside and outside, shall be removed by any suitable mechanical process to a degree such that the resulting radio-

graphic contrast due to any remaining irregularities cannot mask or be confused with that of any objectionable defect. Also, the weld surface shall merge smoothly into the plate surface. The finished surface of the weld bead may be flush with the plate or have a reasonable uniform crown not to exceed 1/16 in.

1.2. Procedure

The weld shall be radiographed using a technique that will indicate the size of defects having a thickness equal to and greater than 2% of the base-metal thickness.

1.2.1 The film during exposure shall be as close to the surface of the weld as practicable. If possible, this distance shall not be greater than one inch.

1.2.2 Identification markers, the images of which appear on the film, shall be placed adjacent to the weld, and their locations shall be accurately and permanently marked on the outside surface near the weld so that a defect appearing on the radiograph may be accurately located.

1.2.3 Sections of welds that are shown by radiography to have any of the following types of imperfections shall be judged unacceptable, shall be removed by mechanical means, shall then be rewelded, and shall again be radiographed as provided in this specification:

(a) Any type of crack or zone of incomplete fusion or penetration.

(b) Any slag inclusions or porosity that will impair the strength, leak-tightness, or corrosion resistance of the joint.

H.1.0 LEAK TESTING FOR VACUUM SERVICE

Testing is to be done on items and assemblies as indicated on the drawings or in this set of technical specifications. Testing is to be done after all machining and finishing operations (grinding, etc.), and all treatments (cleaning, heat treating, pickling, etc.) have been completed. Testing is to be done before any protective coating is applied. All leaks found must be repaired. Before the repaired areas are tested, all machining and finishing operations and treatments must be repeated unless specific permission to omit one or more of the steps is given in writing by the Buyer.

1.1 Leak Testing Method

Testing is to be performed using a mass-spectrometer type helium-leak detector which has been appropriately calibrated

for a leak-rate sensitivity of at least 4×10^{-7} standard cc/sec of helium for a full-scale deflection of the leak-indicator meter.

1.1.1 With the assembly evacuated to 50 microns, the entire exterior surface (including connections) is to be probed with a jet of helium flowing at the rate of approximately 1/2 cfm through the nozzle. The stream of helium shall impinge directly on the joint or surface being examined with the nozzle approximately 1/8 in. from the surface and moved along at a maximum linear speed of 6 in./min. A leak rate in excess of 2×10^{-7} standard cc/sec of helium will be unacceptable.

1.1.2 All welds to be probed shall be clean, dry, and free of scale or grease.

1.1.3 All soldered, brazed, and welded joints designated as "SPEC. CE-H1" on any detail or assembly drawing will be tested as specified above.

H.2.0 LEAK TESTING FOR INTERNAL-PRESSURE SERVICE

Testing is to be performed using a mass-spectrometer type helium-leak detector which has been appropriately calibrated for a leak-rate sensitivity of at least 4×10^{-7} standard cc/sec of helium for a full-scale deflection of the leak-indicator meter.

2.1.1 The assembly is to be pressurized to approximately 1/2-psi gauge with helium. A shielded probe is to be used for the test. The probe is to be moved along at a maximum linear speed of 6 in./min. with the tip of the probe shield held approximately 1/8 in. from the joint or surface being examined. A leak rate in excess of 2×10^{-7} standard cc/sec of helium will be unacceptable.

2.1.2 All joints or surfaces to be probed shall be clean, dry, and free of scale, oil, grease, or other contaminants.

2.1.3 All soldered, brazed, and welded joints designated as "SPEC. CE-H2" on any detail or assembly drawing will be tested as specified above.

H.3.0 LEAK TESTING BY LIQUID-PENETRANT EXAMINATION

Testing is to be done on items and assemblies as indicated on the drawings or in this set of technical specifications. Testing is to be done after all machining and finishing operations (grinding, etc.), and all treatments (cleaning, heat treating, pickling, etc.) have

been completed. Testing is to be done before any protective coating is applied. All leaks found must be repaired. Before testing the repaired areas, all machining and finishing operations and treatments must be repeated unless specific permission to omit one or more or the steps is given in writing by the Buyer.

3.1. Test Method

3.1.1 The testing shall be done with Magnaflux Corporation's Zyglo Z1-1C Fluorescent Penetrant and ZP-4 Developer, or a Buyer-approved equal.

3.1.2 All welds shall be in the finished condition as indicated by the weld symbol (including any finish machining, grinding, etc., where specified).

3.1.3 All welds shall be cleaned until free of all foreign matter and corrosion products before leak testing.

3.1.4 The test procedure is to consist of cleaning, Zyglo penetrant application, rinsing, drying, dry developer application, drying, black-light inspection, and final cleaning. All steps are to be in accordance with the manufacturer's recommendations.

3.1.5 Welded joints consisting of penetration or thru welds shall be inspected for leaks from the side opposite the Zyglo penetrant if at all practical, otherwise these and all other joints shall be inspected from the penetrant side.

3.1.6 All welds designated as "SPEC. CE-H3" shall be tested as specified above.

3.1.7 Any indicated or visible leaks shall be repaired and retested before any protective coating is applied.

K.1.0 COPPER FLAME-SPRAY METAL APPLICATION

The copper-wire flame-spray material specifications, application technique, and procedures shall be as recommended by the flame-spray equipment manufacturer.

1.1 Surface Preparation

The surface of the base metal to be sprayed will be thoroughly cleaned using any standard vapor degreaser. The surface will be roughened using a coarse grit sand blast. Care is to be taken that the sand is clean and the coverage is uniform.

1.2 Spray Metal Covering

The copper spray metal covering shall consist of two layers, namely the base layer, which is bonded to the base metal, and the cover layer, which is bonded to the base layer, and any tubes or other apparatus placed over the base layer. The completed job, when cool, shall be free of visible cracks.

1.2.1 A base layer, built up to a uniform thickness of 1/16 in. maximum, will be added to the prepared surface.

1.2.2 Properly cleaned tubes, heaters, or other apparatus are to be placed, attached, or otherwise held in the positions indicated on the drawings. The cover layer is to be then added and bonded to the base layer and the above-mentioned attachments. The cover-layer thickness is to be 3/32 in. minimum, to 3/16 in. maximum.

1.2.3 The spray-metal application procedures recommended by the manufacturer (gas mixture, relationship of gun to the workpiece, speed, wire feed, etc.) should be followed to minimize the "bridging" effect of incomplete penetration at the underside of the attached tubes. Maximum size of the bridging fillet allowed is one-fourth of the tube diameter. Preheating of the base metal to 350°F before flame spraying increases the bond strength and minimizes the chance for stress concentration cracks or shrinkage cracks. Step-down of the layer build-up at the edges of the spray-metal areas also increases bond strength and minimizes cracking.

1.3 Protective Layer

The protective layer is to consist of material specified on the drawing and metal flame-sprayed to a minimum uniform thickness of 1/64 in. to encase all exposed copper spray-metal surfaces. (Stainless steel Type 308 ELC has been used for the present facility; however, other alloys may be used, and the above alloy is not critical to the technique.)

ACKNOWLEDGMENTS

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Figure 5

LARGE GLOVEBOX FOR ALPHA CONTAINMENT

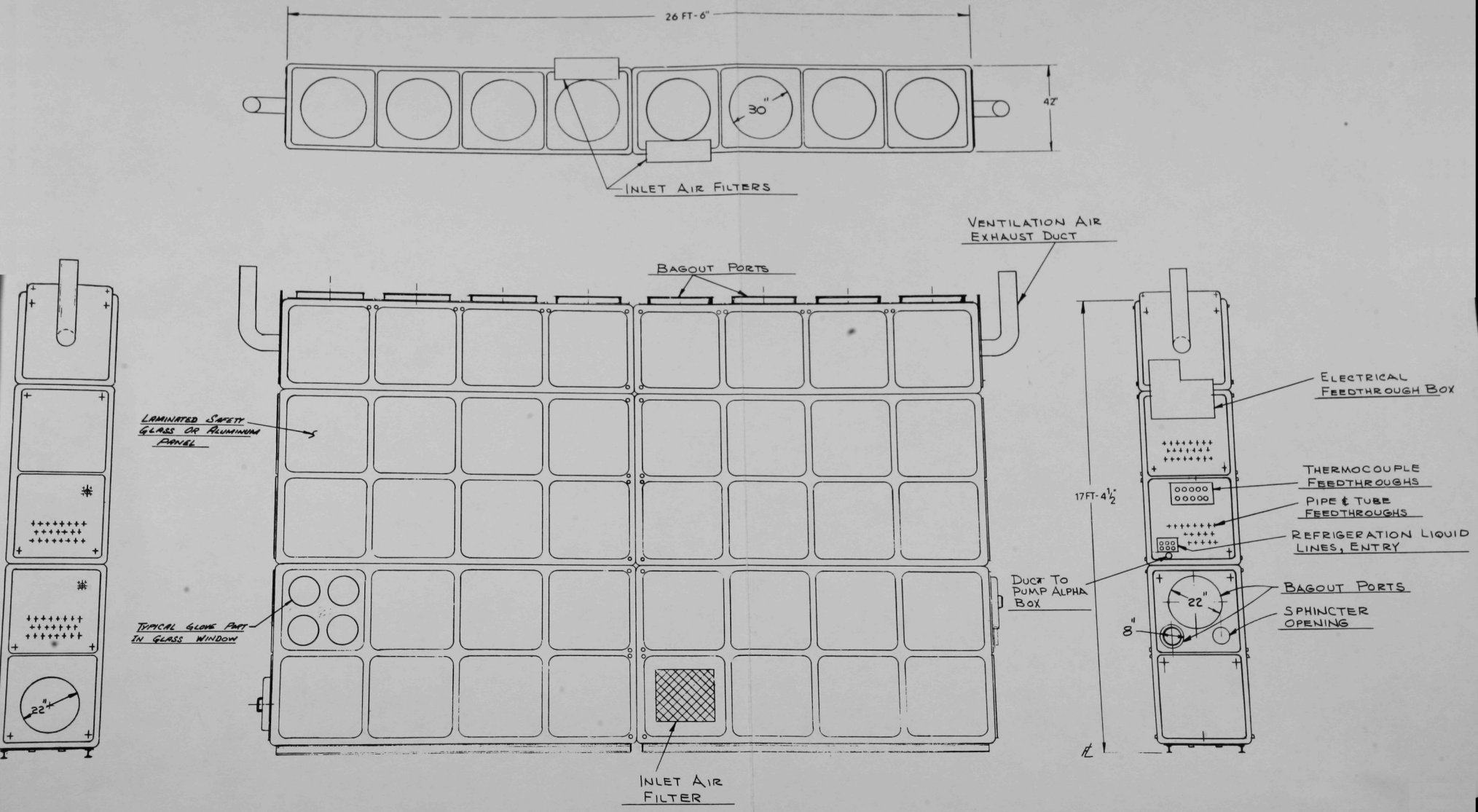


Figure 6

NONLEAK SEAL FOR ELECTRICAL LEADS PASSING THROUGH ALPHA BOX WALL

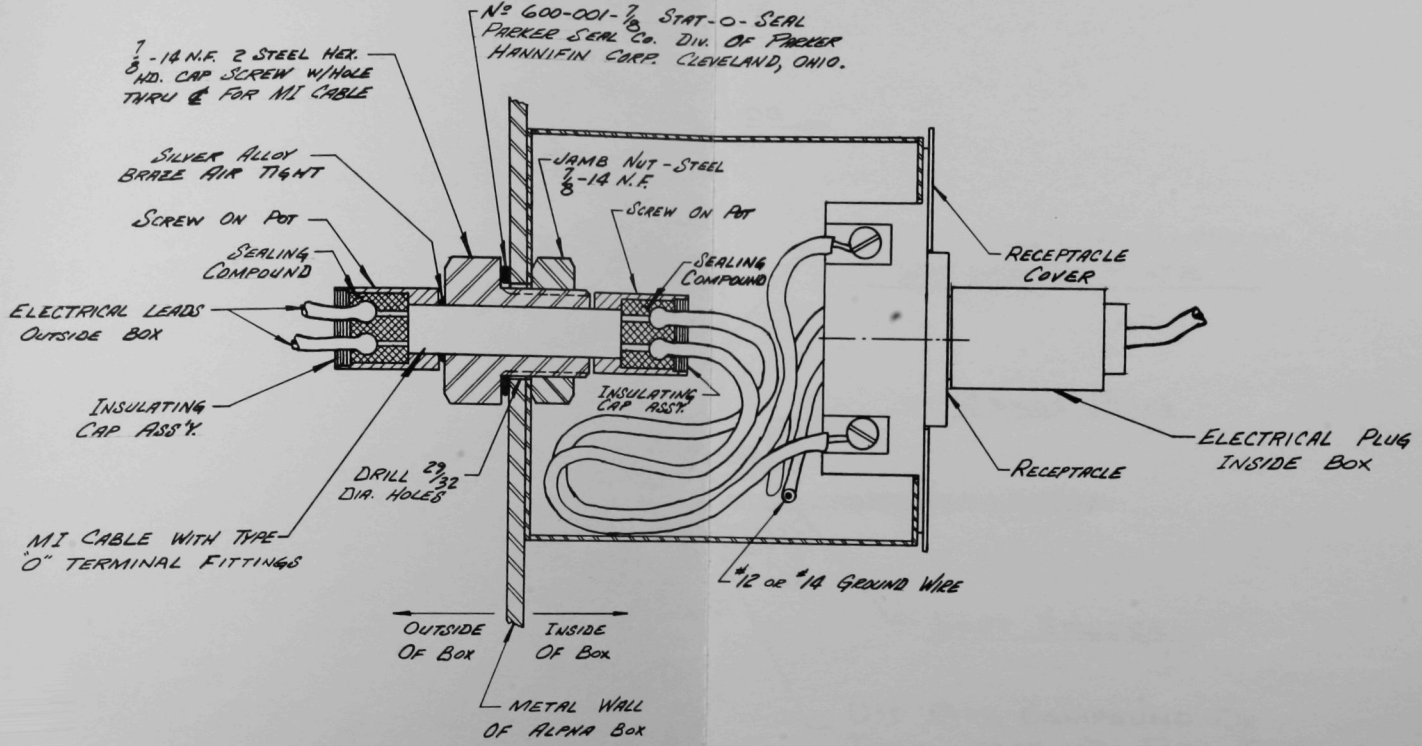


Figure 7

NONLEAK SEAL FOR PIPES AND TUBES ENTERING ALPHA BOX THROUGH WALL

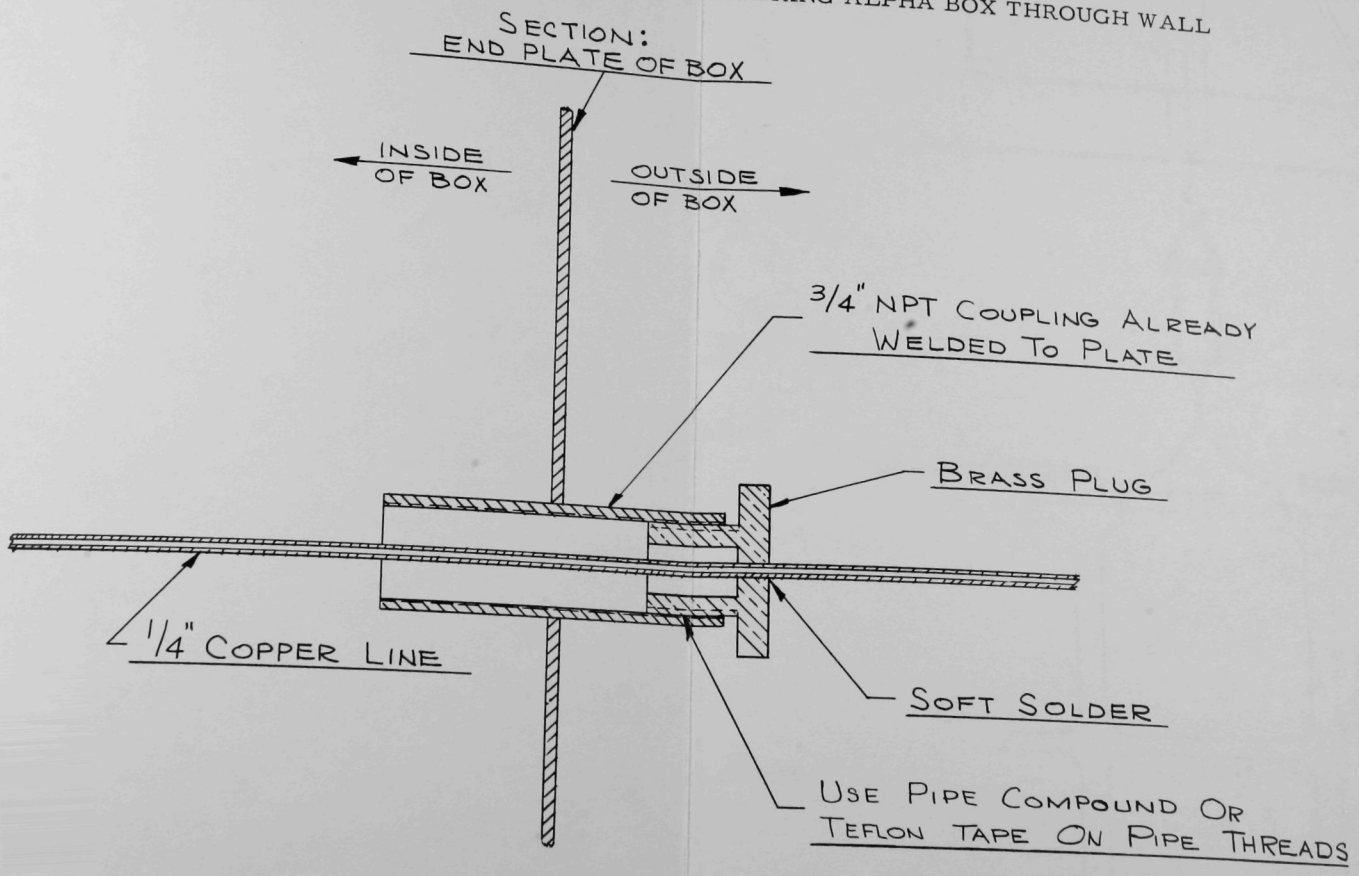
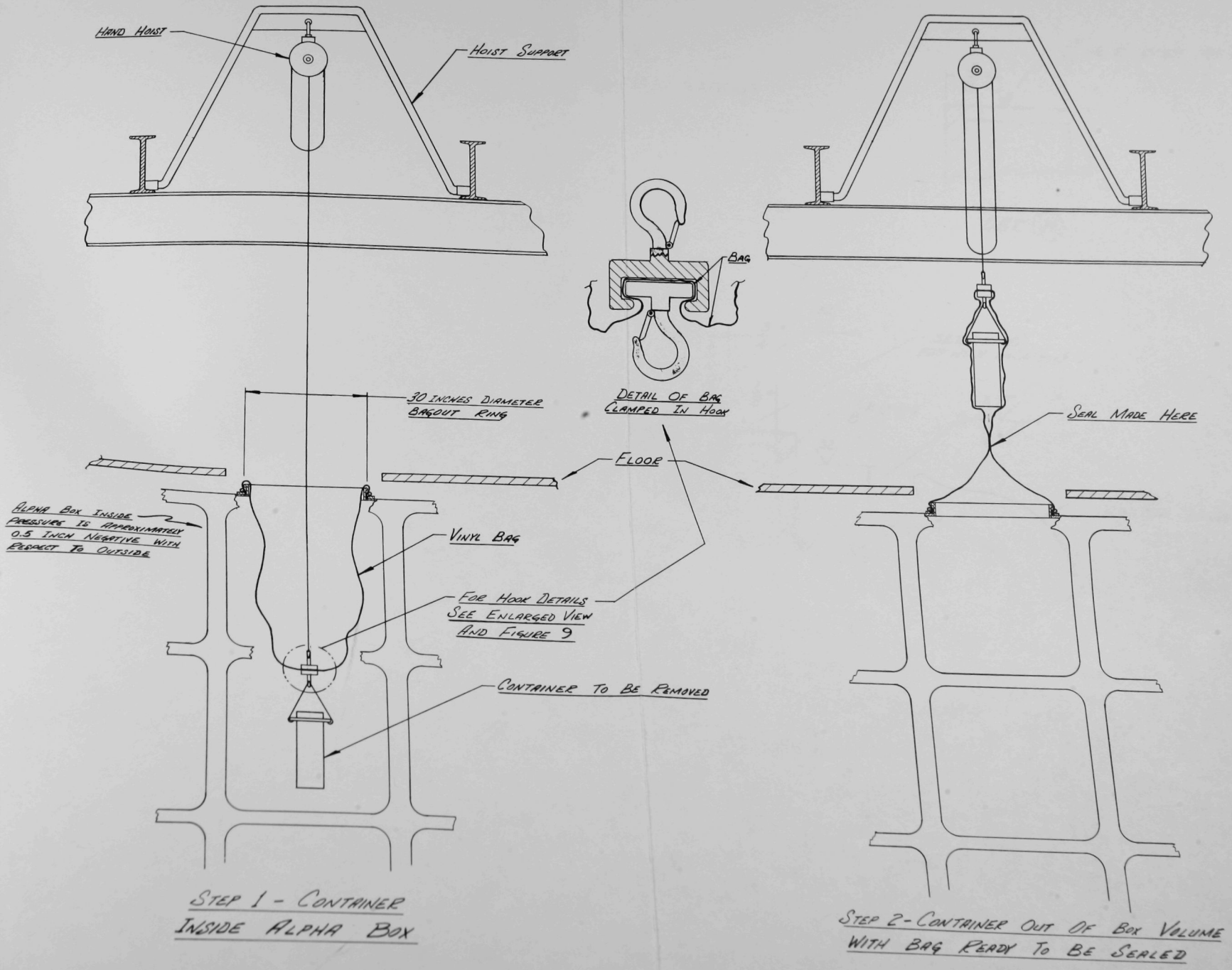


ILLUSTRATION OF METHOD FOR VERTICAL BAGOUT OF EQUIPMENT



STEP 1 - CONTAINER INSIDE ALPHA BOX

STEP 2 - CONTAINER OUT OF BOX VOLUME WITH BAG READY TO BE SEALED

Figure 9

CONSTRUCTION OF HOOK FOR VERTICAL BAGOUT

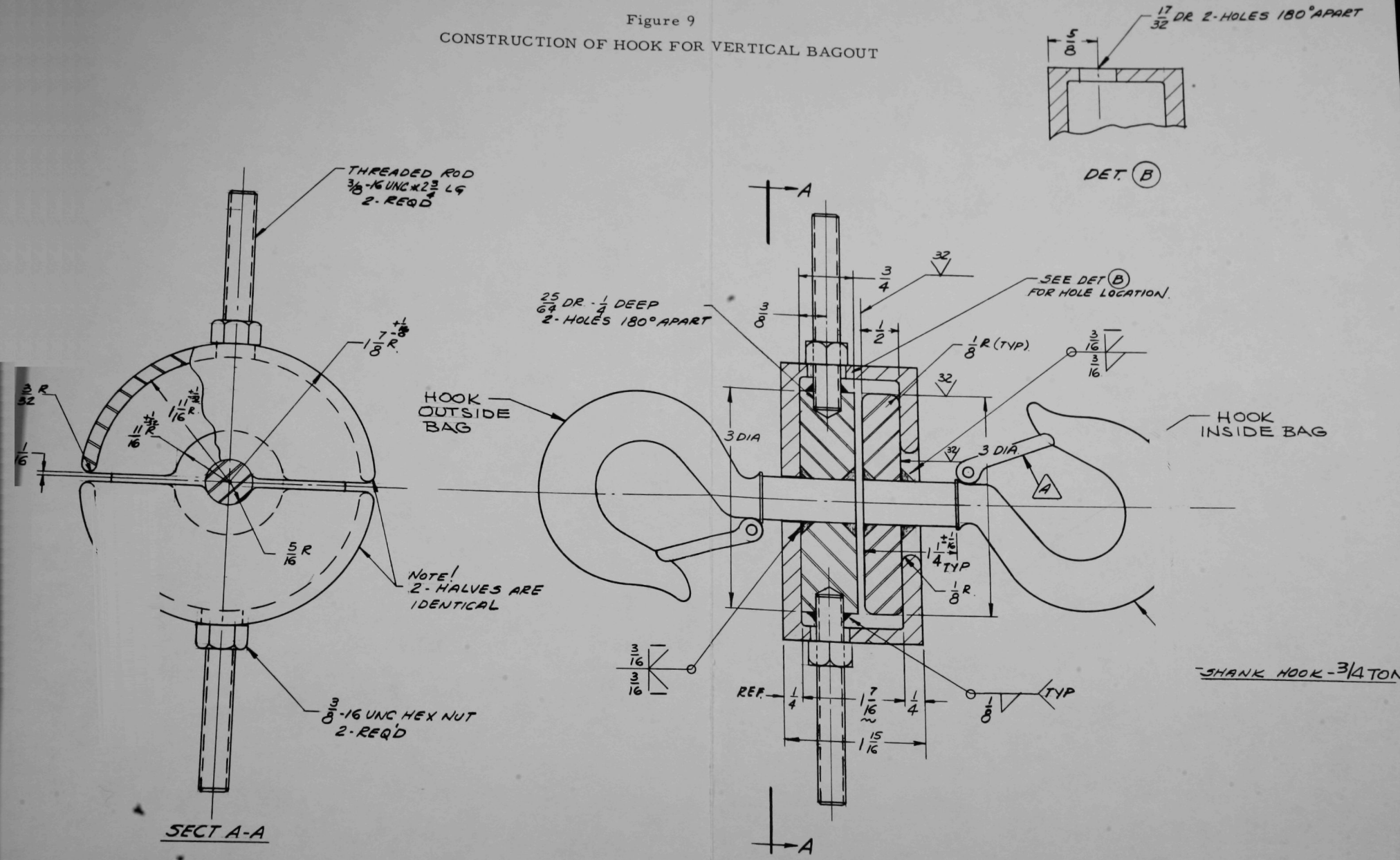


Figure 10

PERSONNEL LIFT FOR OPERATIONS AT THE LARGE GLOVEBOX

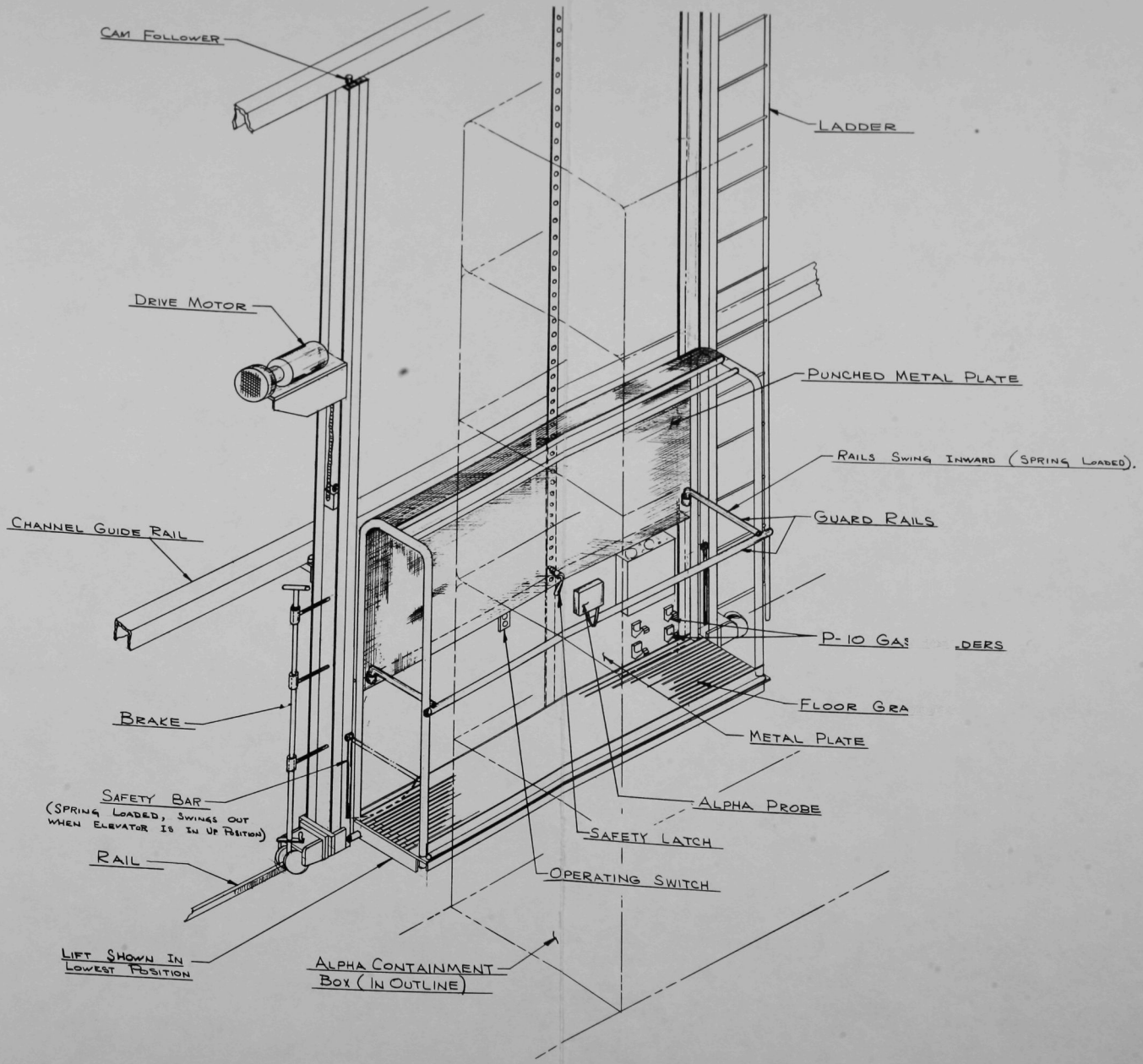


Figure 11

VENTILATION AIR AND PROCESS OFF-GAS FLOWS FOR ALPHA FACILITY

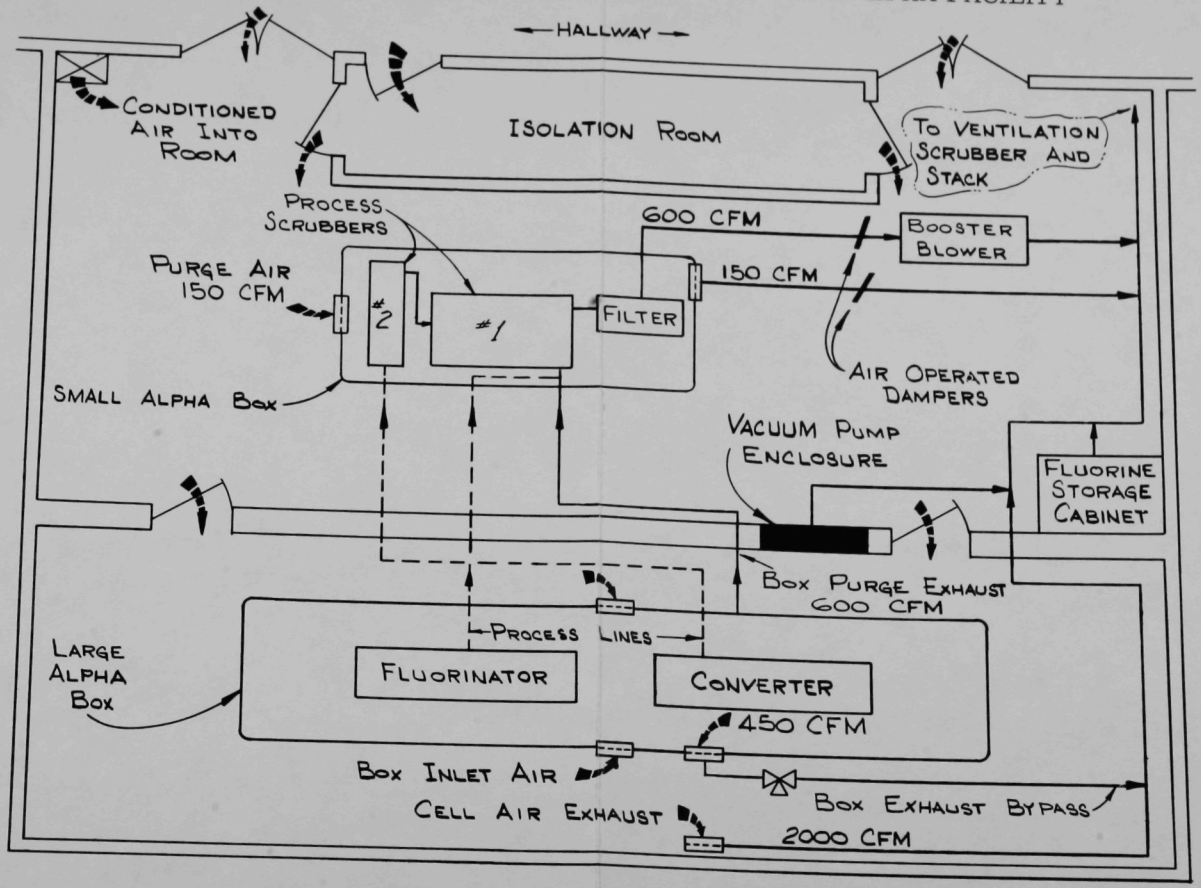


Figure 12

EXHAUST AIR SCRUBBER FOR VENTILATION SYSTEM

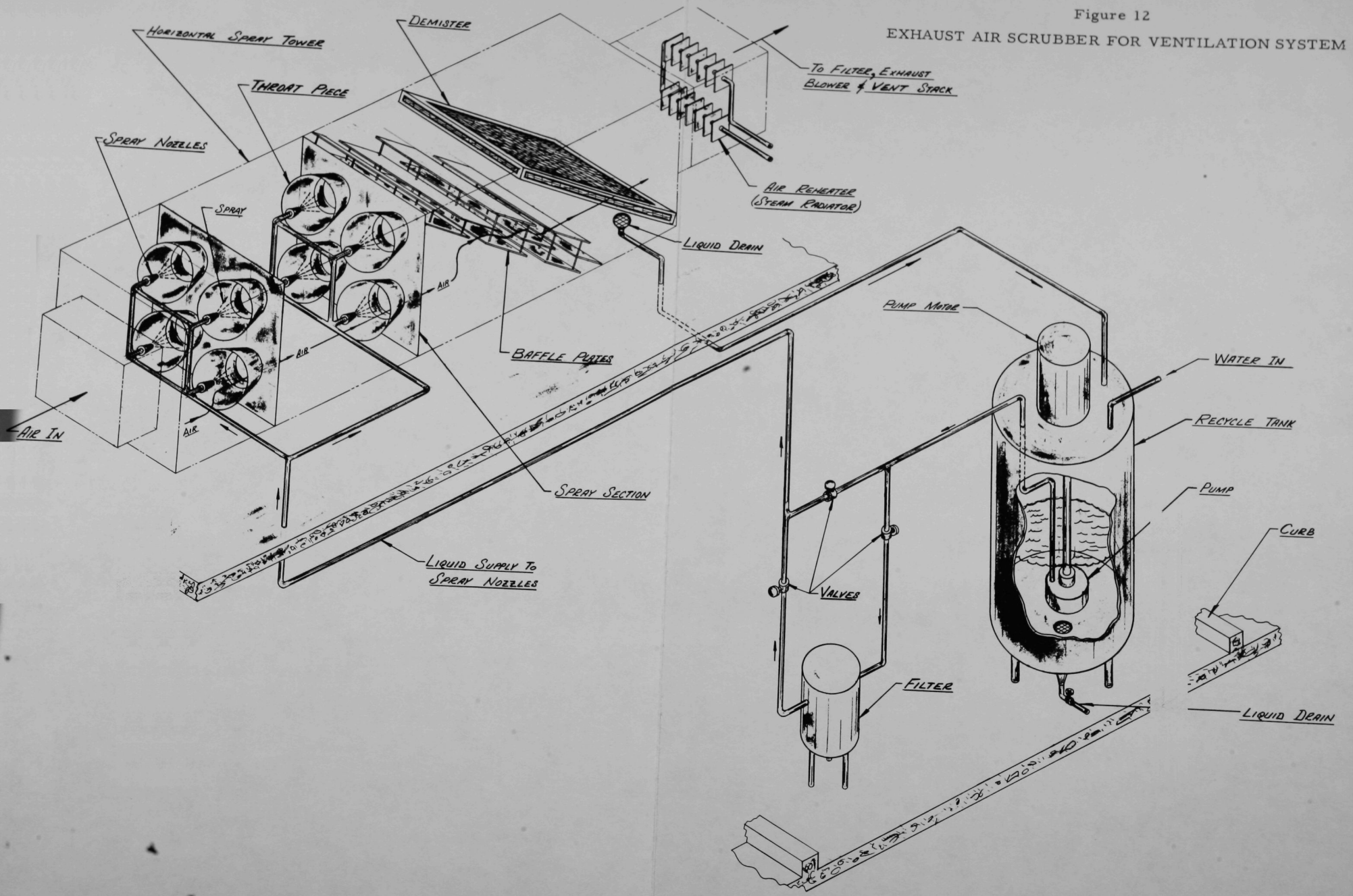


Figure 13

SMALL ALPHA BOX CONTAINING SCRUBBER SYSTEMS FOR LARGE ALPHA BOX EXHAUST AIR AND PROCESS OFF-GAS FROM FLUORINATOR AND CONVERTER

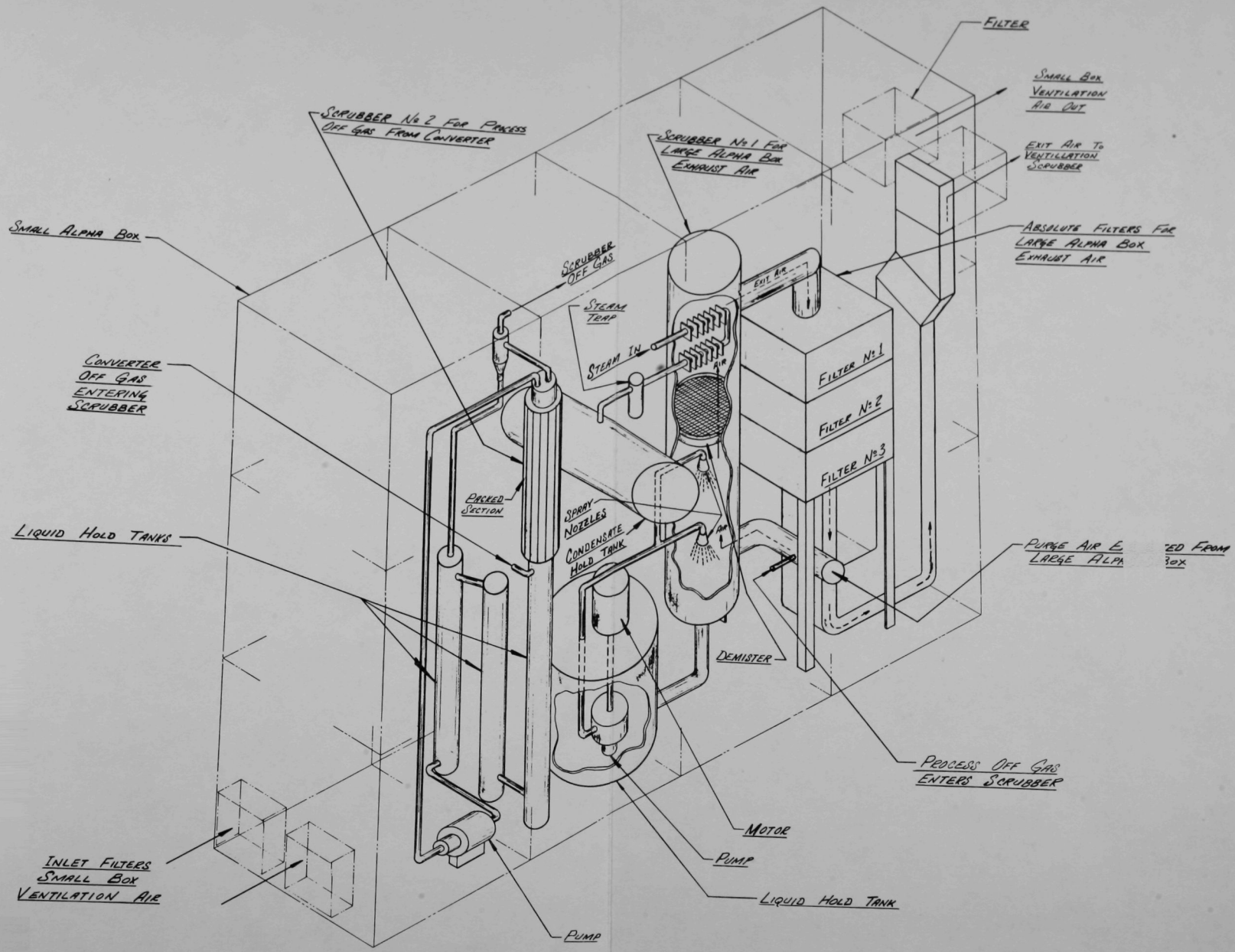


Figure 14

GAS SUPPLY SYSTEM

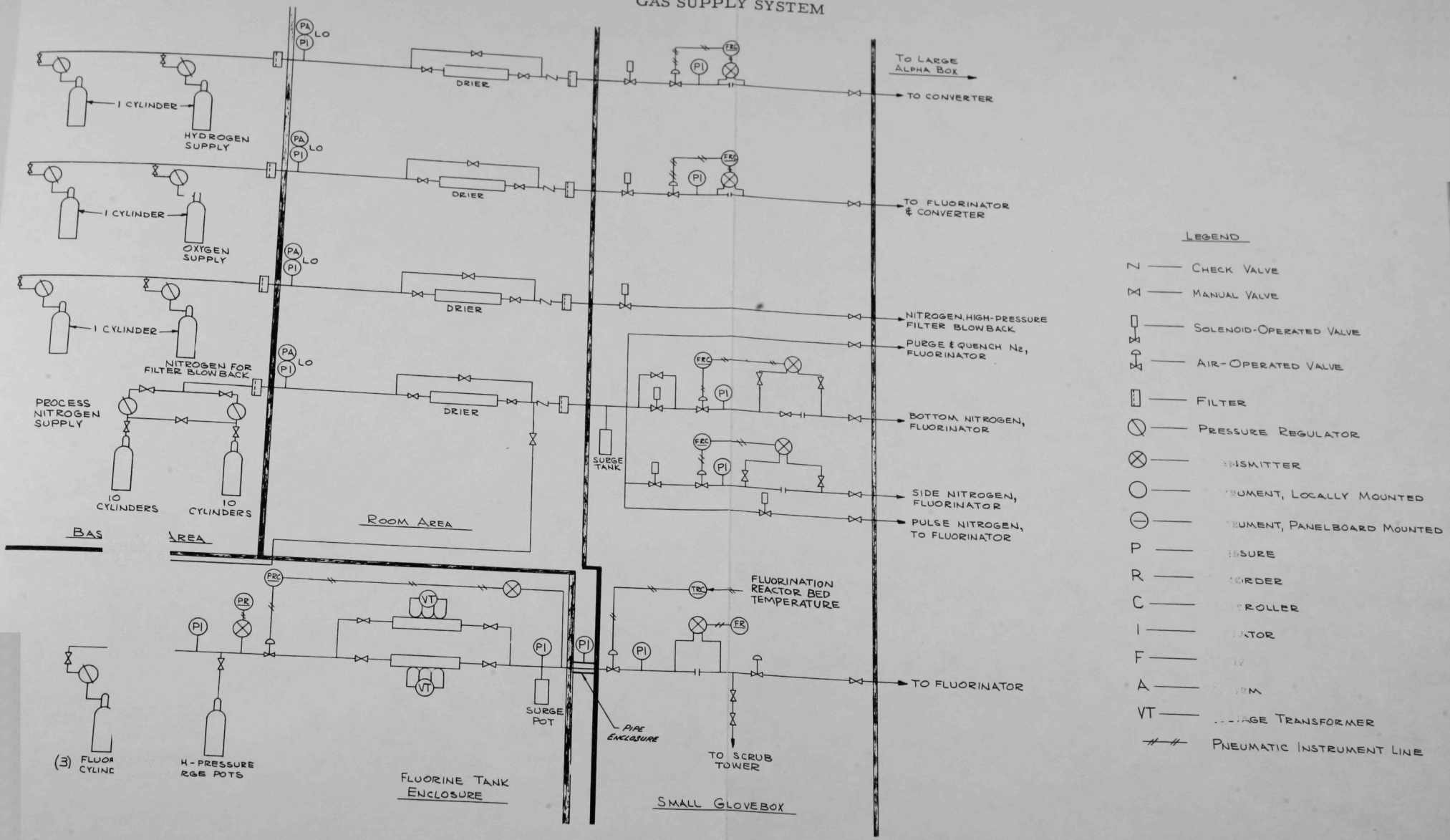
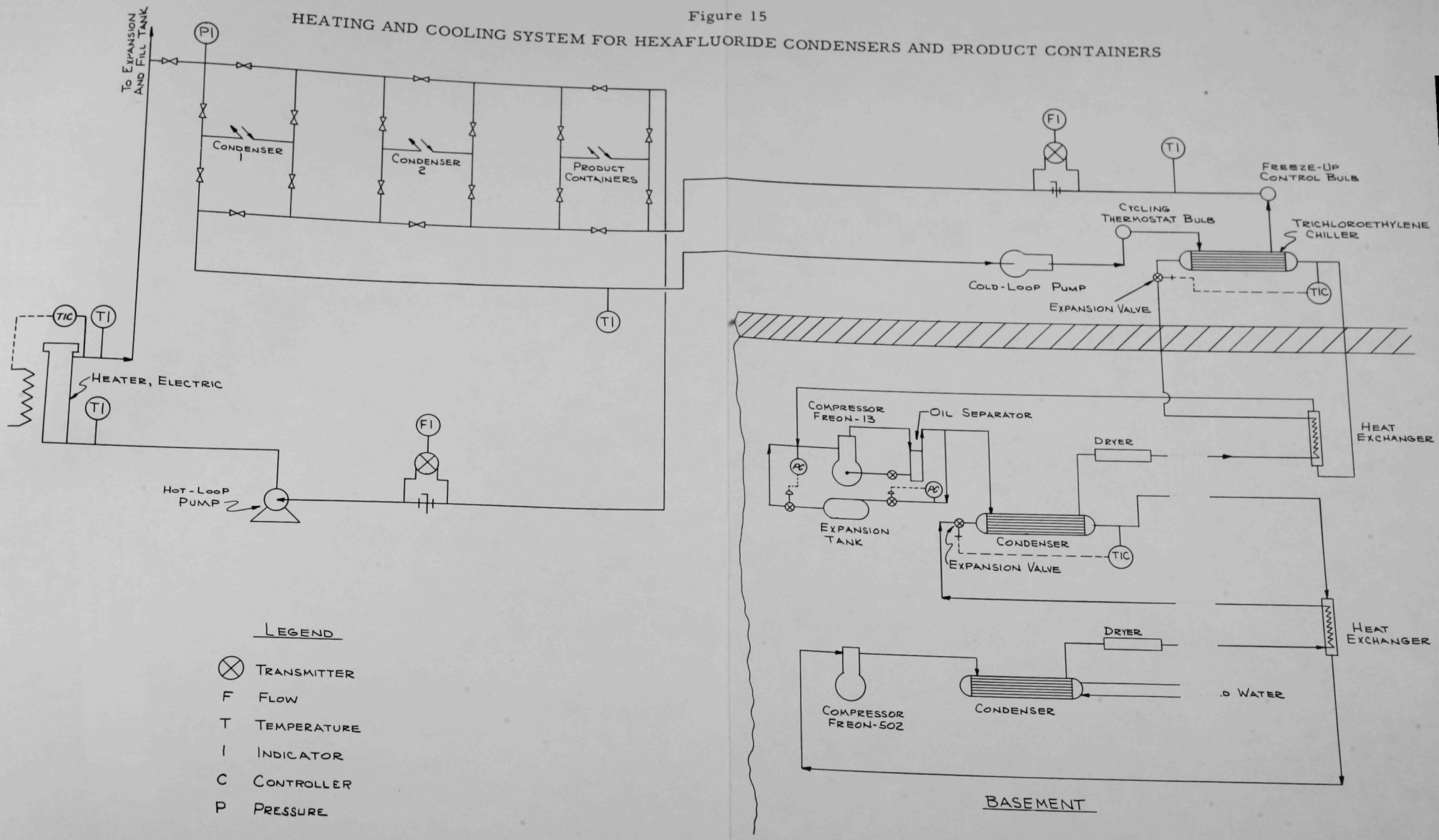
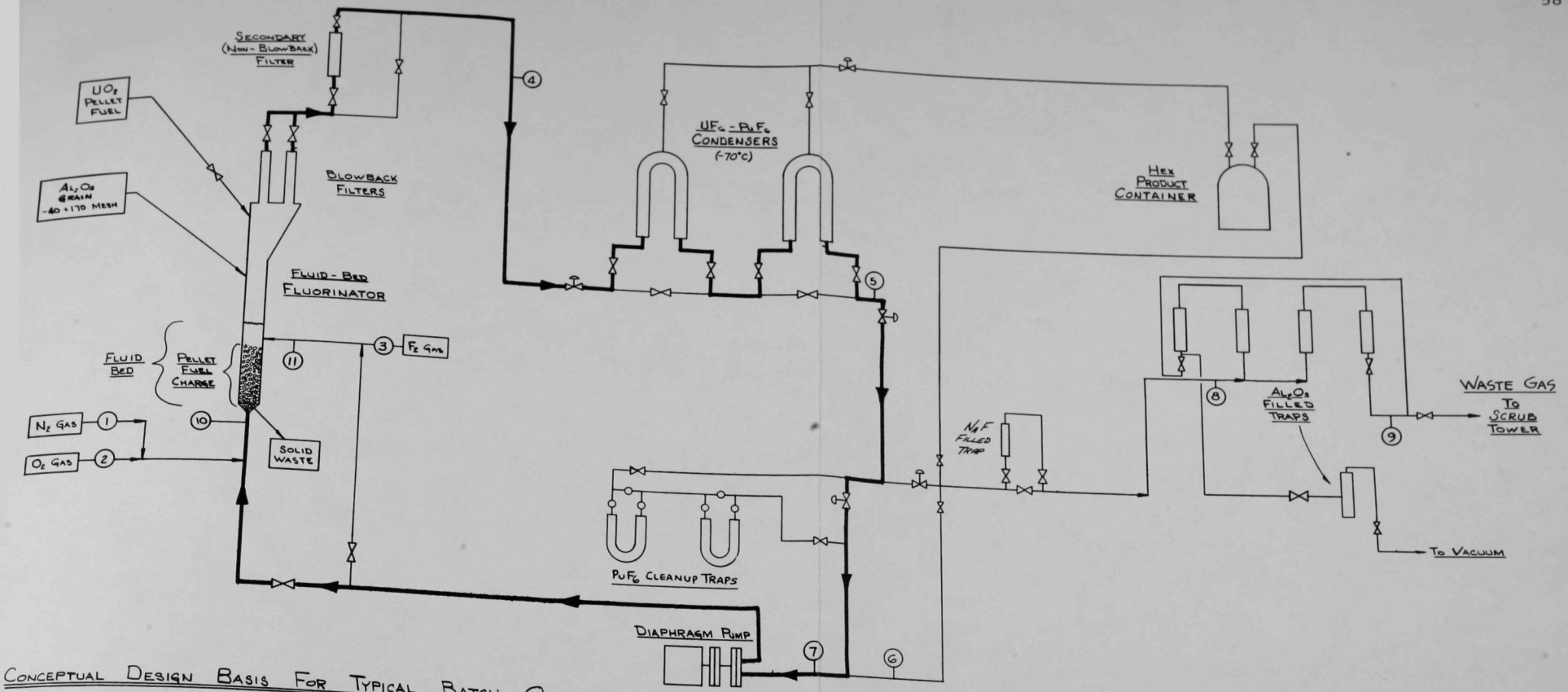


Figure 15

HEATING AND COOLING SYSTEM FOR HEXAFLUORIDE CONDENSERS AND PRODUCT CONTAINERS





Conceptual Design Basis For Typical Batch Operation

Charge (3 inch I.D. Fluorinator)
UO_2-PuO_2 Fuel 10. kg. Total
(PuO_2 Content 50 g Pu)

Batch Reaction Conditions

Primary Fluorination:
- Total Time 10 Hrs.
- Temp. 350°-500° C
- F_2 Inlet Concentration 10-25 M/o
- O_2 Inlet Concentration 2-10 M/o (Balance N_2)

Secondary Fluorination:
- Total Time 5 Hrs.
- Temp. 500°-550° C
- F_2 Inlet Concentration 50-90 M/o (Balance N_2)

Total Al_2O_3 in Reactor 7.0 kg. (Residence Time: 3 Batches)

Process Line		Flow Conditions[a]										
		1	2	3	4	5	6	7	8	9	10	11
Primary Fluorination												
N_2	(Lb. Mol./Hr.)×10³	135	0	0	164	164	135	29	135	135	135[d]	29
	Cu. Ft.[b]/Min.	0.92	0	0	1.1	1.1	0.92	0.19	0.92	0.92	0.92	0.19
UF_6[c]	(Lb. Mol./Hr.)×10³	0	0	0	8.2	Trace	Trace	Trace	0	0	0	Trace
	Cu. Ft./Min	0	0	0	0.055	Trace	Trace	Trace	0	0	0	Trace
F_2	(Lb. Mol./Hr.)×10³	0	0	33	9.9	9.9	8.2	1.7	1.7	0	0	34.7
	Cu. Ft./Min.	0	0	0.22	0.055	0.055	0.045	0.01	0.01	0	0	0.22
O_2[b]	(Lb. Mol./Hr.)×10³	0	12.0	0	24.5	24.5	20.2	4.3	20.2	20.2	12.0	4.3
	Cu. Ft./Min.	0	0.080	0	0.17	0.17	0.14	0.03	0.14	0.14	0.080	0.03
Secondary Fluorination												
N_2	(Lb. Mol./Hr.)×10³	Make-up	0	0	7.5	7.5	Low	7.5	Low	Low	7.5	0
	Cu. Ft./Min.	Make-up	0	0	0.05	0.05	Low	0.05	Low	Low	0.05	0
UF_6[c]	(Lb. Mol./Hr.)×10³	0	0	0	Trace	0	0	0	0	0	0	0
	Cu. Ft./Min.	0	0	0	Trace	0	0	0	0	0	0	0
F_2	(Lb. Mol./Hr.)×10³	0	0	Make-up	75	75	Low	75	Low	0	75	Make-up
	Cu. Ft./Min.	0	0	Make-up	0.50	0.50	Low	0.50	Low	0	0.50	Make-up

a — Note convention on units: e.g., where (Lb. Mol./Hr.)×10³ is given as 8.2, this is to be read as 0.0082 Lb. Mol./Hr.
b — Cu. Ft. at 25°C and 1 Atm.
c — Includes PuF_6 for Pu containing fuel.
d — Gas pulsing may be beneficial.

Figure 16

PROCESS MATERIAL FLOW DIAGRAM FOR FLUORINATIO
OF URANIUM DIOXIDE PELLET FUEL

Figure 17

EQUIPMENT-INSTRUMENTATION FLOWSHEET FOR FLUORINATION SYSTEM

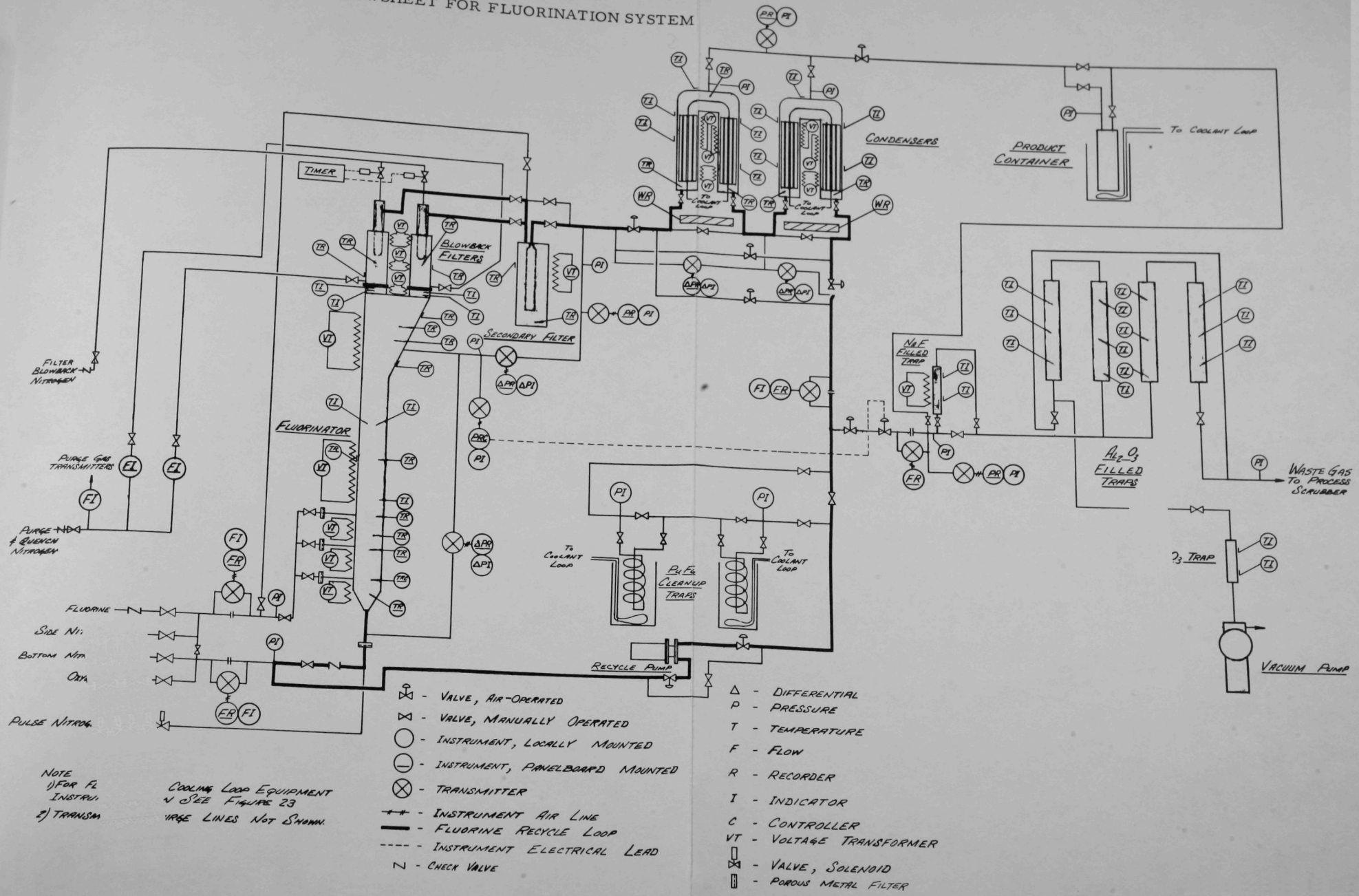


Figure 18

LAYOUT OF FLUORINATION SYSTEM PROCESS EQUIPMENT AND PIPING

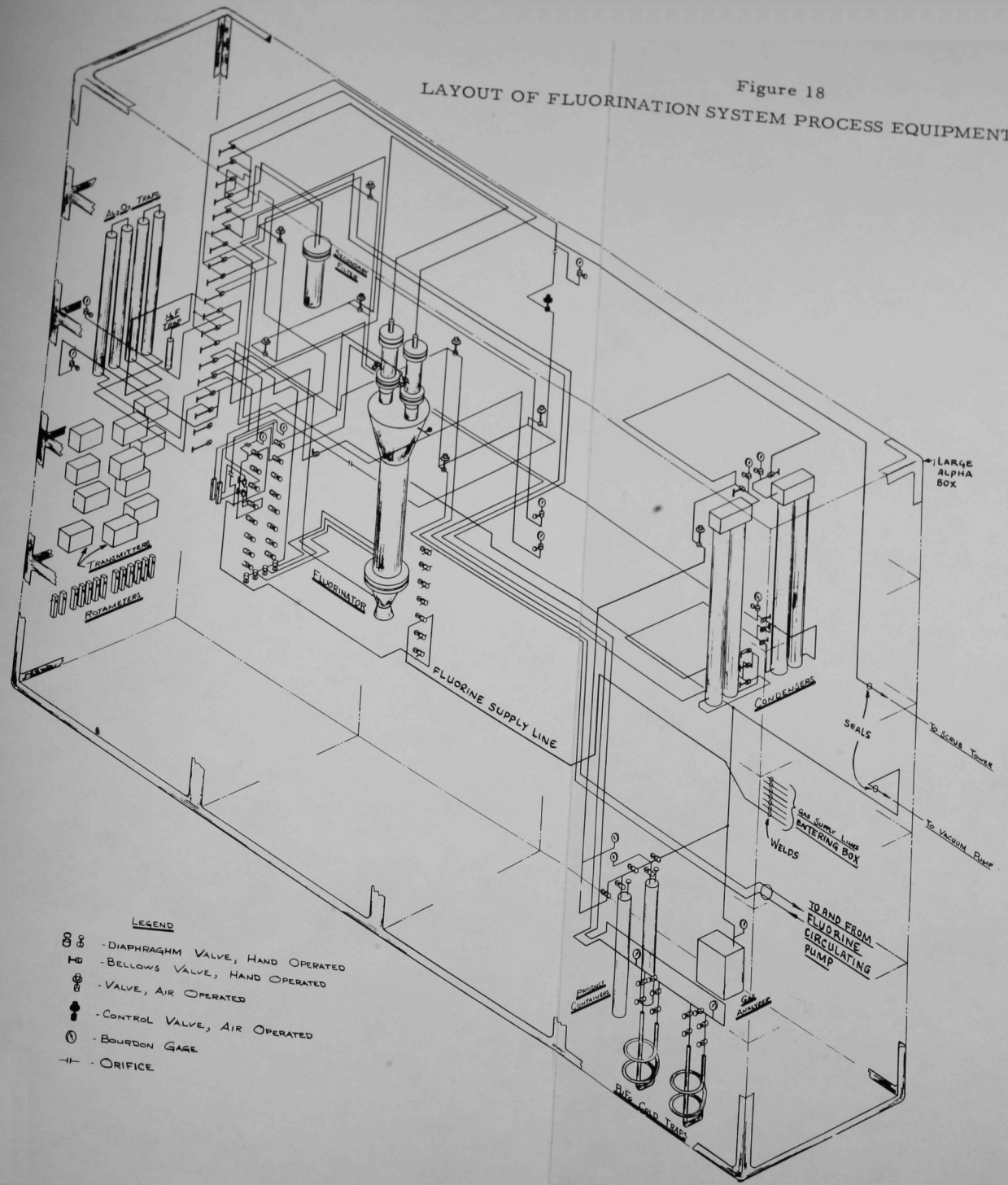


Figure 19
LAYOUT OF FLUORINE CIRCULATION PUMP SYSTEM

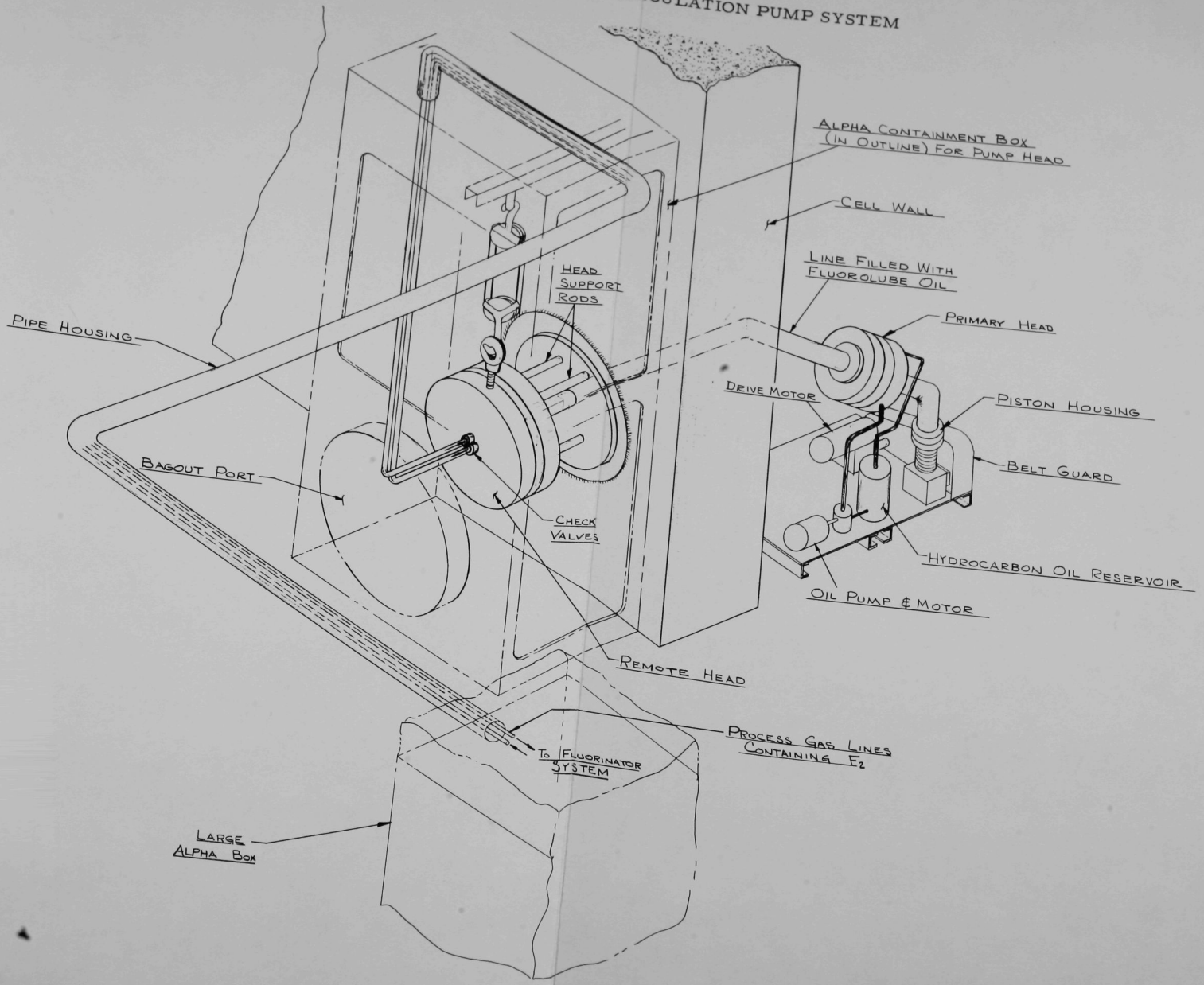
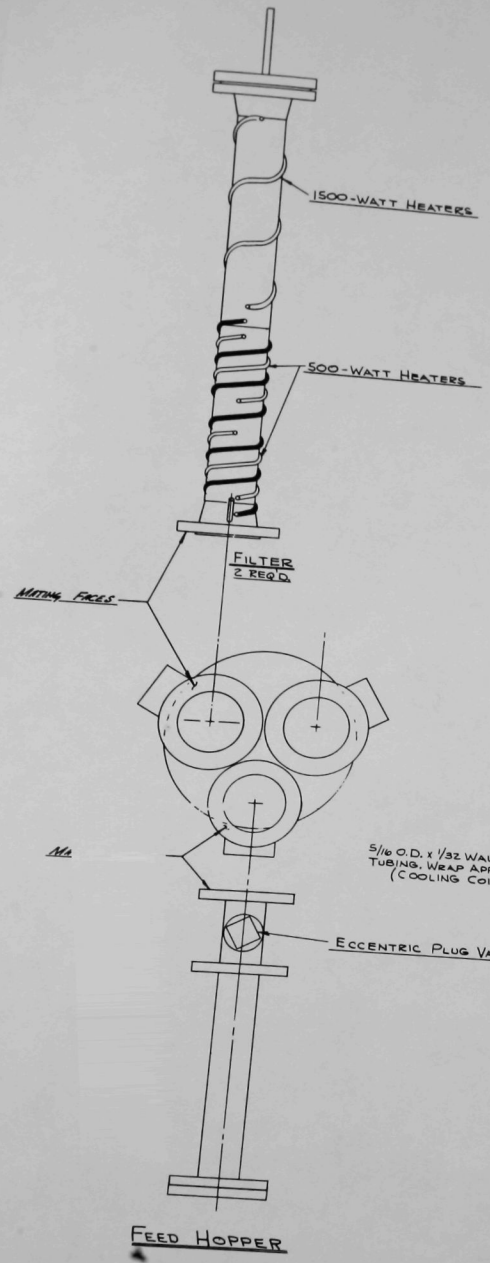
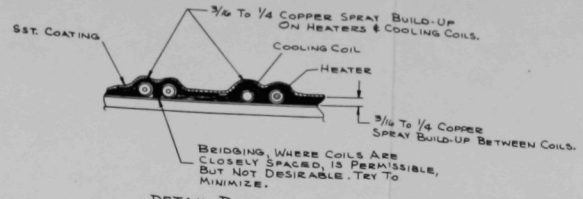


Figure 20

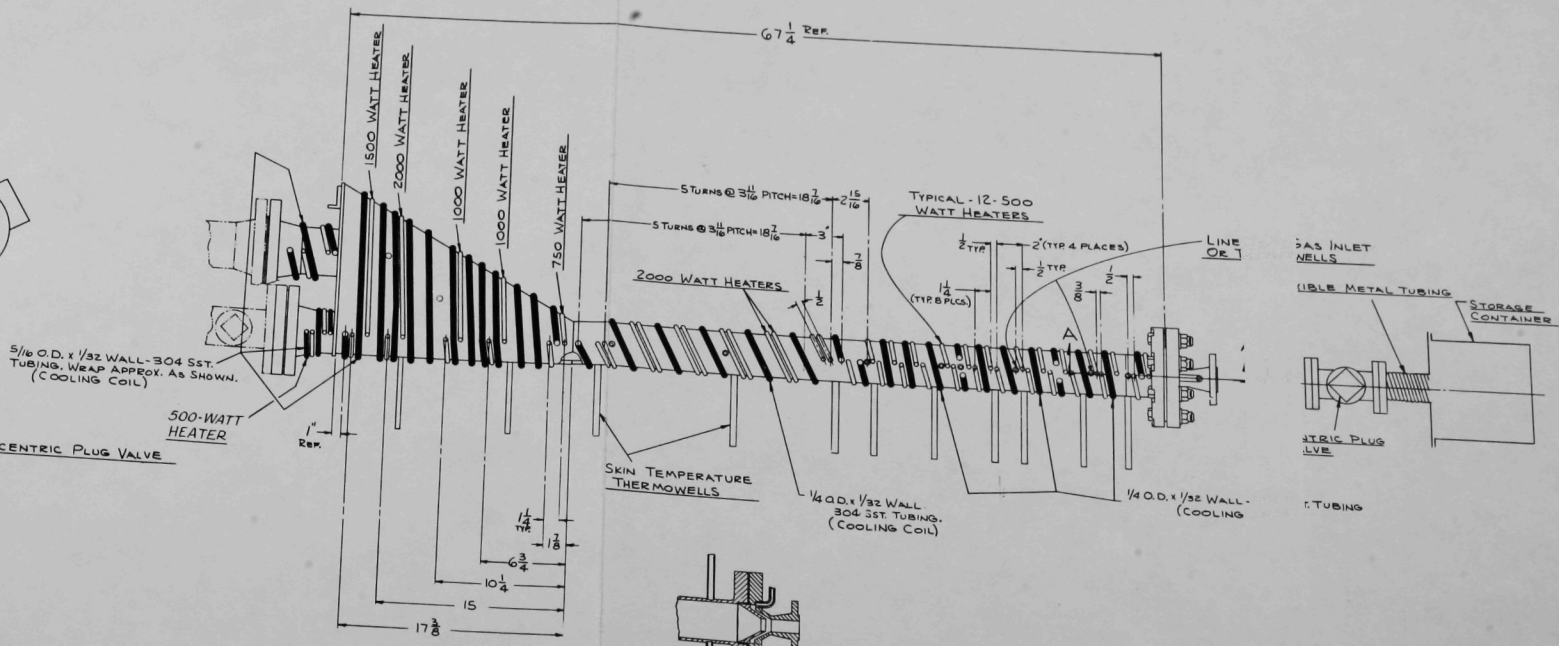
CONSTRUCTION DETAILS OF FLUID-BED FLUORINATOR





DETAIL B

NOTES:
1- HELIUM LEAK TEST WITH MASS SPECTROMETER AS PER SPECS.
2- CLEAN & DEGREASE AS PER SPECS. CE-0102.
3- ALL HEATERS & COOLING COILS TO BE COPPER SPRAYED AS SHOWN IN DETAIL 'B'.
4- ALL COPPER SPRAYED SURFACES TO BE FINALLY COVERED BY STAINLESS STEEL SPRAY.
5- ALL HEATERS & COOLING COILS WILL BE ATTACHED BY TACKWELDING NICKEL CLAMPS.



SECTION A-A

BAFFLED-CONE GAS DISTRIBUTER

Figure 21

CONSTRUCTION DETAILS OF SECONDARY FILTER

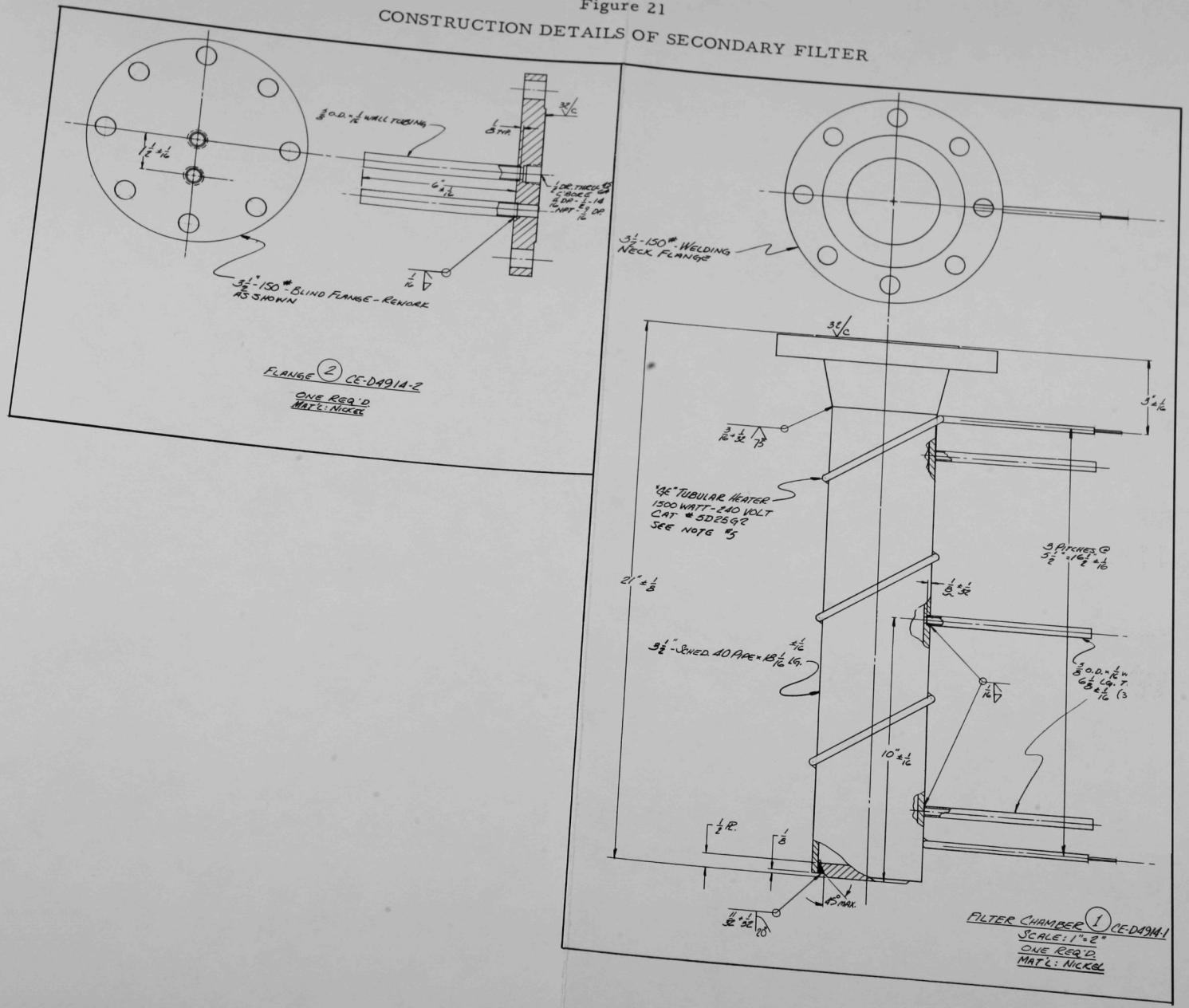
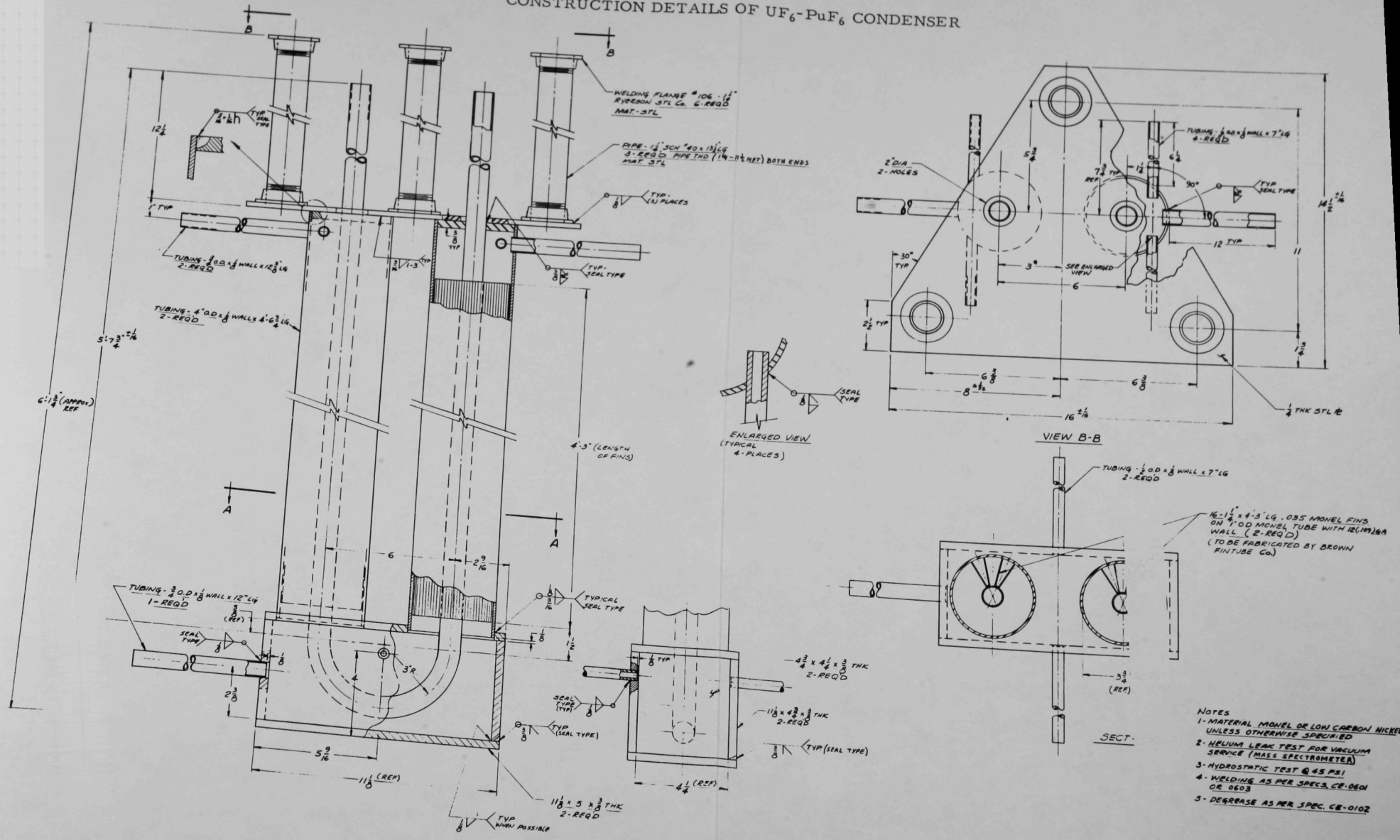


Figure 22

CONSTRUCTION DETAILS OF UF_6-PuF_6 CONDENSER



Notes

1. MATERIAL MONEL OR LOW CARBON NICKEL UNLESS OTHERWISE SPECIFIED
2. HELIUM LEAK TEST FOR VACUUM SERVICE (MASS SPECTROMETER)
3. HYDROSTATIC TEST @ 45 PSI
4. WELDING AS PER SPECS. CE-0601 OR 0603
5. DEGREASE AS PER SPEC. CE-0102

Figure 23

AIR-WATER, TWO-PHASE FLOW COOLING SYSTEM FOR FLUORINATOR

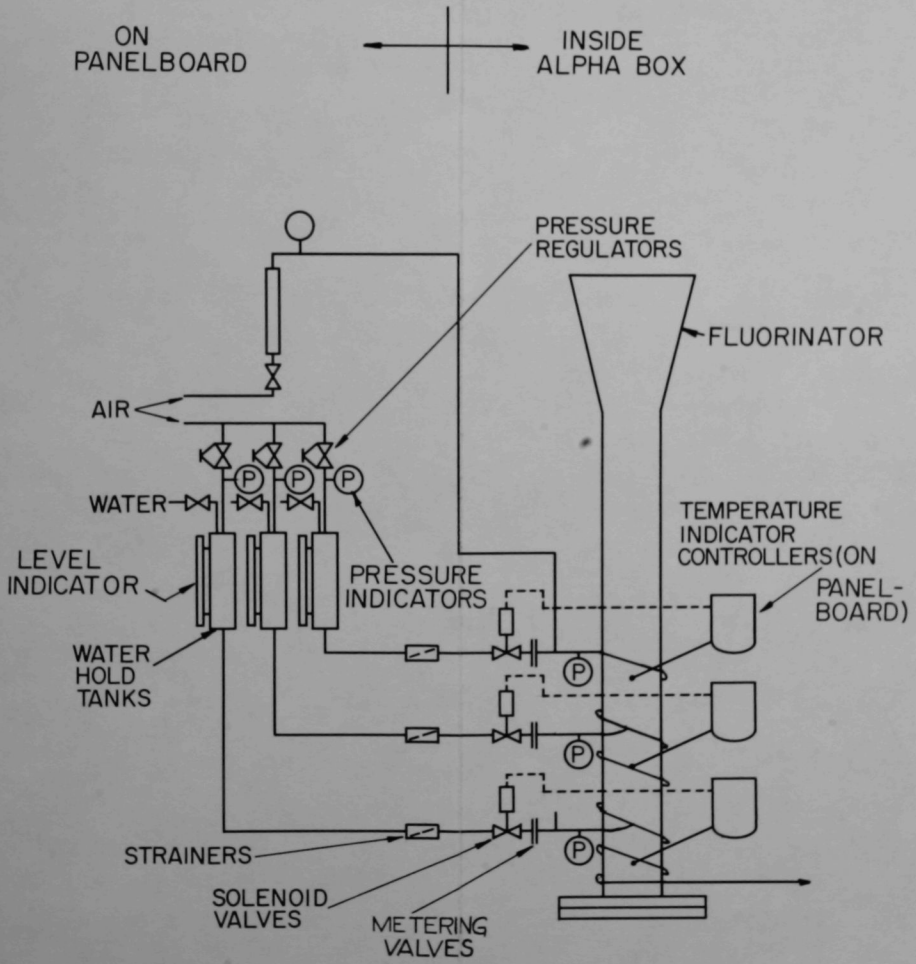


Figure 24

PROCESS GAS CONTINUOUS ANALYZER SYSTEM

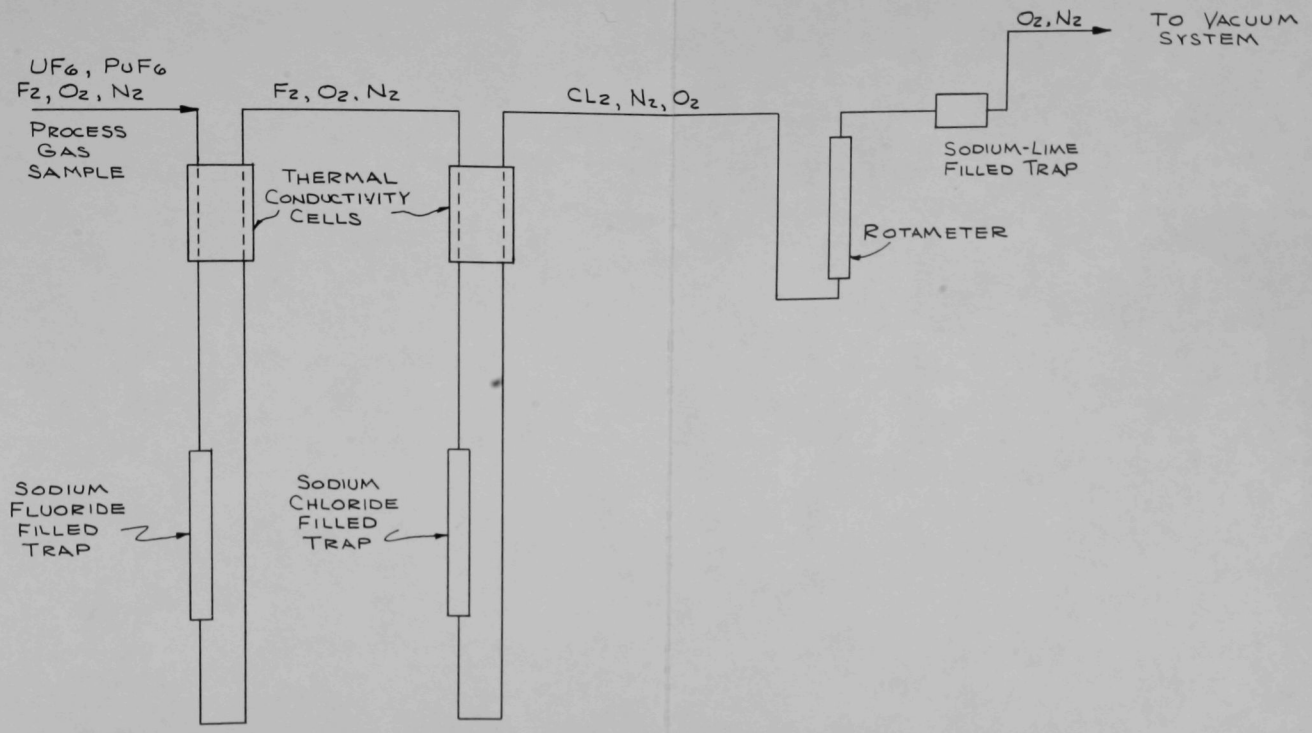


Figure 25

PROCESS VALVE, AIR-OPERATED TYPE

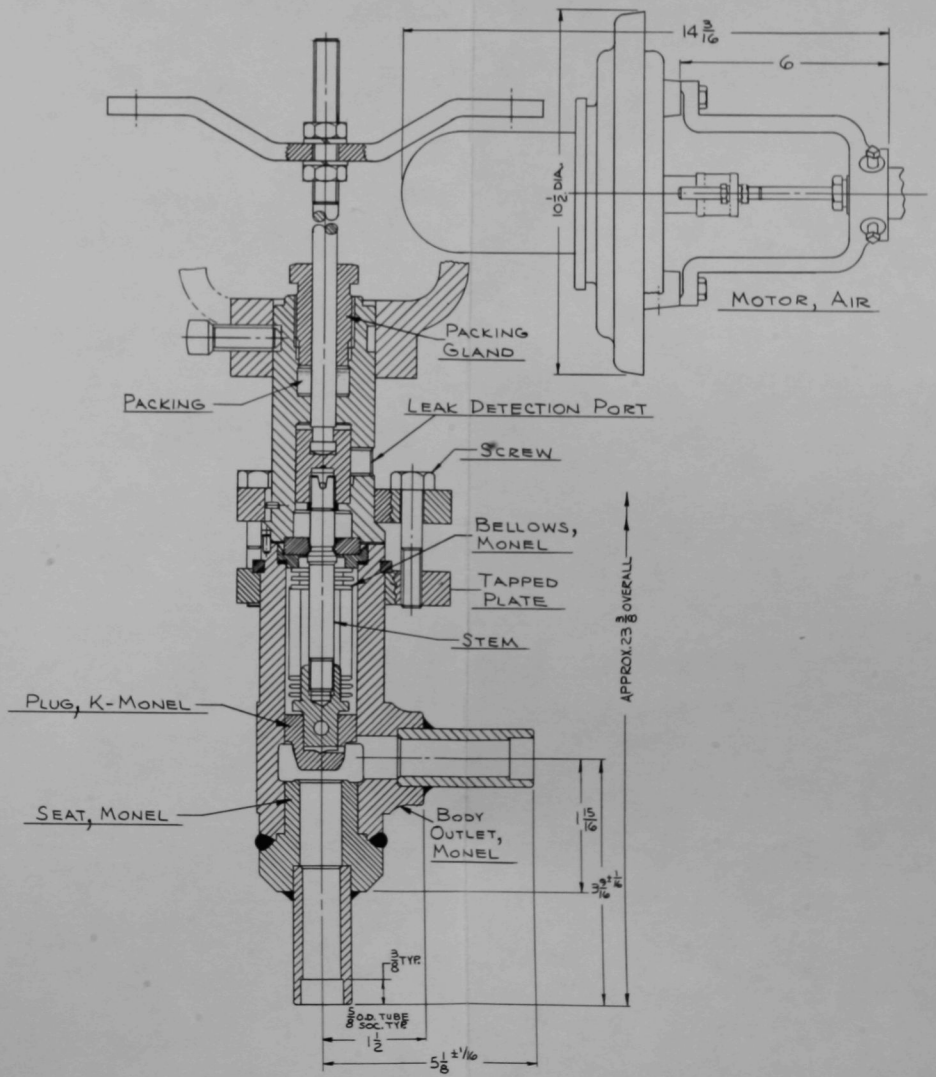


Figure 26

PRIME MOVER UNIT OF FLUORINE CIRCULATION PUMP: VIEW DURING INSTALLATION

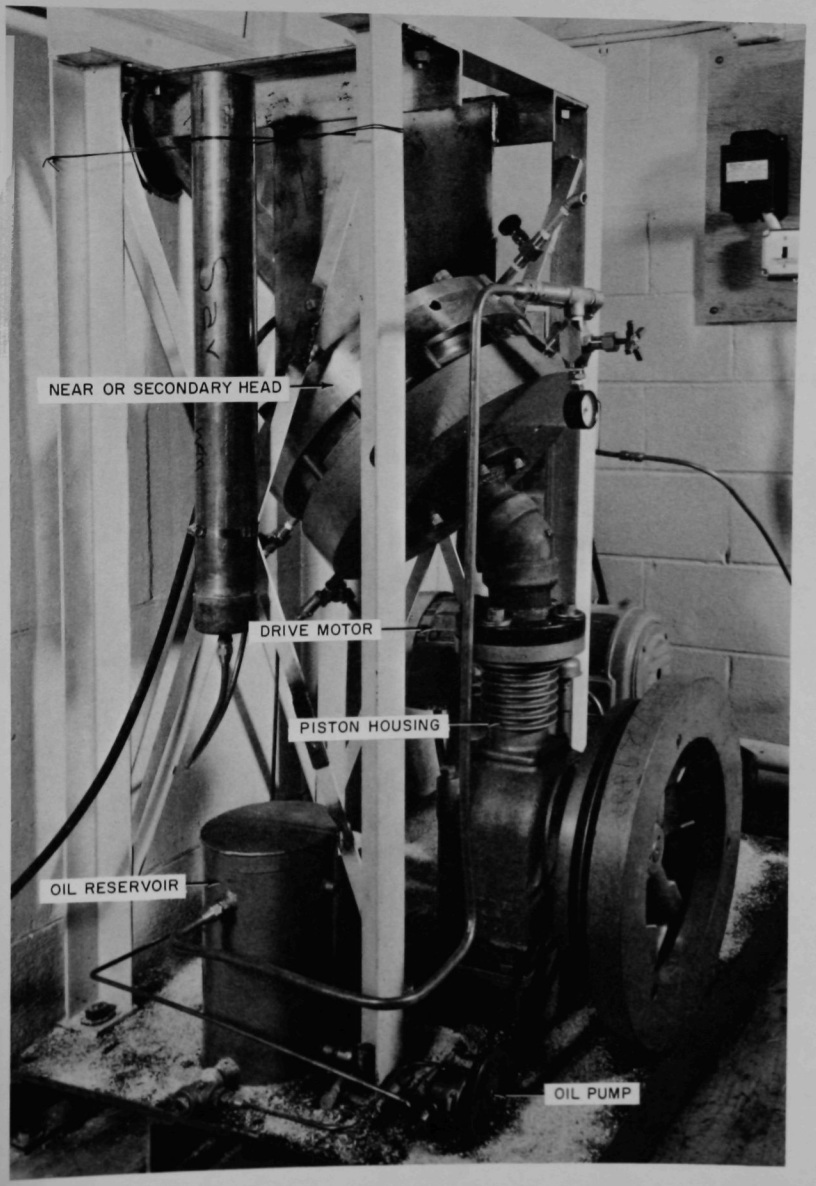


Figure 27

FLUORINATOR BEING LOWERED INTO LARGE ALPHA BOX THROUGH VERTICAL BAGOUT PORT DURING INSTALLATION: VIEW DURING INSTALLATION

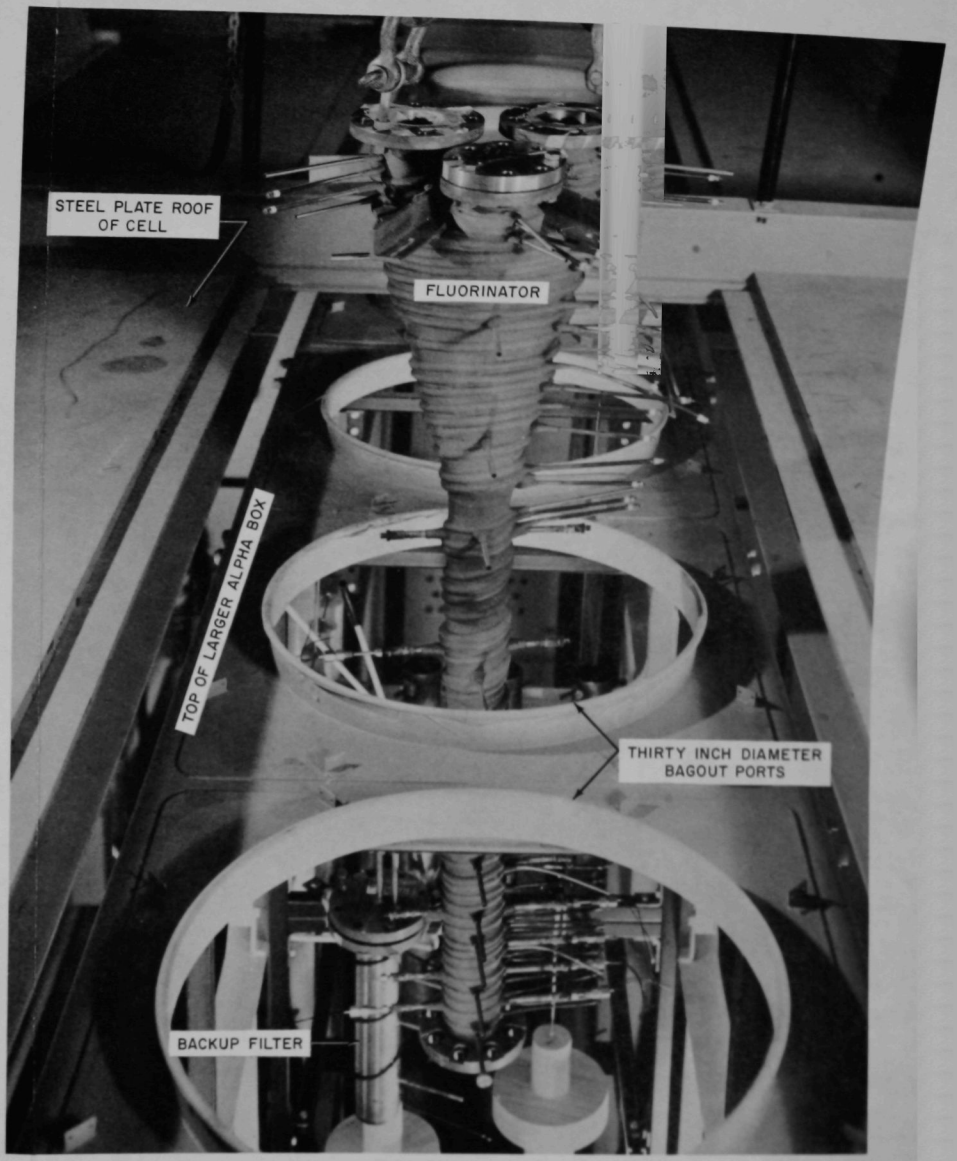


Figure 28

PANELBOARD AREA AND TOP OF SMALL ALPHA BOX: VIEW DURING INSTALLATION

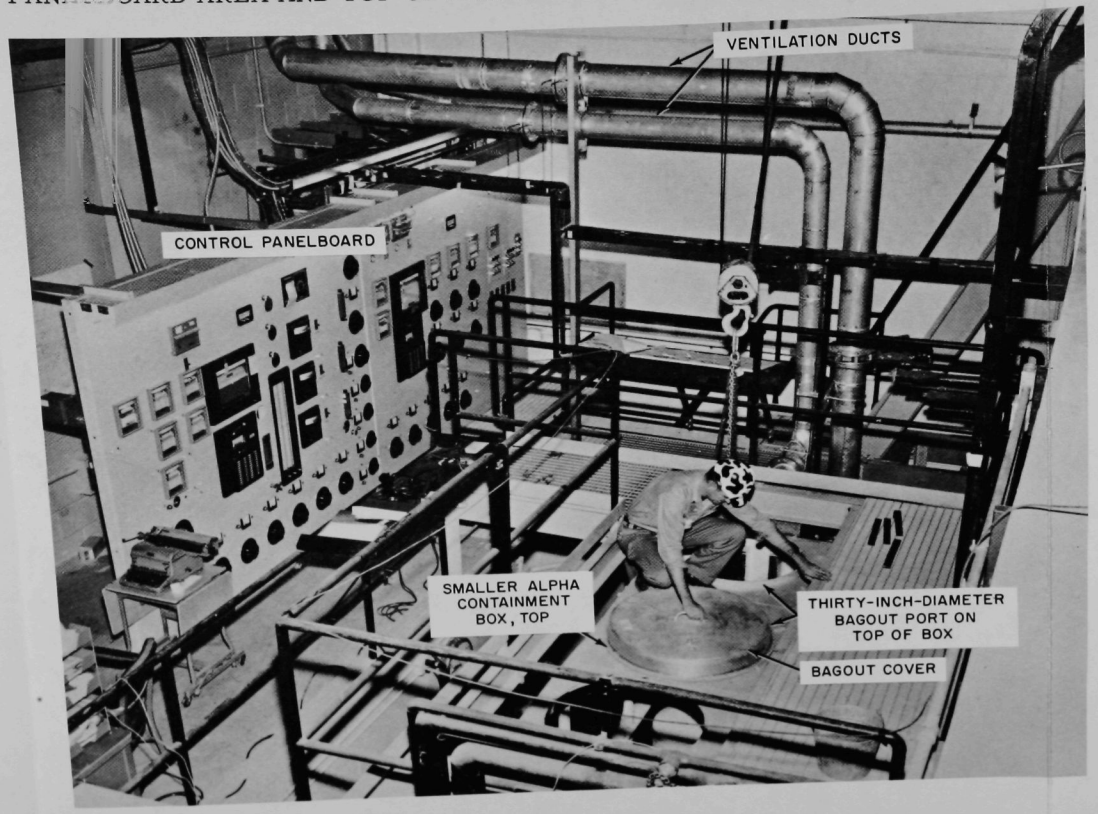
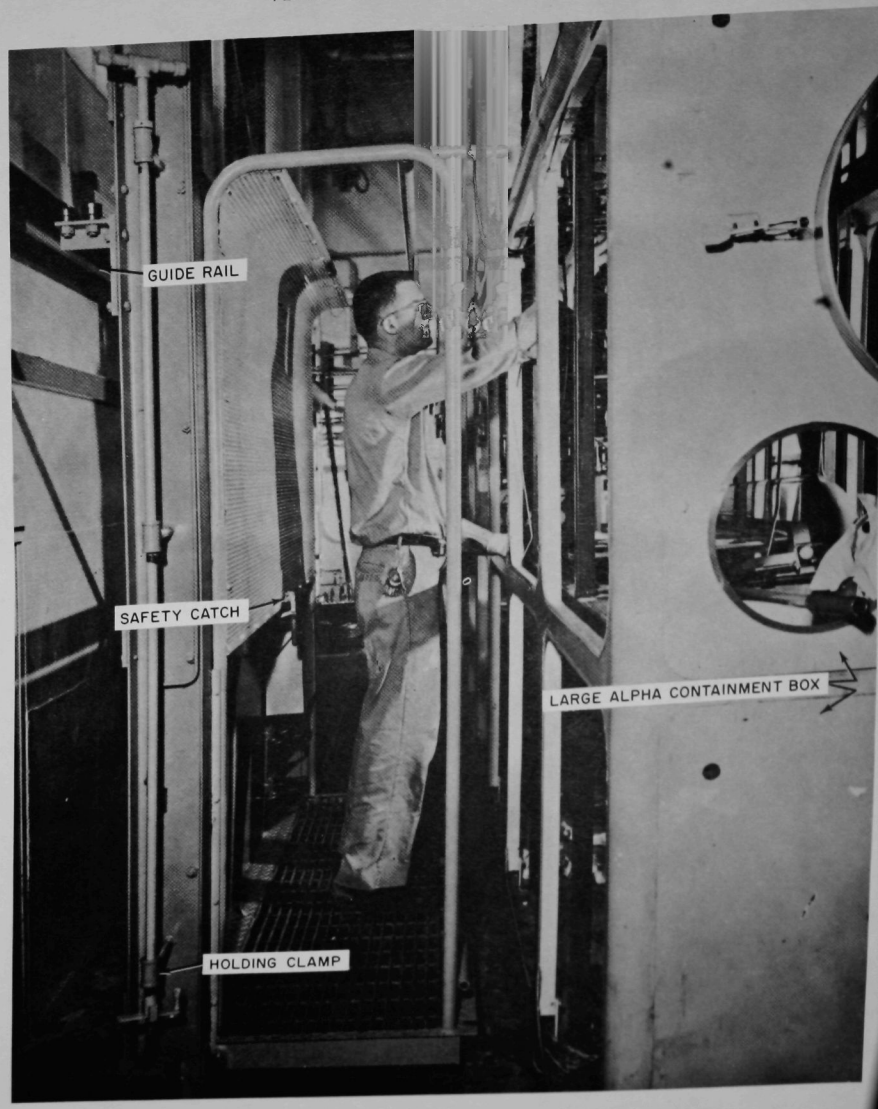
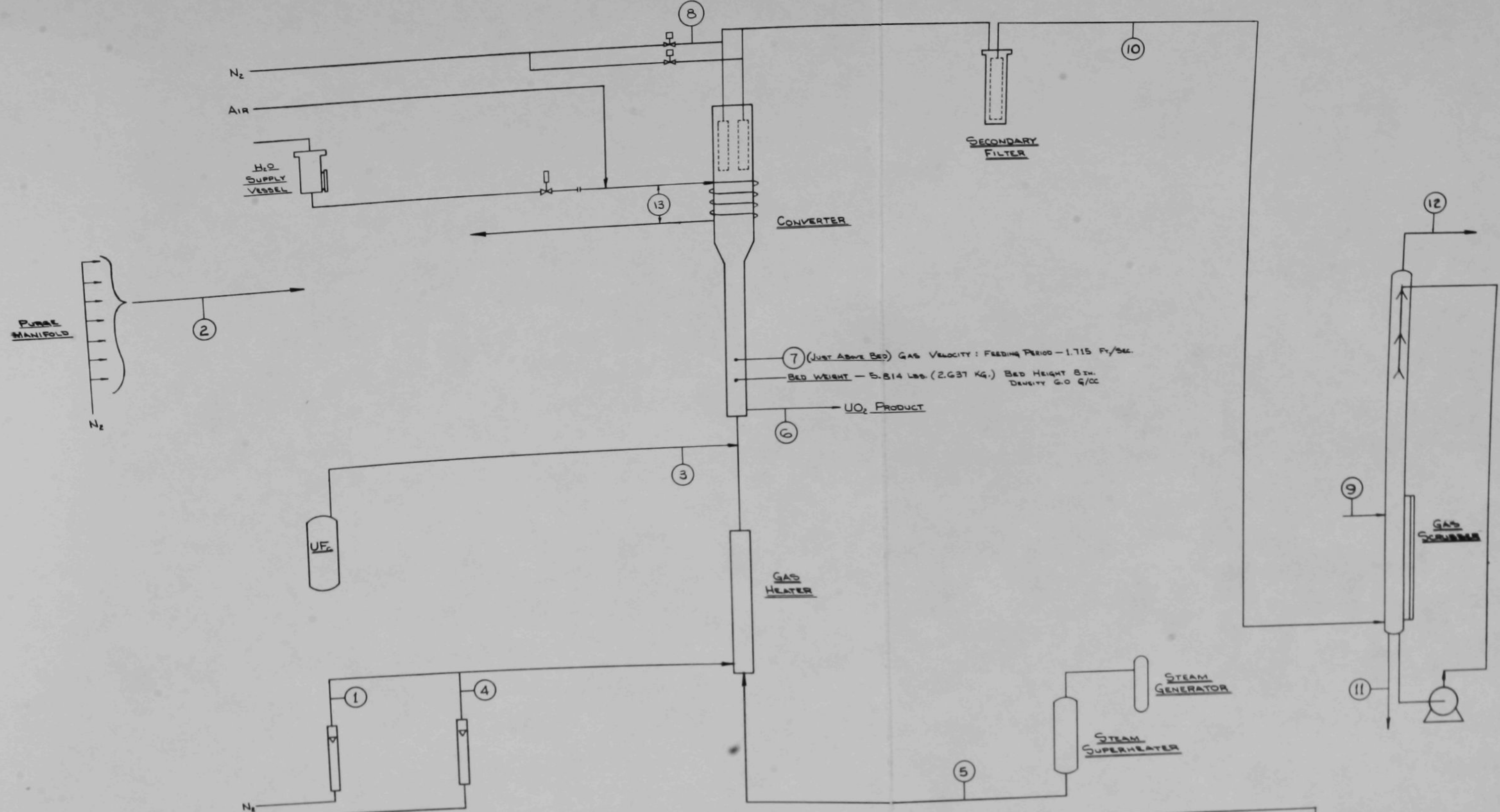


Figure 29

PERSONNEL LIFT FOR ALPHA BOX OPERATION: VIEW DURIN LLATION





FLUIDIZATION START-UP

	Point No.	1	2	3	4	5	6	7	8	9	10	11	12④	13
Temperature, C		25	25					0-650	100				84 (183 F)	
Pressure, PSIA		16.8	Differs 14.7-16.8					14.9	14.8		14.75		14.75	
N_2	LB. MOL./HR.	0.0873	0.0313						0.0276				0.1463	
	LB./HR.	2.449	0.875						0.772				4.096	
	CU. FT./HR.	30.000	12.000						12.670				57.237	
UF_6	LB. MOL./HR.			0										
	LB./HR.													
	CU. FT./HR.				0									
H_2	LB. MOL./HR.												0.0067	
	LB./HR.												0.146	
	CU. FT./HR.					0							2.111	
H_2O	LB. MOL./HR.													
	LB./HR.													
	CU. FT./HR.						0							
UO_2	LB. MOL./HR.													
	LB./HR.													
	CC													
HF	LB. MOL./HR.													
	LB./HR.									0				
	CU. FT./HR.													
KOH	LB. MOL./HR.													
	LB./HR.											0		
	CU. FT./HR.													
KF	LB. MOL./HR.													
	LB./HR.													30
	CU. FT./HR.													Varies
Air	CU. FT./HR.													
H_2O	CC/MIN.													

MATERIAL BALANCES ARE BASED ON THESE REACTIONS OCCURRING DURING THE THIRTY-MINUTE PERIODS —

FEEDING PERIOD:
70% $6UF_6 + 18H_2O \rightarrow 2U_3O_8 + 36HF + O_2$ (ASSUMPTION IS MADE THAT O_2 WILL COMBINE WITH THE H_2 PRESENT TO FORM H_2O) ; 30% $UF_6 + H_2 \rightarrow UF_4 + 2HF$

CLEAN-UP PERIOD:
$U_3O_8 + 2H_2 \rightarrow 3UO_2 + 2H_2O$; $UF_4 + 2H_2O \rightarrow UO_2 + 4HF$

FEEDING PERIOD (30 MINUTES)

	Point No.	1	2	3	4	5	6	7	8	9	10	11	12④	13
Temperature, C			25	70	25	200		650	100		100		45C (113F)	
Pressure, PSIA			Differs 14.7-16.8	16.8	16.8	16.8		14.7	14.8		14.7		14.7	
N_2	LB. MOL.	0	0.0156						0.0138		0.0294		0.0294	
	LB.		0.438						0.386		0.823		0.825	
	CU. FT.		6.000						6.718		14.423		12.298	
UF_6	LB. MOL.			0.0021									0	
	LB.			0.728										
	CU. FT.			0.816										
H_2	LB. MOL.				0.0351						0.0340		0.0340	
	LB.				0.070						0.068		0.068	
	CU. FT.				12.048						16.698		14.285	
H_2O	LB. MOL.					0.0054					0.0015		0.0067	
	LB.					0.097					0.273		0.121	
	CU. FT.					2.923					0.749		1.800	
UO_2	LB. MOL.						0				0			
	LB.													
	CC													
HF	LB. MOL.										0.0099		0	
	LB.										0.198			
	CU. FT.										4.807			
KOH①	LB. MOL.									0.0099				
	LB.									0.557				
	CU. FT.									—				
KF①	LB. MOL.											0.00962		
	LB.											0.5588		
	CU. FT.											—		
Air	CU. FT.													45
H_2O	CC/MIN.													Varies

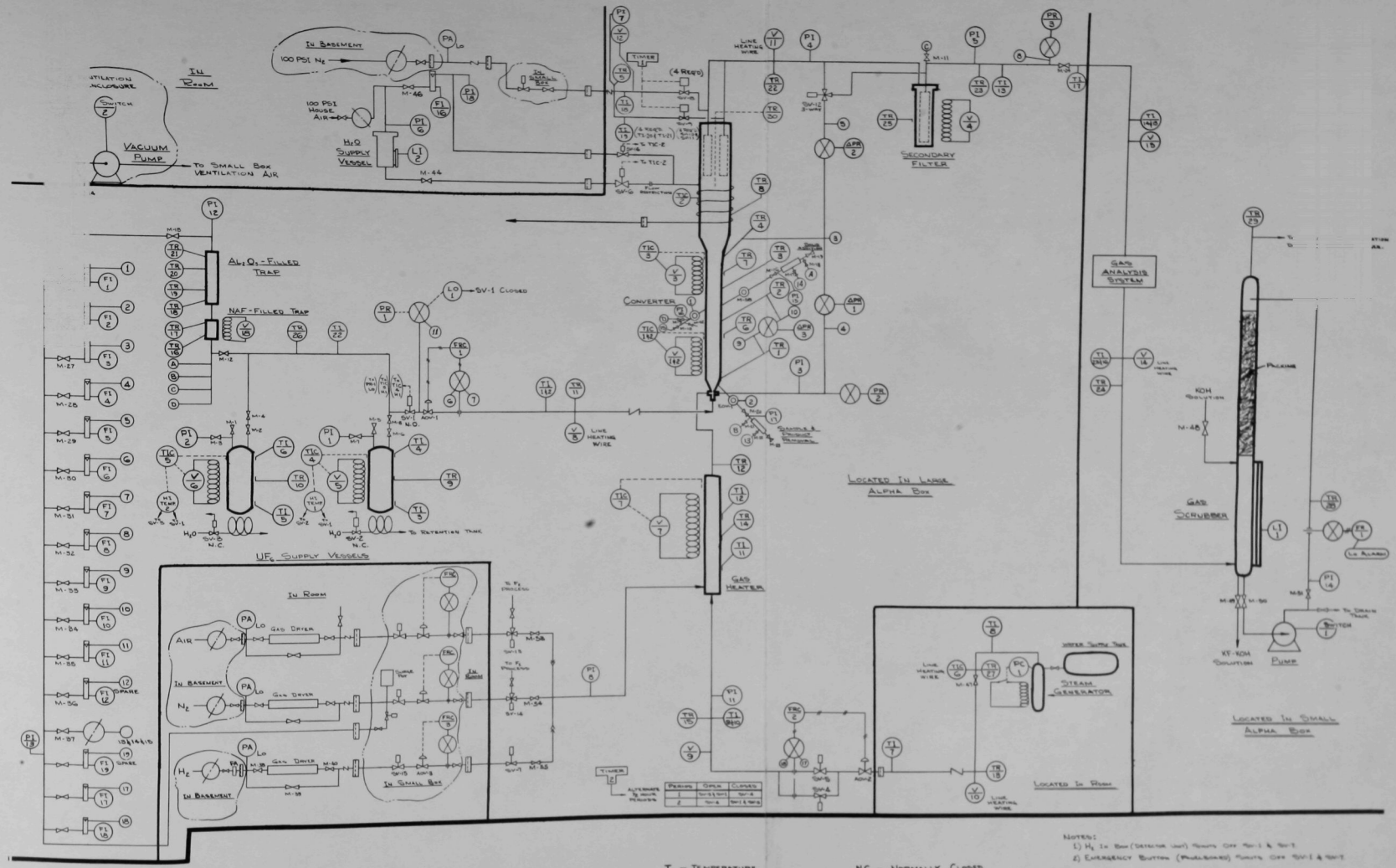
CLEAN-UP PERIOD (30 MINUTES)

	Point No.	1	2	3	4	5	6	7	8	9	10	11	12④	13
Temperature, C			25		25	200		650	100		100		87 (189F)	
Pressure, PSIA			Differs 14.7-16.8		16.8	16.8		14.9	14.8		14.7		14.7	
N_2	LB. MOL.	0	0.0156						0.0138		0.0294		0.0294	
	LB.		0.438						0.386		0.823		0.823	
	CU. FT.		6.000						6.718		14.428		11.981	
UF_6	LB. MOL.			0							0			
	LB.													
	CU. FT.													
H_2	LB. MOL.				0.0351						0.0362		0.0362	
	LB.				0.073						0.068		0.068	
	CU. FT.				12.048						16.762		13.980	
H_2O	LB. MOL.					0.0078②					0.0072		0.0043	
	LB.					0.138					0.129		0.077	
	CU. FT.					4.980					3.515		1.749	
UO_2	LB. MOL.						0.0021				0			
	LB.						0.558							
	CC						42.2③							
HF	LB. MOL.										0.0088		0	
	LB.										0.087			
	CU. FT.										1.216			
KOH①	LB. MOL.									0.0023				
	LB.									0.129				
	CU. FT.									—				
KF①	LB. MOL.											0.0023		
	LB.											0.018		
	CU. FT.											—		
Air	CU. FT.													45
H_2O	CC/MIN.													Varies

NOTES:
① KOH WILL BE ADDED AND KF WITHDRAWN IN A WATER SOLUTION. THIS WATER HAS NOT BEEN CONSIDERED HERE. NET WATER USAGE IN THE SCRUBBER IS 34 cc DURING THE FEEDING PERIOD AND GAIN IN THE CLEAN UP PERIOD IS 44 cc. NET GAIN DURING 1 HOUR IS 10 cc.
② STEAM FLOW INCREASED TO COMPENSATE FOR NON-FEEDING OF UF_6. TOTAL LB. MOL. GAS IN IS THE SAME IN BOTH PERIODS.
③ AT 6.0 G/CC
④ CFH DILUTION AIR REQ'D TO PREVENT CONDENSATION (ASSUME DILUTION AIR AT 25C & 40% R.H.): START UP — 70; FEEDING — 46; CLEAN UP — 71.

Figure 30

PROCESS-MATERIAL FLOW DIAGRAM FOR CONVERTER SYSTEM



LEGEND

- ○ – Instrument, Local Mount
- ⊖ – Instrument, Panelboard Mount
- ⊗ – Transmitter, Local Mount
- ∅ – Pressure Reducer Plus PI
- ⋈ – Valve, Manual
- ⋈ – Valve, Air-Operated
- ⋈ – Valve, Solenoid
- ◎ – Valve, Plug
- ▯ – Flowmeter, Rotameter
- ⊣⊢ – Flowmeter, Orifice
- ▯ – Filter
- N – Valve, Check
- ..... – Signal Line, Electric
- –+–+– Signal Line, Pneumatic
- ▭▭ – Heating Coil
- ▭▭ – Cooling Coil

T – Temperature
P – Pressure
L – Level
F – Flow
W – Weight
I – Indicator
R – Recorder
V – Variac
SV – Solenoid Valve
C – Controller

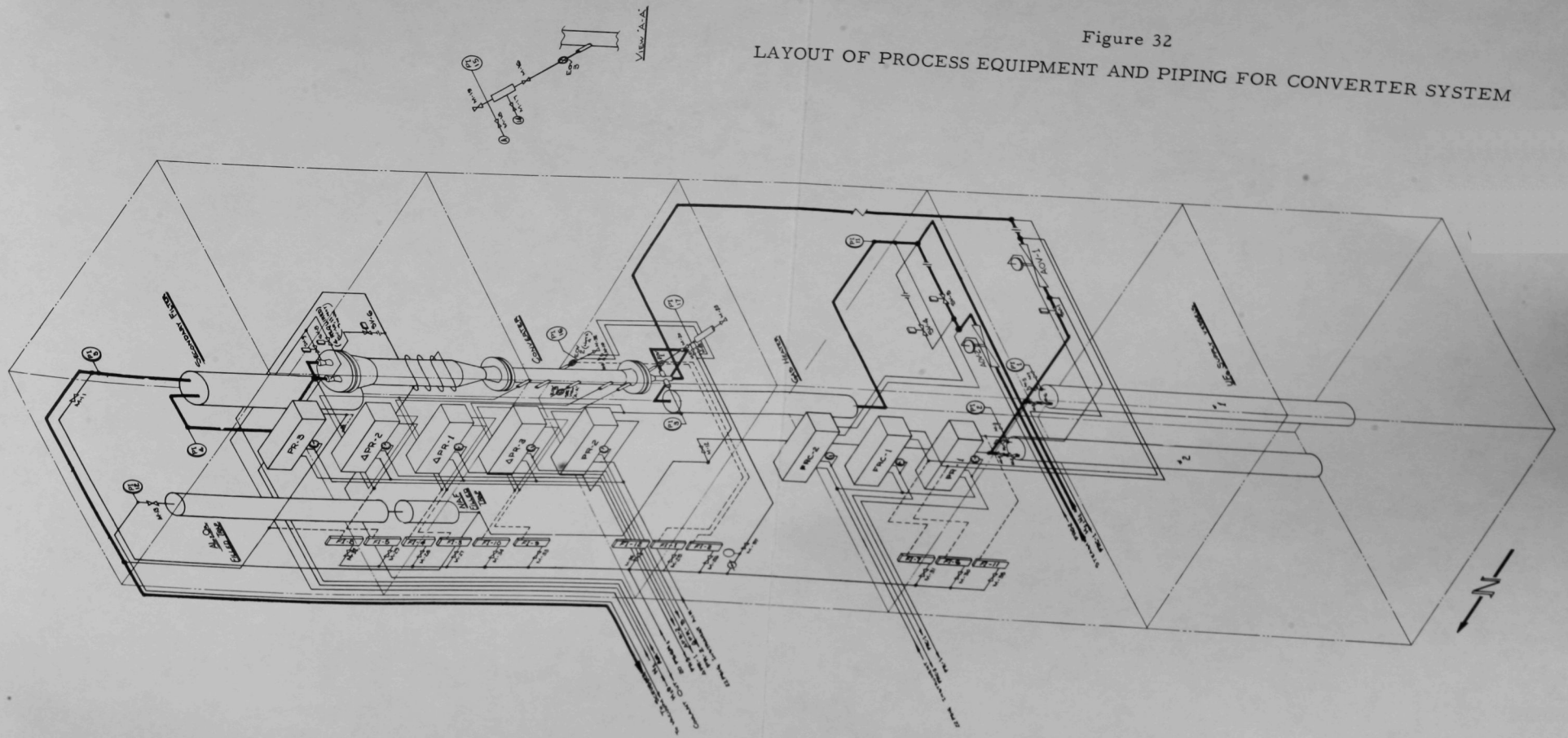
N.C. – Normally Closed
N.O. – Normally Open
M – Manual
A – Alarm
AOV – Air-Operated Valve
EOV – Electrically Operated Valve
FA – Flame Arrestor

Figure 31

EQUIPMENT-INSTRUMENTATION DIAGRAM FOR CONVERTER SYSTEM

Figure 32

LAYOUT OF PROCESS EQUIPMENT AND PIPING FOR CONVERTER SYSTEM



NOTES:

1) LOCATE ROTAMETER MANIFOLD & ROTAMETERS IN S.W. CORNER OF BAY, KEEPING ROTAMETERS AT THE LEVELS SHOWN. (FOR CONVENIENCE ROTAMETERS ARE SHOWN IN N.W. CORNER.)

2) ORIENT ALL GAGES & ROTAMETERS SO THAT INDICATORS CAN BE SEEN FROM SOUTH SIDE OF ALPHA BOX.

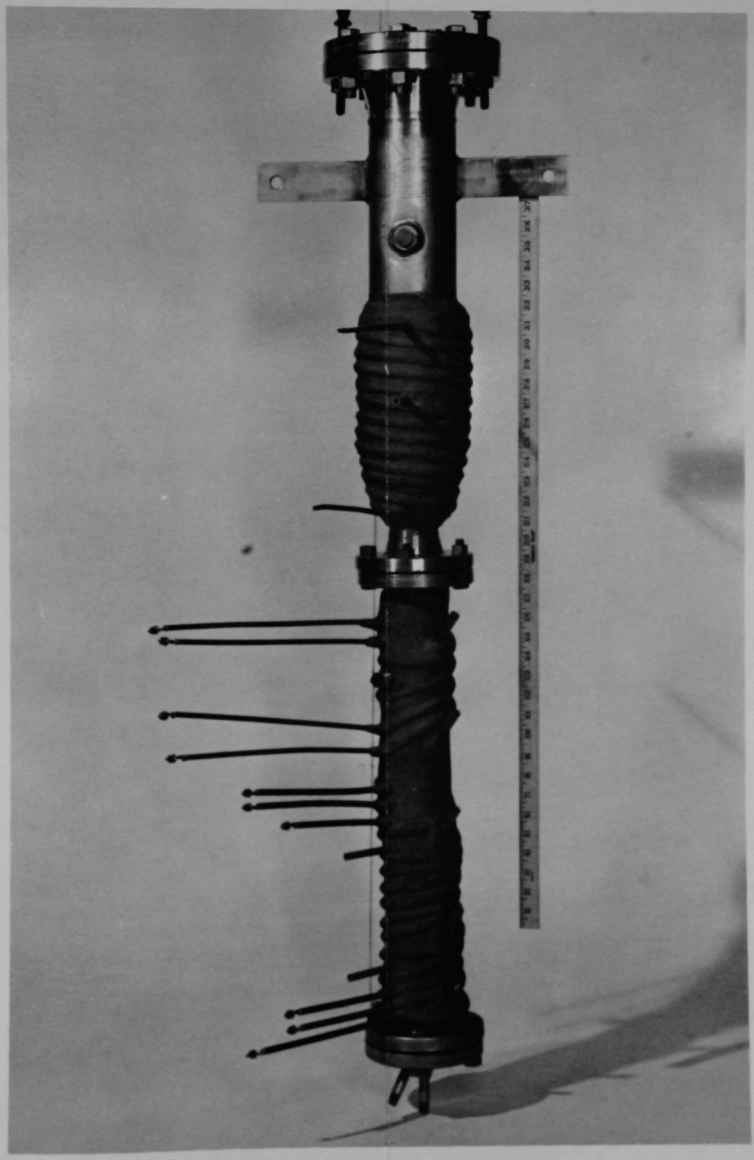
3) ALL LINES EXIT AT 2ND & 3RD LEVEL OF EAST END PLATE.

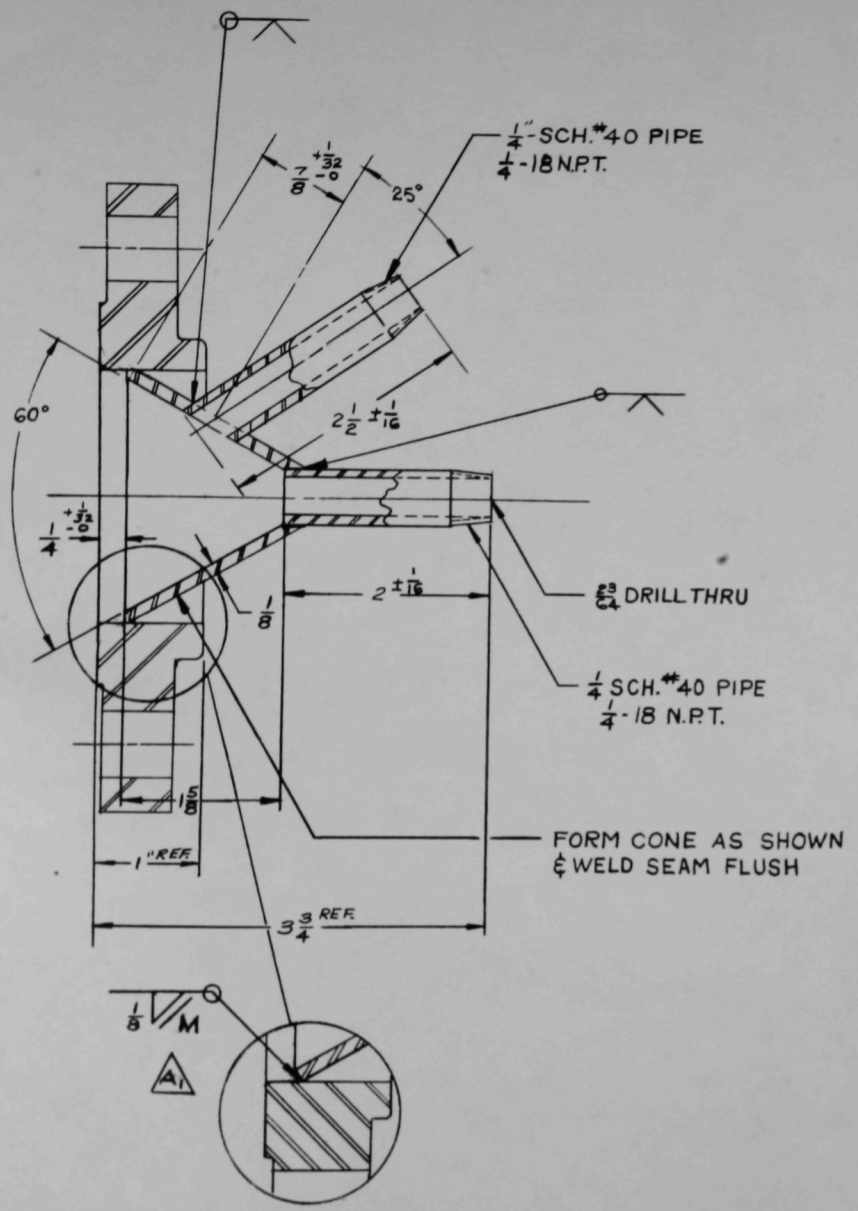
4) VALVES EOV-1, EOV-2, & EOV-3 ARE T. H_2O COOLED. NOTE THAT COOLING LINES ARE NOT SHOWN.

5) MANUAL VALVES M-14, M-15, & M-24 AND SOLENOID VALVES SV-2, SV-8, SV-10, & SV-11 ARE NOT SHOWN ON THIS DWG.

Figure 33

CONVERTER PROCESS UNIT





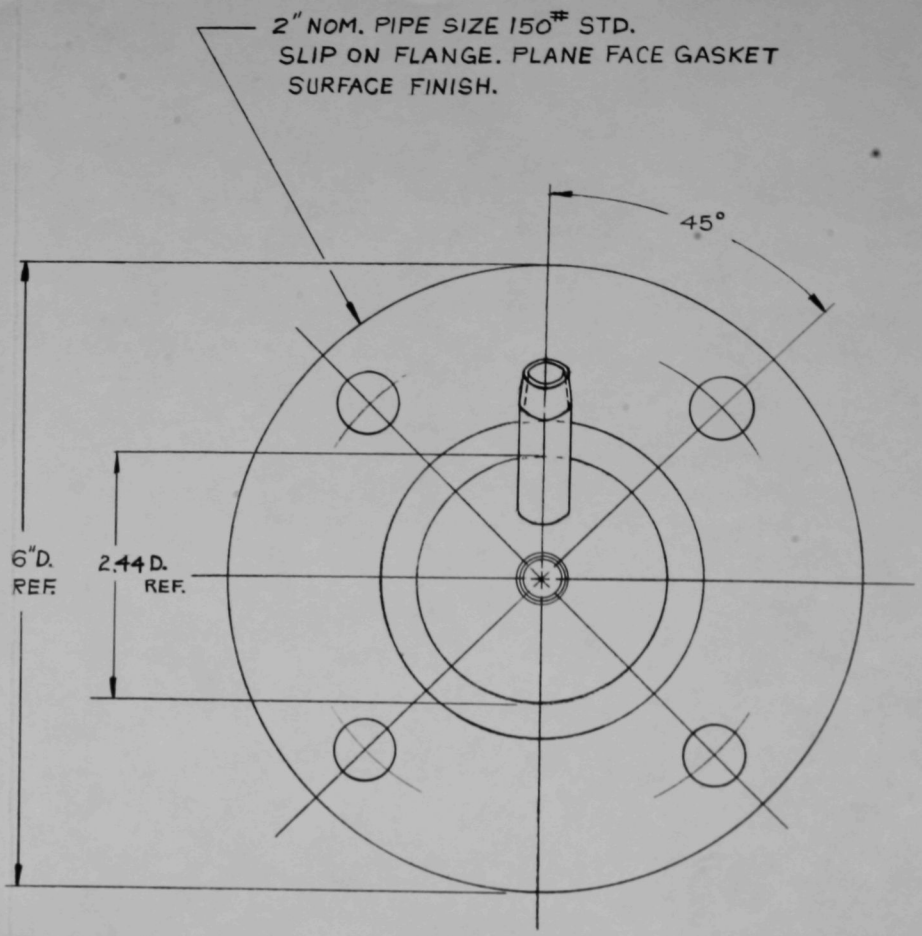


Figure 34

DETAIL OF CONE BOTTOM OF CONVERTER

Figure 35

DETAIL OF FLUID-BED SECTION OF CONVERTER

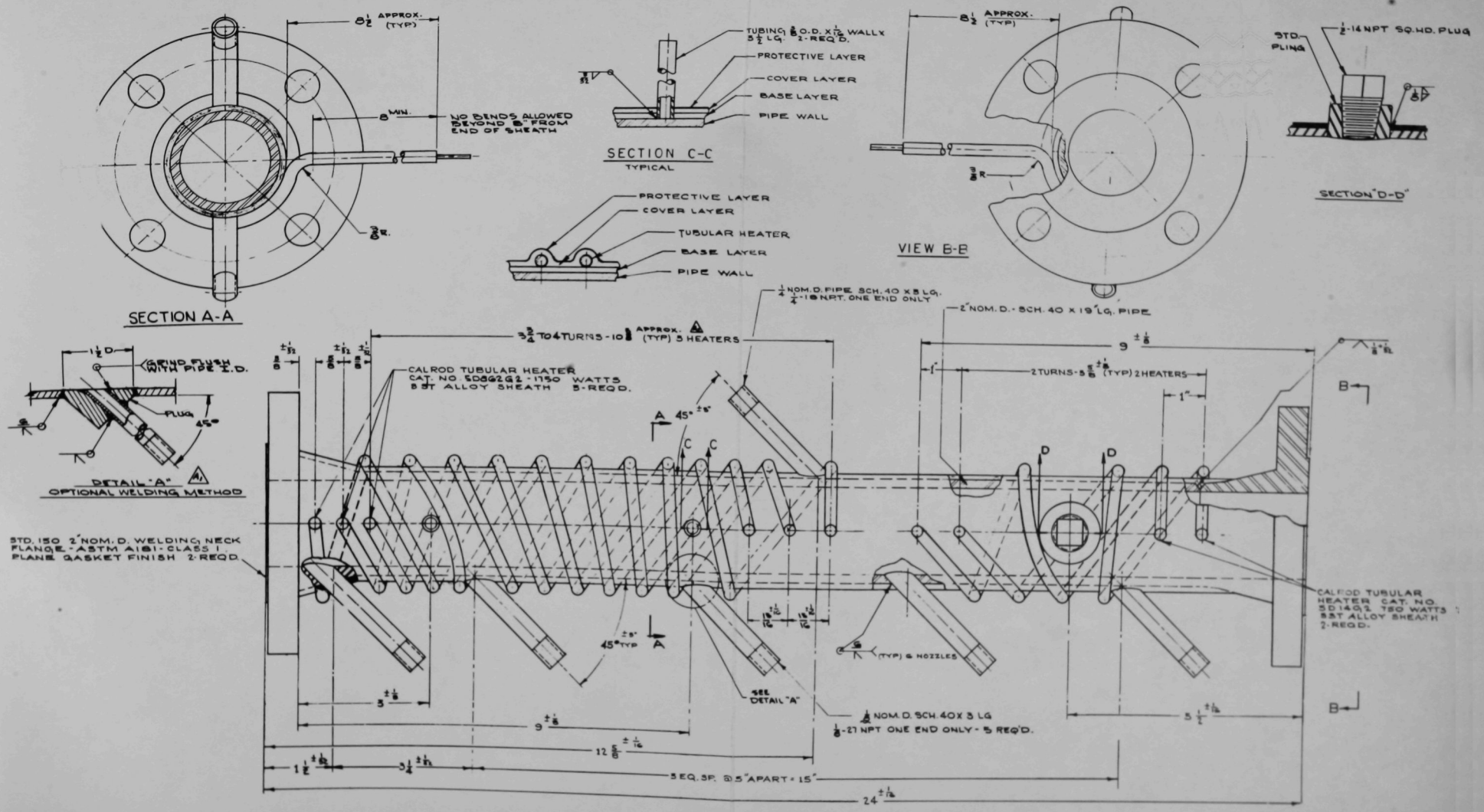


Figure 36

DETAIL OF DISENGAGING SECTION OF CONVERTER

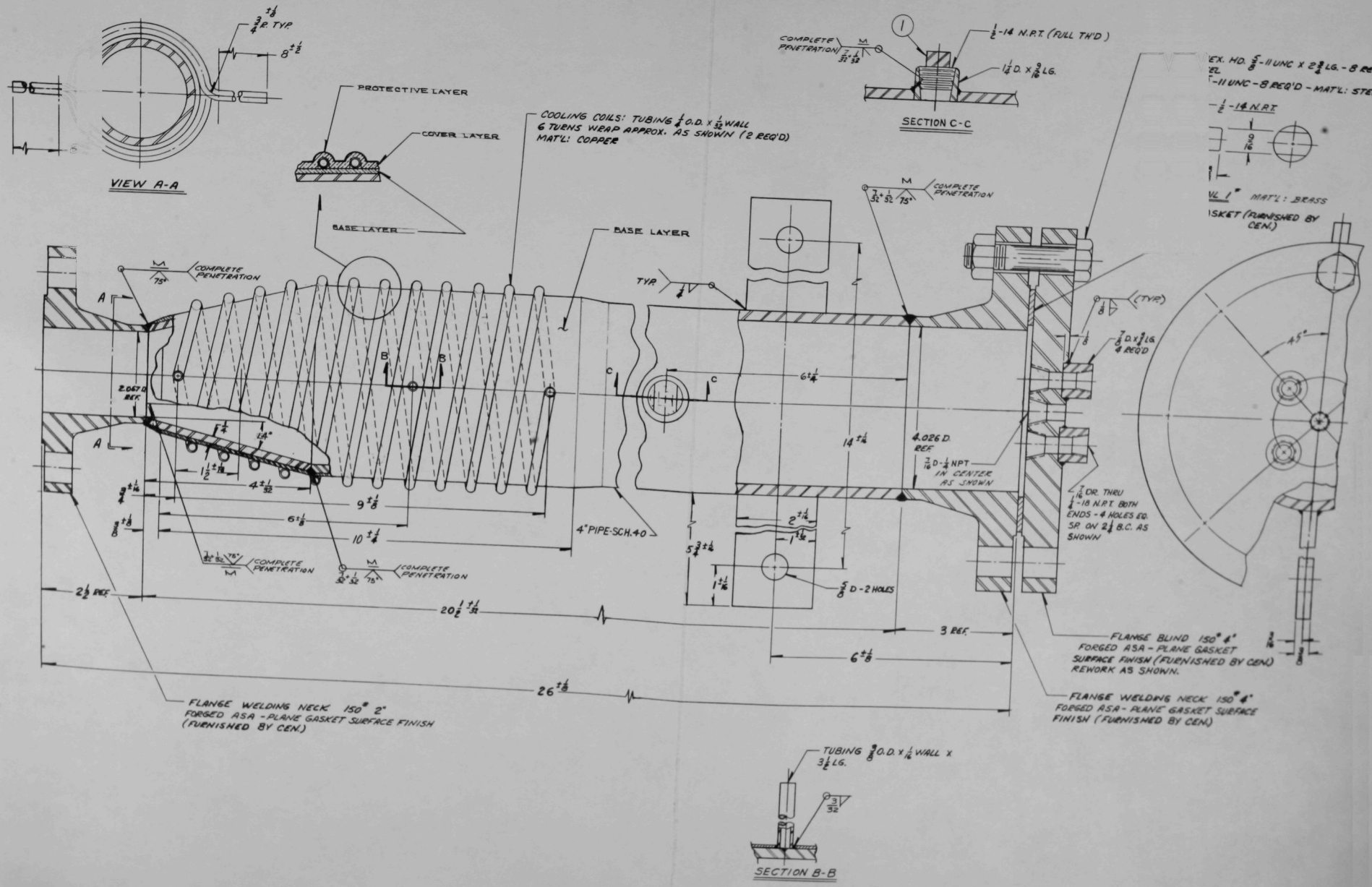


Figure 37

SCRUBBER FOR PROCESS OFF-GAS OF CONVERTER S