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ORNL-564
Chemistry-Separation Processes for Plutonium and Uranium

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ORIENTATION MANUAL FOR 23 PILOT PLANT

OAK RIDGE NATIONAL LABORATORY
OPERATED BY
CARBIDE AND CARBON CHEMICALS DIVISION
UNION CARBIDE AND CARBON CORPORATION
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ORNL-564
Chemistry-Separation Processes for Plutonium and Uranium

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OAK RIDGE NATIONAL LABORATORY

TECHNICAL DIVISION

CHEMICAL TECHNOLOGY DEPARTMENT

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ORIENTATION MANUAL FOR 23 PILOT PLANT

D. E. Ferguson and R. J. Klotzbach

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OAK RIDGE NATIONAL LABORATORY
operated by
CARBIDE AND CARBON CHEMICALS DIVISION
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Oak Ridge, Tennessee

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0.0 ABSTRACT

This report summarizes the status of process chemistry for the recovery of 23 from pile irradiated thorium and describes the 23 Pilot Plant which will be used at Oak Ridge National Laboratory for further process development.

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1.0 INTRODUCTION AND SUMMARY

The objective of this manual is to summarize the status of the Uranium-233 (23) recovery process and to describe the equipment of the 23 Pilot Plant recently completed at Oak Ridge National Laboratory.

Production and recovery of 23 is now an intergal part of the Materials Testing Reactor project. In addition, a highly efficient 23 separation process will be required for future 23 breeding piles. Solvent extraction processes for 23 recovery were developed by the Chemistry Division of Argonne National Laboratory and the Chemistry and Technical Divisions of Oak Ridge National Laboratory. Early in 1948 the Technical Division compared and further investigated all proposed 23 processes. Based on this work, the 23 Pilot Plant was designed and constructed during 1948 and 1949.

The present process consists of the following steps: dissolution of irradiated thorium metal in nitric acid, continuous countercurrent solvent extraction of the 23 in a packed tower, scrubbing the product carrying solvent with aluminum nitrate, and stripping the product with dilute nitric acid in a second tower. Hexone appears to be the most attractive solvent tested, and this solvent will be used in the early pilot plant runs. Work on laboratory and semi-works scales has shown that the process described above will recover 99.95% of the 23 and that the product is separated from thorium, protoactinium, and fission

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Introduction and Summary (continued)

products by a factor of 10^4.

The general features of the 23 Pilot Plant equipment are similar to the pilot plant used for 25 and Redox process investigations at Oak Ridge National Laboratory, except the equipment capacity has been reduced. Twenty-one major equipment units, including tanks, condensers, filters, and two 1-1/2 inch packed columns are used for hot feed preparation and solvent extraction. The average volume of hot process tanks is 40 gallons, and the maximum capacity of the plant is 50 kilograms of irradiated thorium per day. Shielding consists of two feet of concrete, and direct maintenance procedures are used. Eleven 55 gallon tanks with accessories for mixing, pumping, weighing, and filtering are used for cold feed preparation and transfer, and five major equipment pieces are used for solvent recovery.

There is available for the 23 Pilot Plant program 740 kilograms of Hanford irradiated thorium metal. This metal contains about 280 grams of 23 and has cooled six to twenty months. Twenty-five hot runs will be made over a three to four months period, beginning about January 3, 1949. The primary purpose of these runs will be to obtain the engineering data necessary for the design and operation of a plant to recover 23 produced in the Materials Testing Reactor.

[redacted]

2.0 PROCESS CHEMISTRY

Uranium-233 (23) is produced by the neutron bombardment of thorium-232 as follows:

$$_{90}Th^{232}\ (n,\gamma) \rightarrow\ _{90}Th^{233} \xrightarrow[23\ m]{\beta}\ _{91}Pa^{233} \xrightarrow[27.4d]{\beta}\ _{92}U^{233}$$

By this reaction 23 will be produced in gram quantities in the proposed Materials Testing Reactor. The irradiated thorium metal from thispile will contain about 1.5 grams of 23 and protoactinium-233 (13), and 0.04 grams of fission products per kilogram of thorium. This material will be cooled for twelve months to reduce the amount of 13 to 0.01% the amount of 23; otherwise, a significant amount of potential 23 is lost as 13 during chemical processing.

Several solvent extraction processes have been developed to effect the chemical separation of 23 from thorium, 13 and fission products. These processes all consist of two solvent extraction cycles, the first in large,moderately shielded equipment, the second cycle in laboratory equipment with only light shielding. On the basis of cost estimates, continuous solvent extraction was chosed for the first cycle and a batch extraction for the laboratory-scale second cycle. Following this choice, the Technical Division of ORNL tested and compared the four most promising continuous solvent extraction processes on a semi-works scale. Table 2.0 presents the pertinent features of the four process flowsheets.

[redacted]

Table 2.0

PROPOSED 23 RECOVERY FLOWSHEETS

Solvent	Composition of Feed	Composition of Scrub	Composition of Strip	Flowratios Scrub:Feed:Solvent:Strip
Hexone	1.6 M $Th(NO_3)_4$ 0.2 N acid deficient	1.0 M $Al(NO_3)_3$ 0.02 N acid deficient	0.04 N HNO_3	1:4:5:1.
Diisopropyl Ether	2.5 M $Th(NO_3)_4$ 0.1 M HNO_3	2.5 M $Al(NO_3)_3$ 0.75 M NaF 0.4 N acid deficient	Water	1:4:5:1.
Dibutyl Cellosolve	1.25 M $Th(NO_3)_4$ 1.0 M HNO_3	2.0 M $Al(NO_3)_3$ 1.0 M $Ca(NO_3)_2$ ~1 M $Ca(OH)_2$	Water	1:1:2:0.2
Dibutyl Cellosolve	2.0 M $Th(NO_3)_4$ 0.2 M HNO_3	2.4 M $Al(NO_3)_3$ pH 1.8 (~3 N acid deficient)	Water	1:3:4:1

[redacted]

2.1 DIBUTYL CELLOSOLVE

The Chemistry Division at ORNL developed and demonstrated a continuous, countercurrent, extraction process for recovery using dibutyl cellosolve as the solvent. This process was found to give excellent separation of 23 from thorium and fission products; however, it was found that 23 losses as high as 10% occurred in stripping the dibutyl cellosolve because of emulsion formation in the strip column.

A modification of the Chemistry Divisions dibutyl cellosolve process was developed by the Technical Division. This process used a lower HNO_3 concentration in the feed which permitted a lower scrub to feed ratio and a scrub solution containing only $Al(NO_3)_3$ and NH_4OH (see Table 2.0). As with the other dibutyl cellosolve process, 23 losses from the stripping column were excessively high, and it was recommended that dibutyl cellosolve not be considered as a solvent in the 23 Pilot Plant unless the stripping losses could be reduced.

2.2 DIISOPROPYL ETHER PROCESS

The diisopropyl ether 23 process was found to give excellent separation of 23 from thorium and fission products. Thorium reduction factors of 10^6 and beta fission product decontamination factors of 10^5 were obtained consistently with this solvent. However, diisopropyl ether now appears less attractive than hexone for a 23 solvent because of a longer HETS, lower flash point solvent, engineering difficulties in handling salt solution near saturation, and the necessity for close temperature control.

2.3 HEXONE PROCESS

A 23 process using hexone as the solvent will be fully tested in the 23 Pilot Plant. This process has a low HETS, high flooding capacity and utilizes a relatively safe solvent. Another advantage of hexone over the other solvents is that extensive data obtained in the 25 process is directly applicable to the hexone extraction of 23.

In the 25 process, aluminum nitrate is employed as a salting agent in both feed and scrub. Since it was shown that the salting effect of $Th(NO_3)_4$ is almost equivalent, mole for mole, to that of $Al(NO_3)_3$, a modified 25 process for 23 recovery was feasible. The 23 flowsheet substitutes $Th(NO_3)_4$ from the dissolution of the irradiated thorium metal for $Al(NO_3)_3$ as a salting agent in the feed.

At the time of the Technical Division's evaluation of the 23 processes, the 25 hexone flowsheet salt concentration was 1.3 M $Al(NO_3)_3$ in both scrub and metal feed. This has now been changed to 1.0 M $Al(NO_3)_3$ in the scrub and 1.6 M in the feed, and this latter flowsheet will be followed in the 23 program. With the former flowsheet, it was found that one cycle of solvent extraction with hexone would recover 99.98% of the 23 with a decontamination factor of 10^4 for thorium, 13, and fission products. Thus, after one cycle of operation, the product will be

[redacted]

Hexone Process (continued)

sufficiently decontaminated to carry out the laboratory clean up and concentration with only light shielding.

Figure 2.3 gives a schematic flowsheet for the 23 hexone process. In this process the irradiated thorium metal is dissolved in 60% HNO_3 (0.1 M HF as catalyst) with a sufficient excess of metal and in such a manner that the acid concentration of the final metal solution is less than 0.2 N HNO_3. This metal solution is then adjusted with NaOH (or NH_4OH) to 0.2 N acid deficient and filtered. The filtrate is then adjusted to 1.6 M $Th(NO_3)_4$, 0.2 N acid deficient and pumped to the extraction column. The 23 is extracted from this solution with hexone leaving all but traces of thorium, fission products, and 13 in the aqueous phase; $Th(NO_3)_4$ acts as the salting agent. The hexone solution of 23 is scrubbed in the upper section of the extraction column with 0.2 N acid deficient 1.0 M $Al(NO_3)_3$, and is stripped in a second column with 0.04 N HNO_3. The resulting aqueous solution of 23 is then reduced in volume by evaporation and transferred to the laboratory for further clean up and concentration.

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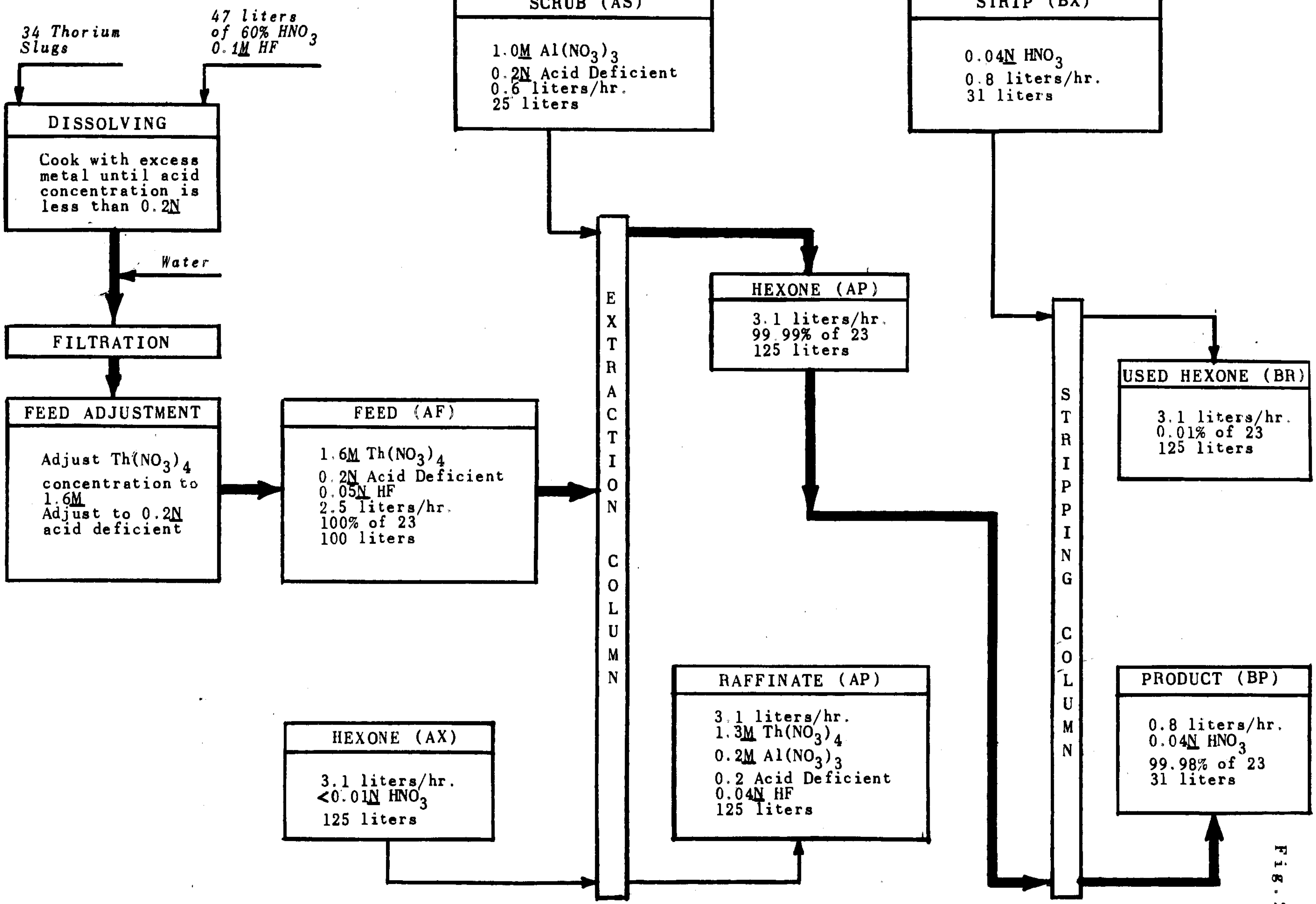

Fig. 2.3

23 PROCESS PILOT PLANT CHEMICAL FLOWSHEET

11-15-49

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2.4 LABORATORY CLEAN UP AND CONCENTRATION

The concentrated 23 strip solution will contain about 2 g/l of thorium, together with traces of aluminum, corrosion products from stainless steel vessels, and decomposition products from hexone. $Al(NO_3)_3$ is added to this solution as salting agent, and a batch extraction cycle with diisopropyl ether is carried out. This extraction cycle will separate further the 23 from fission products and 13, and reduce the other contaminants to undetectable quantities. This operation completes the 23 chemical separation process. A summary of process chemistry for the laboratory cycle, and a description of the equipment to be used, is presented in ORNL 122, Laboratory Purification and Concentration of U^{233} by D. C. Overholt.

3.0 EQUIPMENT AND PROCESS OPERATIONS

There are four major processing areas for one cycle operation: (1) Feed Makeup, (2) Columns and Accessories, (3) Solvent Recovery, and (4) Cold Makeup. The cell tanks have volumes ranging from 25 to 100 gallons and were fabricated of stainless steel to conform to the ASME Code for Unfired Pressure Vessels. They are variously equipped with accessories for mixing, heating and cooling, liquid level and specific gravity determination, overflow alarms, and temperature measurement and control. Solutions are transferred by pumps, steam jets, pressure, vacuum, and gravity, and are sampled by means of recirculating air jet sample units. A general equipment flowsheet, Figure 3.0-1 denotes the main process operations. A detailed schematic flowsheet, Figure 3.0-2, shows all major equipment units, the accessories and instrumentation of each unit, and a detailed process flowstudy. The detailed drawings for the equipment are listed both numerically and functionally on ORNL Drawing C-4391.

3.1 BUILDING AND CELLS

The 23 Pilot Plant is housed in Building 706-HB. The building, 100 ft. long, 50 ft. wide, and 53 ft. high, is constructed of a structural steel frame covered with 20 ga. corrugated aluminum sheet siding. The

Building and Cells (continued)

process areas are two 18 x 18 x 30 ft. concrete cell blocks, each of which contains two 6 x 6 ft. cells and a 14 x 6 ft. cell, having 2 ft. thick concrete walls. Surrounding three faces of each cell block are platforms at elevations of 10, 20, and 30 ft. The detailed drawings for this building are listed on Drawing TD-1318.

3.11 "23" PROCESS AREA

The process and solvent recovery areas are located in the east cell block, Block B, in which there is a 3 x 6 ft. pit, 18 ft. deep, to accommodate the columns. The inactive solution makeup area is at ground level along the south wall of the building. A plan of the building and cell block, Figure 3.11-1, shows the area occupied by the "23" process equipment.

3.12 ELECTRICAL SERVICES

Building 706-HB was constructed to fulfill National Fire Code Class I solvent specifications. All electrical equipment and services in the cells conform to NEMA Class VII specifications and, in general, the electrical equipment and services outside the cells meet or exceed NEMA Class IV specifications.

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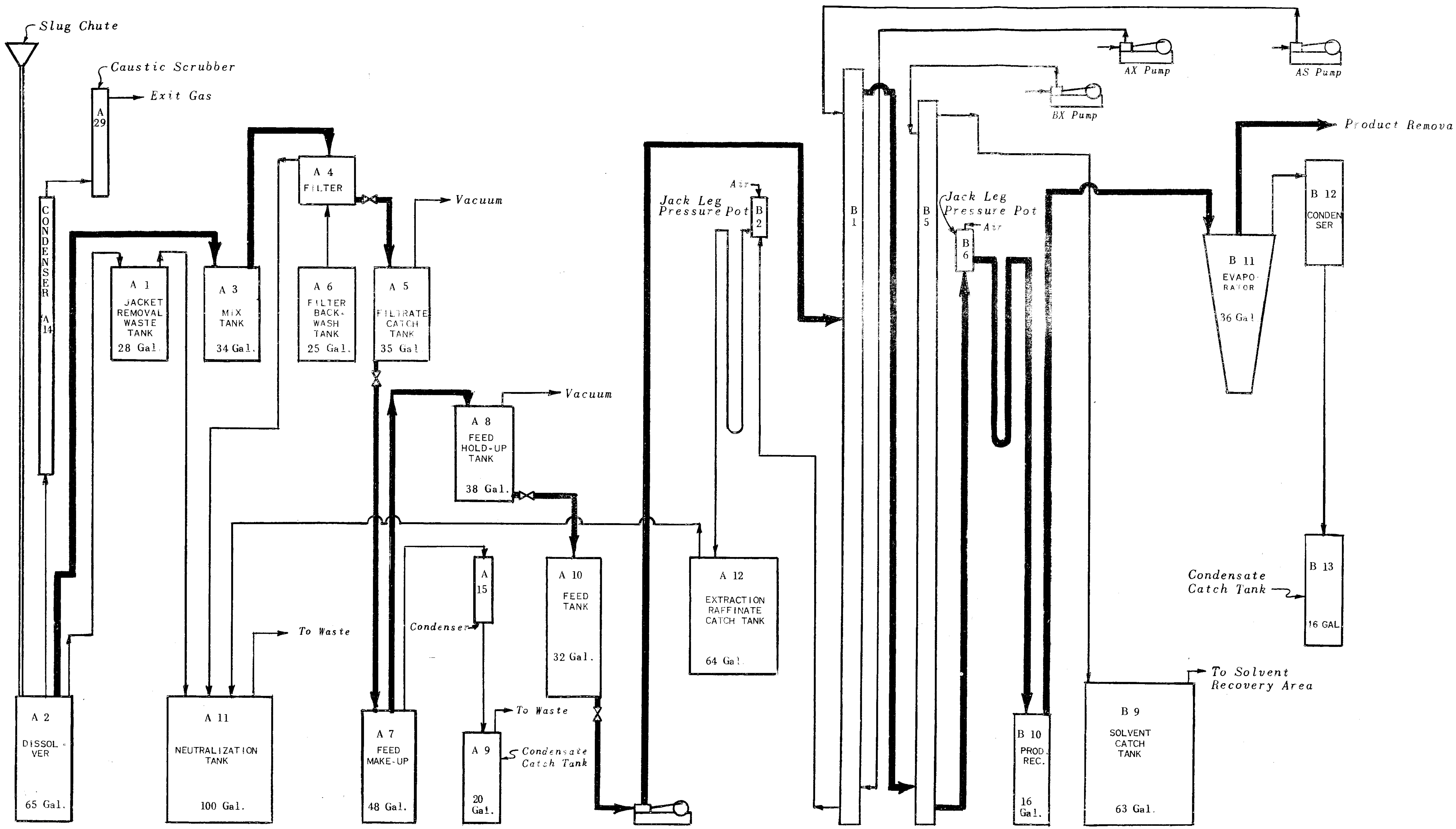
Fig. 3.0-1



706 HB-1300 PRODUCT PILOT PLANT

CONDENSED SCHEMATIC EQUIPMENT FLOWSHEET

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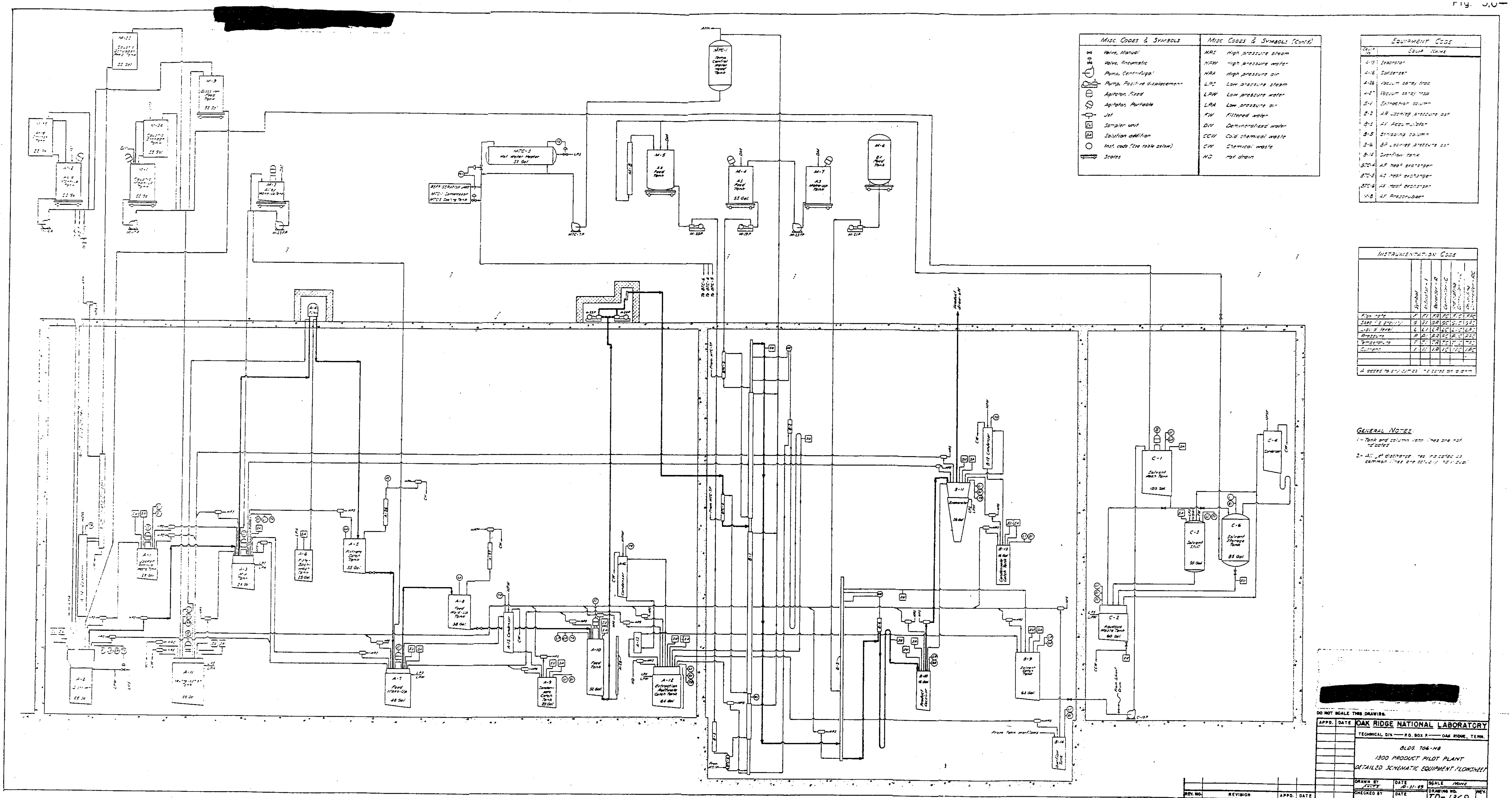
[redacted]
Misc. Codes & Symbols
Valve, Manual
Valve, Pneumatic
Pump, Centrifugal
Pump, Positive displacement
Agitator, Fixed
Agitator, Portable
Jet
Sampler unit
Solution addition
Inst. code (See table below)
Scales
Misc. Codes & Symbols (Cont'd)
HPS High pressure steam
HPW High pressure water
HPA High pressure air
LPS Low pressure steam
LPW Low pressure water
LPA Low pressure air
FW Filtered water
DW Demineralized water
CCW Cold chemical waste
CW Chemical waste
HD Hot drain
Equipment Code
Instrumentation Code
General Notes
Hot Water Heater
Feed Tank
Solvent Wash Tank
Solvent Storage Tank
Aqueous Waste Tank
[redacted]
DO NOT SCALE THIS DRAWING.
APPD. DATE
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TECHNICAL DIV — P.O. BOX P — OAK RIDGE, TENN.
BLDG. 706-HB
1300 PRODUCT PILOT PLANT
DETAILED SCHEMATIC EQUIPMENT FLOWSHEET
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DATE 10-31-49
SCALE NONE
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Fig. 3.6

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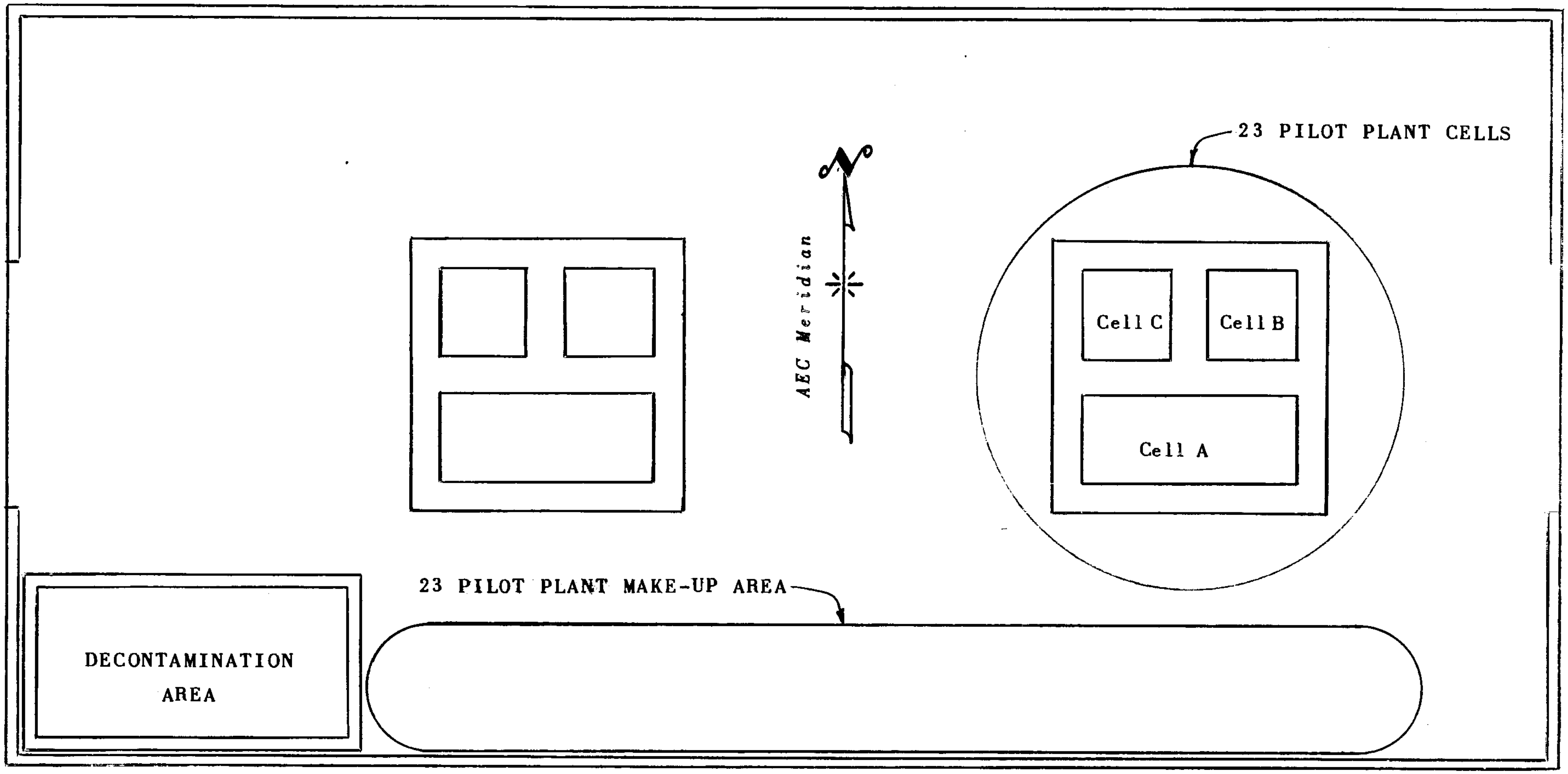


SCHEMATIC PLAN OF BLDG. 706 HB

Fig. 3.11-1

[redacted]

3.2 MATERIALS OF CONSTRUCTION

3.21 TANKS

Most major equipment units are constructed of AISI Type 347 stainless steel. The dissolver, evaporator and feed adjustment tank are the only exceptions. They were constructed of AISI Type 309 SCb heat treated stainless steel.

3.22 PIPE

Process piping is schedule 40, Type 347 stainless steel. Service piping to equipment jackets, coils, etc. is galvanized wrought steel pipe. All thermocouple wires and equipment power lines are run in 3/4 inch galvanized conduit.

3.23 TUBING

Most of the process lines in the cell are constructed of tubing rather than pipe. Seamless, Type 304, stainless steel tube with wall thicknesses of 20 and 22 B and W gauge is used in most cases. Instrument lines outside the cell are constructed of 3/8 inch O.D. x 0.035 inch wall, seamless copper tube; inside the cell the lines are constructed of 3/8 inch x 0.025 inch wall, seamless, Type 304 stainless tube.

3.24 FITTINGS

All tube fittings are Parker Triple Tube couplings, Aircraft 811 type. Type 303 stainless steel fittings are used for stainless steel tubing and brass fittings are used for copper tubing. In most cases, the tube fittings are screwed directly to couplings welded in the tank covers. The screwed joints are tinned to give a leakfree connection. Some flanged joints are used on tanks and these have full face teflon or polythene gaskets.

3.25 VALVES

Three types of bellows sealed valves manufactured by Crane, Fulton Sylphon, and Alloyco are used in the process. All types are constructed of Type 347 stainless steel and have a replaceable teflon disc. The Crane and Fulton valves have screwed ends and the Alloyco valve has flanged ends. All other types of valves in the process are globe valves constructed of 18-8 stainless steel with a replaceable teflon seat and teflon packing. Brass gate and globe valves are used on Panel Board headers and service lines to all equipment pieces.

[redacted]

3.3 CELL EQUIPMENT

Cell block B, in which the 23 Pilot Plant is located, consists of three cells. Cell A, the large 14 x 6 x 30 ft. cell, is used exclusively for metal solution preparation and waste disposal. Cell B, the east 6 x 6 x 30 ft. cell, with an 18 ft. pit, contains the columns and product treatment equipment. Cell C, the west 6 x 6 x 30 ft. cell, contains the solvent recovery unit.

Each equipment identification number is prefixed with the letter A, B, or C, indicating which cell it occupies. Equipment identification numbers outside the cells are prefixed with M.

Panel boards for process control of cell equipment are located at both north and south cell block faces on the 10 ft. elevation platform and at the north face on ground level. Samplers and reagent addition funnels for cell equipment are located at the 20 ft. elevation platform.

3.31 CELL A EQUIPMENT

3.311 DISSOLVING

Irradiated metal slugs are dropped from the top of the cell block to the Dissolver, A2 through the Slug Chute, A17. Caustic and nitric acid solutions are pumped to A2 from the makeup area for dissolution of the slug jackets and metal, respectively. A Reflux Condenser,

[redacted]

Dissolving (continued)

A14, is mounted on the dissolver. Off gases are drawn through the condenser to a packed caustic Scrubber, A29, and discharged to the atmosphere through a Schutte and Koerting air jet exhauster which provides vacuum for the system. Jacket removal solutions are steam jetted to the Jacket Removal Waste Tank, A1. Metal solutions are steam jetted to the Mix Tank, A3.

3.312 FILTRATION

The metal solution is transferred from A3, by vacuum, to the Filter, A4, mounted in a lead blister on the outside cell face. The solids are collected on a Micro-Metallic Corp. sintered stainless steel, star shaped, filter element with a mean pore opening of 10 microns (grade G porosity) and a 1.33 sq. ft. filter area. The filtrate is collected in a vacuum Filtrate Catch Tank, A5, where vacuum for the filtration is provided by an air jet exhauster. A Moore Products Company, Electric Liquid Level Controller, is mounted on A5. A stainless steel ball float, set at maximum allowable liquid level, actuates a micro-switch which operates a warning light and howler on the panel board. Solutions for backwashing the filter plate are transferred from a blowcase, A6, through the Filter, A4, to the Neutralizer, A11. The filtered

Filtration (continued)

solution can be recycled through the filter by steam jetting from A5 to A3. All of the filtration equipment can be by-passed if desired.

3.313 FEED ADJUSTMENT

The filtrate is drained from the Catch Tank, A5, by gravity flow through a bellows sealed valve, to the Feed Makeup Tank, A7. The solution concentration can be adjusted by either diluting or evaporating. The vapor is condensed in Condenser A15 and collected in a Condensate Catch Tank, A9.

3.314 TRANSFER TO COLUMN FEED TANK

The adjusted solution is transferred to a Feed Holdup Tank, A8. Vacuum is provided by an air jet exhauster and overflow is prevented by a Moore Electric Liquid Level Alarm identical to the alarm mounted on Vacuum Tank A5. The solution is drained from A8, by gravity flow through a bellows sealed valve, to the Extraction Column Feed Tank, A10.

3.315 PUMPING OF FEED

The feed solution is drained from A10, by gravity flow through a bellows sealed valve, to a pair of Argonne Bellows Pumps, connected in parallel. They are enclosed in a lead case, located in the east Cell A doorway. The construction is such that it is possible to service one pump while the other is operating. Each pump consists of a Chicago Metal Hose Company bellows which is flexed by a cam driven rocker arm. The length of bellows travel is determined by an adjustable rocker arm pivot, and can be reset at any time while operating. The bellows actuates a Hills-McCanna, 1/4 inch, vertical, composite, double-ball suction and discharge, gravity type, check valve. The pumping rate is controlled by the adjustable pivot and is checked by a thermoelectric flowmeter and a liquid contact, electrode type flow calibrator, located in a Stand Pipe, A28, on the outlet drain of the Feed Tank, A10. The solution is pumped through the cell wall to the extraction column in Cell B.

3.32 CELL B EQUIPMENT

3.321 EXTRACTION COLUMN

The metal solution is pumped through a heat exchanger, BTC4, to the Extraction Column, B1. The column is a 1-1/2 inch IPS stainless steel pipe, packed with 1/4 x 3/8 inch split raschig rings.

Extraction Column (continued)

It has a 30 ft. extraction section, a ten ft. scrub section, and an overall height of approximately 46-1/2 ft. It is divided into nine flanged sections, is equipped with three packing supports and approximately 35 ft. of the extraction and scrub sections are jacketed for temperature control.

3.322 EXTRACTION OPERATIONS

The feed stream enters column B1 approximately 15 ft. below the top flange. Scrub and organic solutions are pumped from the makeup area. The scrub solution is pumped through the Scrub Heat Exchanger, BTC5, to a point on the column 10.5 ft. above the feed inlet. The solvent is pumped through the Solvent Heat Exchanger, BTC6, and the Accumulator, B3, entering the bottom of the column and passing countercurrent to the aqueous stream. The combined aqueous streams flow out of the bottom of the column through the Jackleg Pressure Pot, B2, to the Aqueous Waste Tank, A12, located in Cell A. Tank A12 is equipped with a Condenser, A16, and Separator, A13, for flashing off solvent before transferring the aqueous waste to the underground Thorium Storage Tank. Provisions for recycling the aqueous waste back to the extraction column consists of a steam jet from A12 to the Feed Makeup Tank, A7.

[redacted]

3.323 STRIPPING COLUMN

The solvent containing essentially all of the U^{233} overflows from column B1 and enters the bottom of the Stripping Column, B5. The column is a 1-1/2 inch IPS stainless steel pipe packed with 1/4 x 3/8 inch split raschig rings. The packed height is 18 ft. and the overall length is approximately 23 ft. It is divided into six flanged sections and is equipped with two packing supports.

3.324 STRIPPING OPERATIONS

Strip solution is pumped from the makeup area to a point on the column 45 inches below the top flange. The solution flows down the column removing all the U^{233} from the solvent and leaves the column at the bottom and flows through the Jackleg Pressure Pot, B6, to the Product Catch Tank, B10. The spent solvent overflows from the column to the Solvent Catch Tank, B9.

3.325 INTERFACE CONTROL

The position of the interface in the upper unpacked sections of both the extraction and stripping columns is detected by a set of bubbler probes positioned 24 inches apart. The pressure differential records the interface position on a Taylor

Interface Control (continued)

Fulscope Controller-Recorder which supplies controlled air to the jack-leg pressure pot through a Moore Products Company 5:1 reducing ratio relay. As the interface position varies from the set point, the controlled air supply varies the pressure head on the aqueous exit stream controlling its flow from the column, and returning the interface position to normal.

3.326 PRODUCT EVAPORATION

The stripping aqueous product solution is steam jetted from the Product Catch Tank, B10, to an Evaporator, B11, which is a conical jacketed tank equipped with a Condenser, B12. The solution is evaporated down to a volume of two liters. The condensate is collected in a Condensate Catch Tank, B13, and can be recycled by jetting back to the evaporator. The concentrated product solution is transferred to the laboratory for further cleanup and concentration.

3.327 EXTRACTION COLUMN TEMPERATURE CONTROL

The temperature of the extraction column is controlled by pumping water from a temperature control unit outside of the cell through the jackets of the feed stream heat exchangers, BTC4, BTC5, Extraction Column Temperature Control (continued)

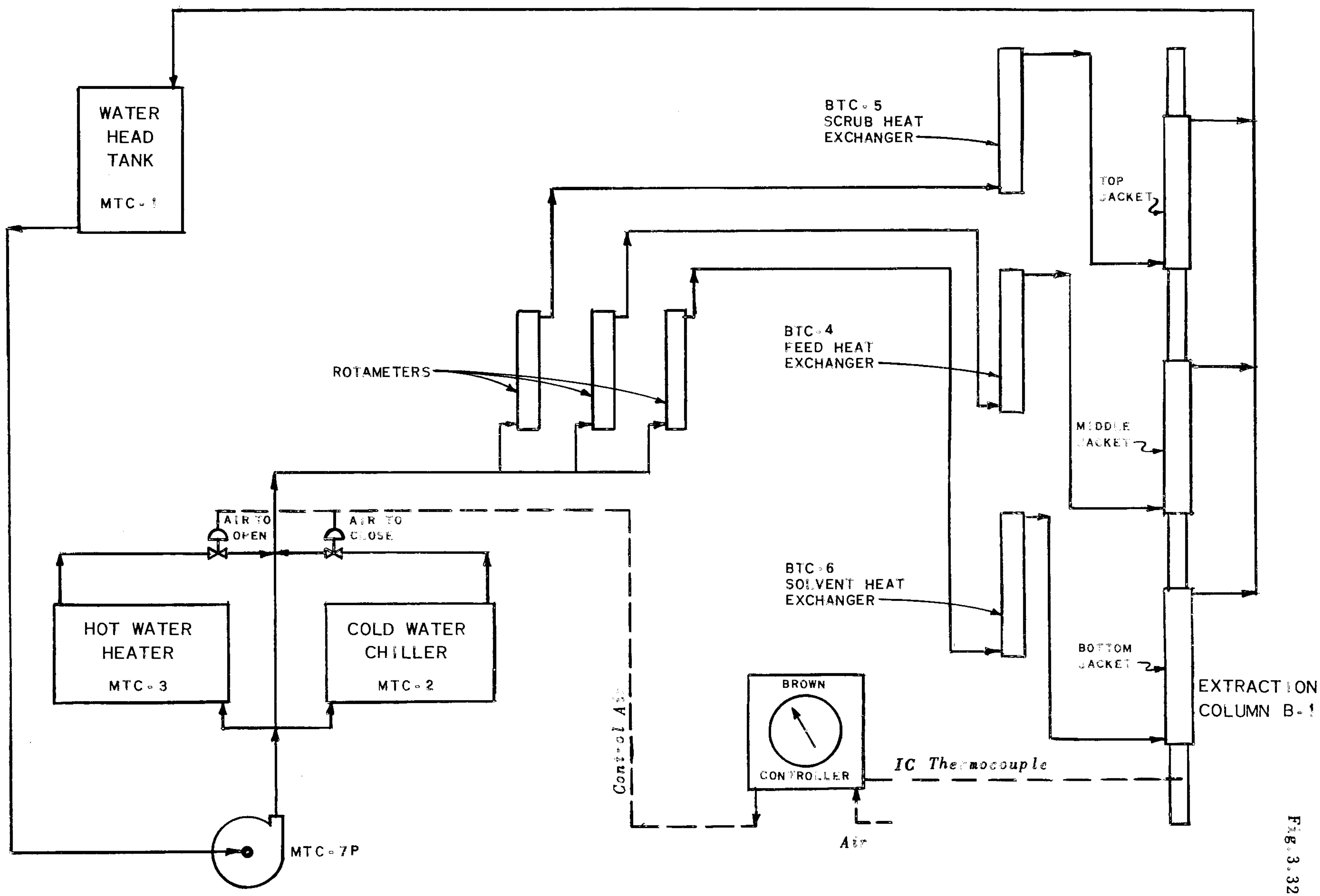
and BTC6, to the three jacketed column sections.

The main stream of water is pumped to the jackets from a constant head Feed Tank, MTC1, by a Durco, Series E-5-MCR-56, Centrifugal Pump, MTC7P. The water temperature is controlled by the addition of chilled water from a refrigeration unit, MTC-2, or hot water from a steam heated water heater, MTC-3, through a Hammel-Dahl air to open valve. A Brown controller measuring the column temperature by means of a iron-constantan thermocouple, controls the air to the hot and cold water valves. The water from the column jackets recirculates to the constant head tank, MTC1, and the hot water heater and refrigeration unit are fed from Pump MTC7P, so that the entire system is closed except for makeup water to the constant head tank. A schematic flowsheet of the temperature control system, Figure 3.327-1, shows the controlled water flow.

3.33 SOLVENT RECOVERY CELL C

3.331 SOLVENT WASHING

The stripped solvent is drained from the Catch Tank, B9, by gravity flow through a bellows sealed valve, to an Eastern Industries Model F centrifugal pump, C10P, in the Cell C. It is pumped to the Solvent Wash Tank, C1, where it is washed by agitation with caustic



23 PILOT PLANT

SCHEMATIC ARRANGEMENT FOR

COLUMN TEMPERATURE CONTROL

Fig. 3.327-1

[redacted]

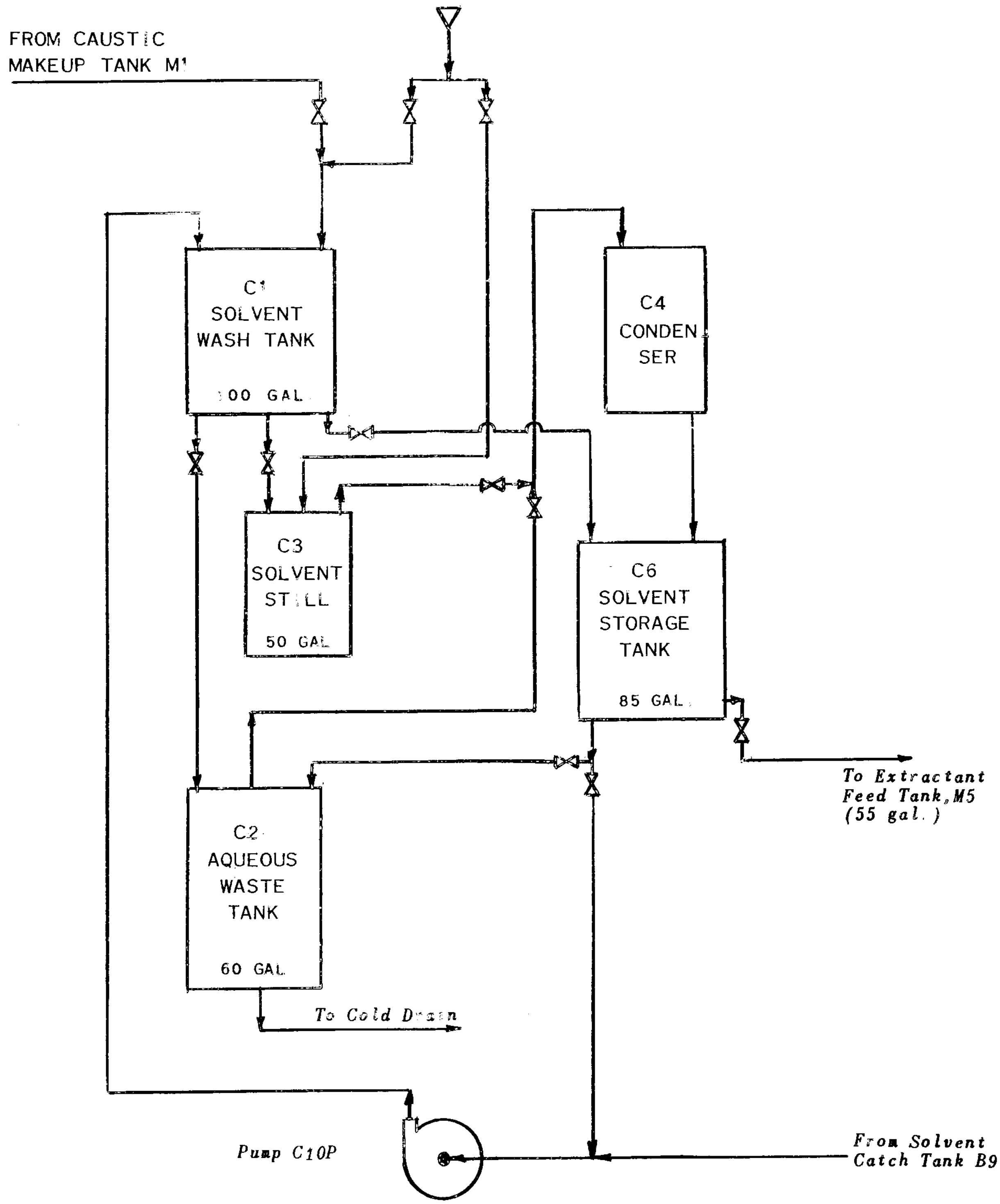
Solvent Washing (continued)

solution. The phases are allowed to separate and the waste wash solution is drained from the bottom of C1, through a bellows sealed valve, to the Aqueous Wash Waste Tank, C2. The outlet drain on C1 is equipped with a liquid contact, electrode type probe which actuates an alarm on the panel board when the contact solution conductivity changes, indicating that drainage of the aqueous phase from the tank is complete.

3.332 STEAM DISTILLATION

The washed solvent can either be drained to the purified solvent Storage Tank, C6, or to the Steam Still, C3, if a steam distillation is desired. Both transfers are made through bellows sealed valves. If a steam distillation is made, the vapors are condensed in Condenser, C4, and the condensate collected in the Storage Tank, C6, which has a bottom drain visually controlled, for removing the water heel. The purified solvent is drained from C6 to the makeup area.

A schematic equipment flowsheet, Figure 3.332-1, shows the Solvent Recovery equipment.



23 PILOT PLANT

SOLVENT RECOVERY SCHEMATIC

EQUIPMENT FLOWSHEET

[redacted]

3.4 MAKEUP AREA EQUIPMENT

The makeup area equipment is located primarily along the south wall of the building at ground level. Several items are spotted in various other locations for facility of operation. In general, all of the tanks have a capacity of approximately 50 gal., are constructed of stainless steel, are mounted on Howe beam type scales, and equipped with a sight glass and a Mixing Equipment Company portable 1/4 HP "Lightin" Mixer, Model C-4.

3.41 CAUSTIC

Caustic solutions are pumped to the process from a caustic makeup tank, M1, with a Viking, all iron, rotary pump, Model ZG2D. Caustic is stored in an iron Caustic Storage Tank, M34, located on the 10 ft. elevation platform. This tank is equipped with a steam heating coil and drains by gravity to M1. Caustic solution for jacket removal is pumped to a Head Tank, M9, on the 30 ft. elevation platform. Caustic scrub solution for the off Gas Scrubber, A29, is pumped to a Head Tank, M33, on the 30 ft. elevation platform.

3.42 ACID

Nitric acid solutions are pumped to the process from Makeup Tank, M2, with a Durco Company, durichlor, open impeller, centrifugal pump, Model X3MDR-72. Nitric acid is stored in tank, M35, on the 10 ft. elevation platform, and drained by gravity to M2. Acid for dissolving operations is pumped to a Head Tank, M9, on the 30 ft. elevation platform.

3.43 THORIUM NITRATE SOLUTIONS

Thorium nitrate solutions, required for salting minimum dissolving batches, are pumped to the process from Alloy Makeup Tank, M3, with an Alsop "Sealed Disc" Filter, Model SD-6-NR-4, equipped with a "centripoise" pump. A stainless steel hood, M32, with a 1000 CFM blower, is mounted on M3 to prevent the spread of alpha activity.

3.44 Scrub Solutions

Scrub solution is made up in Scrub Makeup Tank, M7, and pumped to the Scrub Feed Tank, M4, with an Alsop "Sealed Disc" Filter, Model SD-6-WR-10, equipped with a "centripoise" pump. Scrub solution is pumped to the extraction column, through a surge pot and rotameter, with a Milton Roy Simplex pump, Model MD1-13-24, driven by a 1/6 HP motor and equipped with a micro indicating while running stroke adjustment.

3.45 SOLVENT

Purified solvent is drained from C6 and transferred by gravity flow to the Solvent Feed Tank, M5, through a section of 4 inch pyrex glass pipe, M8, where it is prescrubbed with aluminum nitrate solution. Solvent is pumped to the extraction column, through a surge pot and rotameter, with a Milton Roy Simplex pump, Model MD1-24-48, driven by a 1/4 HP motor and equipped with a micro indicating while running stroke adjustment.

3.46 STRIP SOLUTIONS

Stripping solutions are made up in Strip Makeup Tank, M6, and are pumped to the Stripping column, through a surge pot and rotameter, with a Milton Roy Simplex pump, Model MD1-13-24, driven by a 1/6 HP motor and equipped with a micro indicating while running stroke adjustment.

3.5 INSTRUMENTATION

3.51 PANEL BOARDS

Instruments for process control are mounted on three panel boards. Each panel board is constructed of 1/4 inch steel plate bolted to an angle iron frame. Panel Board No. 1, located on the south 10 ft. elevation platform, is built in the shape of an L and services

Panel Boards (continued)

all equipment in Cell A. It is approximately 25 feet long. Panel Board No. 2, located on the north 10 ft. elevation platform, is approximately 10-1/2 ft. long and services all equipment in Cells B and C. Panel Board No. 3, located on the north ground level, services the extraction column temperature control unit.

3.52 PRESSURE

Pressure gauges for panel board headers, process services, and jets are flush mounted, bourdon tube type gauges, manufactured by the Ashcroft Gauge Division of Manning, Maxwell, and Moore Company.

3.53 Liquid Level and Specific Gravity

Both recording and indicating type instruments are used for liquid level and specific gravity determination. Two pen, double bellows recorders of the aneroid manometer type, manufactured by the Taylor Instrument Company, Catalog No. 76JD282, are used for recording both liquid level and specific gravity on the three column Catch Tanks, the Dissolver, the Feed Makeup Tank, and the Extraction Column Feed Tank. All other liquid level and specific gravity instruments are well type, flush mounted, manometers manufactured by Meriam

Liquid Level and Specific Gravity (continued)

Instrument Company, Part No. A203FF. These instruments are connected, on both high and low pressure sides, with Fisher and Porter "C" clamp rotameter air bubblers and Moore Products Company, type 62 constant differential relays. Meriam Instrument Company manometer check valves, Part No. M-104, are connected only to the low pressure side.

3.54 TEMPERATURE

Two Leeds and Northup Company indicating and recording potentiometers, Model S Micromax, are connected by IC thermocouple leads to tanks in Cells A and B. A Brown Instrument Company Electronik Recording Resistance Thermometer, Part No. 153X60XW6X42A, records temperatures of the extraction column feed and discharge lines. A Brown Electronik Series 156X63 precision indication, with selector switch, is used to indicate temperatures of the extraction column temperature control system and equipment items in Cell C. The air operated valves on the column temperature control system are actuated by a Brown Electronik temperature controller, Part No. 152P14P-63-11A.

3.55 INTERFACE CONTROL

Taylor Fulscope aneroid flow recording controllers, Catalog No. 122RD182, are used for column interface control. The bubbler lines have the same accessories as the liquid level and specific gravity instruments. The controlled air output is reduced with a Moore Products Company 5:1 Reducing Ratio Relay.

3.56 OVERFLOW ALARMS

Electric Liquid Level Controllers, manufactured by Moore Products Company, are mounted on Tanks A5 and A8, and operate warning lights and a howler on Panel Board No. 1.

3.57 VACUUM CONTROL

A mercury filled Cartesian Manostat, manufactured by Emil Greiner Company, operating on the cartesian diver principle, controls the amount of vacuum on the star type filter.

3.6 SAMPLERS

Tank solutions are sampled by recirculating jet type sample units each enclosed in a separate lead case mounted on the cell faces at about the 25 ft. elevation. A Cl-5 air jet with auxiliary air lift is used to circulate the solutions.

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Samplers (continued)

Column streams, except for the "AP" stream, are sampled with re-circulating jet type units from overflow sample pots. The "AP" stream is sampled by a direct tap into the process line.

3.7 JETS

Jets used for process transfers are Schutte and Koerting, 3/4 inch, Figure 217, steam jets. Vacuum for the Vacuum Transfer Tank A8 and the process vent system is supplied with 3/4 inch Schutte and Koerting, Part No. 431, Steam Jet Exhausters which are operated with air instead of steam. Vacuum for the filter is provided with a one inch jet of the same type.

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