REPÚBLICA ARGENTINA

COMISION NACIONAL DE
ENERGIA ATOMICA

PILOT PLANT FOR THE REPROCESSING OF IRRADIATED NUCLEAR FUELS

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REPUBLICA ARGENTINA

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Pilot Plant for the Reprocessing of Irradiated Nuclear Fuels

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Buenos Aires, 1967.

PILOT PLANT

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PILOT PLANT FOR THE REPROCESSING OF IRRADIATED NUCLEAR FUELS

1) OBJECTIVES

The present report describes the small pilot plant the Argentine Atomic Energy Commission has built at the Ezeiza Atomic Center near Buenos Aires. The installation, equipment and flow-sheets are briefly detailed.

The objectives in mind for designing this plant were the following:

1) Obtaining experience in the chemical process and remote operation techniques needed for the reprocessing of irradiated fuels.
2) Formation of personnel with the necessary preparation to design future plants on a larger scale.
3) Reprocessing the spent fuel elements from the first core of the RA 1 reactor.

The installation cost of this plant could be kept very low without jeopardizing the security of the process due to the low activity of the fuel elements and the limited objectives mentioned.

2) FUEL ELEMENTS DESCRIPTION

The material to reprocess are 119 fuel elements in the form of aluminium cladded sheets belonging to the Argonaut-type RA 1 reactor. Each sheet is 600 mm long, 72 mm wide and 2.7 mm thick. The fuel is 20 % enriched uranium in the form of U_3O_8 dispersed in aluminium. The mean weight of each fuel element is 347 g and contains an average of 117 g U_3O_8 with 19.45 g U-235.

Since the start of the reactor these fuel elements have been irradiated with times and fluxes as indicated in Table I.

date	irradiation time hs	power kW	flux $n/cm^2.s$
8/3/60-12/6/60	573	10	$9.5x10^{10}$
I/11/61-6/22/61	725	10	$9.5x10^{10}$
6/26/61-11/8/62	2373	20	$1.9x10^{11}$
11/12/62-4/30/63	1796	15	$1.4x10^{11}$

TABLE I. Irradiation times and fluxes for first core of RA 1.

The fuel elements were removed from the reactor between 10/15/63 and 11/22/63. They are now stored in the pool of the reprocessing plant.

3) PROCESS DESCRIPTION

To perform the proposed objectives we have chosen a liquid-liquid extraction process using tri-butyl-phosphate as extractant.This decision was based on the widespread use of TBP in reprocessing plants resulting in an extended literature being available. Also, local experience with this solvent had been acquired, since TBP is used for the purification of uranium salts.

The process starts with the complete dissolution of the fuel elements in nitric acid. Because of the high aluminium content, the aluminium nitrate solubility is the limiting factor that maintains the uranyl nitrate concentration at low values. Consecuently the processing volumes and the active waste volumes are high compared to the weight of recovered uranium. Because of the small amount of fuel to reprocess and for simplicity reasons, we preferred the complete dissolution in nitric acid instead of the usual previous caustic aluminium dissolution.

The dissolver solution is very turbid because of the presence of coloidal SiO_2. It is necessary to clarify the solution by filtration through a porous stainless steel filter prior to the solvent extraction step.

To obtain experience in the U-Pu partition process we will try to recover the Pu present in the solution in spite of its very small amount. For this purpose, two

solvent extraction cycles are provided. Each one has an adequate number of extraction, scrubbing and stripping stages. The chemical variables are adjusted in the first fuel cycle to produce the codecontamination of U and Pu from the bulk of the fission products and the aluminium. In the second cycle, the partition of Pu from U is performed as well as an additional decontamination of the U.

The uranium is precipitated as peroxide, filtered, dryed and calcined to obtain U_3O_8. The Pu solution which still contains fission products is concentrated and decontaminated by ion exchange. After adecuate adjustment of the solution, the anion exchange resin column retaines Pu and separates Fe, U and fission products. The adsorbed Pu is eluted and precipitated as oxalate. All the active waste solutions will be stored in an underground stainless steel tank until definite disposal in the ground.

4) DESCRIPTION OF FLOW SHEET

The flow sheet is detailed in Fig. 1. All amounts are adjusted to the treatment of batches of 8 fuel elements to maintain the total mass of U-235 within safe limits.

Dissolution

In a previous work (1) the nitric acid consumption of the RA 1 fuel elements dissolution has been studied. The obtained values had been compared with data found in the literature.

The aluminium is dissolved in nitric acid according to the following reactions:

a) mercury catalyzed Al dissolution in 4N HNO_3

$$Al + 3.75\ HNO_3 \longrightarrow Al(NO_3)_3 + 0.225\ NO + 0.15\ N_2O + 0.1125\ N_2 + 1.825\ H_2O$$

b) mercury catalyzed Al dissolution with air bubbling. Slow addition of acid

$$Al + 3.18\ HNO_3 \longrightarrow Al^{3+} + 2.56\ NO_3^- + 0.44\ HO^- + 0.023\ NO_2 + 0.23\ NO + 0.03\ H_2 + 0.05\ N_2O + 0.13\ N_2 + 1.34\ H_2O$$

The second equation gives a nitrate deficient solution but coincides with the first in establishing that 20 % of the HNO_3

consumed is reduced. The equations differ only in the composition of the evolved gases.

The U_3O_8 present in the fuel plates contributes a negligible amount to the acid consumption.

This acid consumption was compared with experimental results obtained dissolving samples of fuel elements in HNO_3. The Hg concentration was 0.05M and the gases were absorbed in a NaOH scrub column. The results are the following:

	NO_3 in the dissolver	Retained in the absorption column	Lost
Calculated	78.5%	10.8%	10.6%
Obtained	77.7%	10.5%	11.8%

To avoid hydrolysis and assure a high recovery of Pu, the nitric acid concentration is kept at 1N. At this acidity the $Al(NO_3)_3$ saturation concentration is 2.3M at 19 °C. To avoid the risk of crystallization the dissolver solution is kept at 2M $Al(NO_3)_3$. Since the average Al/U weight in the fuel plates is 2.33 the U concentration will be approximately 23.2 g/litre.

The mass balance indicates that the dissolution of 8 fuel plates requires: 20.7 litre 14N HNO_3, 13.4 litre H_2O and 34 g Hg. 34.2 litre of a 2M $Al(NO_3)_3$, 1N HNO_3, 23.2 g U/litre and 0.005M $Hg(NO_3)_2$ solution will be obtained.

Filtration

The dissolver solution is very turbid because of the presence of colloidal SiO_2. The clarification by direct filtration is practically impossible. However, by addition of 100 ppm of gelatin in the form of a 1% solution, the floculation is produced in a few hours (2). The resulting solution is easily filtered through a sintered stainless steel filter covered by a filter aid like Hyflow Supercel (Celite). After filtering, the filter cake is washed with 5 litre 1N HNO_3. The final solution is analyzed and adjusted to obtain 45.6 litre of a solution 1.5M $Al(NO_3)_3$, 1N HNO_3, 17.8 g U/litre and 0.0037M $Hg(NO_3)_2$.

The filter aid retains an appreciable amount

of activities, particularly zirconium and niobium.

The filter cake is separated by counter-current washing with water and directed to the waste tank.

Solvent extraction

The system is formed by two decontamination cycles. The first one separates U and Pu from the bulk of tho fission products and the aluminium. The second one partitions Pu from U and produces an additional decontamination step for U. Each cycle is formed by an adequate number of extraction, scrubbing and stripping stages. In the first cycle extraction and scrubbing stages, the nitric acid concentration is adjusted to 1N and the Pu valence state to IV to assure its extractability by TBP. $Al(NO_3)_3$ is the salting agent which displaces U and Pu to the organic phase.

As solvent, 5% TBP in kerosene is used. The aromatic and unsaturated hydrocarbons present in the diluent are previously eliminated by washing with concentrated sulphuric acid to avoid the formation of nitro-derivatives by agitation with the feed solution.

The 5% TBP concentration was choosen for the convenience of obtaining a high percentage of U saturation in the solvent and at the same time to maintain the flow rate of acqueous to organic phases at a value near unity to improve the efficiency of the solvent extraction step.

The U saturation in the solvent is adjusted to 70% at the outlet of the scrubbing section. This is high enough to maintain the extractability of fission products at low values and offers sufficient margin of security to avoid losses of U due to accidental changes in pumping rates or concentrations.

In the scrubbing section the washing solution is 1.5M $Al(NO_3)_3$ and 1N HNO_3, with an aqueous/organic flow ratio of 1/3. This ratio assures a good scrubbing efficiency and the high salt concentration avoids an excessive reflux. With these concentrations and flow ratios the U recovery is higher than 99.9% and sufficient flexibility is provided to avoid any U loss due to minor changes in the flow rates or

concentrations. This may be appreciated in the corresponding Varteressian-Fenske diagram (Fig. 2).

For stripping, a 0.01N HNO_3 solution is used and the aqueous/organic ratio is 1/4. The spent solvent is washed with 5% Na_2CO_3 solution prior to recycling.

The U-Pu solution from the first cyclo is adjusted for its feeding to the second cycle. The Pu is reduced to valence III by means of ferrous sulphamate and concentrated HNO_3 is added as salting agent to final concentrations of 0.05M Fe^{++} and 3N HNO_3. In this condition U is extracted by TBP while Pu III remains in the aqueous phase.

In the second cycle 20% TBP is used. The solvent is scrubbed with 3N HNO_3 at an aqueous/organic flow ratio of 1/3. The stripping is performed with 0.01N HNO_3 solution at a 1/1 aqueous/organic flow ratio. The solvent is washed with 5% Na_2CO_3 and recycled.

By this procedure Pu is separated from U. The Pu remains in the raffinate from the 2nd cycle in a solution with high nitric acid concentration along with the fission products separated in this cycle.

Pu recovery

The Pu decontamination and concentration is performed by ion exchange using Permutit SK resin. The process is based on the selective absorption of Pu IV as anion complex in such conditions that the other components like Fe III, U and fission products are not retained by the resin (3).

For this the Pu is stabilized at valence IV by making the solution 0.2M in $NaNO_2$. This is enough to oxidize all the $(SO_3NH_2)_2Fe$ present. The nitric acid concentration is raised to 7N which corresponds to the highest Pu distribution coefficient. Permutit SK resin is chosen because of its good elution kinetics and its resistance to chemical damage due to HNO_3.

After absorption, the resin is washed with 7N HNO_3 and the Pu eluted with 0.3N HNO_3.

U recovery

The $UO_2(NO_3)_2$ solution obtained by the solvent extraction process is treated with H_2O_2 to precipitate UO_4. The pH is maintained at a value of 2 during the process by addition of NH_4OH and the temperature is adjusted to approximately 50 °C to form a precipitate which is easy to filter.

The peroxide is filtered, dryed and calcined at 800 °C to form U_3O_8. The filtered solutions are neutralized with NH_4OH and allowed to stand for a day to precipitate ammonium diuranate, which is recycled to the solvent extraction process.

5) DESCRIPTION OF EQUIPMENT

Location

The plant is located in a building which was formerly a uranium production plant at Ezeiza (Fig. 3). The room in which it is contained measures 23 m by 8 m, with a height of 8 m. The plant units are contained in three concrete cells. The first cell contains the dissolution equipment with its accessories, the second one the storage tanks of solutions with high activities and the third one the solutions of intermediate activities. The first two cells have 60 cm concrete wall thicknesses with a height of 2 m while the third one has a 30 cm concrete wall. Adjoining the dissolution cell is the storage and decay pool for the fuel elements. The hoods, which contain the solvent extraction equipment and the filter, are located on the outside of the cells (Fig. 4).

Storage pool

This is a long and deep tank with a capacity of 3 m^3. The tank is buried and open at the top so that its upper part is at ground level. It is made of 2 mm stainless steel sheet and rests on a concrete layer of about 30 cm. The width of the tank is 40 cm with a depth of 210 cm. At one end it has a discharge section with a width of 80 cm and 265 cm depth. The storage pool continues under the concrete wall of the dissolution cell, so that fuel elements can be loaded into the dissolver by means of a chute in the cell.

The water is cleaned by circulation through a filter and the conductivity is kept low by means of a mixed bed ion exchanger.

Dissolution equipment

The discontinuous dissolver consists of a cylindrical tank with 95 litre capacity. It has two condensers, heating coils, a feeding chute and accessories for measurements and sampling (Fig. 5). The tank is made of 3 mm type 304 stainless steel and has a coil of 12.7 mm O.D. with a heat interchange surface of 0.32 m^2. The coil can be used for steam heating as well as cooling by water circulation. Connections are provided for air injection, thermocouples, density and level indicators, and the sampler. To avoid direct contact of the fuel elements with the heating coil and other connections, a perforated bask has been provided which is made out of 3 mm stainless steel sheet. The fuel elements which are loaded through the chute fall into this basket.

The dissolver has two condensers. The first one is of the water jacket type with a heat exchange surface of 0.31 m^2. It has a sliding lid by which it is connected to the loading chute. The second condenser, which is parallel to the first, is of the shell and tube type with an interchange surface of 1 m^2. Both are totally constructed of stainless steel. A constant negative pressure of 15 mm water column is kept in the dissolver so that gases cannot escape via the sliding lid, which is not airtight. The off gases are evacuated through the plant ventilation system.

The dissolution process is controlled by means of one immersed thermocouple and five thermistors located on different parts of the condensers and gas extraction circuit.

Storage tanks

Storage tanks with 70 litre capacity contain the solutions from different parts of the process. They are built of stainless steel 304 with 3 mm thickness (Fig. 6) and are provided with density and level probes, connections for sampling with a Thorex-type sampler, solutions entrances, air entrance for agitation, and a

connection for ventilation or vacuum. Decontamination with a recirculating solution is provided which works with a steam jet. This directs the washing solution against the top and walls of the tank.

Four of these tanks are contained in the second cell. They are used for: storing the solution coming from the dissolver, storing the filtered solution, storing the feed solution of the first solvent extraction cycle, and storing the active residue from this cycle.

In the third cell five tanks are located for the following purposes: storing of the TBP solutions of both solvent extraction cycles, storing of the U and Pu solution from the first extraction cycle, storing of the residual plutonium solution from the second cycle, and the spent 5 % Na_2CO_3 solution after its use in the washers.

Filter for the dissolver solution

A porous stainless steel filter (Pall Corp., Micromotallic Div.) is used. The filtering medium is a cylinder with a filtration surface of 0.1 m^2 with a mean pore size of 5 microns, capable of retaining 98 % of 2 micron particles. The filter is mounted in such a way that the following operations can be carried out without dismantling (Fig. 7): covering the filter with a filter-aid layer, filtering the solution, washing the cake, and disengaging and removing the cake. The filter is placed in a recess in the plant with its front covered by a 5 cm lead shield. The shield is perforated in places to let through the connecting rods to the valves by which the filter circuit is operated.

Solvent extraction equipment

Four pump-mix multistage mixer-settlers of the type developed by Knolls Atomic Power Laboratories form the TBP extraction equipment. This type of mixer-settler is a horizontal continuous contactor in which the phases flow co-currently in the individual steps and countercurrently between the different steps and in the overall process. Its principal feature is that the impeller of the mixer provides the necessary pressure difference for moving the heavy phase and

regulates the mixing intensity as well as the interphase level. This eliminates the necessity of an interphase controller and indicator for each individual step, and it makes unnecessary tilting the unit to obtain a level difference.

A mixer-settler unit consists of an appropiate number of horizontally disposed steps, each of which has one mixing and one settling section. The design and the flow of the phases are indicated in Fig. 8. The mixing section has 2 chambers connected by a hole in a horizontal plate. The upper chamber contains the turbine which reaches the lower chamber by means of its suctioning tip. In this way the turbine at the same time lifts and disperses the heavy phase. The upper mixing chamber is in contact with the settling chamber by means of a connection at the top which avoids turbulence and causes no appreciable pressure drop. The mixing section is also connected to the settling sections of the adjacent steps by openings for the heavy and light phases. These ports are designed to oppose backward flows but without causing a significant pressure drop. The turbine has a much higher pumping rate than the flow of the heavy phase. This produces an efficient mixing of the phases and maintains the interphase in the mixing chamber at a lower level than the height of the aspiration tip. The interphase level in the adjacent settling chamber is automatically regulated through the heavy phase port. It is necessary only to control manually the level in the last discharge step of the heavy phase which can be done by means of an air regulated outlet weir.

The mixing chamber is 3 x 3 cm by 5.5 cm height and the settling chamber is 3 x 12.5 cm by 6.5 cm height. Two 16 steps mixer-settlers are used in the first cycle and the second cycle uses one of 22 steps and one of 10 steps. The first unit in each cycle is used for extraction and scrubbing and the second one for stripping. A stainless steel tray is provided for each mixer-settler and all of them are shielded with 5 cm lead on the bottom and sides. The four mixer-settlers are located in a cell with sliding glass doors, the air of

which is withdrawn through a prefilter and then extracted through the general air extraction circuit (Fig. 9).

Metering pumps

The remote pumping of the active solutions (aqueous or solvent) of each cycle is made by Lapp Pulsafeeder metering pumps, model CPS-1. These pumps are a combination of piston and diaphragm pumps. Dosage is established by means of a precise regulation of the stroke length of the piston. Pulsation is not directly transmitted to the liquid to be pumped but by hydraulic transmission through two teflon diaphragm heads. In this way the stroke is transmitted in a uniform way and without losses to the remote reagent head located behind the concrete wall. With this arrangement the same precision is obtained as with a piston pump and the problems of direct pumping of active solutions are eliminated.

For pumping and dosage of inactive solutions (washing and stripping) piston pumps with variable stroke lengths are used. The pump head is in direct contact with the solution in this case. The heads have a double set of ball valves for entrance and outlet to insure precision in dosage.

Solvent washers

During solvent extraction, TBP undergoes a hydrolysis process with formation of di- and mono-butyl phosphates. At the same time the nitric acid present produces nitrate derivatives with the diluent. Radioactivity of the solutions, although low in this case, also causes degradation of the solvent. These reactions, while unimportant from the quantitative point of view, have a strong effect on the uranium decontamination and the plutonium recovery. It is therefore necessary to wash the TBP continuously with a solution capable of retaining the acid decomposition products.

To this effect, two solvent washers have been installed in the TBP circuits of each cycle. Each washer consists of an agitation section and a settling section, separated by deflecting plates so that turbulence cannot be transmitted between them (Fig. 10). The agitation section is a cylinder with 10 cm diameter and 3.5 litre capacity, where

continuous central agitation is produced by a central axis with five impellers. Each washer contains 2 litre of a 5 % Na_2CO_3 solution. The mean retention time of the solvent in the agitation section is half an hour for each washer, with a flow of 3 litre/hour. The depleted Na_2CO_3 solution is transferred to a storage tank for analysis. If it contains uranium, the solution can be acidified and solvent extracted; if not, the solution is transferred to the waste disposal tank for active residues.

Samplers

Samples of the dissolver and storage tanks can be taken with a sampler similar to the type used in Oak Ridge National Laboratory's Thorex pilot plant. In this sampler a circulation of the solution is established from the tank to the sampler's bottle and back to the tank (Fig. 11). The circulation pipes cross the concrete shielding to the sampling bottles, which are on the outside of the shielding. The sample is only a few cc in volume and can easily be transported in a lead shielded container.

Circulation of the active solution is induced by means of an airlift aided by a negative pressure in the sampler. The system only works if air is injected in the air lift and if a negative pressure is established at the same time. The solution circulates through the sampling bottle by means of two hypodermic needles which perforate the rubber stopper. If the bottle is not in its place the system does not operate since the vacuum causes air to be taken in through the needles.

Air and solution coming from the sampling bottle go to a container above the tank where they are separated and the solution returns to the tank. The container is also under negative pressure. To avoid possible losses of active solution through the vacuum line, the container possesses a floater which closes the vacuum and opens the air admission when the solution reaches a dangerous level.

The sampling system consists of two units,

each of which can sample from five different locations. The first one samples the dissolver and the four storage tanks in the second cell; the second sampler samples the five tanks in the third cell.

Tank interconnection system

Vacuum is used to transfer solutions from one tank to another. They are connected by means of stainless steel piping and stainless steel valves with teflon diaphragms. The valves are located on three racks placed at 1.2 m height above the top of the tanks. The valves themselves are inside the cells and their stems cross the shielding wall, so that they can be manually operated from the outside. Solutions are transferred evacuating the tank to be filled and opening the appropiate valve. Connections have also been provided for relaying back solutions to a former step in the process, since it may happen that an operation is not running at the required efficiency.

The following solution transfer operations are possible:

Valve number	from	to
1	dissolver	storage tank
2	1st cycle product	,, ,,
3	filtered solution	,, ,,
4	filtered solution	feed adjustment
5	1st cycle waste	storage tank
6	1st cycle waste	intermediate waste decay
7	1st washer 1st cycle	washer treatment
8	2nd washer 1st cycle	,, ,,
9	1st washer 2nd cycle	,, ,,
10	2nd washer 2nd cycle	,, ,,
11	2nd cycle waste	1st cycle product
12	2nd cycle waste	Pu recovery
13	washer treatment	1st cycle product
14	washer treatment	intermediate waste decay

Waste disposal of active residues

A 12 m^3 tank is provided for decay of the active waste from the plant. This is sufficient to contain all

liquid residues produced during reprocessing of the core of the RA 1 reactor. This tank is made from 2.5 mm stainless steel type 316 sheet and is located underground in a concrete chamber. Between this storage tank and the plant a 120 litre capacity intermediate tank is located, also underground and in a concrete chamber (Fig. 12). This tank is made from 3 mm stainless steel sheet and it can be evacuated, so that it can be filled with solutions from the plant. From the intermediate tank the solutions flow to the storage tank by gravity alone.

It is intended to use absorption in the ground as the ultimate waste disposal method. For this the geophysical characteristics are appropiate and a special location near the plant will be reserved for this purpose (4).

An underground stainless steel centrifugal pump is used for discharging the liquids from the storage tank for ultimate disposal. Double piping is provided as connection between the tanks and the pump. One of these lines is remotely operated by solenoid valves and the other manually.

Plant decontamination system

All storage tanks have been linked to a decontamination system to provide access to the plant for repairs or modifications. By means of a steam ejector, a decontaminating solution can be recirculated in each tank thrcugh a spray nozzle which wets the top and the walls of the tank. The dissolver can be decontaminated by boiling a nitric acid solution in it and the solvent washers by agitation with the appropiate decontamination solutions.

To control the activity inside the plant as well as to measure the degree of contamination of each equipment, a monitor is placed near each one with a scale ranging from 0.1 mR/h to 10 R/h.

Ventilation system

The room containing the pilot plant is provided with an air ventilation and filtration system which maintains a 2 mm water column negative pressure. The fresh air renewal rate is 8 times per hour.

The injection system consists of a battery

of glass fiber pre-filters, a pre-heater, a humidifier, a heater and a centrifugal injection ventilator. Fresh air enters the plant room through 10 inlets on the roof and is injected at a temperature of 17.1 °C and a relative humidity of 50 %. The extraction system collects the air through 10 outlets located on the walls after which it circulates through a battery of pre-filters and absolute filters, a centrifugal ventilator and a 12 m high chimney (Fig. 13). The absolute filtration battery consists of 4 glasswool pre-filters with 10 cm thckness and 4 units of Vokes absolute filters, modol CG6, which are fireproof and acid resistant. The filters and pre-filters can be exchanged and enclosed in polythene bags without danger of contamination through an exchange device.

The cells and hoods of the plant, as well as the dissolver, have their own ventilation and air filtration system. Here a depression of 10 mm water column is maintained by means of a centrifugal ventilator and the fresh air renewal rate is 10 times per hour. The air enters the third cell through a pre-filter after which it circulates through the second cell and the dissolution cell. It is then mixed with air coming from the solvent extraction hoods and the gases from the dissolver. A 15 mm water column depression is maintained in the dissolver by means of controls so that gases produced during dissolution cannot escape through its sliding lid which is used for loading. Before the exhaust air is released, it is heated by electrical resistances so that condensation of water and nitric acid in the filters is avoided. It then passes a glass fibre pre-filter with a [illegible] and an absolute Vokes filter, type [illegible] through a 12 m high stainless steel

[illegible] mixed bed ion exchange demineralizers [illegible] them provides pure water for the make-up [illegible] the plant. The other one keeps pure water [illegible] the fuel elements are kept.

[illegible] a vertical boiler, fueled by gas oil, [illegible] the plant. Its heating surface is 4 m^2.

6) CRITICALITY CONTROL

Nuclear safety will be kept by mass control of total U-235 in the plant. The reprocessing will be carried out batch-wise, using 8 fuel elements at a time, with a mean mass of 157 g U-235. This amount is 41 % of the maximum safe limit of 384 g U-235 established by the nuclear safety regulations of the Argentine Atomic Energy Commission for the case of solutions with 20 % enriched uranium with total reflection. The control of the amount of uranium in the plant at one time will be carried out by mass balances based on chemical analyses of samples of the solutions in the tanks and by measurement of their volumes.

The most dangerous parts of the plant in regards to nuclear safety are the dissolver and the solvent washers.

The dissolver could accumulate scrap from undissolved fuel plates. To prevent this possibility, dissolution of fuel elements will be continued for a time considered as safe (6 hours) while keeping sufficient free acidity to insure a high reaction rate. Mass balance control at the end of the dissolution step will show if a significant deviation exists from the mean weight of 8 fuel elements. The dissolver will be cleaned out with hot nitric acid after each five dissolution processes or after an anomalous mass balance.

In the solvent washers, an accumulation of uranium could happen through malfunction of the stripping mixer-settlers. If the TBP at the mixer-settler outlet still contains uranium, it will be complexed by the Na_2CO_3 in the washers and retained there. To avoid this danger control will be kept on the mass balances of the extraction cycles and the Na_2CO_3 solution used in the washers will be changed after each five batches or after an anomalous mass balance.

The mixer-settlers do not present criticality problems since their volume and shape are always safe. The control of all other steps of the process are carried out by chemical analysis and measurement of the volumes of the solutions in the tanks.

7) LABORATORIES

In another section of the plant building are the chemical laboratories with one section for analytical chemistry and one for alpha active elements. The analytical laboratory is equipped to carry out the routine analyses needed for the plant. The following analyses are made:

Uranium: Samples with over 0.1 mg/ml are titrated by coulombimetric analysis. The method has been adapted from the one described in (5), method 1.302.

For samples with concentrations between $5 \cdot 10^{-5}$ mg/ml and 0.1 mg/ml a fluorimetric method described in (5) has been adapted, method 1.402.

Free acidity in the presence of hydrolyzable ions:

The same method is used as described in (6), Suppl. 4, H^+-Pot-4, Mod-5.

Aluminium: The method followed is the one described in (6), Suppl. 1, Al-1, Mod-2.

Tributyl phosphate:

TBP concentration in kerosene is found by titrating the amount of HNO_3 extracted by the TBP. The method follows the one described in (6), Suppl. 4, TBP-2.

Fission products: Total activity is determined by 4 pi beta counting. The composition of gamma emitting fission products is found by gamma spectrometry, for which a 512 channel analyzer (Nuclear Data) is available.

Plutonium: Plutonium is determined by specific absorption on a Dowex 1-X4 anion exchange resin from 7.2 N HNO_3. After elution it is counted in a calibrated alpha counter (3) (7).

Ferrous sulphamate:

Ferrous ion is titrated by coulombimetry. To determine sulphamate, it is first oxidized to sulfate by nitrite, then weighed as barium sulfate.

These methods together with some others have been collected in an Analytical Guide for use in the laboratory.

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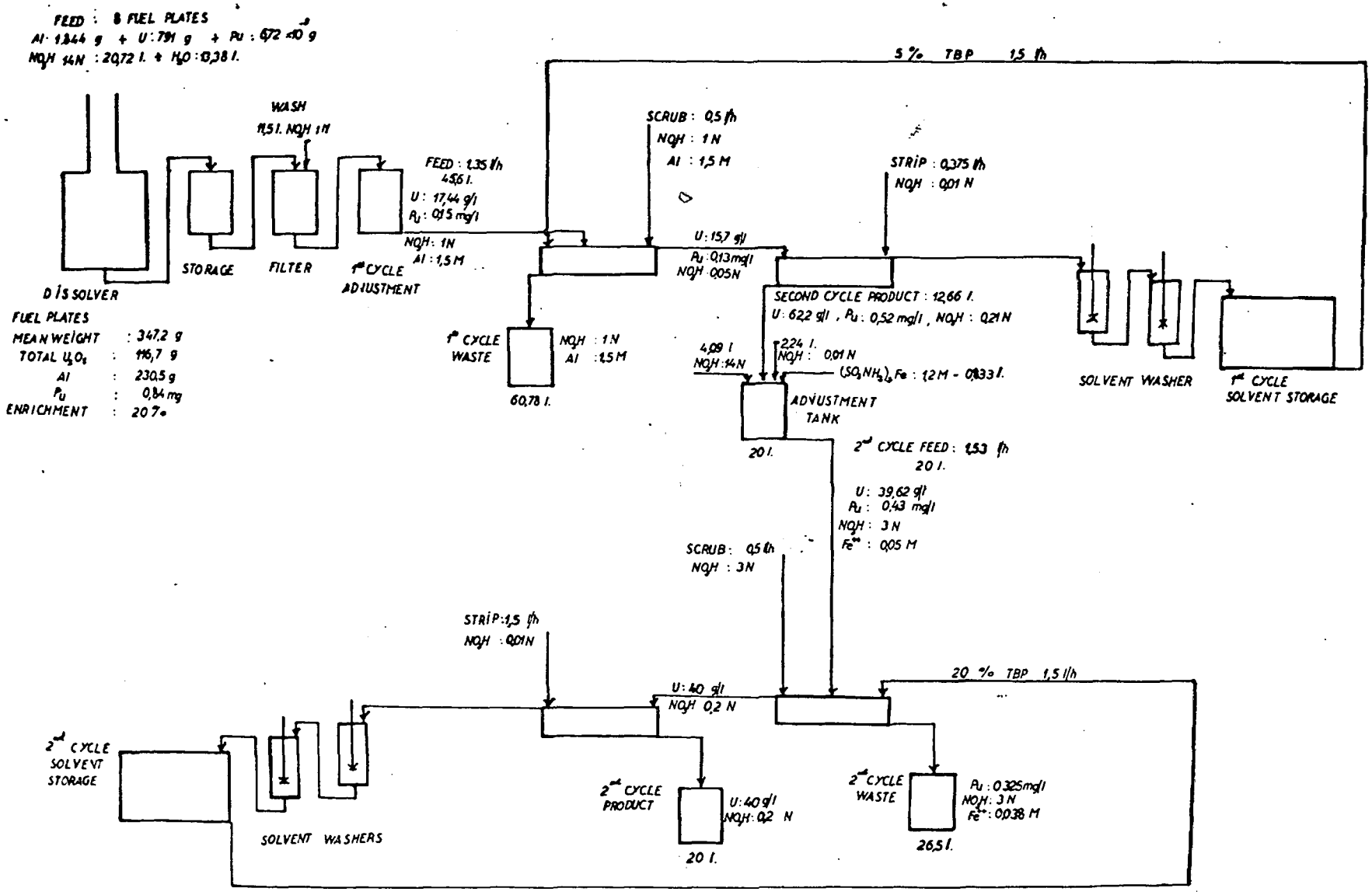


FIGURE 1 FLOW SHEET

	U g/l	Al M	H N	flow rate
feed	17,4	1,5	1,0	1,35 l/h
scrub	-	1,5	1,0	0,50 l/h
solvent	-	-	-	1,50 l/h

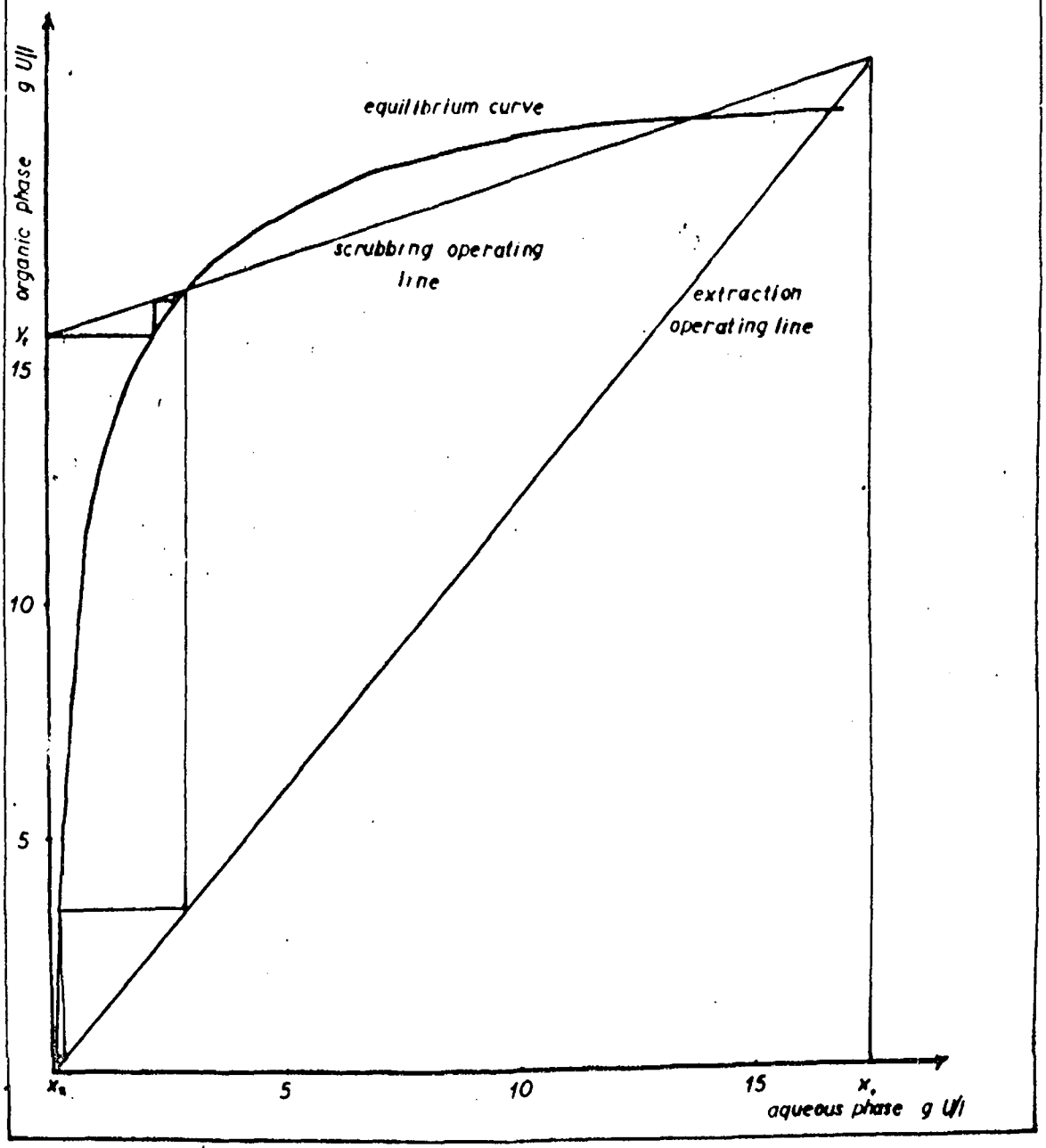


FIGURE 2

VARTERESSIAN-FENSKE DIAGRAM

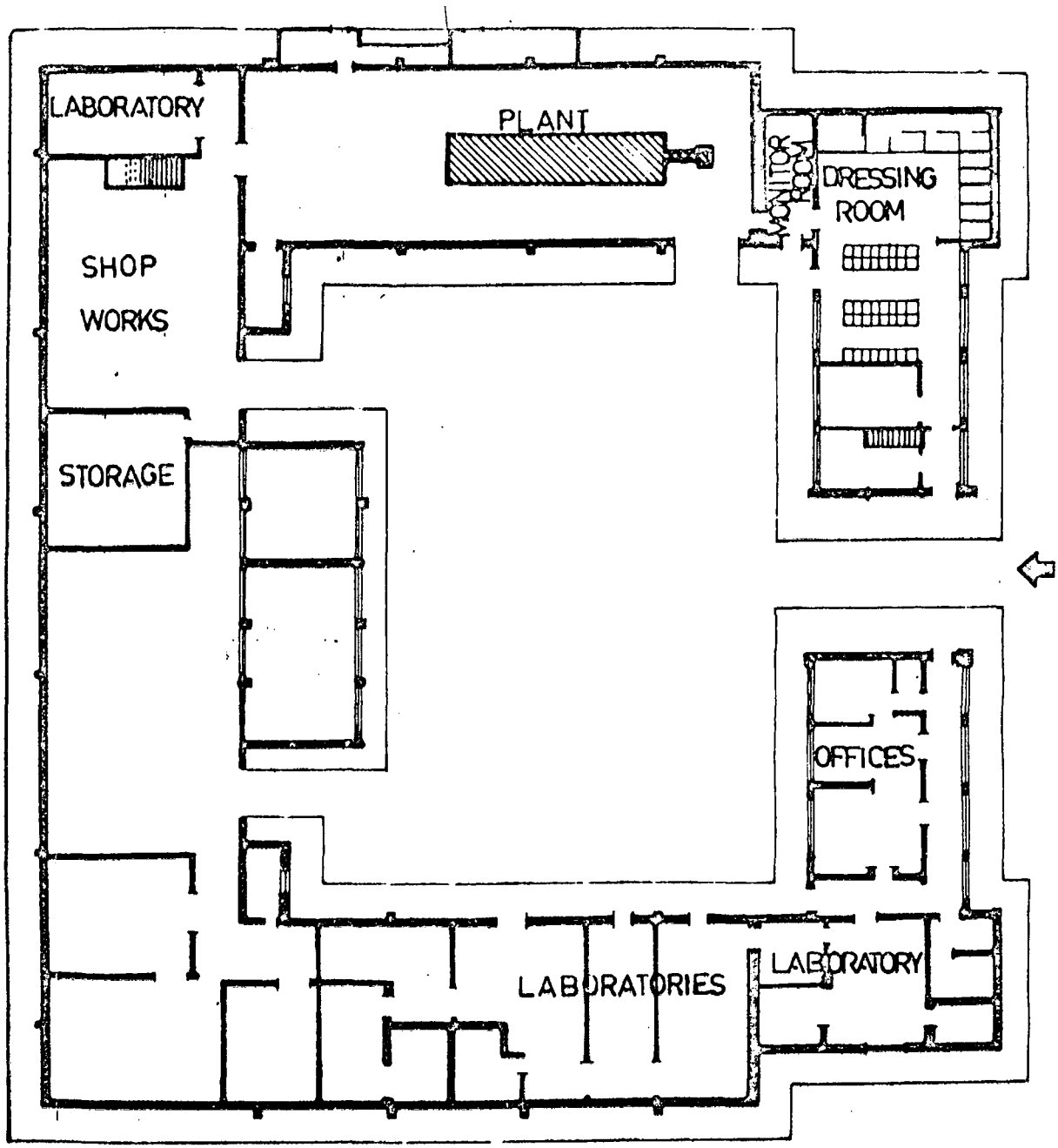


FIGURE 3.

CHEMICAL REPROCESSING BUILDING

FIGURE 4

PLANT DISTRIBUTION

1. Storage pool
2. Dissolver
3. Dissolver storage tank
4. Hot cell air filter
5. Filtered solution tank
6. Feed solution adjustment tank
7. 1st cycle waste tank
8. 1st cycle solvent tank
9. Filter
10. Solvent washers
11. 1st cycle hood
12. 2nd cycle solvent tank
13. 1st cycle product tank
14. 2nd cycle waste tank
15. Washer waste treatment tank
16. 2nd cycle hood

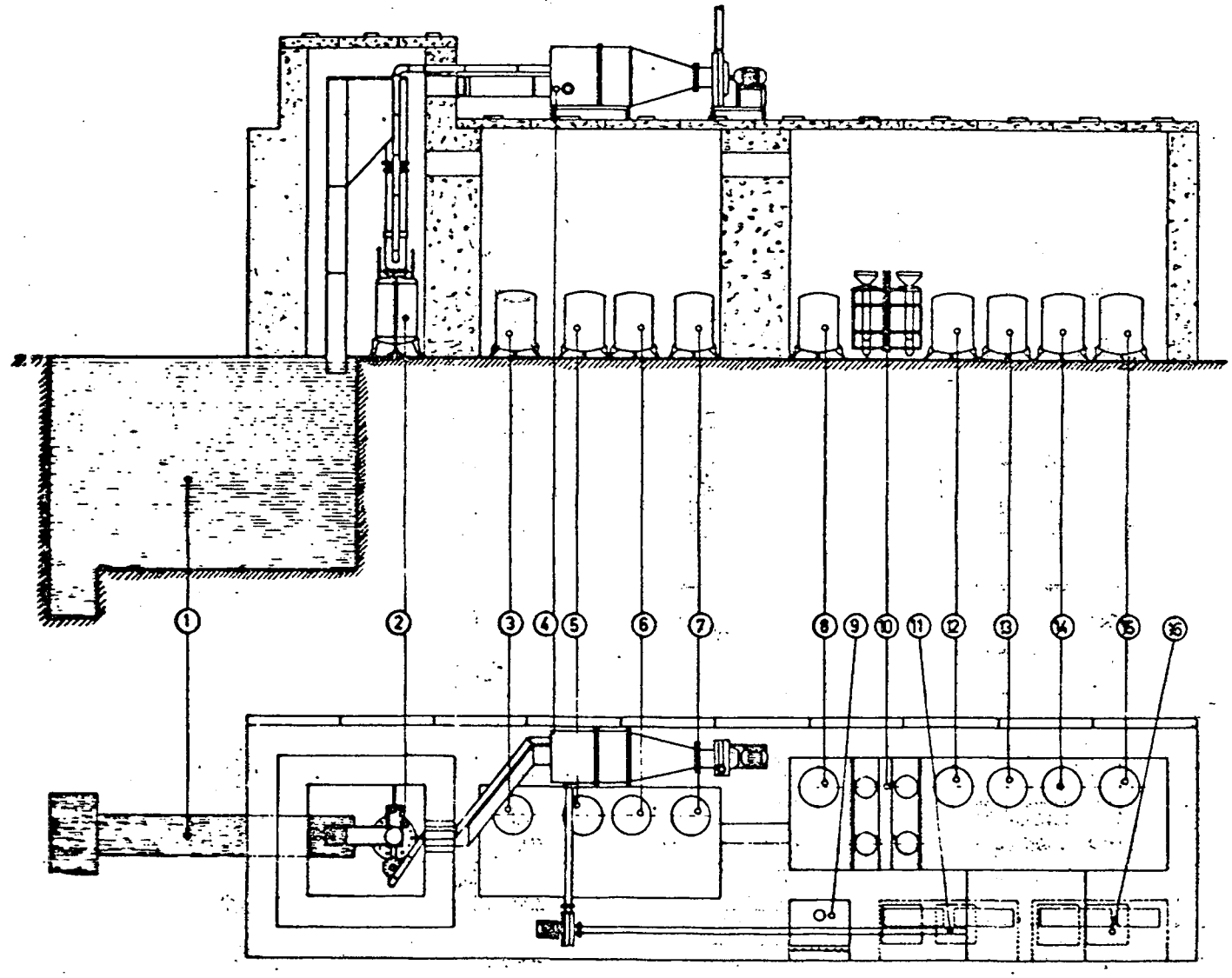
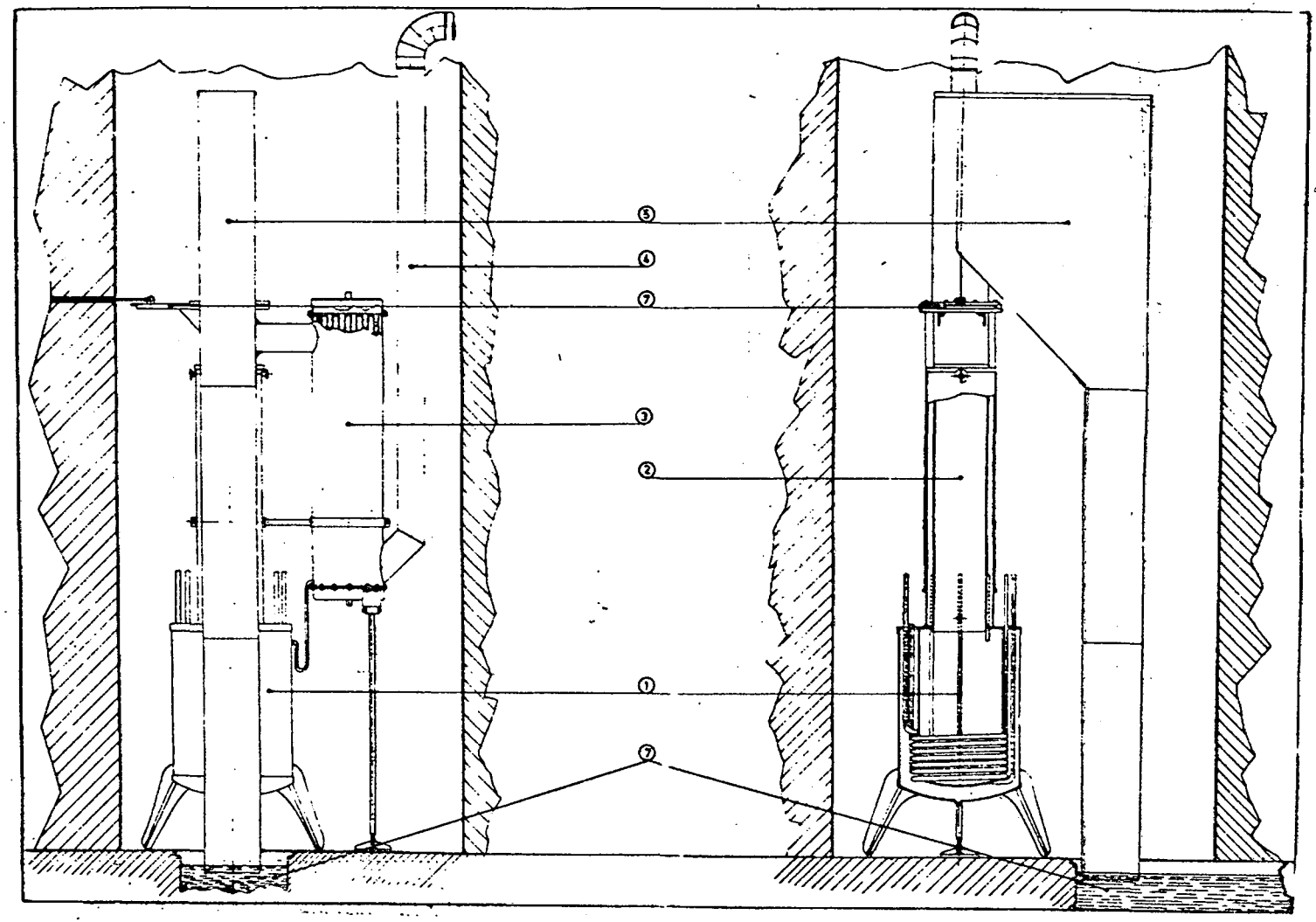


FIGURE 5

DISSOLVER CELL

1. Dissolver
2. Jacket condenser
3. Shell and tube oondenser
4. Exhaust gases duct
5. Charging chute
6. Sliding lid
7. Storage pool



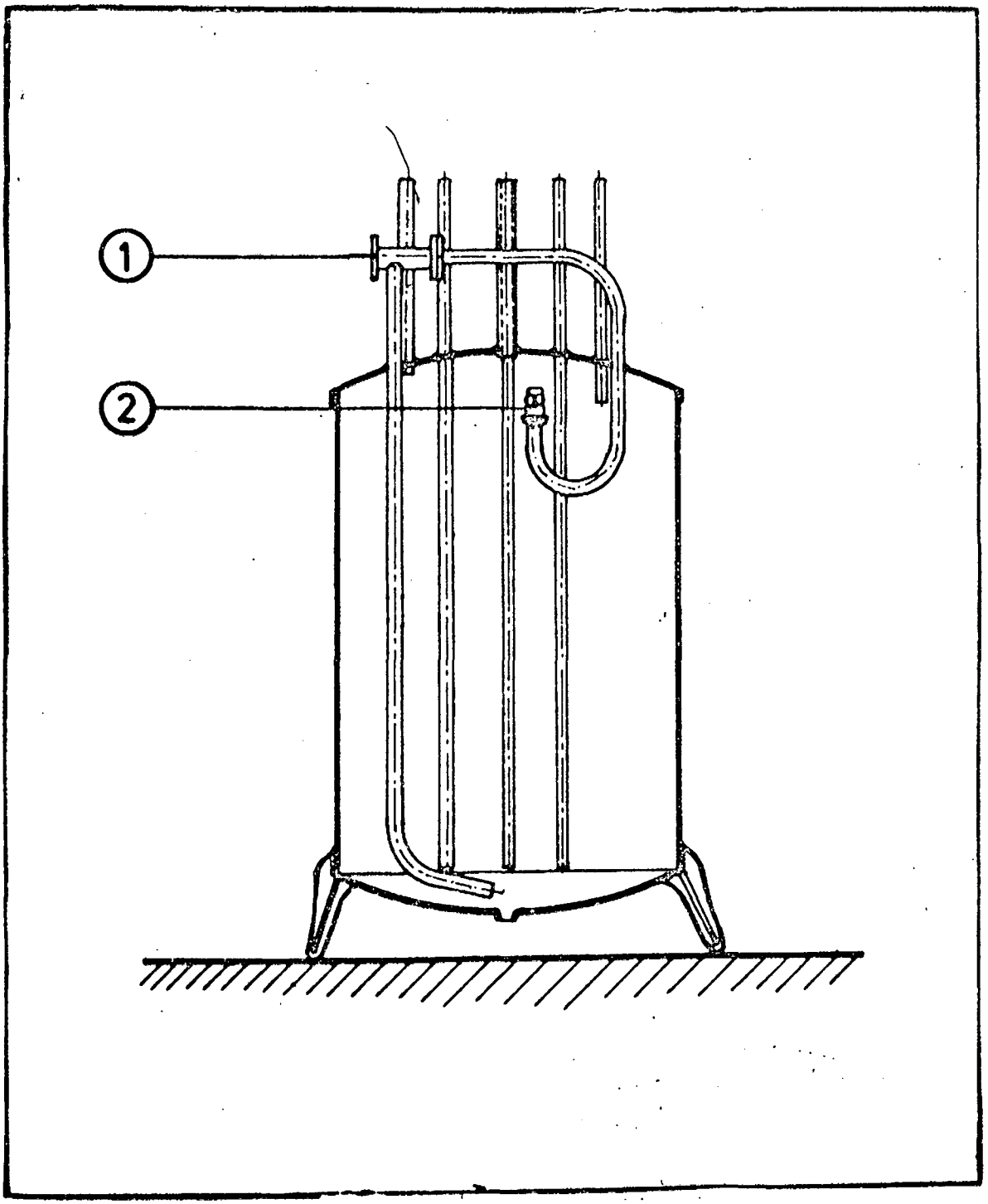


FIGURE 6

STORAGE TANK

1. Steam jet

2. Spray nozzle

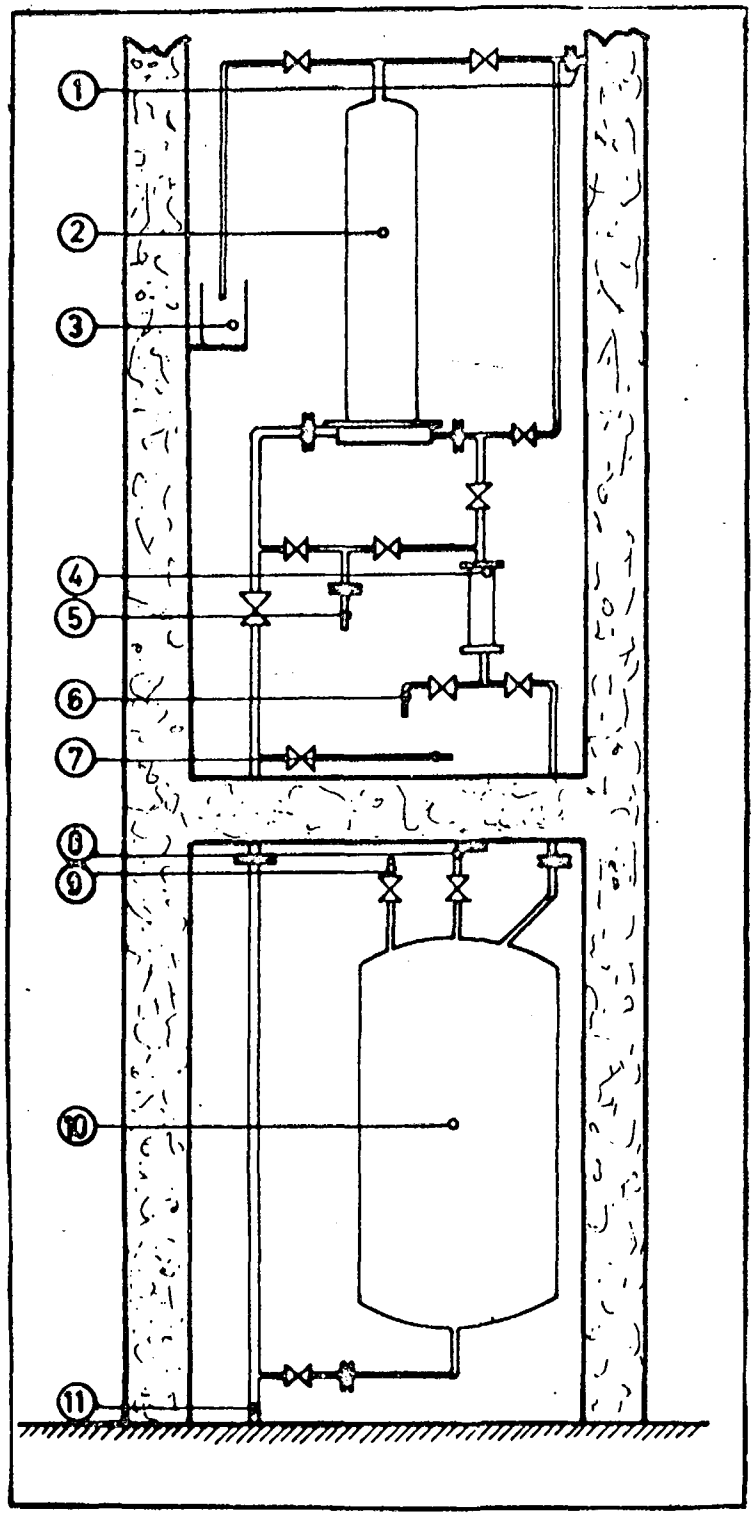


FIGURE 7
FILTER CIRCUIT

1. Wash water line
2. Filter
3. Filter-aid make up
4. Observation glass
5. Inlet from dissolver storage tank
6. Filtered solution outlet
7. From 1st cycle waste tank
8. Vacuum line connection
9. Vent
10. Filter auxiliary tank
11. To waste disposal intermediate tank

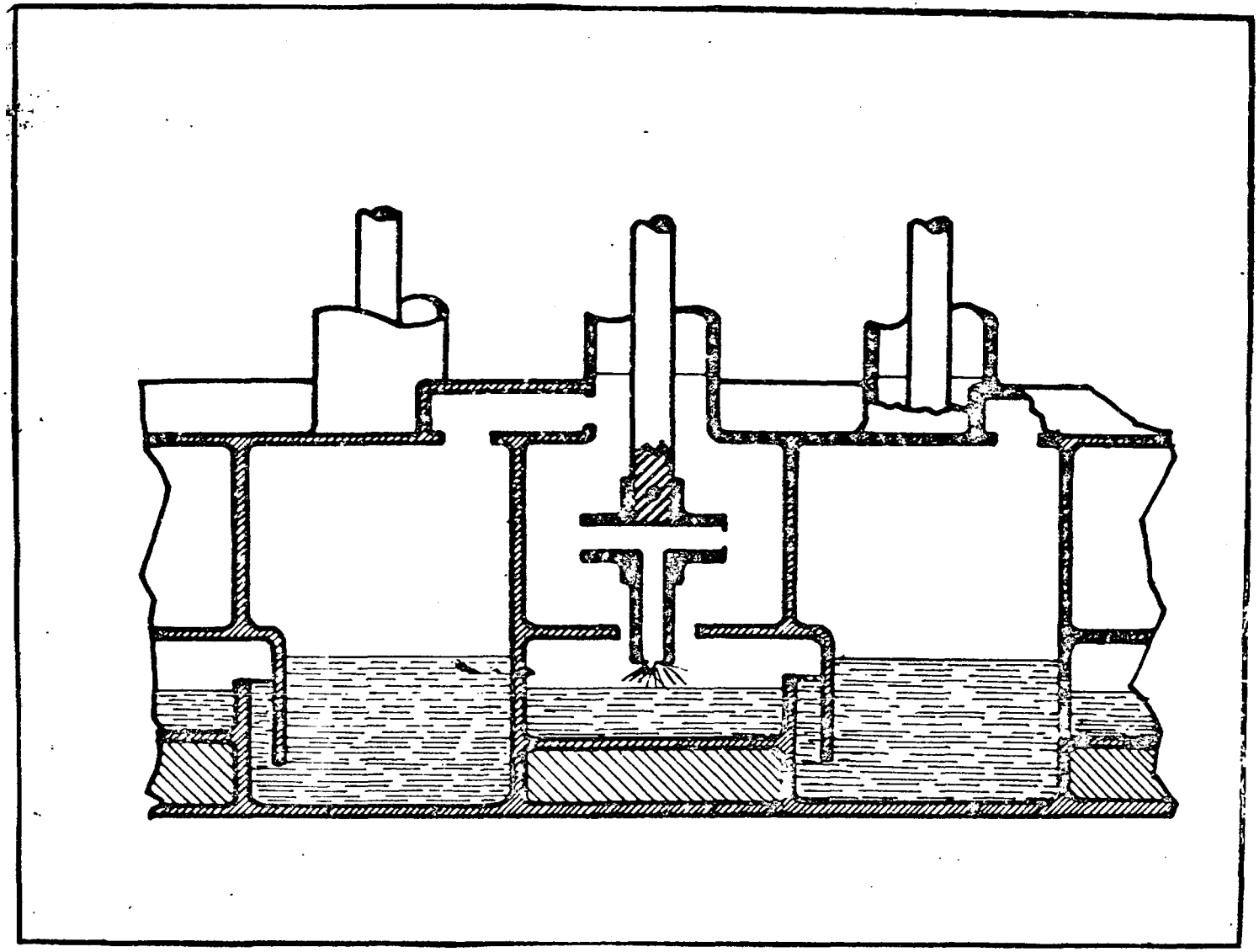


FIGURE 8

PUMP MIX MIXER SETTLER

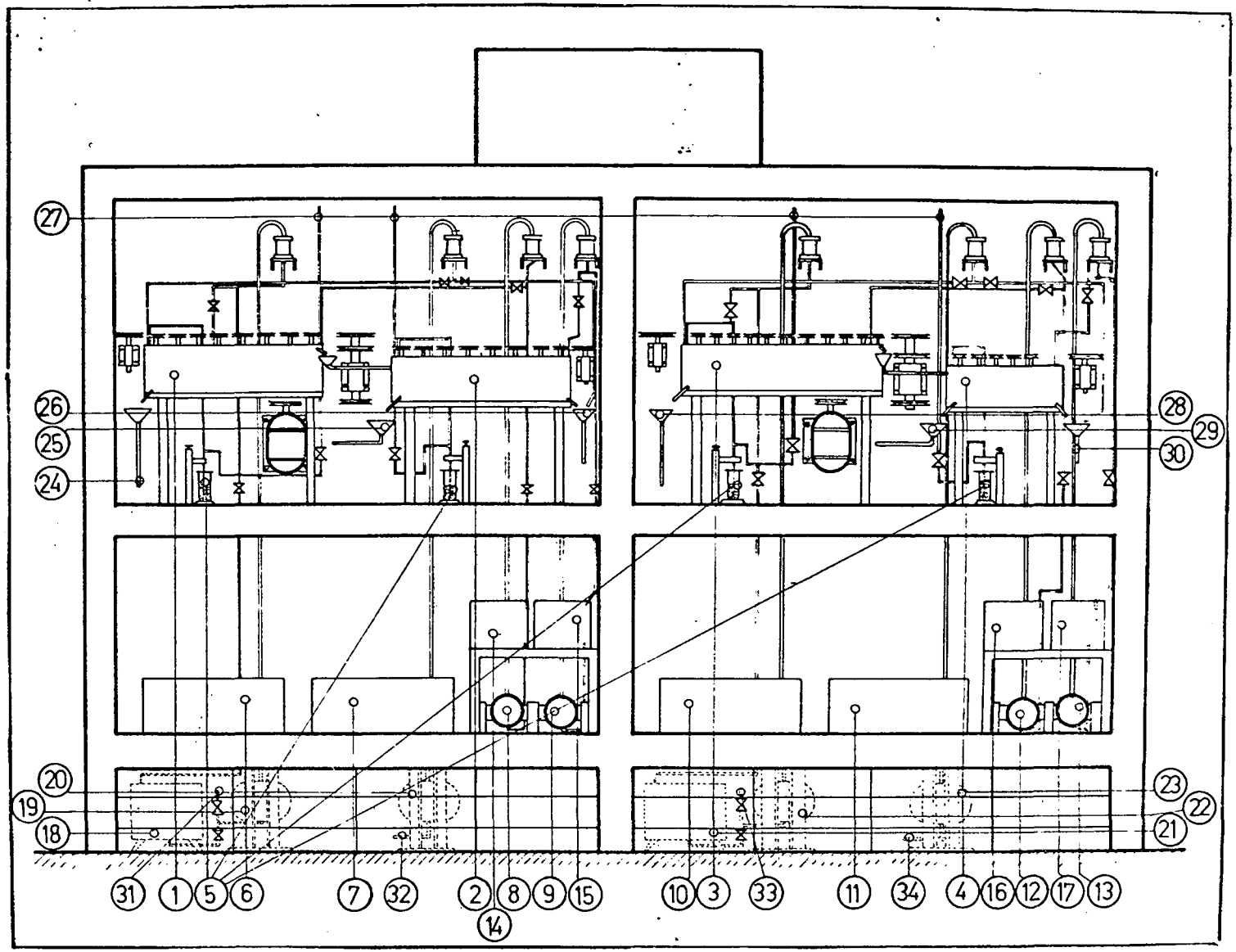


FIGURE 9

SOLVENT EXTRACTION EQUIPMENT

LEGENDS TO FIGURE 9

SOLVENT EXTRACTION EQUIPMENT

1. 1st cycle extraction and scrubbing mixer-settler
2. 1st cycle stripping mixer-settler
3. 2nd cycle extraction and scrubbing mixer-settler
4. 2nd cycle stripping mixer-settler
5. Air pressure regulators for mixer-settler heavy phase outlet control
6. 1st cycle feed solution pump
7. 1st cycle solvent pump
8. 1st cycle scrubbing solution pump
9. 1st cycle stripping solution pump
10. 2nd cycle feed solution pump
11. 2nd cycle solvent pump
12. 2nd cycle scrubbing solution pump
13. 2nd cycle stripping solution pump
14. 1st cycle scrubbing solution tank
15. 1st cycle stripping solution tank
16. 2nd cycle scrubbing solution tank
17. 2nd cycle stripping solution tank
18. 1st cycle feed solution pump recycling tank
19. 1st cycle feed solution pump remote head
20. 1st cycle solvent pump remote head
21. 2nd cycle feed solution pump recycling tank
22. 2nd cycle feed solution remote head
23. 2nd cycle solvent pump remote head
24. To first cycle waste tank
25. To first cycle product tank
26. To first cycle solvent washer
27. To pressurized air line
28. To 2nd cycle waste tank
29. To 2nd cycle product tank
30. To 2nd cycle solvent washer

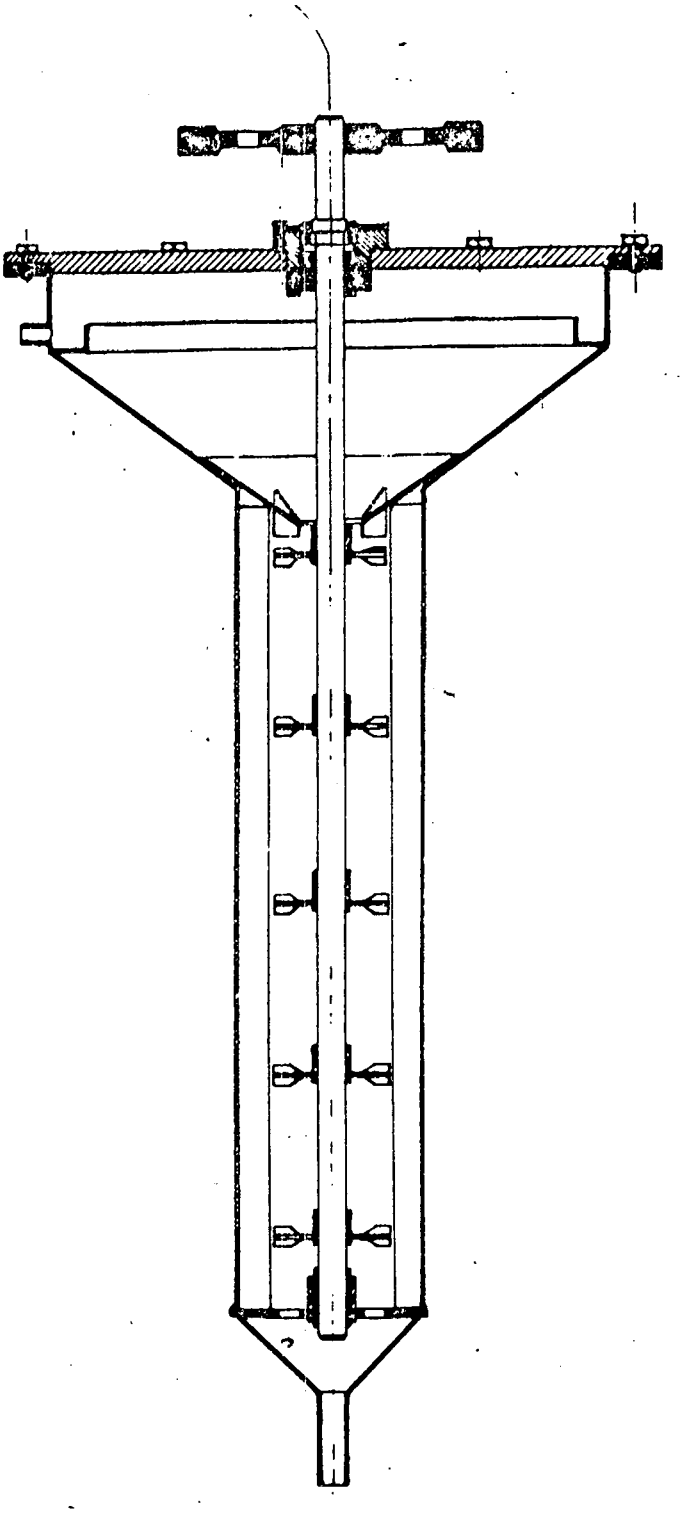


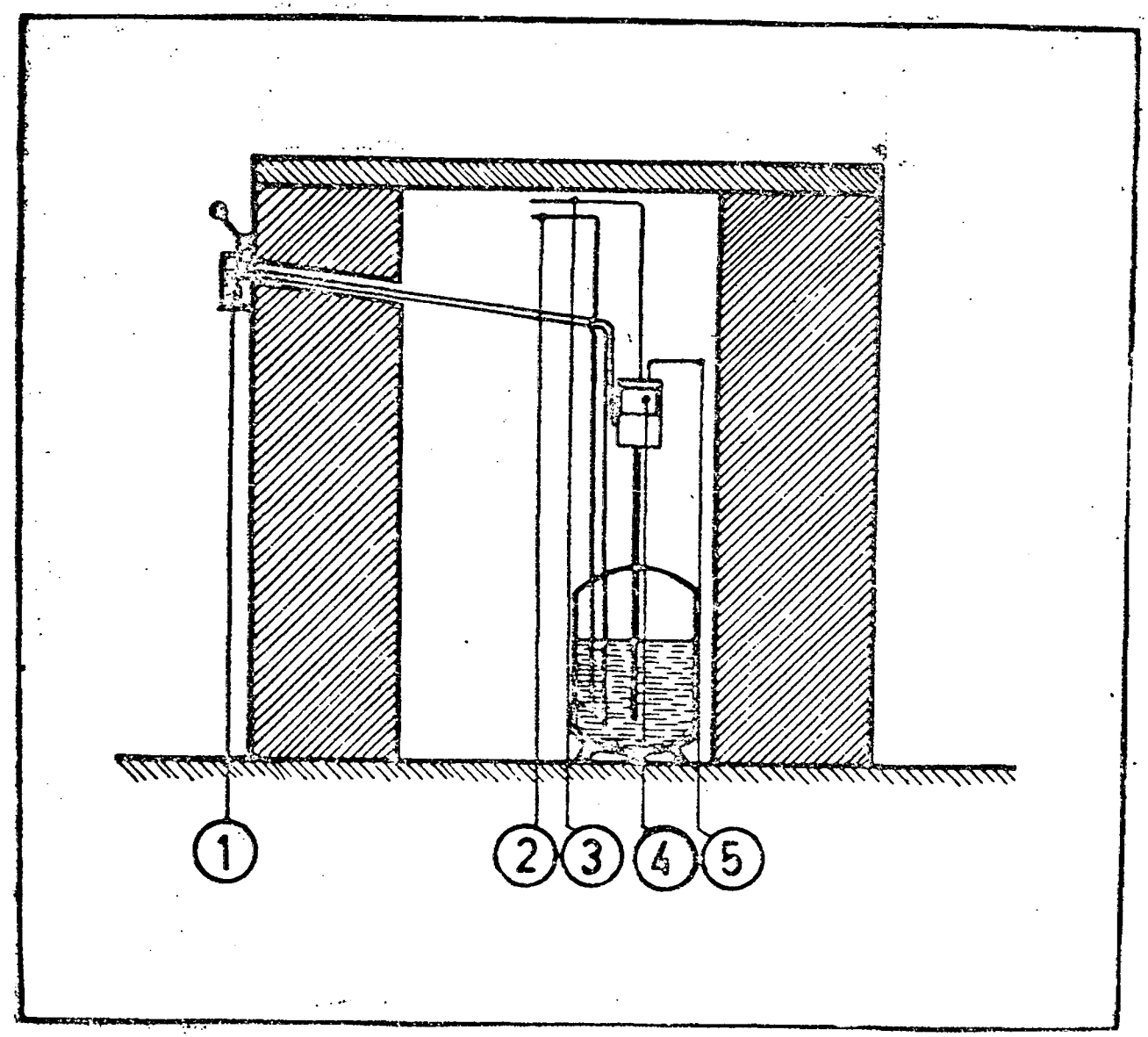
FIGURE 10

SOLVENT WASHER

FIGURE 11

SAMPLER CONNECTIONS

1. Sampler bottle
2. Pressurized air line
3. Vacuum line
4. Security float
5. Security vent



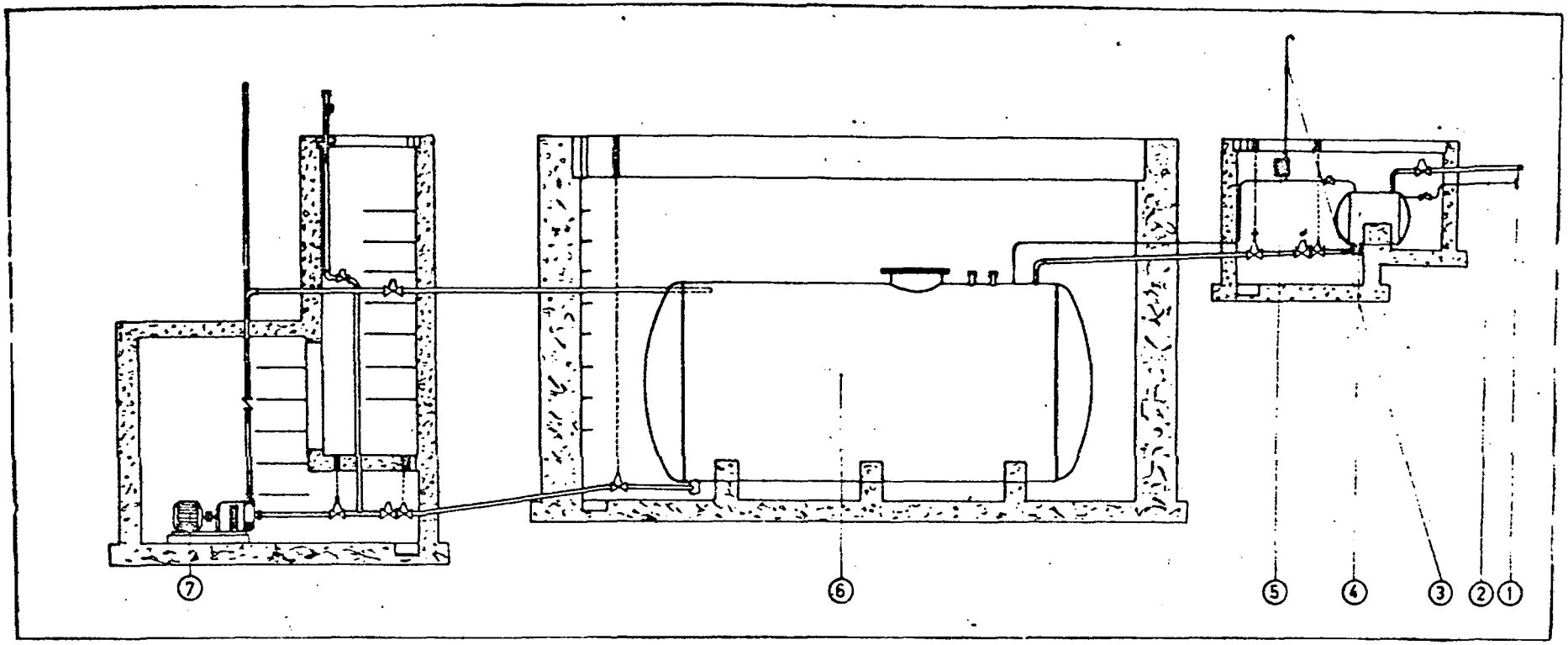


FIGURE 12 WASTE STORAGE FACILITY

1. Waste solution line from plant
2. Vacuum line
3. Vent
4. Waste solution intermediate tank
5. Absolute air filter
6. Waste solution decay tank
7. Centrifugal pump

FIGURE 13

PLANT BUILDING VENTILATION SYSTEM

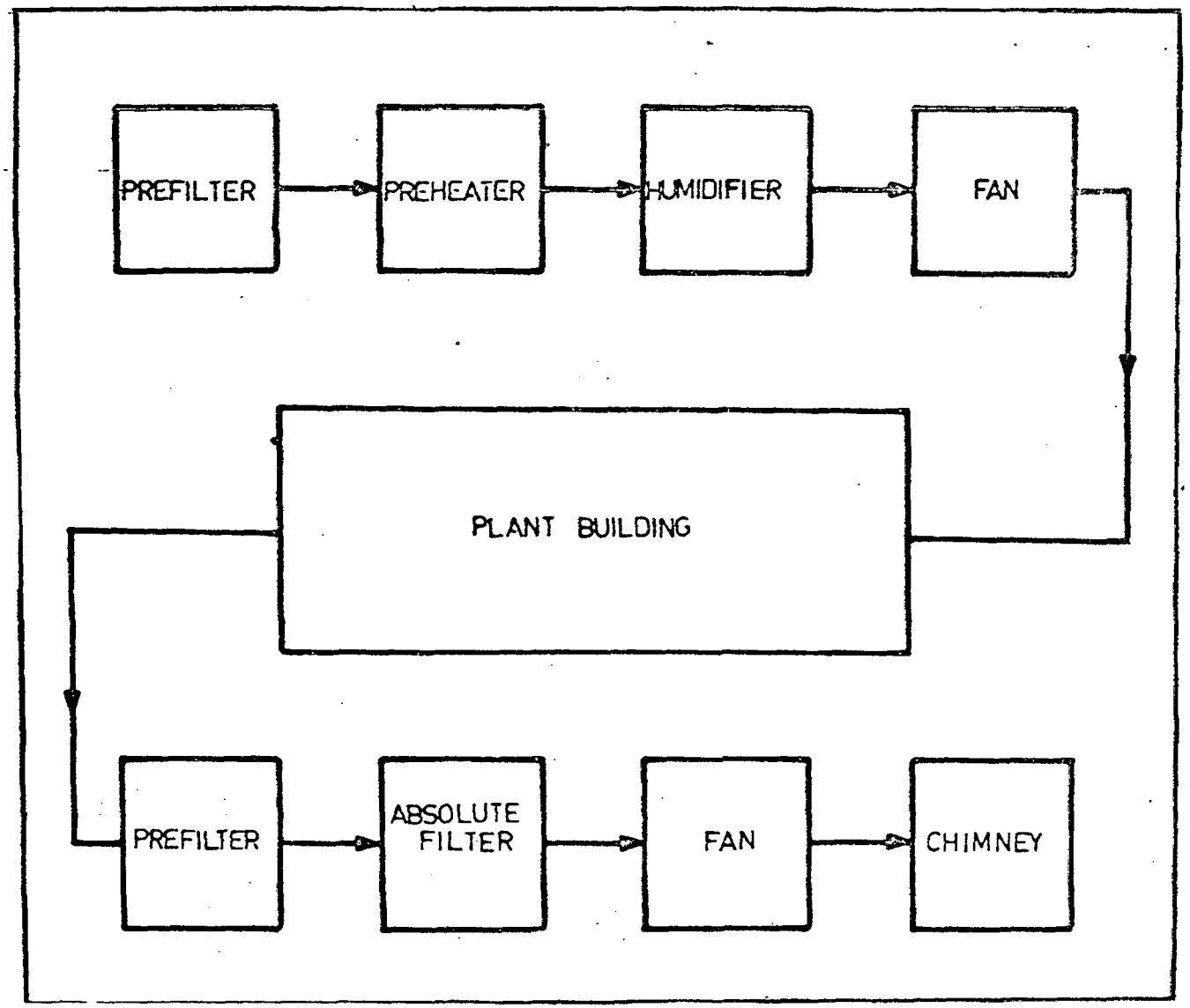


FIGURE 14

PLANT HOT CELL AND HOOD

VENTILATION SYSTEM

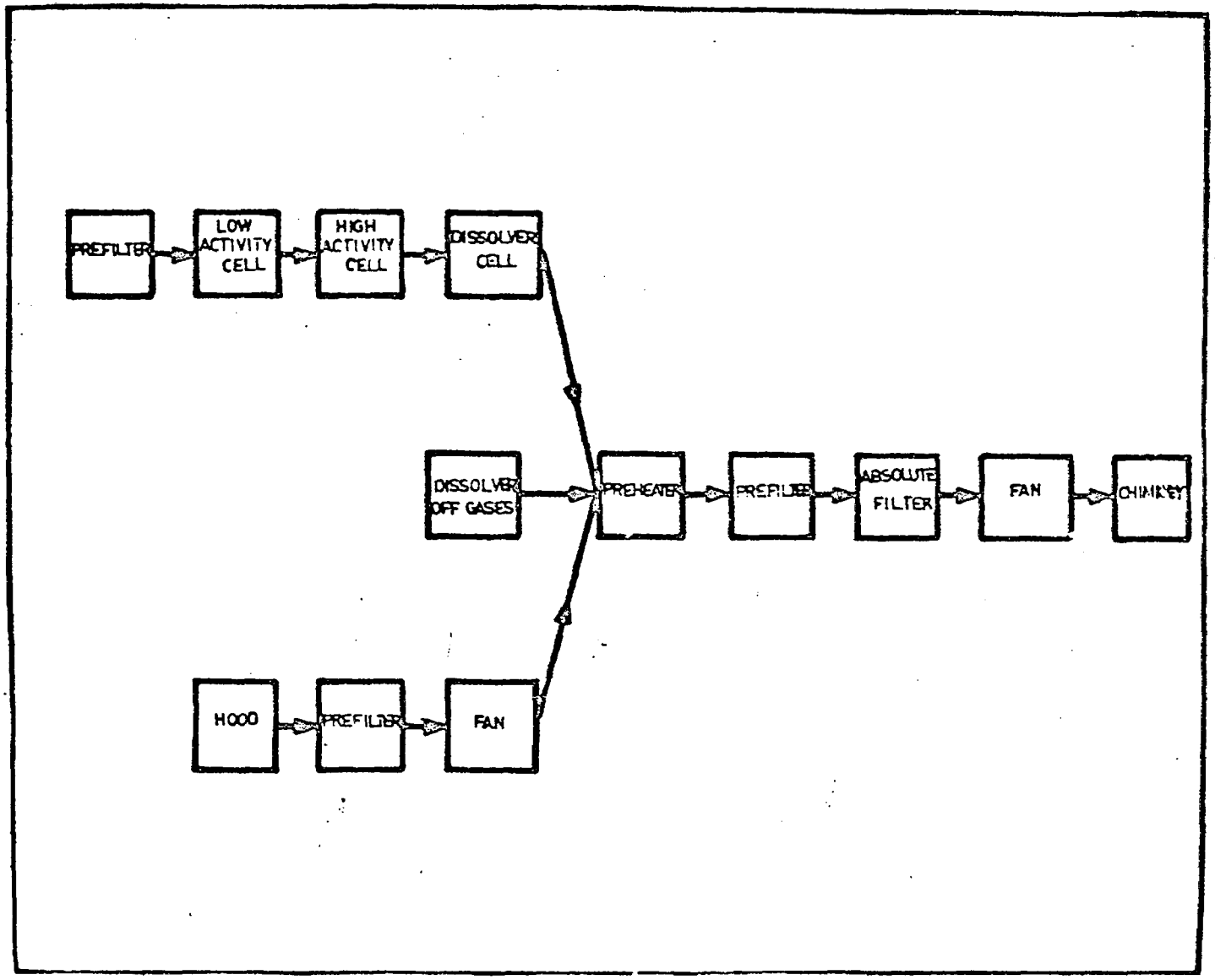


FIGURE 15

TANK CONTROL BOARD CONNECTIONS

1. Ventilation line
2. Feeding line to tank
3. Feeding funnel
4. Vacuum line
5. Vacuum gauge
6. Air line
7. Ventilation or vacuum line to tank
8. Air agitation line to tank
9. and 11. Connection to density probes in tank
10. Density indicator
12. and 14. Connections to level probes in tank
13. Level indicator

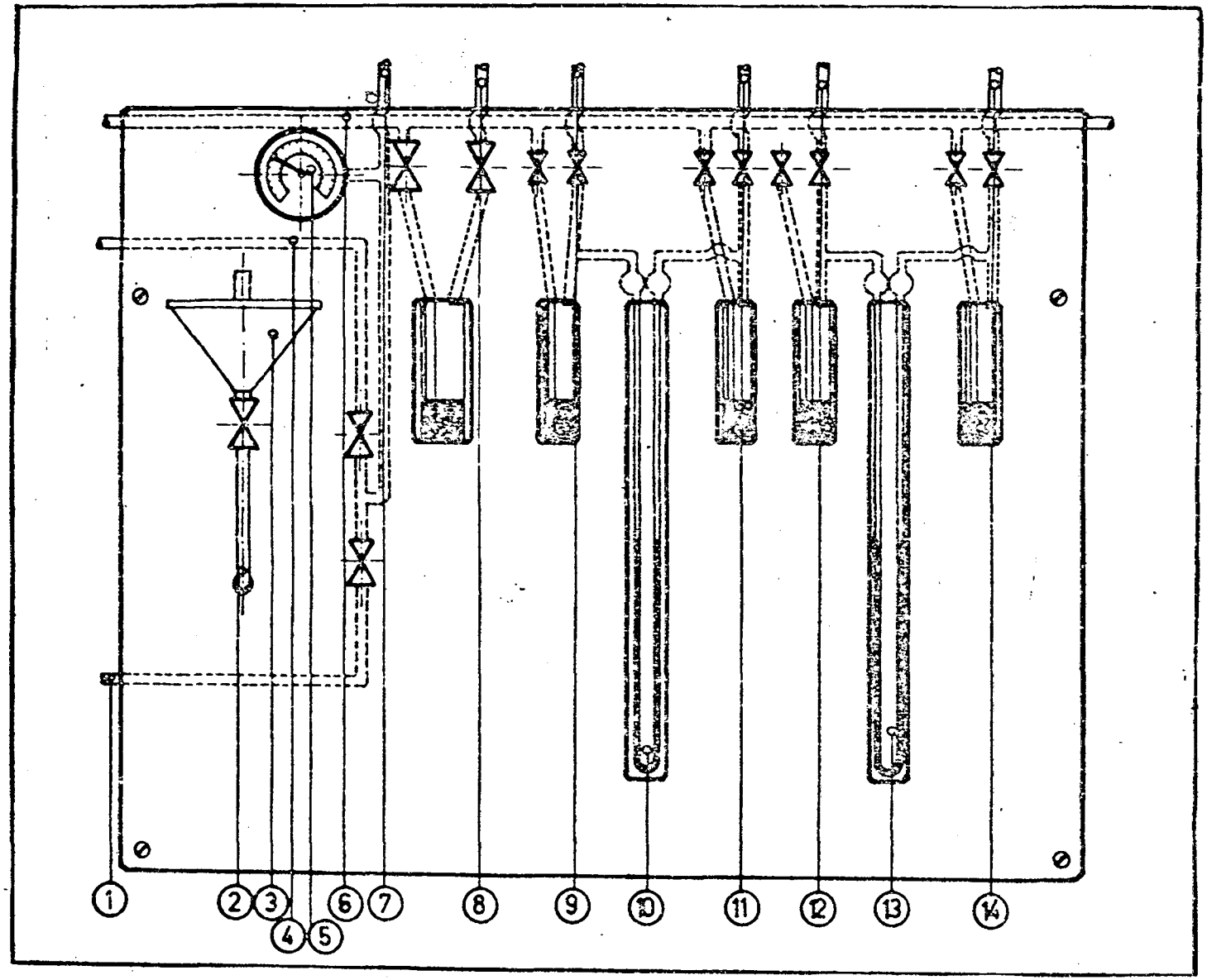
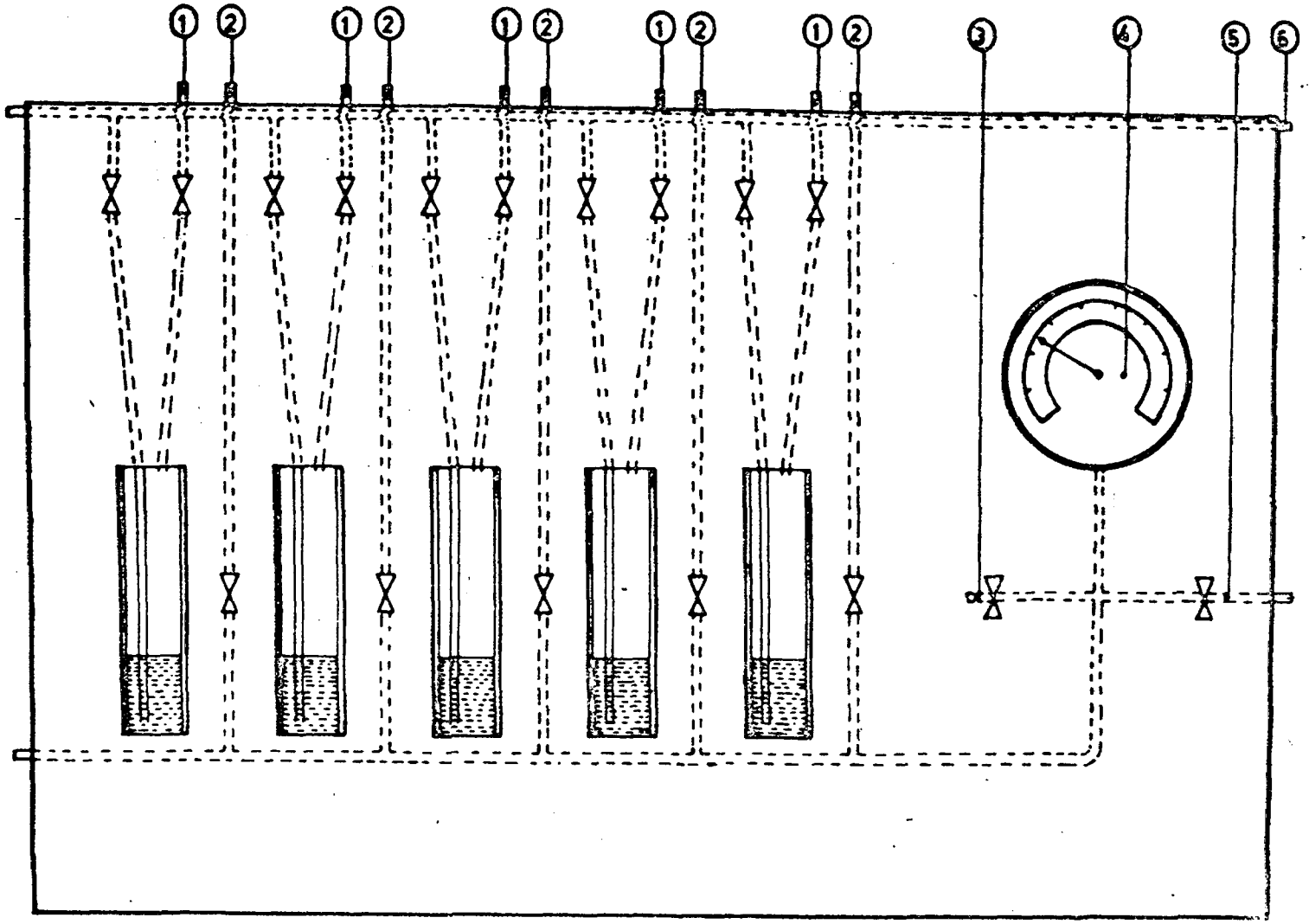


FIGURE 16
SAMPLER CONTROL BOARD
CONNECTIONS

1. Sampler air lift connection
2. Sampler vacuum connection
3. Vent
4. Vacuum gauge
5. Vacuum line
6. Air line



COMISION NACIONAL DE ENERGIA ATOMICA

Buenos Aires

ARGENTINA

May 1967

PILOT PLANT FOR THE REPROCESSING OF IRRADIATED NUCLEAR FUELS

OPERATION MANUAL

TABLE OF CONTENTS

LIST OF FIGURES

DISSOLVER CHARGING

1) Control performance of dissolver control board (see Control Board Operation Instructions)
2) Measure volume (see Control Board Operation Instructions)
3) If volume is more than 1.0 litre suspend operation
4) Measure activity indicated by dissolver monitor
5) Control that fuel element charging lid is closed
6) Verify depression of 15 mm water column in dissolver
7) If depression is not 15 mm, adjust register in plant ventilation system
8) Add 13.5 litre of demineralized water (see Control Board Operation Instructions)
9) Measure volume
10) Open dissolver cell lid
11) Open dissolver charging lid
12) Place crane with attached tong in position for submergence in storage pool
13) Lower tong into storage pool
14) Fasten one fuel element to tong
15) Lift tong and place in position for charge in dissolver
16) Open tong
17) Measure raise in activity by reading dissolver monitor
18) Register annotation in fuel element control book
19) Repeat operations 12 to 18 until there are 3 fuel elements in the dissolver
20) Close dissolver charging lid
21) Control depression of 15 mm water column in dissolver
22) Take out tong and replace in his site
23) Close dissolver cell lid

FUEL ELEMENT DISSOLUTION

1) Control performance of dissolver control board
2) Measure volume
3) Measure activity
4) Control depression of 15 mm water column in dissolver
5) Open cooling water inlet valve to jacket condenser
6) Purge air in jacket condenser
7) Open cooling water inlet valve to shell and tube condenser
8) Purge air in shell and tube condenser
9) Open cooling water outlet valve from jacket condenser
10) Open cooling water outlet valve from shell and tube condenser
11) Connect temperature indicators
12) Connect air filter preheaters
13) Control minimum temperature difference of 10 °C between inlet and outlet of filter
14) Fill feed tank with commercial 14 N HNO_3 acid
15) Open steam inlet valve
16) Open slowly and regulate coil condensate outlet valve
17) Shut steam inlet valve when thermocouple indicator reads 100 °C or temperature at outlet of first condenser is higher than 50 °C
18) Start air bubbling
19) If temperature at outlet of first condenser (indicator N° 1) is above 50 °C, wait until it reaches this temperature
20) Add 2 litre HNO_3 from feed tank
21) Add 170 ml $Hg(NO_3)_2$ 1 M
22) Observe temperature raise at outlet of first condenser and second condenser. Temperature at outlet of first condenser should not pass 80 °C and at outlet of second condenser should not pass 30 °C
23) If temperature goes above these values open cooling water inlet valve to dissolver coil until outlet temperature of first condenser descends below 80 °C
24) If there is no temperature raise and thermocouple indicates less than 100 °C, or temperature at outlet of first condenser is below 50 °C, repeat operations 15, 16 and 17
25) Wait until temperature of outlet of first condenser descends to 50 °C

26) Repeat operation 19 to 25 four times to add 10 litre HNO_3 total
27) Repeat operation 19 to 25 but adding 4 litre HNO_3
28) Repeat operation 19 to 25 but adding 7 litre HNO_3
29) Stop air bubbling
30) Open steam inlet valve
31) Regulate coil condensate outlet valve to keep slow boiling. Temperature at outlet of first condenser should not pass 60 °C
32) Keep boiling for one hour
33) Measure volume
34) If volume is less than 34.2 litre add demineralized water to make up to this volume
35) Take out sample (see Sampler Operation Instructions)
36) Send sample to laboratory for free acid and U analysis
37) If free acid is less than 1 N add concentrated HNO_3 to make up to this normality
38) Keep boiling for two hours
39) Repeat operations 33 to 37
40) Keep boiling for two hours
41) Repeat operations 33 to 36
42) If free acid is less than 0.5 N add concentrated HNO_3 to make up to 1 N and return to point 40
43) If free acid is more than 0.5 N close steam inlet valve
44) Open cooling water inlet valve to dissolver coil
45) When thermocouple indicates 30 °C close cooling water inlet valve to dissolver coil
46) Close shell and tube condenser outlet valve
47) Close shell and tube condenser inlet valve
48) Close jacket condenser outlet valve
49) Close jacket condenser inlet valve
50) Disconnect air filter preheater
51) Take out sample
52) Send sample to laboratory for free acid, U, Al, and total gamma determination
53) Measure density
54) Measure volume
55) Measure activity
56) Calculate total mass of U and Al

FILTRATION

1) Control performance of storage tank control board
2) Measure density in storage tank
3) Measure volume in storage tank
4) Measure activity in storage tank
5) Control performance of filtered solution tank control board
6) Measure density in filtered solution tank
7) Measure volume in filtered solution tank
8) Measure activity in filtered solution tank
9) If sum of volumes of both tanks is more than 50 litre, do not filter
10) Verify if all valves corresponding to filter equipment are closed (valves 1 - 13)
11) Verify if filter auxiliary waste tank is empty
12) Start air bubbling in density meter of filtered solution tank (open slowly valves 6 and 8 of control board)
13) Start air bubbling in volume meter of filtered solution tank (open slowly valves 10 and 12 of control board)
14) Start mixing in filtered solution tank (open slowly valve 4 of control board)
15) Start air bubbling in filtered solution tank sampler (open slowly valve A of sampler control board)
16) Open valve N° 1 on filter
17) Open valve N° 3 until level in filter aid preparation tank reaches red mark
18) Close valve N° 3 on filter
19) Close valve N° 1 on filter
20) Start mixing of filter aid preparation tank
21) Add 30 g Hyflow Supercel in filter aid preparation tank
22) Start vacuum pump
23) Open valve N° 11 on filter
24) Open valve N° 9 on filter
25) Open valve N° 2 on filtered solution control board
26) Close valve N° 3 on filtered solution control board
27) Open valve N° 1 on filter
28) Open valve N° 4 on filter
29) When water passage through observation glass stops, open valve N° 8 and close valve N° 9
30) Stop mixing in filter aid preparation tank

31) Open valve N° 5 on filter
32) Close valve N° 1 on filter
33) Write down filtration starting time
34) Close valve N° 11 on filter
35) Open valve N° 12 on filter
36) Control progress of filtration through observation glass and volume indicator of filtered solution tank control board
37) Write down time of end of filtration when solution flow through observation glass stops
38) Measure density in filtered solution tank
39) Measure volume in filtered soltion tank
40) Measure activity in filtered solution tank
41) Calculate difference between initial volume in storage tank and volume increase in filtered solution tank
42) If difference is less than 1 litre add 2.5 litre HNO_3 1 N to storage tank
43) If difference is more than 1 litre wait 15 minutes and return to point 38
44) Repeat point 36 to 40
45) Calculate difference between initial volume in storage tank plus 2.5 litre and volume increase in filtered solution tank
46) If difference is less than 1 litre add 2.5 litre HNO_3 1 N to storage tank
47) If difference is more than 1 litre wait 15 minutes, repeat point 38, 39 and 40, and return to point 45
48) Repeat points 36 to 40
49) Calculate difference between initial volume in storage tank plus 5 litre and volume increase in filtered solution tank
50) If difference is less than 1 litre open valve N° 1 and wait 15 minutes to dry filter cake
51) If difference is more than 1 litre wait 15 minutes and repeat points 38, 39 and 40, and return to point 49
52) Open slowly valve N° 3 on filtered solution tank control board
53) Close slowly valve N° 2 on filtered solution tank control board
54) Stop air bubbling in density meter of filtered solution tank (close valves 6 and 8 of control board)

55) Stop air bubbling in volume meter of filtered solution tank (close valves 10 and 12 of control board)
56) Stop mixing in filtered solution tank (close valve 4 of control board)
57) Stop air bubbling in filtered solution tank sampler (close valve A of sampler control board)
58) Close valve N° 4 on filter
59) Close valve N° 8 on filter
60) Close valve N° 5 on filter
61) Close valve N° 1 on filter
62) Verify if all valves on waste disposal control board are closed
63) Open valve N° 2 on waste disposal control board
64) Open valve N° 1 on waste disposal control board
65) Open valve N° 7 on filter
66) Open valve N° 3 on filter for 1 minute
67) Close valve N° 3 on filter
68) Open valve N° 2 on filter for 1 minute
69) Close valve N° 2 on filter
70) Open valve N° 4 on filter
71) Open valve N° 9 on filter for 1 minute
72) Close valve N° 9 on filter
73) Close valve N° 4 on filter
74) Close valve N° 7 on filter
75) Open valve N° 13 on filter
76) When filter auxiliary waste tank is empty close valve N° 13 on filter
77) Close valve N° 2 on waste disposal control board
78) Close valve N° 1 on waste disposal control board
79) Close valve N° 12 on filter
80) Open valve N° 3 on waste disposal control board
81) Open valve N° 4 on waste disposal control board
82) Wait 15 minutes
83) Close valve N° 4 on waste disposal control board
84) Close valve N° 3 on waste disposal control board

PROCEDURE TO FOLLOW WHEN FILTER CLOGGS BETWEEN OPERATIONS 36 AND 52

1) Open slowly valve N° 1 on filter
2) Wait 15 minutes
3) Close valve N° 5 on filter
4) Measure density in filtered solution tank
5) Measure volume in filtered solution tank
6) Measure activity in filtered solution tank
7) Measure density in storage tank
8) Measure volume in storage tank
9) Measure activity in storage tank
10) Add 1 litre HNO_3 1 N to filter aid preparation tank
11) Control rate of passage through observation glass
12) Wait until all of the solution is through, or at least one hour
13) Open valve N° 5 on filter
14) Wait 5 minutes
15) Repeat operations 3 to 14
16) Continue with point 52 of filtration instructions

FIRST CYCLE FEED ADJUSTMENT

1) Control performance of feed solution control board
2) Mix solution in tank for 5 minutes
3) Take out sample
4) Send sample to laboratory for U, Al and free acid determination
5) Measure density
6) Measure volume
7) Measure activity
8) Add HNO_3 14 N and demineralized water to adjust concentrations to 1.5 M $Al(NO_3)_3$ and 1 N HNO_3
9) Mix solution in tank for 5 minutes
10) Take out sample
11) Send sample to laboratory for U, Al and free acid determination
12) Measure density
13) Measure volume
14) Measure activity
15) If results of analysis are between 1.4 - 1.6 M $Al(NO_3)_3$ and 0.95 - 1.05 N HNO_3 stop adjustment
16) If results of analysis are different from indicated in point 15 return to point 8

FEED SOLUTION FLOW RATE ADJUSTMENT

1) Verify if valve N° 1 is closed
2) Verify if valve N° 6 is open
3) Verify if valve N° 10 is closed
4) Verify if valve N° 9 is open
5) Verify if level in feed pump recycling tank reaches red mark
6) If level in feed pump recycling tank is below red mark, fill up with scrub solution:
   1.5 M $Al(NO_3)_3$ + 1 N HNO_3 for first cycle pump
   3 N HNO_3 for second cycle pump
7) Calculate flow rate according to formulae:
   flow rate in litre/h = 23.5/U concentration in g/litre in feed solution for first cycle
   flow rate in litre/h = 60/U concentration in g/litre in feed solution for second cycle
8) Read on pump calibration chart the plunger amplitude indicator value corresponding to feed solution flow rate
9) Set plunger amplitude regulator to corresponding value
10) Start pumping
11) Wait 3 minutes
12) Close valve N° 6 and start chronometer timing
13) When level in calibration tube reaches mark, stop chronometer and open valve N° 6
14) Write down measured time
15) Repeat operations 12, 13 and 14 twice
16) Calculate average time
17) Calculate measured flow rate according to calibration chart
18) If difference between measured flow rate and calculated flow rate is more than 5%, prime pump according to fabricators instructions
19) If difference between measured flow rate and calculated flow rate is between 2 and 5%, adjust plunger amplitude regulator accordingly and return to point 12
20) If difference between measured flow rate and calculated flow rate is less than 2%, stop pumping

SOLVENT FLOW RATE ADJUSTMENT

1) Verify if valve N° 2 is closed
2) Verify if valve N° 3 is open
3) Set plunger amplitude regulator to value 14, corresponding to a flow rate of 1.5 litre/h
4) Start solvent washer N° 1
5) Start solvent washer N° 2
6) Start pumping
7) Wait 3 minutes
8) Close valve 3 and start chronometer timing
9) When level in calibration tube reaches mark, stop chronometer and open valve N° 3
10) Write down measured time
11) Repeat operations 8, 9 and 10 twice
12) Calculate average time
13) Calculate measured flow rate according to calibration chart
14) If measured flow rate is below 1.4 litre/h, prime pump according to fabricators instructions
15) If measured flow rate is between 1.40 and 1.47 litre/h or above 1.53 litre/h, adjust plunger amplitude regulator accordingly and return to point 8
16) If measured flow rate is between 1.47 and 1.53 litre/h, stop pumping

SCRUBBING AND STRIPPING FLOW RATE ADJUSTMENTS

1) Verify if corresponding recycle valve is open:
   scrub solution, valve N° 4
   strip solution, valve N° 5
2) Verify if corresponding mixer-settler feed valve is closed:
   scrub solution, valve N° 7
   strip solution, valve N° 8
3) Verify if level of corresponding tank reaches red mark
4) If level of solution does not reach red mark fill up with corresponding solution:
   first cycle scrub, 1.5 M $Al(NO_3)_3$ + 1 N HNO_3
   first cycle strip, 0.01 N HNO_3
   second cycle scrub, 3 N HNO_3
   second cycle strip, 0.01 N HNO_3
5) Set flow rate adjustment screw to the following values:
   first cycle scrub, 0.5 litre/h
   first cycle strip, 0.375 litre/h
   second cycle scrub, 0.5 litre/h
   second cycle strip. 1.5 litre/h
6) Start pumping
7) Wait 3 minutes
8) Close corresponding recycling valve and start chronometer timing
9) When level in corresponding calibration tube reaches mark, stop chronometer and open recycling valve
10) Write down measured time
11) Repeat operations 8, 9 and 10 twice
12) Calculate average time
13) Calculate measured flow rate according to corresponding calibration chart
14) If measured flow rate is different from following values, regulate adjustment screw accordingly and return to point 12:
   first cycle scrub, 0.48 - 0.52 litre/h
   first cycle strip, 0.35 - 0.40 litre/h
   second cycle scrub, 0.48 - 0.52 litre/h
   second cycle strip, 1.45 - 1.55 litre/h
15) If measured flow rate is between the preceding values, stop pumping

FIRST CYCLE SOLVENT EXTRACTION

1) Control performance of feed adjustment tank control board
2) Measure density in feed adjustment tank
3) Measure volume in feed adjustment tank
4) Measure activity in feed adjustment tank
5) Take out sample from feed adjustment tank
6) Send sample to laboratory for U, Pu, Al, free acid and total gamma determination
7) Control performance of first cycle solvent tank control board
8) Measure density in first cycle solvent tank
9) Measure volume in first cycle solvent tank
10) Measure activity in first cycle solvent tank
11) Take out sample from first cycle solvent tank
12) Send sample to laboratory for TBP, U, Pu and total gamma determination
13) Control performance of first cycle product tank control board
14) Measure density in first cycle product tank
15) Measure volume in first cycle product tank
16) Measure activity in first cycle product tank
17) Take out sample from first cycle product tank
18) Send sample to laboratory for U, Pu and total gamma determination
19) If volume in first cycle product tank + $\frac{1}{3}$ of volume in feed adjustment tank is more than 50 litre, suspend operation
20) If total amount of U in first cycle product tank and feed adjustment tank is more than 1000 g, suspend operation
21) Control performance of first cycle waste tank control board
22) Measure density in first cycle waste tank
23) Measure volume in first cycle waste tank
24) Measure activity in first cycle waste tank
25) Take out sample from first cycle waste tank
26) Send sample to laboratory for Al, U, Pu and total gamma determination
27) If volume in first cycle waste tank is more than 1.5 litre, suspend operation
28) Adjust pumping rate of first cycle solvent pump to 1.5 litre/h (see instructions for solvent flow rate adjustment)

29) Adjust pumping rate of first cycle scrub pump to 0.5 litre/h (see instructions for scrubbing and stripping flow rate adjustments)
30) Adjust pumping rate of first cycle stripping pump to 0.375 litre/h (see instructions for scrubbing and stripping flow rate adjustments)
31) Adjust pumping rate of first cycle feed solution pump to 23.5/U concentration in g/litre in feed solution (see instructions for feed solution flow rate adjustment)
32) Control if level in feed pump recycling tank is above red mark
33) If level in feed pump recycling tank is below red mark, fill up with scrub solution 1.5 M $Al(NO_3)_3$ and 1 N HNO_3
34) Control if level in scrub solution storage tank is above red mark
35) If level in scrub solution storage tank is below red mark, fill up with scrub solution 1.5 M $Al(NO_3)_3$ and 1 N HNO_3
36) Control if level in stripping solution storage tank is above red mark
37) If level in stripping solution storage tank is below red mark, fill up with stripping solution 0.01 N HNO_3
38) Control if valves N° 1, 2, 4, 5 and 9 are closed
39) Control if valves N° 3, 6, 7, 8 and 10 are open
40) Control if heavy phase outlet of extraction mixer-settler is closed
41) Control if heavy phase outlet of stripping mixer-settler is closed
42) Control if extraction and stripping mixer-settlers are full
43) If mixer-settlers are full, pass points 44 to 63 and continue with point 64
44) Start first cycle solvent pump
45) Open valve N° 2
46) Close valve N° 3
47) Wait until solvent overflows stripping mixer-settler's outlet weir
48) Start first solvent washer
49) Start second solvent washer
50) Start mixer-settler agitation
51) Start scrubbing solution pump
52) Open valve N° 4
53) Close valve N° 7

54) Start stripping solution pump
55) Open valve N° 5
56) Close valve N° 8
57) When aqueous level in interphase level indicator of extraction mixer-settler is close to mark, connect air pressure regulator
58) Open heavy phase outlet of extraction mixer-settler
59) Start controlling interphase level of extraction mixer-settler with air regulator
60) When aqueous level in interphase level indicator of stripping mixer-settler is close to mark, connect air pressure regulator
61) Open heavy phase outlet of stripping mixer-settler
62) Start controlling interphase level of stripping mixer-settler with air regulator
63) Pass points 64 to 81 and continue with point 82
64) Start first solvent washer
65) Start second solvent washer
66) Start solvent pump
67) Open valve N° 2
68) Close valve N° 3
69) Connect extraction mixer-settler interphase level air regulator
70) Connect stripping mixer-settler interphase level air regulator
71) Start scrub solution pump
72) Start strip solution pump
73) Open valve N° 4
74) Open valve N° 5
75) Close valve N° 7
76) Close valve N° 8
77) Start mixer-settler agitation
78) Open heavy phase outlet of extraction mixer-settler
79) Open heavy phase outlet of stripping mixer-settler
80) Start controlling interphase level of extraction mixer-settler by air regulator
81) Start controlling interphase level of stripping mixer-settler by air regulator

PERFORM OPERATIONS 71 TO 81 AS FAST AS POSSIBLE

82) Start feed solution pump
83) Open valve N° 1

84) Close valve N° 6
85) Wait until both mixer-settlers reach steady state (interphase level keeps steady without change of air pressure in regulator)
86) Stop feed solution pump
87) Close valve N° 10
88) Open valve N° 9
89) Start feed solution pump

PERFORM OPERATIONS 86 TO 89 IN LESS THAN 10 SECONDS

90) Observe increase in volume in first cycle waste tank and first cycle product tank, and decrease in volume in feed, scrub and strip solution tanks. Write down density and volume in each tank at one hour intervals
91) At end of feed solution (stop of flow in feed solution observation glass) open valve N° 10
92) Close valve N° 9
93) Wait 15 minutes
94) Stop feed solution pump
95) Close valve N° 1
96) Open valve N° 6
97) Wait two hours
98) Open valves N° 3, 7 and 8
99) Close valves N° 2, 4 and 5
100) Close heavy phase outlet of extraction mixer-settler
101) Close heavy phase outlet of stripping mixer-settler
102) Stop mixer-settler stirrer motor
103) Stop first cycle solvent pump
104) Stop scrubbing solution pump
105) Stop stripping solution pump
106) Stop air supply to interphase regulators
107) Stop first solvent washer
108) Stop second solvent washer
109) Measure density in first cycle feed adjustment tank
110) Measure volume in first cycle feed adjustment tank
111) Measure activity in first cycle feed adjustment tank
112) Take out sample from first cycle feed adjustment tank
113) Send sample to laboratory for U, Al, Pu, free acid and total gamma determination
114) Measure density in first cycle solvent tank
115) Measure volume in first cycle solvent tank
116) Measure activity in first cycle solvent tank
117) Take out sample from first cycle solvent tank
118) Send sample to laboratory for TBP, U, Pu and total gamma determinations

119) Measure density in first cycle product tank
120) Measure volume in first cycle product tank
121) Measure activity in first cycle product tank
122) Take out sample from first cycle product tank
123) Send sample to laboratory for U, Pu and total gamma determination
124) Measure density in first cycle waste solution tank
125) Measure volume in first cycle waste solution tank
126) Measure activity in first cycle waste solution tank
127) Take out sample from first cycle waste solution tank
128) Send sample to laboratory for Al, U, Pu and total gamma determination
129) Calculate mass balances for U and Pu:

before extraction	after extraction
volume feed sol. x U concent. in feed sol.	
volume 1st cycle solvent x U conc. in 1st cycle solvent	
volume 1st cycle product x U conc. in 1st cycle product	
volume 1st cycle waste x U oonc. in 1st cycle waste	
etc.	

SECOND CYCLE FEED ADJUSTMENT

1) Control performance of first cycle product tank control board
2) Measure density
3) Measure volume
4) Measure activity
5) Take out sample
6) Send sample to laboratory for U, Pu, free acid and total gamma determination
7) If volume is more than 15 litre, suspend operation
8) Add 4.3 litre 14 N HNO_3
9) Add 0.83 litre 1.2 M $Fe(SO_3NH_2)_2$
10) Add water to complete 20 litre
11) Measure density
12) Measure volume
13) Measure activity
14) Take out sample
15) Send sample to laboratory for U, Pu, free acid, Fe^{++}/Fe^{+++} and total gamma determination

SECOND CYCLE SOLVENT EXTRACTION

1) Control performance of first cycle product tank control board
2) Measure density in first cycle product tank
3) Measure volume in first cycle product tank
4) Measure activity in first cycle product tank
5) Take out sample from first cycle product tank
6) Send sample to laboratory for U, Pu, free acid, Fe^{++}/Fe^{+++} and total gamma determination
7) If free acid concentration is different from 2.8 - 3.2 N or Fe^{++}/Fe^{+++} is less than 10, suspend operation
8) Control performance of second cycle solvent tank control board
9) Measure density in second cycle solvent tank
10) Measure volume in second cycle solvent tank
11) Measure activity in second cycle solvent tank
12) Take out sample from second cycle solvent tank
13) Send sample to laboratory for TBP, U, Pu and total gamma determination
14) Control performance of second cycle waste tank control board
15) Measure density in second cycle waste tank
16) Measure volume in second cycle waste tank
17) Measure activity in second cycle waste tank
18) Take out sample from second cycle waste tank
19) Send sample to laboratory for U, Pu, free acid, Fe^{++}/Fe^{+++} and total gamma determination
20) If volume in second cycle waste tank + twice the volume in first cycle product tank is more than 50 litre, suspend operation
21) Control if second cycle product tank is clean and empty
22) Adjust pumping rate of second cycle solvent pump to 1.5 litre/h (see instructions for solvent flow rate adjustment)
23) Adjust pumping rate of second cycle scrubbing pump to 0.5 litre/h (see instructions for scrubbing and stripping flow rate adjustments)
24) Adjust pumping rate of second cycle stripping pump to 1.5 litre/h (see instructions for scrubbing and stripping flow rate adjustments)
25) Adjust pumping rate of second cycle feed solution pump to 60/U concentration in g/litre in feed solution (see instructions for feed solution flow rate adjustment)
26) Control if level in feed pump recycling tank is above red mark
27) If level in feed pump recycling tank is below red mark, fill

up with scrub solution 3 N HNO_3

28) Control if level in scrub solution storage tank is above red mark

29) If level in scrub solution storage tank is below red mark, fill up with 3 N HNO_3

30) Control if level in strip solution storage tank is above red mark

31) If level in strip solution storage tank is below red mark, fill up with strip solution 0.01 N HNO_3

32) Control if valves N° 1, 2, 4, 5 and 9 are closed

33) Control if valves N° 3, 6, 7, 8 and 10 are open

34) Control if heavy phase outlet of extraction mixer-settler is closed

35) Control if heavy phase outlet of stripping mixer-settler is closed

36) Control if extraction and stripping mixer-settlers are full

37) If mixer-settlers are full, pass points 38 to 57 and continue with point 58

38) Start second cycle solvent pump

39) Open valve N° 2

40) Close valve N° 3

41) Wait until solvent overflows stripping mixer-settler's outlet weir

42) Start first solvent washer

43) Start second solvent washer

44) Start mixer-settler agitation

45) Start scrub solution pump

46) Open valve N° 4

47) Close valve N° 7

48) Start strip solution pump

49) Open valve N° 5

50) Close valve N° 8

51) When aqueous level in interphase level indicator of extraction mixer-settler is close to mark, connect air pressure regulator

52) Open heavy phase outlet of extraction mixer-settler

53) Start controlling interphase level of extraction mixer-settler with air regulator

54) When aqueous level in interphase level indicator of stripping mixer-settler is close to mark, connect air pressure regulator

55) Open heavy phase outlet of stripping mixer-settler

56) Start controlling interphase level of stripping mixer-settler

with air regulator

57) Pass points 58 to 75 and continue with point 76
58) Start first solvent washer
59) Start second solvent washer
60) Start solvent pump
61) Open valve N° 2
62) Close valve N° 3
63) Connect extraction mixer-settler interphase level air regulator
64) Connect stripping mixer-settler interphase level air regulator
65) Start scrub solution pump
66) Start strip solution pump
67) Open valve N° 4
68) Open valvo N° 5
69) Close valve N° 7
70) Close valve N° 8
71) Start mixer-settler agitation
72) Open heavy phase outlet of extraction mixer-settler
73) Open heavy phase outlet of stripping mixer-settler
74) Start controlling interphase level of extraction mixer-settler by air regulator
75) Start controlling interphase level of stripping mixer-settler by air regulator

PERFORM OPERATIONS 65 TO 75 AS FAST AS POSSIBLE

76) Start feed solution pump
77) Open valve N° 1
78) Close valve N° 6
79) Wait until both mixer-settlers reach steady state (interphase level keeps steady without change of air pressure in regulator)
80) Stop feed solution pump
81) Close valve N° 10
82) Open valve N° 9
83) Start feed solution pump

PERFORM OPERATIONS 80 TO 83 IN LESS THAN 10 SECONDS

84) Observe increase in volume in second cycle waste tank and second cycle product tank, and decrease in volume in first cycle product tank and scrub and strip storage tanks. Write down density and volume in each tank at one hour intervals
85) At end of feed solution (stop of flow in feed solution

observation glass) open valve N° 10
86) Close valve N° 9
87) Wait 15 minutes
88) Stop feed solution pump
89) Close valve N° 1
90) Open valve N° 6
91) Wait two hours
92) Open valves N° 3, 7 and 8
93) Close valves N° 2, 4 and 5
94) Close heavy phase outlet of extraction mixer-settler
95) Close heavy phase outlet of stripping mixer-settler
96) Stop mixer-settler stirrer motor
97) Stop second cycle solvent pump
98) Stop scrub solution pump
99) Stop strip solution pump
100) Stop air supply to interphase regulators
101) Stop first solvent washer
102) Stop second solvent washer
103) Measure density in first cycle product tank
104) Measure volume in first cycle product tank
105) Measure activity in first cycle product tank
106) Take out sample from first cycle product tank
107) Send sample to laboratory for U, Pu, free acid, Fe^{++}/Fe^{+++} and total gamma determination
108) Measure density in second cycle solvent tank
109) Measure volume in second cycle solvent tank
110) Measure activity in second cycle solvent tank
111) Take out sample from second cycle solvent tank
112) Send sample to laboratory for TBP, U, Pu and total gamma determination
113) Measure density in second cycle product tank
114) Measure volume in second cycle product tank
115) Measure activity in second cycle product tank
116) Take out sample from second cycle product tank
117) Send sample to laboratory for U, Pu and total gamma determination
118) Measure density in second cycle waste solution tank
119) Measure volume in second cycle waste solution tank
120) Measure activity in second cycle waste solution tank
121) Take out sample from second cycle waste solution tank
122) Send sample to laboratory for U, Pu, free acid and total

gamma determination

123) Calculate mass balances for U and Pu:

before extraction	after extraction
vol. feed sol. x U conc. feed sol.	
vol. 2nd cycle solv. x U conc. 2nd cycle solv.	
vol. 2nd cycle prod. x U conc. 2nd cycle prod.	
vol. 2nd cycle waste x U conc. 2nd cycle waste	
etc.	

DRAINING OF FIRST AND SECOND CYCLE SOLVENT WASHERS

1) Control performance of corresponding solvent tank control board
2) Measure density
3) Measure volume
4) Measure activity
5) Take out sample
6) Send sample to laboratory for U, Pu and total gamma determination
7) Calculate total content of U in solvent tank
8) Calculate total content of Pu in solvent tank
9) Verify if valve N° 2 in corresponding mixer-settler hood is closed
10) Verify if valve N° 3 in corresponding mixer-settler hood is open
11) Adjust corresponding solvent pump to flow rate between 1 - 3 litre/h
12) Start solvent pump
13) Add 500 ml water to corresponding recycle funnel
14) Wait one minute
15) Observe increase in volume in corresponding solvent tank
16) Close valve N° 3 until some liquid is build up in the corresponding calibration tube
17) Observe if there is aqueous layer in the liquid in the calibration tube
18) Open valve N° 3
19) If there was no aqueous layer, return to point 13
20) If there was aqueous layer, regulate valve N° 3 in order to recycle aqueous layer but no solvent for 10 minutes
21) Stpp solvent pump
22) Close valve N° 3
23) Control performance of solvent washer waste treatment tank control board
24) Measure density in solvent washer waste treatment tank
25) Measure volume in solvent washer waste treatment tank
26) Measure activity in solvent washer waste treatment tank
27) Take out sample from solvent washer waste treatment tank
28) Send sample to laboratory for U, Pu, total gamma and pH determination
29) If volume is more than 25 litre, or pH is below 7, suspend operation

30) Open valve N° 4 of solvent washer waste treatment tank control board until there is strong air bubbling in corresponding observation tube

31) Open slowly and simultaneously valves N° 6 and N° 8 on solvent washer waste treatment tank control board until there is strong air bubbling in corresponding observation tubes. Take care not to produce fast level changes in density indicator.

32) Open slowly and simultaneously valves N° 10 and N° 12 on solvent washer waste treatment tank control board until there is strong air bubbling in corresponding observation tubes. Take care not to produce fast level changes in volume indicator

33) Open corresponding interconnection valve:
valve N° 7 for first cycle
valve N° 9 for second cycle

34) Start vacuum pump

35) Open valve N° 2 on solvent washer waste treatment tank control board

36) Close slowly valve N° 3 on solvent washer waste treatment tank control board until vacuum gauge indicates a depression of 2.5 m of water column. Regulate valve N° 3 to keep this pressure

37) Observe through volume indicator the progress and end of the solution transfer

38) Control on observation tube the end of solution transfer

39) Open slowly valve N° 3 on solvent washer waste treatment control board

40) Close slowly valve N° 2 on solvent washer waste treatment control board

41) Close valve N° 4 on solvent washer waste treatment control board

42) Close simultaneously valves N° 6 and N° 8 on solvent washer waste treatment control board

43) Close simultaneously valves N° 10 and N° 12 on solvent washer waste treatment control board

44) Drain all the aqueous layer in solvent pump calibration tube through valve N° 3

45) Start corresponding solvent pump and simultaneously regulate valve N° 3 in order to drain only aqueous phase but no solvent

46) When organic phase in calibration tube reaches red mark, stop solvent pump and close valve N° 3
47) Repeat operations 30 to 43
48) Measure density in solvent washer waste tank
49) Measure volume in solvent washer waste tank
50) Measure activity in solvent washer waste tank
51) Take out sample from solvent washer waste tank
52) Send sample to laboratory for U, Pu and alkalinity determination
53) Calculate U content in solvent washer waste tank
54) Calculate Pu content in solvent washer waste tank
55) Calculate volume of 14 N HNO_3 necessary to neutralize content in solvent washer waste tank
56) Add by slow increments the volume of 14 N HNO_3 calculated by point 55
57) If amount of U in tank is more than 1.0 g, transfer solution to first cycle product storage tank
58) If amount of U in tank is less than 1.0 g, transfer solution to intermediate waste decay tank

STORAGE TANK CONTROL BOARD OPERATION

A) Control of performance

1) Verify if valves N° 1, 2, 4, 6, 8, 10 and 12 are closed
2) Verify if valves N° 3, 5, 7, 9, 11 and 13 are open
3) Start air pump
4) Open slowly valve N° 4 until air bubbles in corresponding observation tube
5) Close valve N° 4
6) Verify if liquid level in density indicator is close to half of scale
7) Open slowly and simultaneously valves N° 6 and 8 until air bubbles in corresponding observation tubes, taking care not to produce fast liquid level changes in density indicator
8) Close valves N° 6 and 8 simultaneously
9) Verify if liquid level in volume indicator is close to half of scale
10) Open slowly and simultaneously valves N° 10 and 12 until air bubbles in corresponding observation tubes, taking care not to produce fast liquid level changes in volume indicator
11) Close simultaneously valves N° 10 and 12

B) Addition of solutions to tanks

12) Take off funnel cover
13) Control if inside of funnel is clean
14) Open valve N° 1
15) Add slowly to funnel and wait until funnel is empty
16) Close valve N° 1
17) Cover funnel

C) Mixing of solution in tank

18) Open valve N° 4 for strong air bubbling in corresponding observation tube
19) Wait 3 minutes
20) Close valve N° 4

D) Measurement of density

21) Open slowly valve N° 8 until slow air bubbling in corresponding observation tube and observe if liquid level

in density indicator changes. Close immediately valve N° 8

22) If no change in liquid level was observed, there is not enough solution in the tank and it is not possible to measure density

23) If a change in liquid level was observed, open slowly and simultaneously valves N° 6 and 8 until air bubbles slowly in corresponding observation tubes, taking care not to produce fast liquid level changes in density indicator

24) Measure liquid level difference between both arms of density indicator

25) Close valves N° 6 and 8

26) Calculate density using formulae:

$$d = K_1 \cdot h_d$$

where d = density in g/cm^3

K_1 = characteristic constant for each tank written on control board

h_d = level difference in cm in density indicator

E) Measurement of volume

For the measuremont of volume it is necessary to know previously the density of the solution contained in the tank, or h_d as measured in step 24

27) Open slowly and simultaneously valves N° 10 and 12 until air bubbles slowly in corresponding observation tubes, taking care not to produce fast liquid level changes in volume indicator

28) Measure liquid level difference between both arms of volume indicator

29) Close valves N° 10 and 12

30) If h_d as measured in step 24 is known, calculate volume by formulae:

$$V = K_2 \frac{h_v}{h_d} + V_o$$

where V = volume of solution in litre contained in tank

K_2 = characteristic constant for each tank written on control board

h_v = level difference in cm in volume indicator

h_d = level difference in cm in density indicator

V_o = dead volume in tank; characteristic for each tank, written on control board

31) If density of solution is known, calculate volume by formulae:

$$V = K_3 \frac{h_v}{d} + V_o$$

where K_3 = characteristic constant for each tank written on control board

32) If no level difference is observed between both arms of volume indicator, the volume of solution in the tank is less than V_o

SAMPLER OPERATION

1) Verify if valves A, B, C and E corresponding to sampler control board are olosed
2) Verify that valve D corresponding to sampler control board is open
3) Control performance of control board corresponding to tank to be sampled
4) Place sample bottle in conveyor
5) Place conveyor in sampler equipment
6) Start vacuum pump
7) Start air pump
8) Open valve C corresponding to tank to be sampled
9) Open valve E corresponding to tank to be sampled
10) Close valve D corresponding to tank to be sampled until vacuum gauge indicates 50 mm nercury column depression
11) Open valve B
12) Open slowly valve A until a strong air bubbling is observed in observation glass
13) Wait 3 minutes
14) Lower sampler handle to introduce needles in sampler bottle
15) When enough sample has been collected, rise sampler handle
16) Open valve D
17) Close valve C
18) Close valve E
19) Close valve A
20) Close valve B
21) Stop vacuum pump
22) Stop air pump
23) Take out sample conveyor

TRANSFER OF SOLUTIONS BETWEEN TANKS

The present instructions apply to the operations described in Table I.

from tank	to tank	interconnection valve N°
dissolver	storage	1
first cycle product	storage	2
filtered solution	storage	3
filtered solution	feed adjustment	4
first cycle waste	storage	5
second cycle waste	first cycle product	11
second cycle waste	ion exchange feed	12
solvent washer waste treatment	first cycle product	13

TABLE I. Transfer operations between tanks

The transfer of solutions from solvent washers to the solvent washer treatment tank (interconnection valves N° 7, 8, 9 and 10) are described in the solvent washer draining instructions. The transfer of waste solutions the first cycle, solvent washer treatment and ion exchange to the waste storage tank (interconnection valves N° 6, 14 and 15) are described in the waste storage instructions.

1) Control performance of feeder tank control board
2) Control performance of receiver tank control board
3) Measure density in feeder tank
4) Measure volume in feeder tank
5) Measure activity in feeder tank
6) Measure density in receiver tank
7) Measure volume in receiver tank
8) Measure activity in receiver tank
9) If sum of volumes in feeder and receiver tank is more than 50 litre, suspend operation
10) Open valve N° 4 on receiver tank control board until air bubbles strongly in corresponding observation tube
11) Open slowly and simultaneously valves N° 6 and 8 on receiver tank control board until air bubbles strongly in corresponding observation tubes, tanking care not to produce fast level changes in corresponding density meter

12) Open slowly and simultaneously valves N° 10 and 12 on receiver tank control board until air bubbles strongly in corresponding observation tubos, taking care not to produce fast level changes in corresponding volume meter
13) Open corresponding interconnection valve corresponding to Table I
14) Start vacuum pump
15) Open valve N° 2 on receiver tank control board
16) Close slowly valve N° 3 on receiver tank control board until vacuum gauge indicates a depression of 150 mm of Hg column
17) Observe through volume indicator in feeder tank the progress and end of operation
18) Control with the observation glass corresponding to the used interconnection valve the end of passage of solution
19) Open slowly valve N° 3 on receiver tank control board
20) Stop vacuum pump
21) Close corresponding interconnection valve according to Table I
22) Close valve N° 2
23) Close valve N° 4
24) Close simultaneously valves N° 6 and 8
25) Close simultaneously valves N° 10 and 12
26) Measure density in feeder tank
27) Measure volume in feeder tank
28) Measure activity in feeder tank
29) Measure density in receiver tank
30) Measure volume in receiver tank
31) Measure activity in receiver tank
32) Calculate volume balance

WASTE STORAGE

The present instructions apply to the transfer of solutions from the first cycle waste tank, the solvent washer treatment tank and the ion exchange waste storage tank to the waste decay tank. The interconnection valves are numbered in Table II.

from tank	to tank	interconnection valve N°
first cycle waste	intermediate waste	6
solvent washer treatment	intermediate waste	14
ion exchange waste	intermediate waste	15

TABLE II. Transfer operations to waste.

1) Verify if all valves on waste storage control board are closed
2) Measure level in waste storage tank
3) Verify if intermediate waste tank is empty
4) Verify performance of feeder tank control board
5) Measure density in feeder tank
6) Measure volume in feeder tank
7) Measure activity in feeder tank
8) Open valve N° 10 on filter equipment
9) Open valve N° 2 on waste storage control board
10) Open interconnection valve according to Table II
11) Open valve N° 1 on waste storage control board
12) Start vacuum pump
13) Observe through volume indicator in feeder tank the progress and end of operation
14) Control with observation glass corresponding to the used interconnection valve the end of passage of solution
15) Stop vacuum pump
16) Close valve N° 2 on waste storage control board
17) Close valve N° 1 on waste storage control board
18) Close valve N° 10 on filter equipment
19) Close corresponding interconnection valve according to Table II

20) Measure level in intermediate waste tank
21) Measure density in feeder tank
22) Measure volume in feeder tank
23) Measure activity in feeder tank
24) Open valve N° 3 on waste storage control board
25) Open valve N° 4 on waste storage control board
26) Wait 15 minutes
27) Close valve N° 3 on waste storage control board
28) Close valve N° 4 on waste storage control board
29) Measure level in intermediate waste tank
30) Measure level in waste storage tank

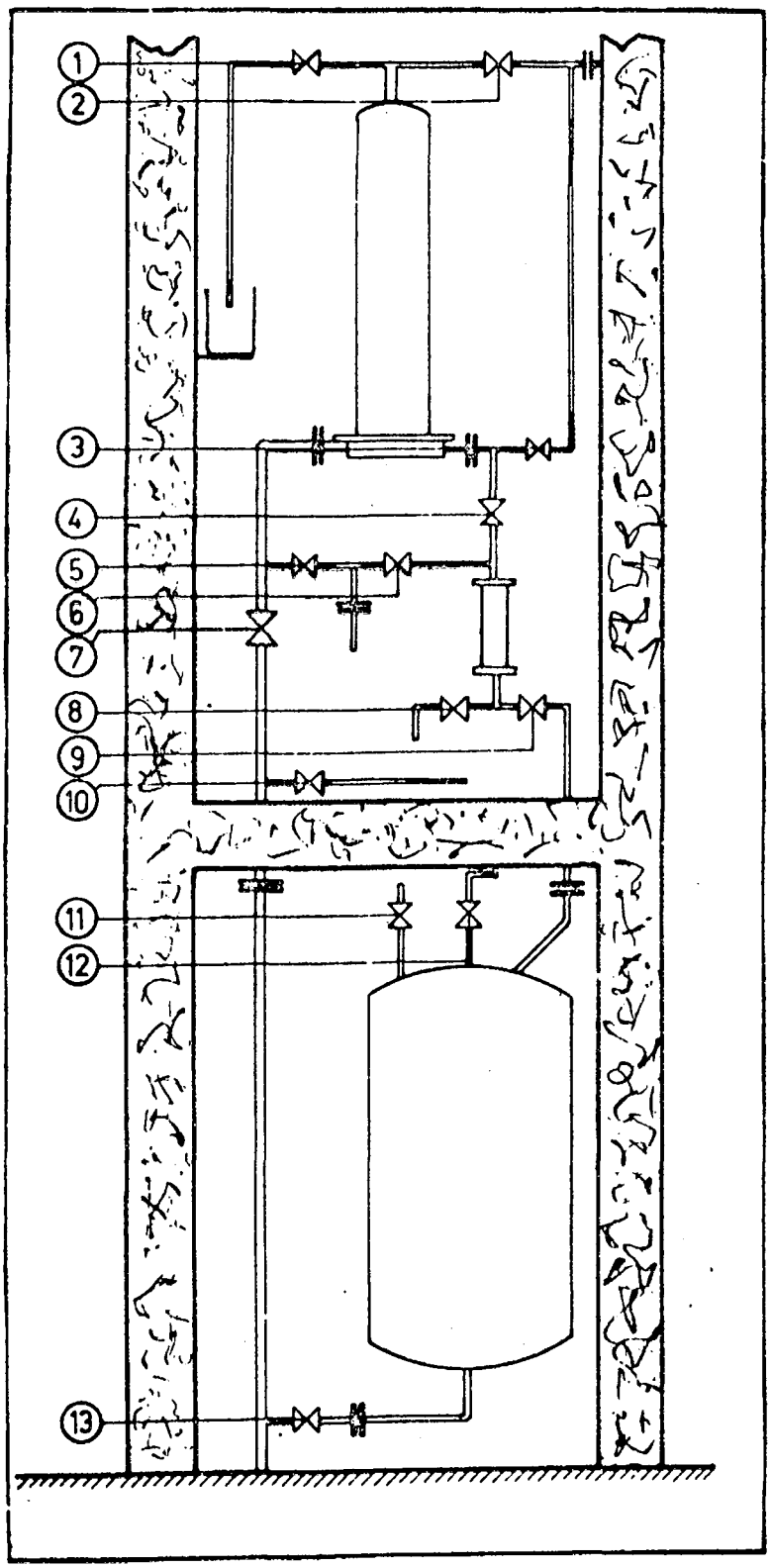


FIGURE 1

FILTER EQUIPMENT VALVE NUMERATION

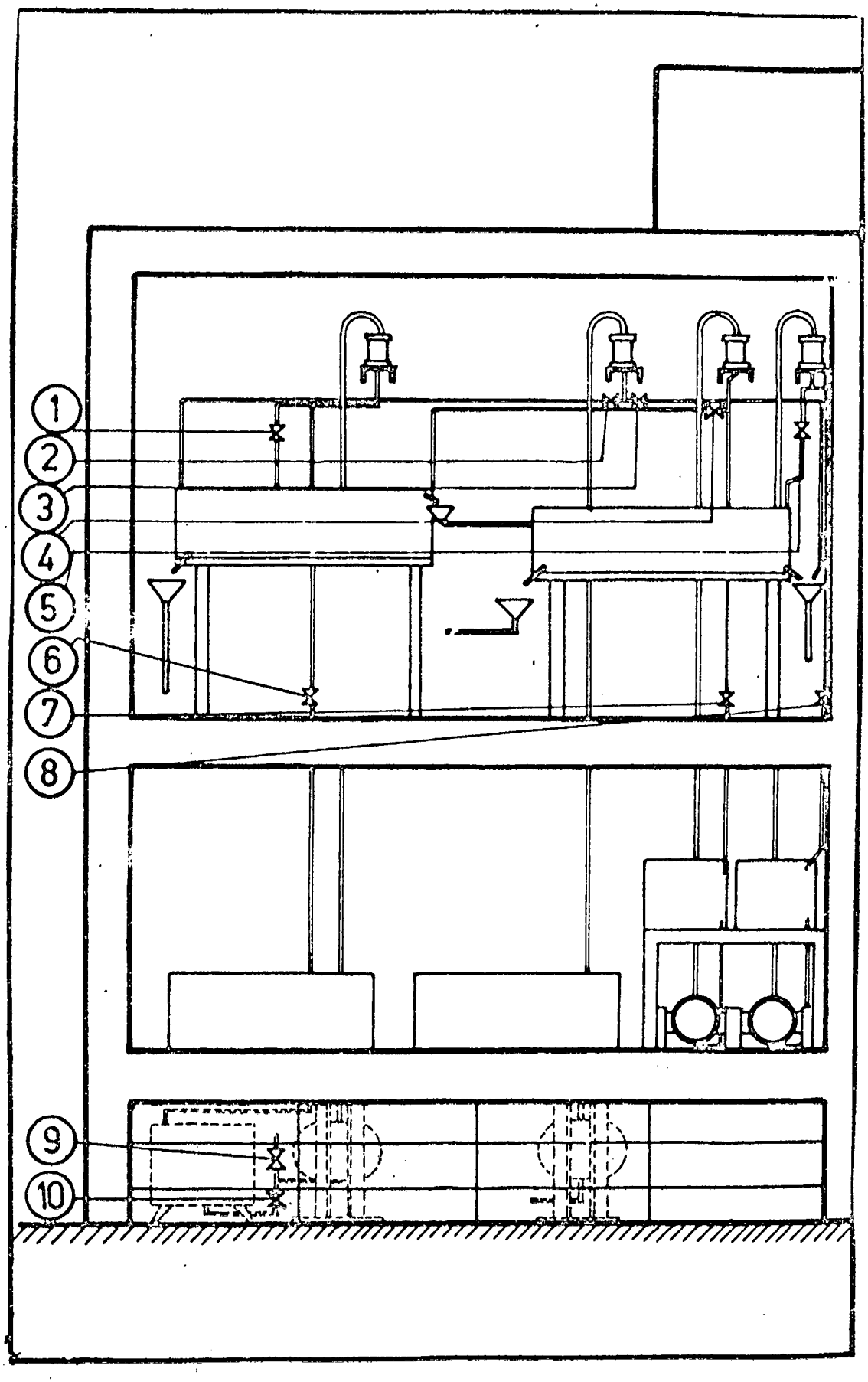


FIGURE 2

SOLVENT EXTRACTION EQUIPMENT VALVE NUMERATION

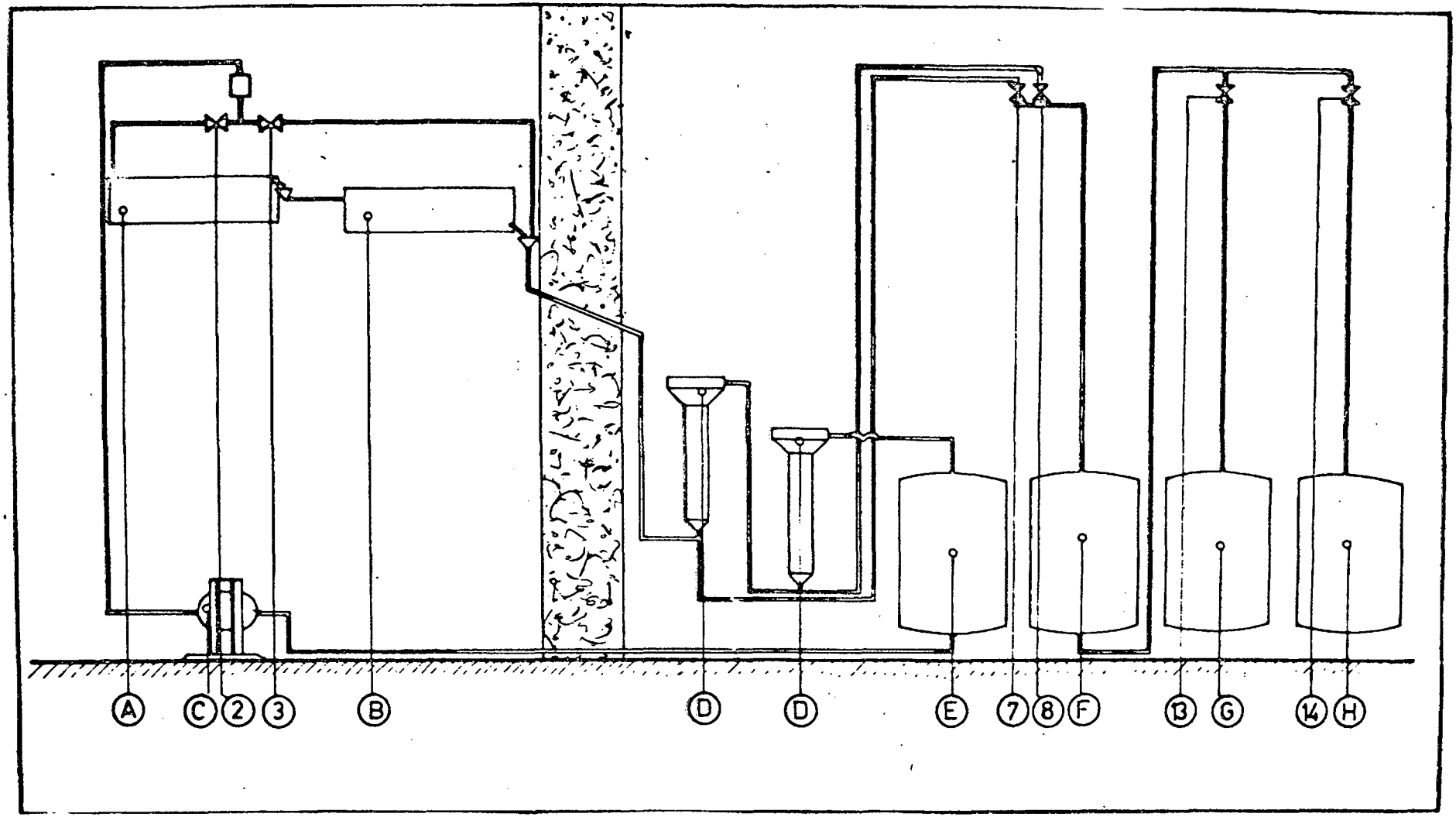
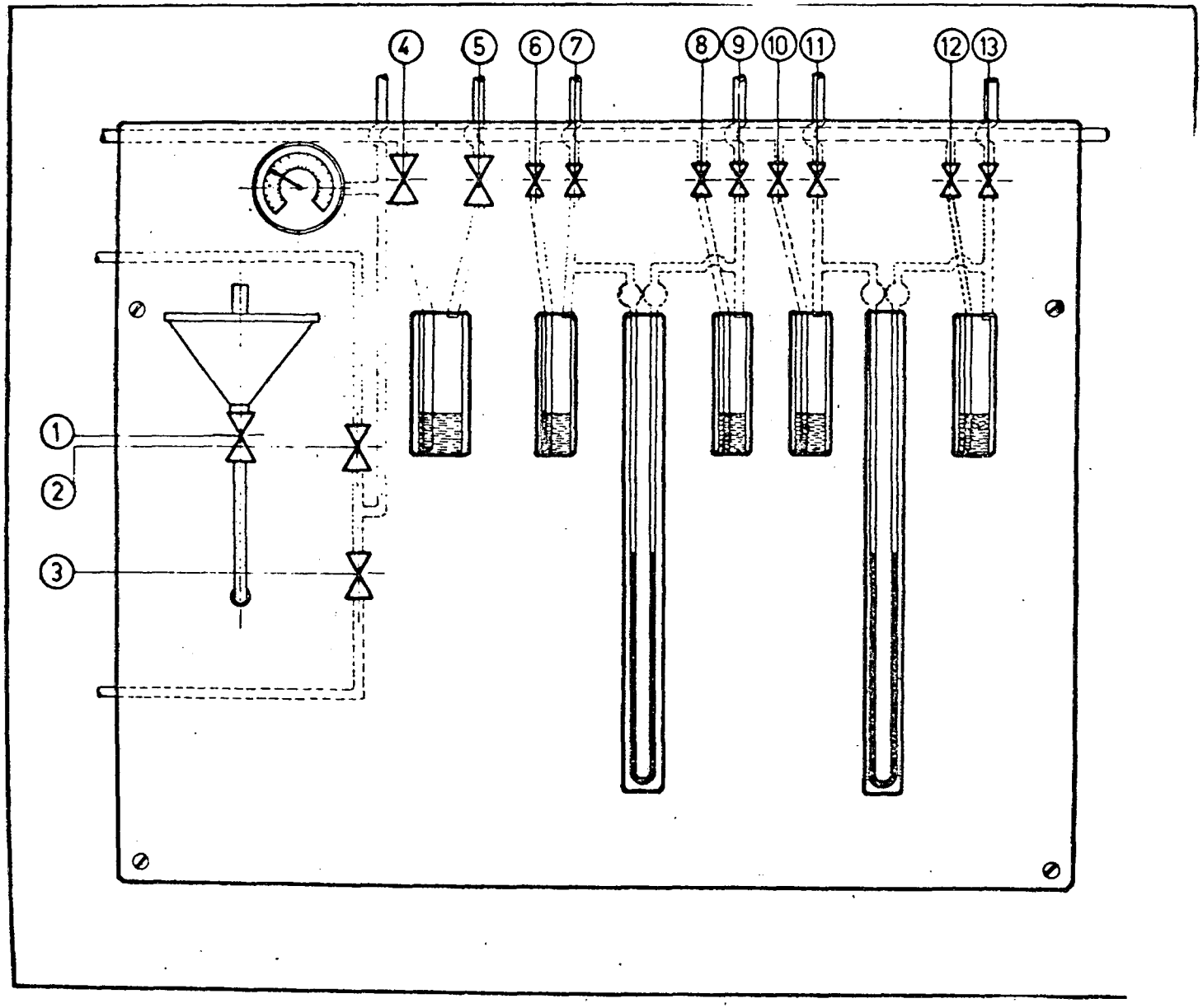


FIGURE 3 SOLVENT CIRCUIT AND SOLVENT WASHER WASTE TREATMENT - VALVE NUMERATION

A. Extraction mixer-settler
B. Stripping mixer-settler
C. Solvent pump
D. Solvent washers
E. Solvent tank
F. Washer waste treatment tank
G. 1st cycle product tank
H. 1st cycle waste tank

FIGURE 4

STORAGE TANK CONTROL BOARD
VALVE NUMERATION



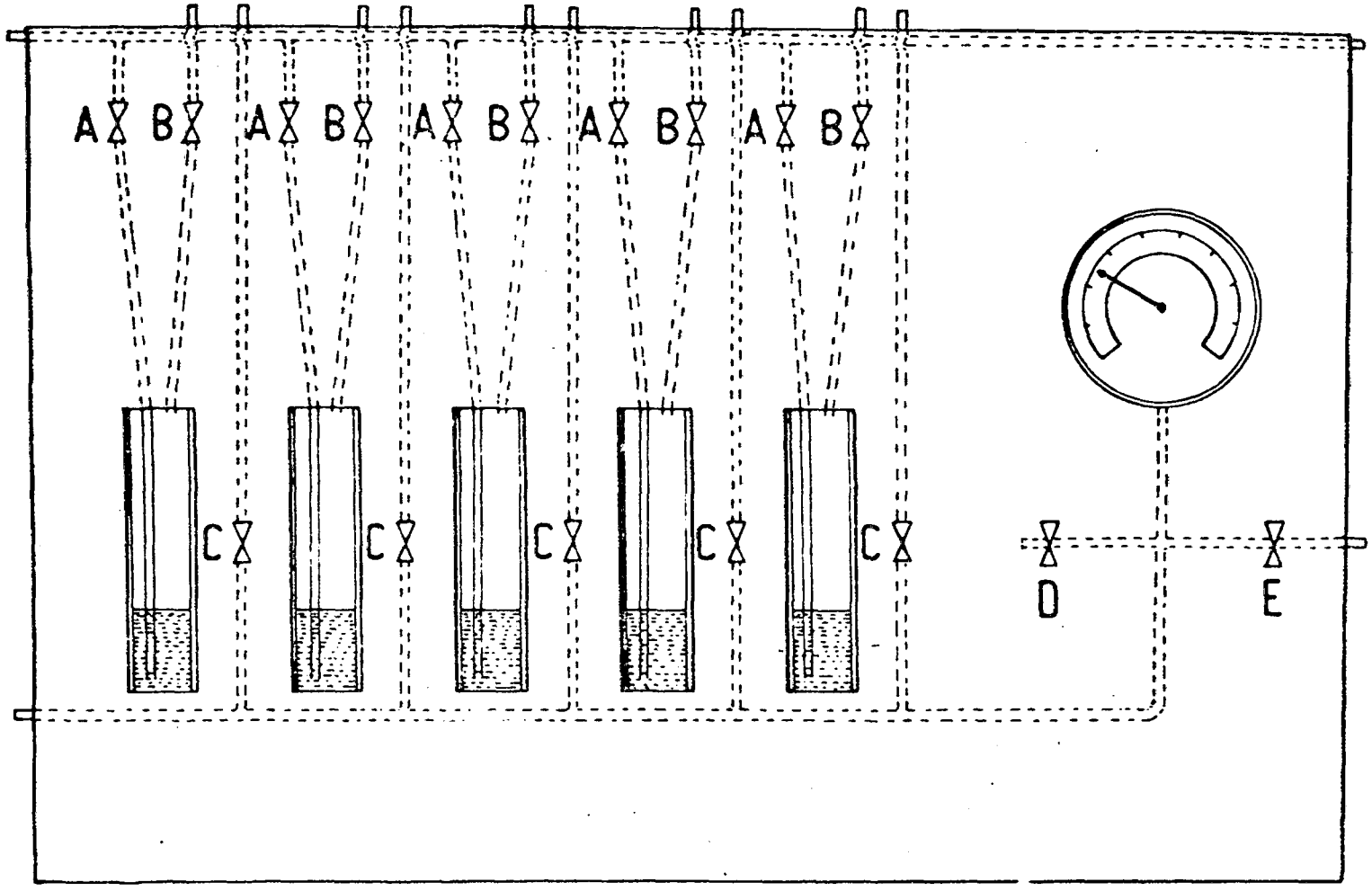


FIGURE 5 SAMPLER CONTROL BOARD - VALVE NUMERATION

NUCLEAR SAFETY EVALUATION

NUCLEAR SAFETY EVALUATION OF A PILOT PLANT FOR THE REPROCESSING OF IRRADIATED FUEL ELEMENTS .

INTRODUCTION

In order to gain experience in the chemical processes and techniques used in spent fuel reprocessing, the CNEA has built a small pilot plant at the Ezeiza Atomic Center. The present study is an evaluation of the nuclear safety of this pilot plant.

Plant description.

The plant is composed of several units [1], of which the following are relevants to this study (fig. 1):

Unit 2. Dissolver

Unit 3. Storage tank, for solution from dissolver

Unit 5. Filter tank, for solution from filter

Unit 6. Adjustement tank, for solutions going to mixer-settlers (Unit 11)

Unit 7. Active waste tank, for first cycle waste (coming from Unit 11)

Unit 8. Storage tank for solvent (TBP), used in the first cycle.

Unit 9. Filter for solutions from storage tank 3.

Unit 10. Solvent washer formed by four subunits, two for solvent from first cycle hood (Unit 11), which are connected to Unit 8, and two for solvent from second cycle hood (Unit 16), which are connected to Unit 12.

Unit 11. First cycle hood, mixer-settlers, when fission products are separated.

Unit 12. Storage tank for solvent (TBP), used in the second cycle.

Unit 13. First cycle product tank where solution coming from mixer settler (Unit 11) is adjusted before going into second cycle.

Unit 14. Waste tank for low level waste from second cycle which contains the plutonium.

Unit 15. Waste tank for Unit 10.

Unit 16. Second cycle hood, mixer-settlers, where U and Pu are separated.

The uranium solution from the second cycle ($UO_2(NO_3)_2$) will be converted batch by batch to U_3O_8 and sent to the special fissionable material storage, at the Metallurgy Department, in Constituyentes Center.

Basis of the safety evaluation.

The operating procedures adopted in the plant [1] imply that under normal conditions not more than 210 grams of U^{235} will be processed at a time. Under these conditions the operation is intrinsecally safe. However, the plant is studied under more general conditions to estimate safety margins in view of possible unforseen events or future changes in operating procedures.

The evaluation of the nuclear safety of the plant is based on published experimental data [2][3][4].

The values of critical concentrations as a function of the H/U ratio correspond to 93, 5% enriched uranium [2]. Estimations based on these values are conservative, since they ignore the presence of other prissons such as HNO_3, etc.

A correction factor of 1. 3 was used in our case (20% enrichment) as prescribed in the basic safety standards used by CNEA [5]; these safety standards are conservative, since the maximum allowed values ("MAV") are . 45 of the critical values. Thus, criticality is avoided even with double batching.

SAFETY EVALUATION.

Maximum allowed concentrations for each unit were calculated. Furthermore, interaction between units was calculated by the solid angle method [5].

Evaluation of individual units.

Dissolver (Unit 2)

This is a 95 liter cylinder, which is not geometrically safe. A water coil for refrigeration is placed inside; therefore, the unit is completely reflected. In each batch 8 fuel elements are processed, together with 13. 38 liters of water and 20. 72 liters of 14N HNO_3 which gives a total volume of 35 liters. We assume that the 8 elements are those with maximum uranium content, which implies 206. 5 grams of U^{235}. Thus maximum possible concentration under homogeneous conditions is 5. 9 gram U^{235}/liter, while maximum allowed concentration for a 35 liter of solution of U(20) is 17. 5 grams U^{235}/liter.

If the 206.5 grams U^{235} are dissolved in 95 liters the safety margin is even larger, since maximum allowed concentration for this volume is 11.7 grams U^{235}/liter and the actual value is 2.2 grams U^{235}/liter.

The dissolver is placed inside a cell with thick (60 cm) concrete walls, so that interaction with other units can be neglected.

Tanks (Units 3, 5, 6, 7, 13, 14, 15)

All these units have a 70 liter capacity. They are not geometrically safe. Maximum allowed concentration for this volume is 12.2 grams U^{235} /liter. A mass of 206.5 grams U^{235} in 70 liters results in a concentration of 2.95 grams U^{235}/liter.

Units 3, 5, 6 and 7 are inside a 240 x 110 cm. rectangular concrete cell, so that interaction between this group of units and the rest of the system can be neglected.

In the improbable case that the solution leak from one of the tanks and is retained by the cell, it would have a slab configuration with a thickness of 2.6 cm, which is geometrically safe.

Units 13, 14 and 15 are placed in a concrete cell which is larger than that of units 3, 5, 6 and 7.

Filter (Unit 9)

This unit is a cylinder 10 cm. in diameter and 50 cm. high which

First cycle hood (mixer-settler) (Unit 11)

The mixer-settlers are of the multistage pump-mix type and are quoted as "ever safe" [6]. It is formed by two sections 8 x 14 x 55 cm. each, with an internal volume of 4. 25 liter, which is geometrically safe for solutions.

Second cycle hood (Mixer-settlers) (Unit 16)

This mixer settler is of the same type as that of the first cycle hood but it has different dimensions. The sections are 78 x 8 x 14 cm. and 35 x 8 x 14 cm. with internal volumes of 5. 6 and 2. 5 liter respectively. These volumes are also geometrically safe for solutions.

Solvent washers (Unit 10)

This unit receives no fissionable material unless the mixer-settlers work incorrectly.

It is formed by four containers placed on the vertices of a 45 x 55 cm. rectangle. The upper part is a truncated cone of 7 cm. height and 12. 7 and 4. 8 cm. radii with a volume of 1. 75 liter, the lower part is a cylinder of 4. 8 cm. radius and 42. 2 cm. height with a volume of 3. 25 liters. The total volume of the unit is 5 liters. Consequently, the unit is geometrically safe by volume and by dimensional criteria.

Table I synthesizes the values obtained in unit evaluations.

TABLE I

UNIT		Maximum allowed concentration (gr U^{235} / l)	Actual concentration (gr U^{235}/l)
N°	Vol. (l)		
2	95	11.7	2.2
3) 5) 6) 7) 13) 14) 15)	70	12.2	2.9
9 10 11 11 16 16	3.9 5.0 4.2 4.2 5.6 2.5	Criticality not obtainable with solutions in these volumes.	

Evaluation of the interaction between units.

Interaction between units was estimated by the solid angle method (5). In view of the operating procedures only two units at a time have to be considered. Maximum interaction takes place between the units that are closest. If this case gives safe values the rest need not be considered. From fig. 1 we conclude that the worst case is that of units 5 and 6.

Since these units are interconnected (Unit 6 is used for adjusting the solution coming from Unit 5), maximum interaction takes place when the tanks are half filled. In this case the solid angle is $\Omega = \frac{2d}{h} \sin \theta = 1.31$ ster. (fig. 2).

The multiplication factor k_{eff} for the solution is calculated taking into account the amount of U^{235} and the presence of U^{238}, aluminium, nitrogen and hydrogen.

With these considerations, $k_{eff} = 0.44$ [7].

Therefore $\Omega_{max} = 9\text{-}10\ k_{eff} = 4.6$ ster., and the interaction between units can be considered negligible.

CONCLUSIONS:

The plant is nuclearly safe. To reach criticality, load must be increased by a factor of about 6 in the dissolver and above 10 in the others which, in practice, is impossible due to operating conditions and administrative controls.

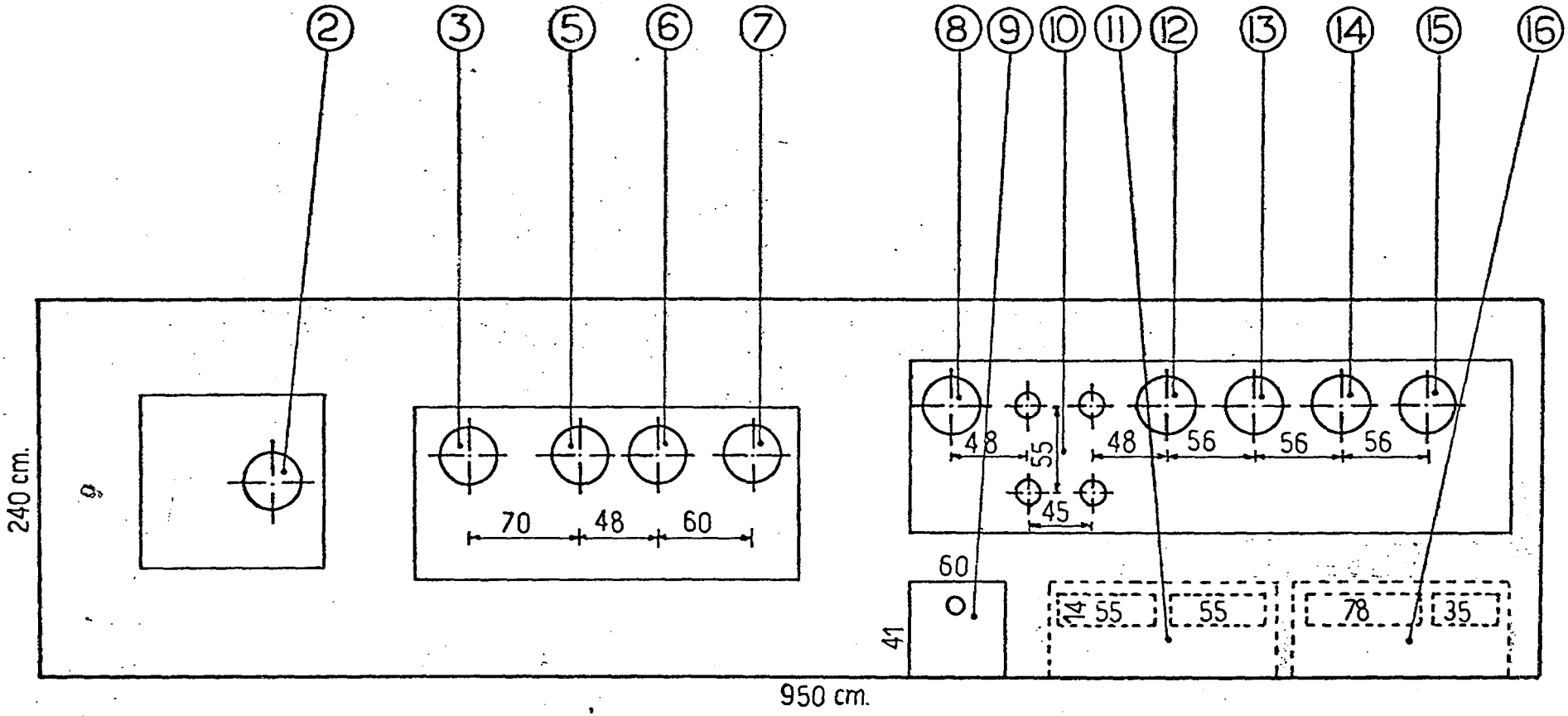


Fig.1 PLANT LAYOUT

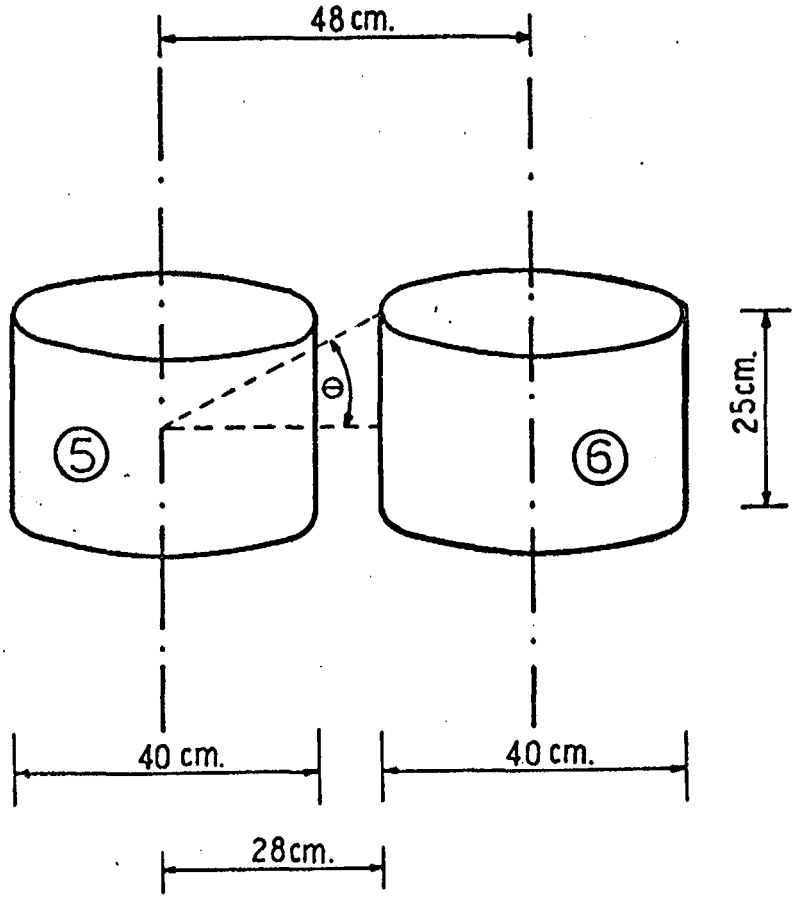


Fig. 2

REFERENCES

(1) Pilot Plant for the Reprocessing of Irradiated Nuclear Fuels - CNEA - 1967.

(2) Critical Dimensions of Systems Containing U^{235}, Pu^{239}, and U^{233}. H.C. Paxton et al. (TID - 7028) - 1964.

(3) Criticality Control in Operations with Fissile Material - H. C. Paxton (LA-3366) - 1966.

(4) Nuclear Safety Guide - (TID-7016, Rev. 1) - 1961.

(5) Normas Básicas de Seguridad Radiológica y Nuclear - CNEA - (S. I. N° 11) - 1966.

(6) Critically Safe Mixer-Settlers - J. Centeno (ETR-138 Rev.) - 1962.

(7) Handbook of Nuclear Safety - H. K. Clark (DP-532) - 1961.

ANNEX 1

RADIATION PROTECTION

The plant was designed so that operations would comply with the basic safety standards of CNEA [5], a copy of which is enclosed with this paper. Health physics services will be provided for the plant.

COMPLEMENTARY INFORMATION

The following instructions are complementary to the Report on the Pilot Reprocessing Plant and its Operation Manual:

1. Safety instructions which are normally adhered to in other establishments handling radioactive materials will apply in this plant. These cover the following matters:

   a) Responsibility for the operations
   b) Access to the area
   c) Protective clothing
   d) Prohibition of eating, drinking and smoking
   e) Film badges and urine samples
   f) Monitoring of the working area
   g) Criticality procedures
   h) Other emergencies

2. It is intended to reprocess the first charge of the RA-1 reactor in shifts of 8 hours (effective time 7 hours) a day, operating one shift a day, with a shift leader and one other fully responsible person (see 4.).

3. At least two persons concerned with the reprocessing operation shall normally be present in the pilot plant room while the plant is operating.

4. Full responsibility towards the plant implies:

   a) Orders for starting or finishing any operation in the plant in which fissionable material is moved.
   b) Orders for carrying out operations according to the Operation Manual.
   c) Allowance of access to the plant of other persons not directly concerned with operation of the plant.
   d) The handling of situations which are not contemplated in the Operation Manual or which might be considered accidents.

   It is privative of the full responsibility category to take decisions concerning points a) to d).

5. Persons with full responsibility towards the plant are:

   F. Kaufmann
   J. Flegenheimer
   N. Venturini
   S. Morazzo

   Other persons concerned with operation of the plant are:

Technician:	A. Bratt
Skilled workers:	J. Bordazahar
	A. Briga
Analysts:	O. Cristallini
	..... (technician, to be named)

6. A logbook will be kept in the plant room on all operations carried out in the plant and a separate record on all movement of enriched uranium. For all samples taken out of the room for analytical purposes, one of the analysts must sign the appropiate entry in the logbook. The reprocessed uranium of each batch of 3 fuel elements will be taken away by the "Gerencia de Tecnología" and the appropiate entry signed in the record by its responsible officer. The logbook will be signed and agreed to be correct at the end of each shift by one of the fully responsible persons.

DETAILS ON PERSONNEL

Ferdy KAUFMANN: Born in 1928. Studied at the School of Sciences, Buenos Aires, where he obtained his degree of Licenciate in Chemistry, specialized in industrial and technological chemistry, in 1955. After private industry work, he entered the Chemistry Department of the CNEA in 1957, carrying out research on uranium extraction and related problems. As head of the Solvent Extraction group and later as head of the Pilot Plant at Constituyentes, he has had a varied experience in plant installations for the obtention of nuclear grade uranium from minerals, including ore mills, lixiviators, thickeners, pulsed columns, mixer-settlers, and ion exchange processes. In 1962 he engrossed the the starting Reprocessing Department at Ezeiza where he has been in charge of the design, installation and operation of the pilot plant. During 1963 Kaufmann was at the Argonne National Laboratory with an IAEA fellowship where he worked out the flow sheet for the RA-1 fuel elements under the direction of Dr. K. A. Varteressian. During the experimental work, distribution coefficients and decontamination factors were obtained with simulated RA-1 fuel elements. Three reports have been produced at Argonne on these results.

Juan G. FLEGENHEIMER: Born in 1927. Studied at the School of Sciences, Buenos Aires, where he obtained his degree of Doctor in Chemistry in 1954. Entered the Radiochemistry group of the CNEA in 1952 which at the time was directed by Prof. W. Seelmann-Eggebert. He was at Cambridge, England, from 1956 to 1959 where he obtained his Ph. D. carrying our research work on protactinium chemistry under the direction of Dr. A. G. Maddock. As head of the CNEA Radiochemistry Division and later of Radioisotope Production, he has had a wide experience in the radiochemical field. He started and headed the Reprocessing Department in Ezeiza since 1962 which is also concerned with research work on the actinides. During 1965 he carried out research in Karlsruhe, Germany, with a von Humboldt fellowship. He has represented his country on international meetings and has several publications in his name.

Néstor R. VENTURINI: Born in 1937. Studied at the School of Sciences, University of Buenos Aires, where he received his degree of Licenciate in Chemistry, specialized in technological chemistry, in 1966. Forms part of the Reprocessing Department since July 1966 having participated actively in the installation and operation of the pilot plant.

Santiago M. MORAZZO: Born in 1941. Studied at the School of Sciences, University of Buenos Aires, where he received his degree of Licenciate in Chemistry, specialized in physical chemistry, in 1966. Forms part of the Reprocessing Department since September 1966 and has been engaged in trial running the plant as well as with investigations concerning modifications.

Osvaldo A. CRISTALLINI: Born in 1934. Studied at the School of Sciences, University of Buenos Aires, receiving his degree of Licenciate in Chemistry in 1957. After a varied experience in the private industry entered the CNEA in 1957 and was analyst at the Malargüe uranium plant until 1961. He took the radiochemistry course at CNEA and entered the radiochemistry group in 1961, where he concerned himself mainly with alpha emitters and general radiochemical separations. Forms part of the Reprocessing Department since its origin in 1962 and has adapted or developed the methods contained in the analytical Guide of the pilot plant. He was at Argonne during part of 1964 and 1965 carrying out research on analytical chemistry in the Chemistry Division headed by Dr. D. C. Stewart. During his stay at Argonne he followed several of the courses at the I. N. N. S. E. and had a varied training program. He has a wide experience in the analytical field and several publications in his name.

Alberto N. BRATT: Obtained his certificate of Chemical Technician at the Technical School, Buenos Aires, in 1966. He has been working at the Department since November 1966 and has operated the plant regularly.

José M. BORDAHAZAR and Aníbal I. BRIGA: Both have been working at the Department since its origin in 1962 and have assembled the plant. They have constructed parts of the plant and are familiar with its operation.
