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APPARATUS FOR REMOTE CHEMICAL OPERATIONS

by

M. D. Snyder

Separations Chemistry Division

June 1956

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by

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ABSTRACT

Laboratory apparatus for performing remote chemical operations was developed around interchangeable glass components and master slave operation.

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INTRODUCTION

Laboratory-scale apparatus was required for dissolving irradiated canned slugs of uranium and further processing the solutions. Design criteria included portability, reliable remote operation, strict shielding and containment of radioactive materials, simplicity of operation; and low cost of construction. No known design met all of these requirements. The criteria were met by designing a containment box to house the apparatus within a high level cave. This report covers design details of the apparatus; other aspects were reported at the fourth annual symposium on hot laboratories (see AEC unclassified document TID-5280).

SUMMARY

A system was developed for processing materials exhibiting multicurie levels of radiation. Apparatus was built at low cost using interchangeable glass components and master slave manipulators. The design of the apparatus permits extensive modifications to be made with ease. All design criteria were met.

A variety of operations were carried out including precipitation, filtration, solvent extraction and ion exchange. For example, irradiated slugs of uranium were dissolved and processed to yield decontaminated plutonium. Six of these operations were performed in one month with no measurable contamination of the cell or significant exposure of personnel.

The apparatus embodies the following special features:

1. The plywood isolation box has a "Plexiglas" front for visibility and accommodates master slave manipulators through sealed ports at the top. All disconnections for removal of the box from the cave cell can be made remotely.

2. The all-glass apparatus consists of components with ball joints for easy interconnection and replacement. Many of these ball joints serve as valves in the regulation of fluid flow.

3. The apparatus is designed and built for maximum freedom from breakage and operating error. All fluid transfers are made by evacuation through a roving vacuum connector which can serve only one vessel at a time. Likewise, vessels can receive but one reagent at a time through a roving feed line. The construction of the apparatus prevents suck-backs. Reverse transfers are made by special adapters.

4. Contamination of the box is minimized by operating under a negative pressure and by keeping disconnect joints free of radioactive liquids. The usual lubricants are carefully avoided, water being applied when necessary.

5. Vessels are cooled by external application of liquid Freon-11, which boils at 23.5°C.

6. Manipulations can be performed with one hand, and complete visibility provides essentially benchtop observation and control of the operations.

DISCUSSION

GENERAL

A system for processing slugs that exhibit multicurie levels of radiation was designed with safety, simplicity and flexibility as prime criteria. It was believed that these requirements could best be realized by the use of easily assembled components contained in an isolation box and that a high degree of reliability would be provided by manual operation through master slaves. Accordingly, a major feature of the apparatus was the use of glass vessels fitted with ball joints and interconnected by plastic tubing. The system is simple to operate and capable of producing dependable results quickly. While many special modifications of the equipment are possible, only the components that have actually been operated will be discussed. The figures attached show most of the apparatus in detail sufficient for an understanding of the principal features.

EQUIPMENT

Isolation Box

The isolation box, which is 62 inches wide, 38 inches high and 36 inches deep, is constructed of painted 3/4-inch plywood with a 1/2-inch "Plexiglas" front. A stainless steel tray two inches deep covers the entire bottom, including the entry airlock section; and enamel pans are provided at strategic points to localize possible spills. Polyethylene film covers the remaining plywood areas, and polyethylene boots isolate the master slave mechanism from contamination. External service lines for liquid reagents and gases are equipped with ball joints so that they can be disconnected from the box by a General Mills Manipulator. Likewise the drain line, two electrical lines, and the three flexible wire cables used for lifting operations can be remotely disconnected. The entire box is positioned in and removed from the cell by a crane. If desired, the entire contents of the isolation box, exclusive of reagent feed lines and mounting hardware, can be removed and replaced remotely. Figures 1, and 12 through 17 show the general layout of the system.

Ball Joint Connectors

The ball joints allow interconnection of any two vessels. In the 12/2 size, both regulating and on-off valve action are provided since the opening in the joint is restricted in proportion to the degree of angular displacement. Figures 2 through 11 show some of the details. For assembly with one hand, the joints are either bent in a close gooseneck or are provided with a pilot tube as shown in Figure 10. The pilot tube also serves to keep the bearing surfaces of joints free of radioactive liquids. Lubricants are carefully avoided, except for infrequent application of water. Spring clamps are provided with channeled handles to facilitate accurate and positive manipulation with the master slaves; some clamps, infrequently used, are unmodified except for weakened springs. A clamp with one-piece extended handles has been designed, and could be mass produced if justified by demand. Likewise, a size 18/5 joint has been designed which would provide valve action for flows exceeding the capacity of 12/2 joints.

Standardization

Vessels, connections, and mountings are standardized as much as practical in order to simplify replacement. For instance, the vessel shown as the trap in Figure 3 serves as a trap in the off-gas scrubber, as a receiver for transfers into the system, and as a trap in the vacuum system.

Feed System

Solutions are drawn into the apparatus from polyethylene bottles that serve as reservoirs. These bottles are located inside the cell and are filled from the outside. Corrosive reagents are followed by a rinse. Design of the equipment allows any vessel to be charged through any external feed line, but with only one solution at a time.

Dissolver

Figures 2, 16, and 17 show the features of the dissolver assembly. The dissolver can accommodate a dissolution yielding as much as 2000 ml of solution, and the dissolving liquid can be trickled over the slug or added batch-wise. A complete slug would be dissolved in two or more steps. The inherent flexibility of the system was illustrated by the remote replacement of the Glas-Col heater in the middle of a dissolution in less than an hour and without contamination of the equipment.

Scrubber System

The exhaust system operates under sufficient vacuum to keep the dissolver below atmospheric pressure. The off-gases are scrubbed by dispersal in appropriate media. The off-gas scrubbers are equipped with "permanent" feed and withdrawal ("slurp") lines fitted at each end with valve-joints. Figures 3 and 14 show some of the details.

Filtration

The filtration equipment shown in Figure 4 operates with two types of fritted glass filters. The filter stick is used for ordinary purposes such as the clarification of dissolver liquid, while the pancake filter, which is precoated with asbestos, is required for such material as MnO_2.

Ion Exchange

Figure 6 shows the column system for ion exchange, including the column feed vessel that is used for make-up and storage.

OPERATIONS

Liquid Storage

Nearly every vessel can be used for storage of liquids. Up to 4500 ml can be stored in the product storage vessel. The lines and vessels are connected in such a manner that each line normally carries flow in only one direction, so that suck-backs cannot occur.

Sampling

Sampling is accomplished by the diaphragm bottle technique. The bottle is first evacuated by impaling it on a hypodermic needle and withdrawing air with a syringe. The evacuated bottle is then impaled on another hypodermic needle which extends into the liquid to be sampled. Figure 8 shows the details of a sample needle.

Radioactive Transfers

Radioactive solutions are transferred to and from shielded containers, or "casks", by a combination of evacuation and liquid displacement. Figure 5 shows the equipment used inside the isolation box.

Cooling

The condenser is cooled conventionally with water. The concentric bulb construction, evident in Figure 2, assures efficient removal of vapors from the noncondensable off-gases. All other cooling is provided by Freon-11, which boils at 23.5°C. The Freon can be directed in the liquid phase to any part of the box, and where necessary the secondary container provides a bath for the vessel to be cooled.

Vacuum

Vacuum is supplied by a small pump and by the building off-gas exhaust system. Only one vessel can be evacuated at a time with the roving line, unless special but simple provision is made for unusual requirements.

Waste Disposal

Liquid waste is first drawn into the slurp vessel either directly from the dissolver or via the slurp manifold which includes roving connections for reaching any part of the box. The slurp vessel empties by gravity to the building high-level drain. Figure 7 shows the details.

Solid waste, usually discarded equipment, is placed in cartons and polyethylene bags and finally in a concrete drum for burial.

Contamination Control

The basic principles of containment have been realized, as indicated by surveys of the cell during and after seven dissolutions and associated operations. Personnel exposure did not approach the tolerance level and no contamination of the cell could be found, except on the covered jaws of the General Mills Manipulator. With no source material present, the general activity level of the cell was 100-200 mr/hr with one spot of 1 R/hr at the slurp vessel.

Following the first period, five runs were made in which about 5000 curies of mixed beta-gamma activity were processed in two and one-half months. For the first time it was necessary to perform batch solvent extractions in open centrifuge cones. Even though internal contamination of the box was greatly increased, a survey following this second period showed a maximum of only 8000 counts per minute of smearable beta-gamma contamination in the cell. After three days of remote cleanup inside the box, the radiation field outside was brought down to 1 R/hr at one foot from the side of the box.

After eighteen months of use, the apparatus was removed so that the cell could be put to other use. Since interior contamination would hamper the necessary repairs and the addition of new features to meet future requirements, the entire assembly was sent to the burial ground. During the lifetime of the apparatus, a total of 13,000 beta-gamma curies of radioactive materials had been converted into solutions and processed as required. At the time the box was removed from the cell, the surface read 5 R/hr at contact and had a maximum of 3000 counts/minute of smearable contamination. No special precautions were required for final disposal to the burial ground, and complete cell cleanup was achieved in one week.

Breakage

No breakage of glassware occurred during the first seven dissolution runs followed by other work in the apparatus. Two heaters that were improperly used were burned out. Radiation caused a slight browning of some vessels, and vessels made of sections of graduated cylinders were more susceptible to browning than Pyrex ware. A few glass-plastic seals on replaceable connectors loosened somewhat but were still operable. There was no apparent damage of the plastic from the general exposure of about 10^6R.

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APPENDIX

COMMONLY USED MATERIALS OF CONSTRUCTION

Ball Joints and Clamps

Arthur H. Thomas design. Size 12/2 for general use; joints should be specially selected for valve action, otherwise about ten per cent may leak slightly.

Size 12/5 for off-gas system, disconnect points, vessel entry holes.

Size 18/7 and 18/9 for dissolver and drain connections.

Polyethylene Tubing

Auburn Button Works is one supplier.

0.114" O.D. by 0.062" I.D. "spaghetti" for connecting hypodermic tubing.

0.114" O.D. by 0.082" I.D. "spaghetti" for general use.

0.175" O.D. by 0.125" I.D. "macaroni" for reagents, slurp.

0.375" O.D. by 0.250" I.D. for drain line.

Tygon Tubing

Type B-44-3 for acids, R-3603 for alkali, either for general use.

1. 7/32" O.D. by 3/32" I.D. for inside fitting "spaghetti" to glass.
2. 1/4" O.D. by 1/8" I.D. for use with (3) below in connecting "macaroni" to glass.
3. 3/8" O.D. by 1/4" I.D. for use with (2) above and for pinch clamping.
4. 5/16" O.D. by 3/16" I.D. for off-gas lines, general use, and with (2) above.

Stainless Steel Tubing

J. Bishop and Company is one supplier.

0.065" O.D. by 0.009" wall, hardened, for hypodermic tubing

3/16" O.D. by 0.035" wall for dissolver dip tubes; with (2) above fits 18/7 joints.

3/32" O.D. by 0.010" wall for "stiff spaghetti".

Dissolver Parts

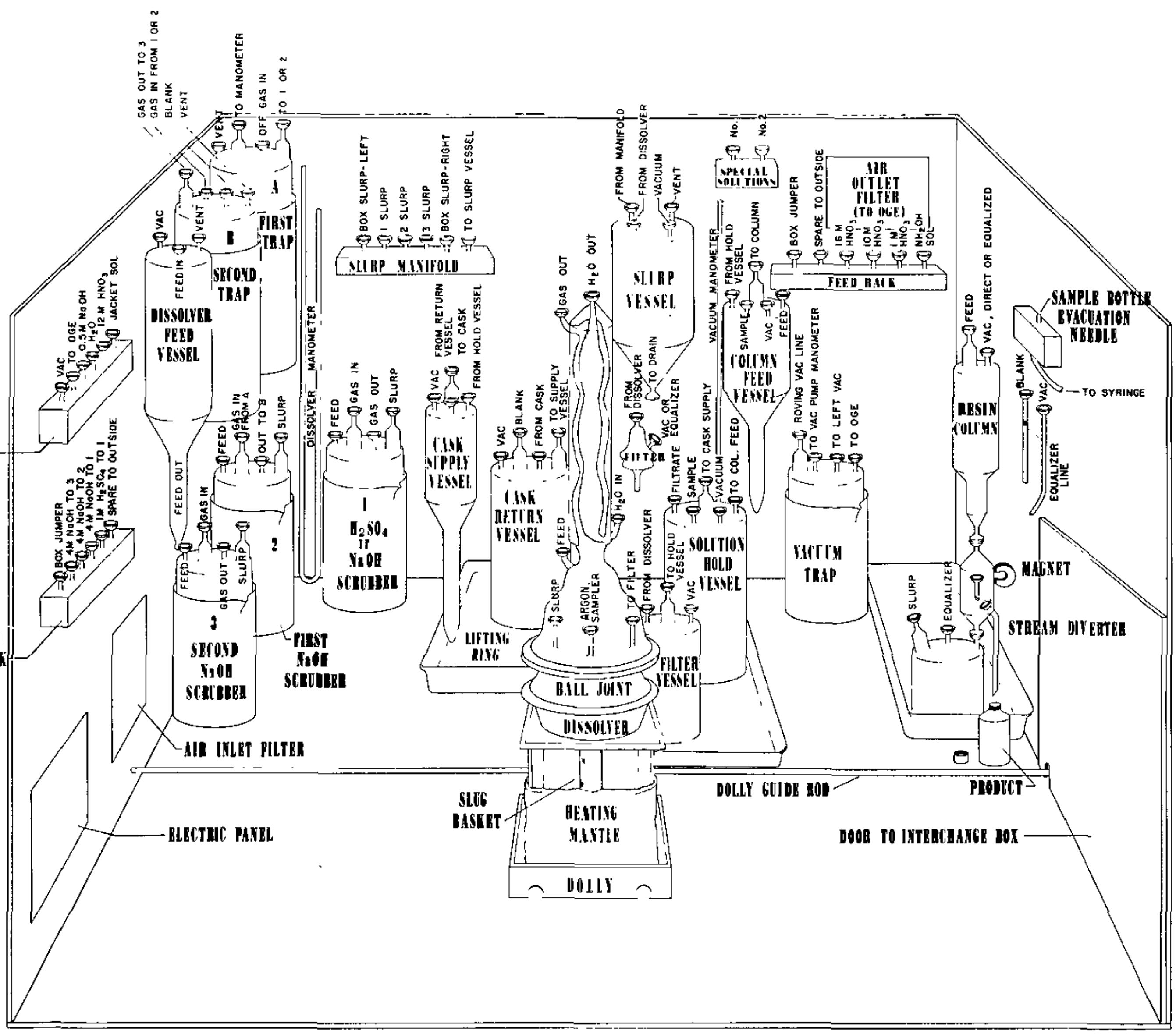
Glassware - Ace Glass Company Drawing number F-2463; vessel is Part A, top section is Part B, double-walled vessel for vapor heating (not used or discussed herein) is Part C. Lifting ring is Ace Glass catalogue number 6496.

Standard 1-liter Glass-Col heater fits a stainless steel beaker of eight inches diameter.

Lifting Cables

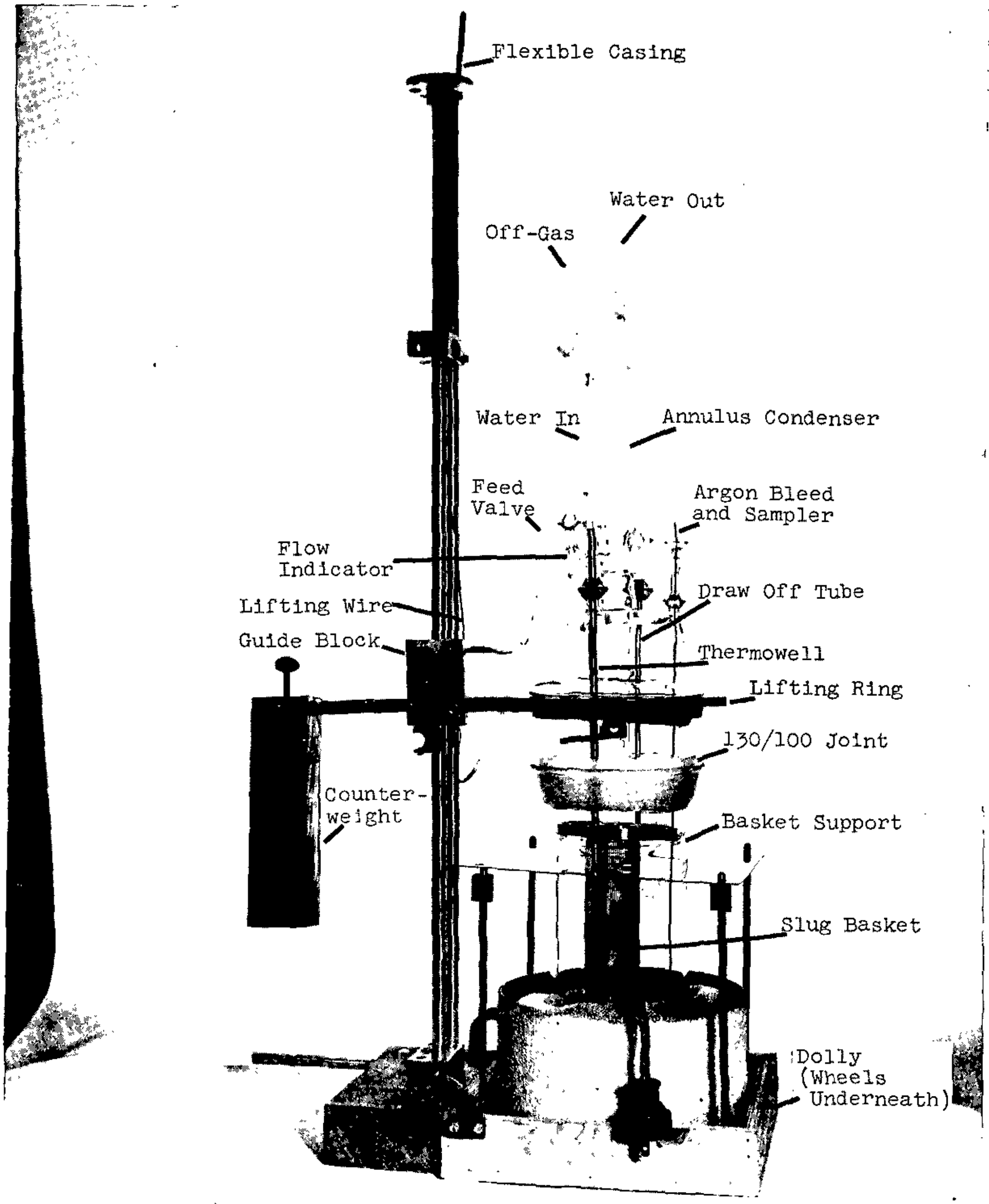
These are locking throttle control cables made by Buffalo Springfield Roller Company, catalogue number 19260267.

FIGURE 1



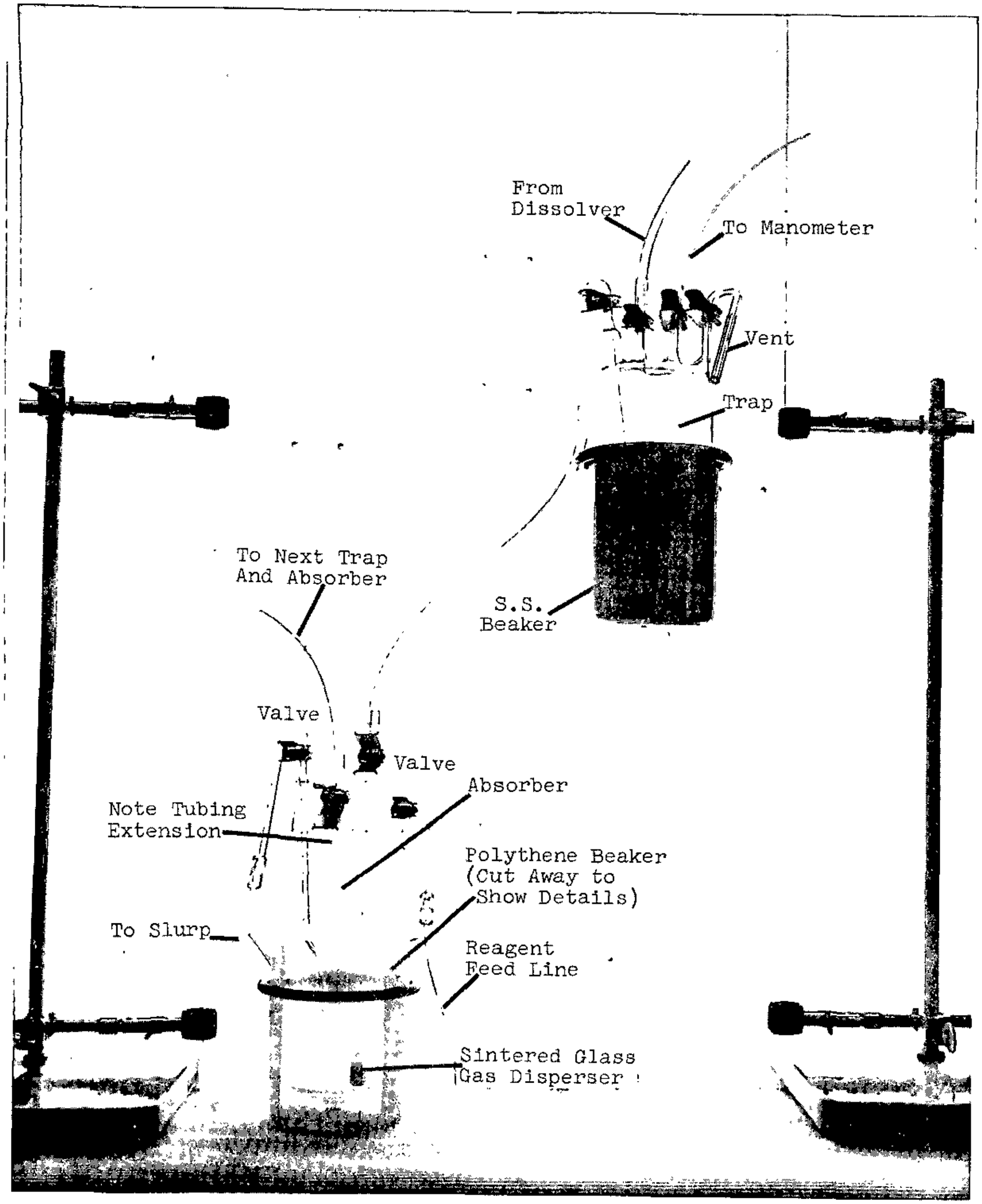
EQUIPMENT LAYOUT

FIGURE 2



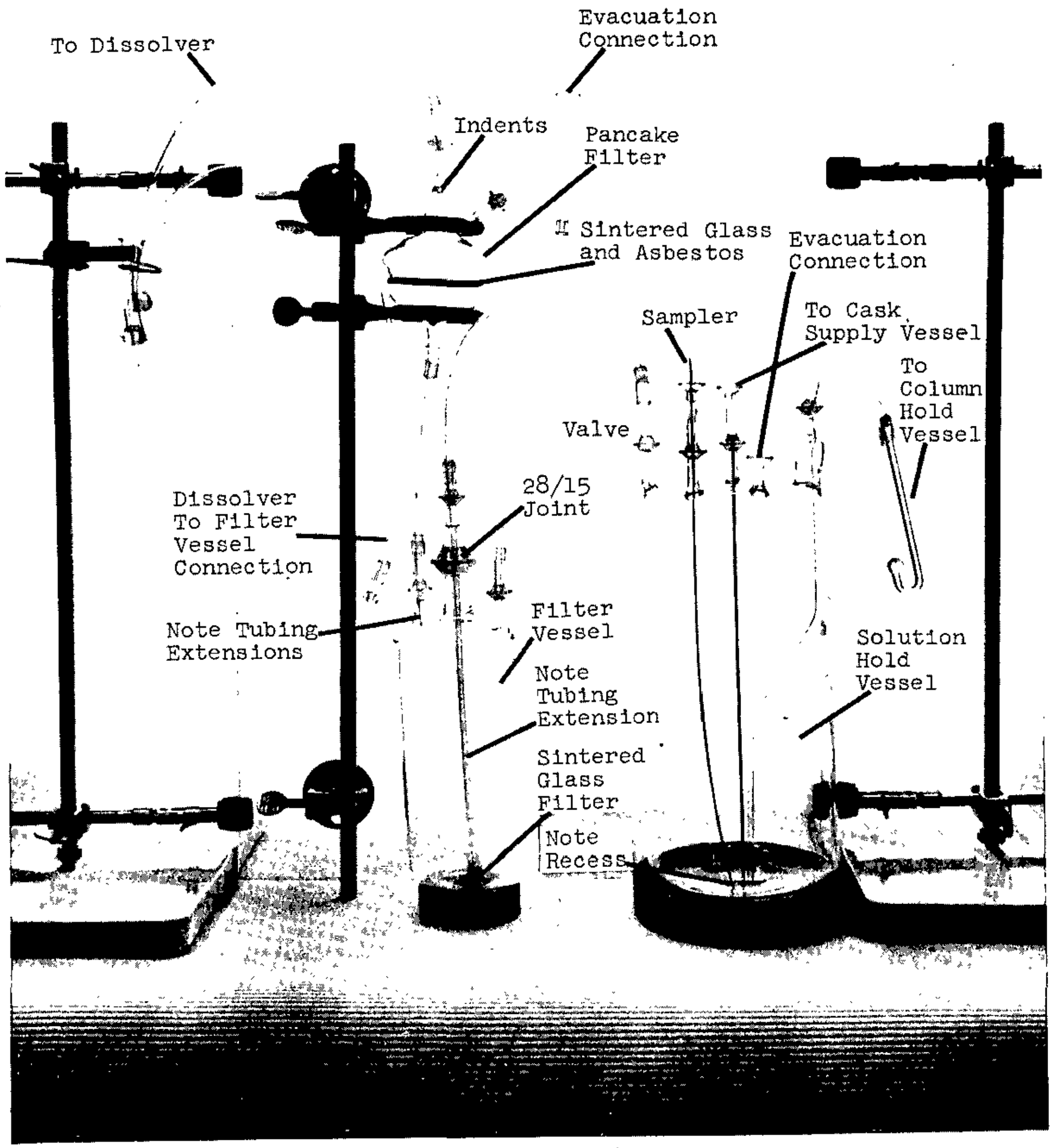
DISSOLVER ASSEMBLY

FIGURE 3



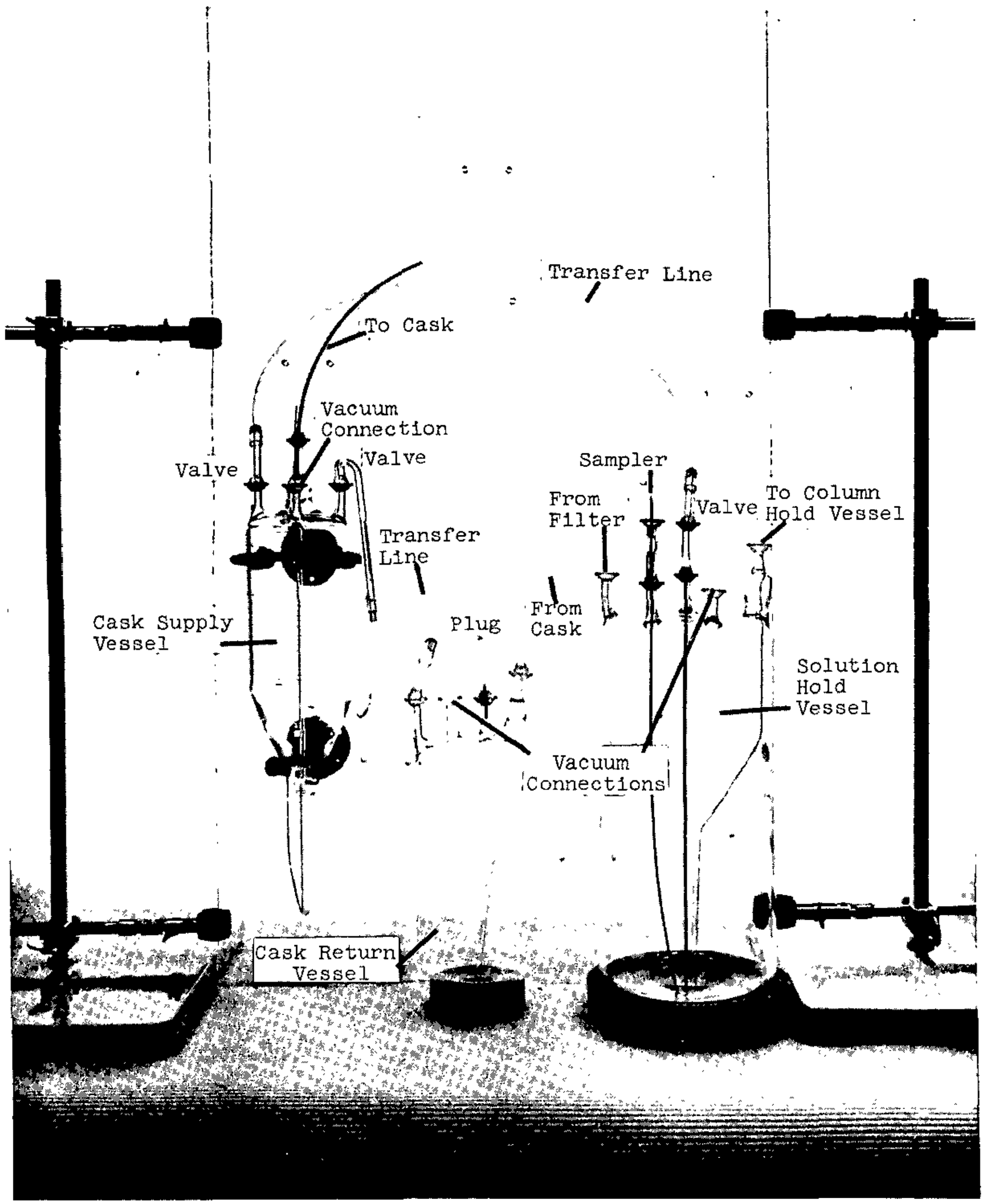
OFF-GAS SCRUBBER

FIGURE 4



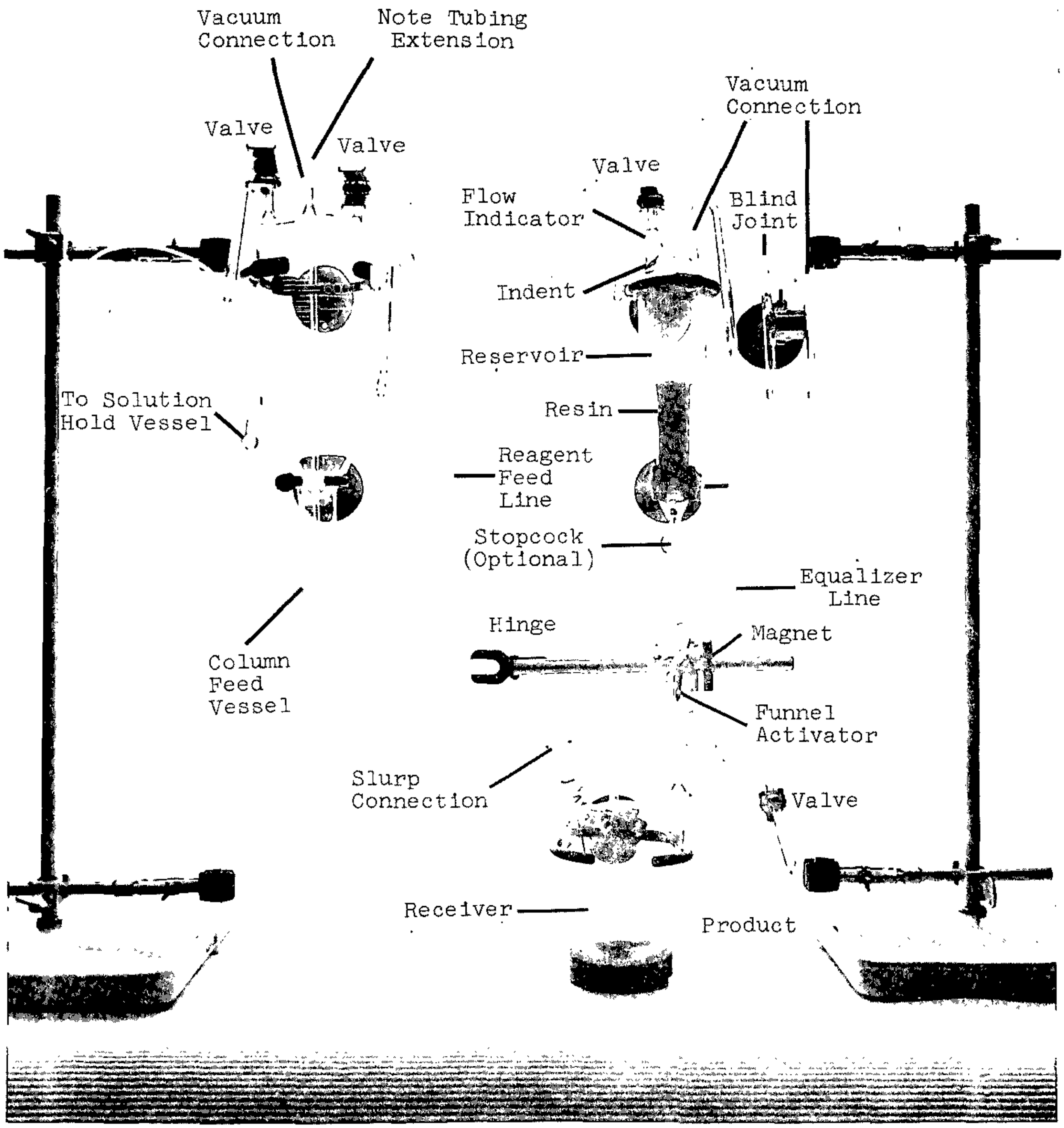
FILTRATION ASSEMBLY

FIGURE 5



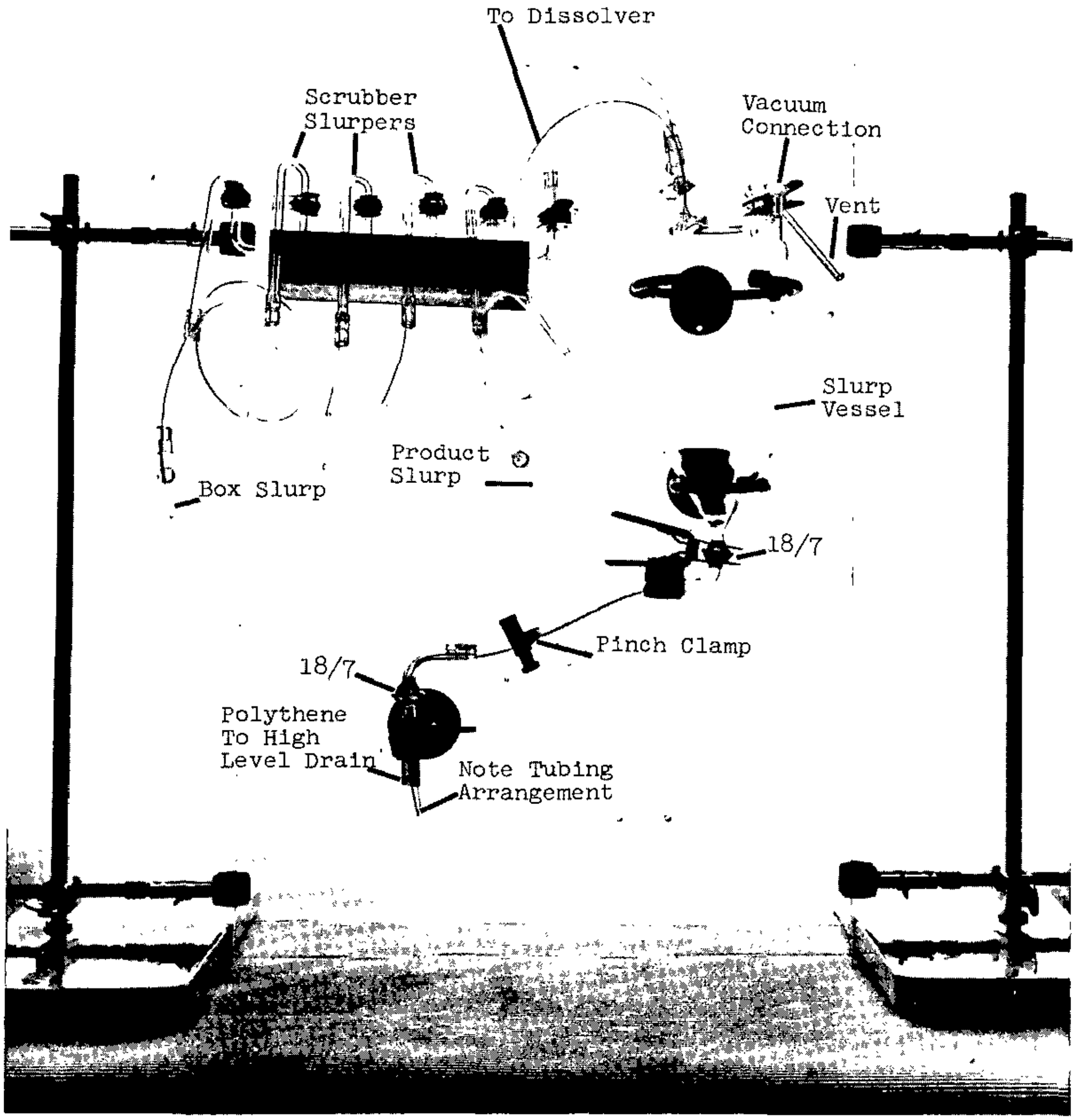
CASK TRANSFER ASSEMBLY

FIGURE 6



ION EXCHANGE ASSEMBLY

FIGURE 7

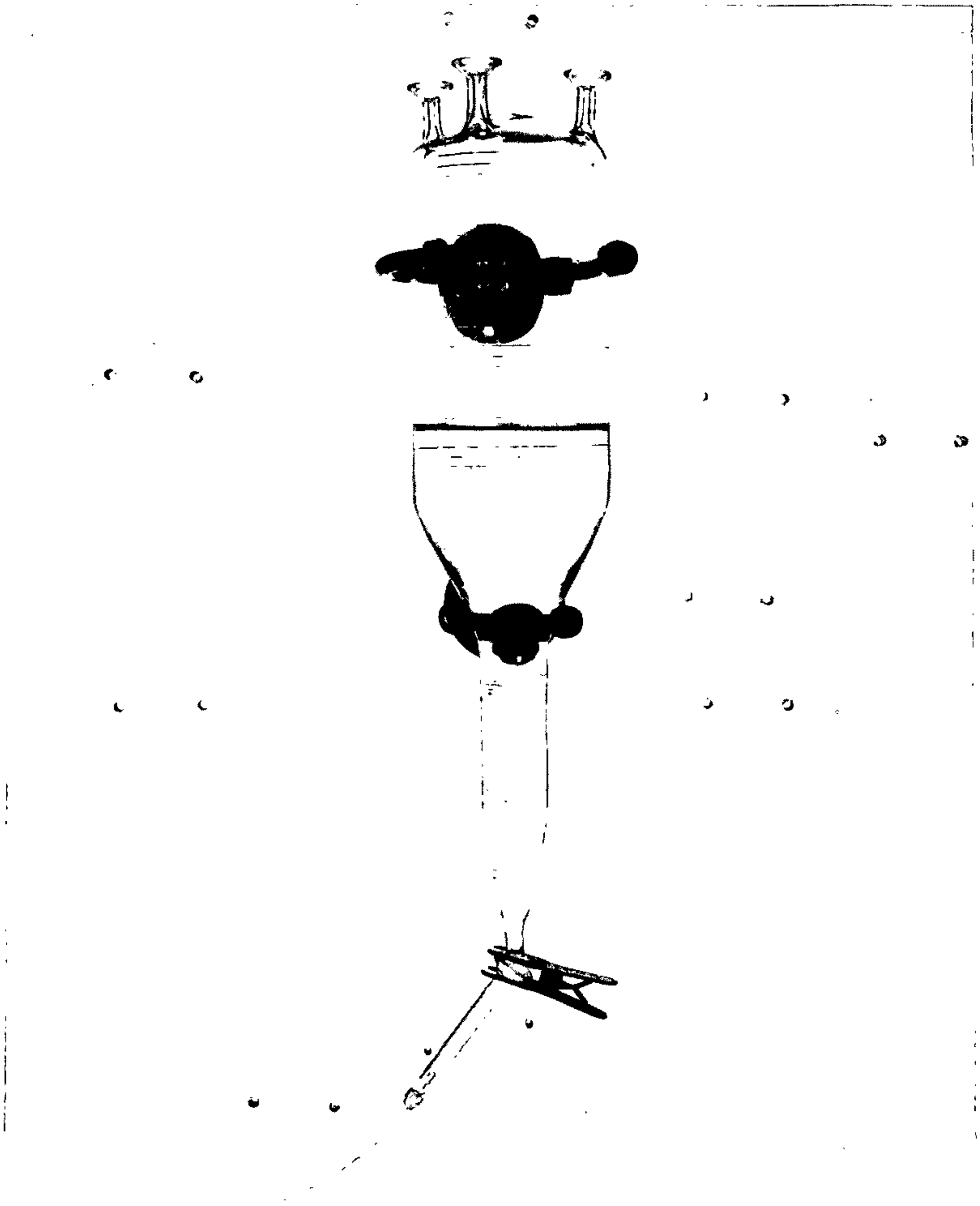


LIQUID WASTE DISPOSAL SYSTEM

FIGURE 8

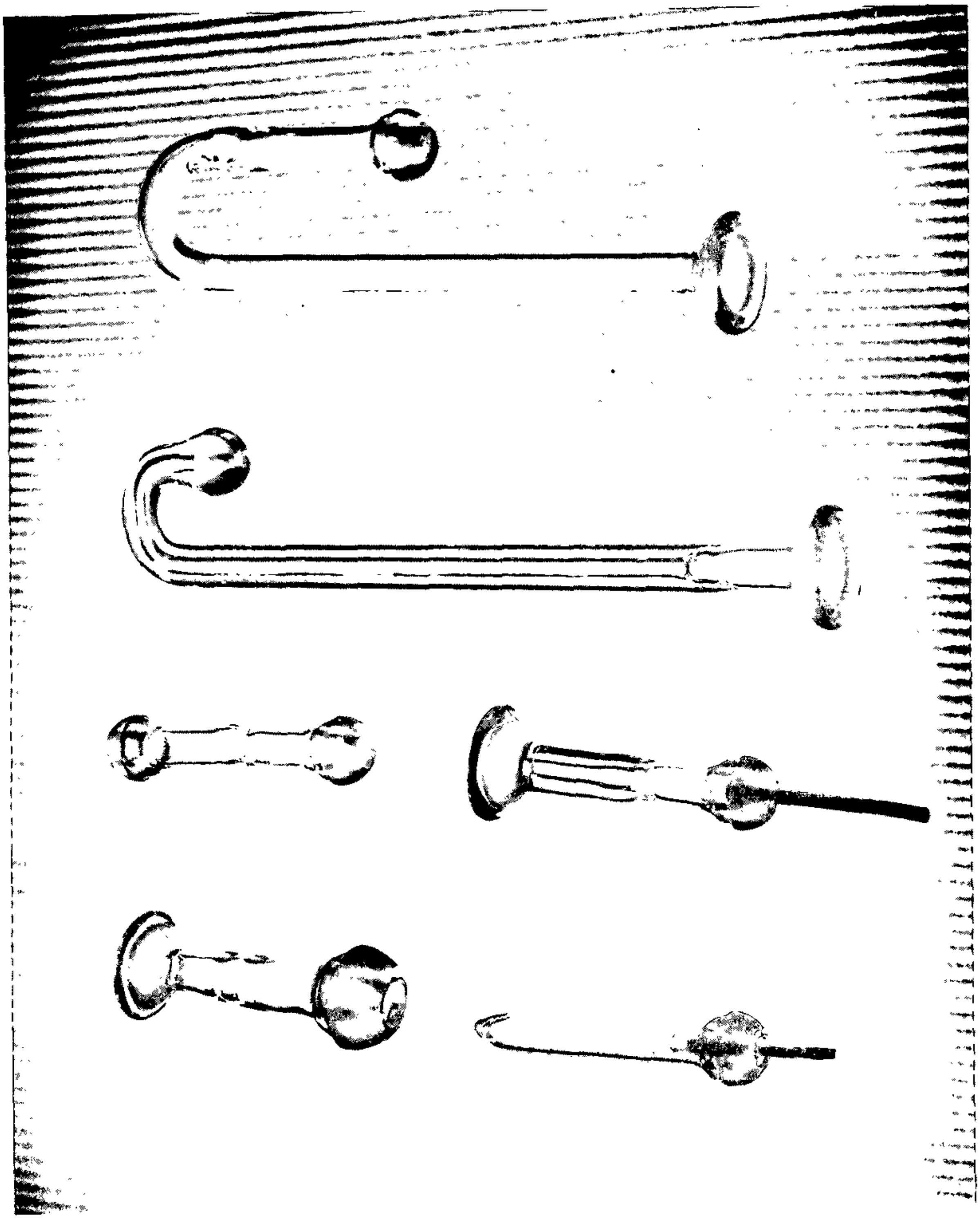
SAMPLER

FIGURE 9



VALVE ACTION OF 12/2 JOINTS

FIGURE 10

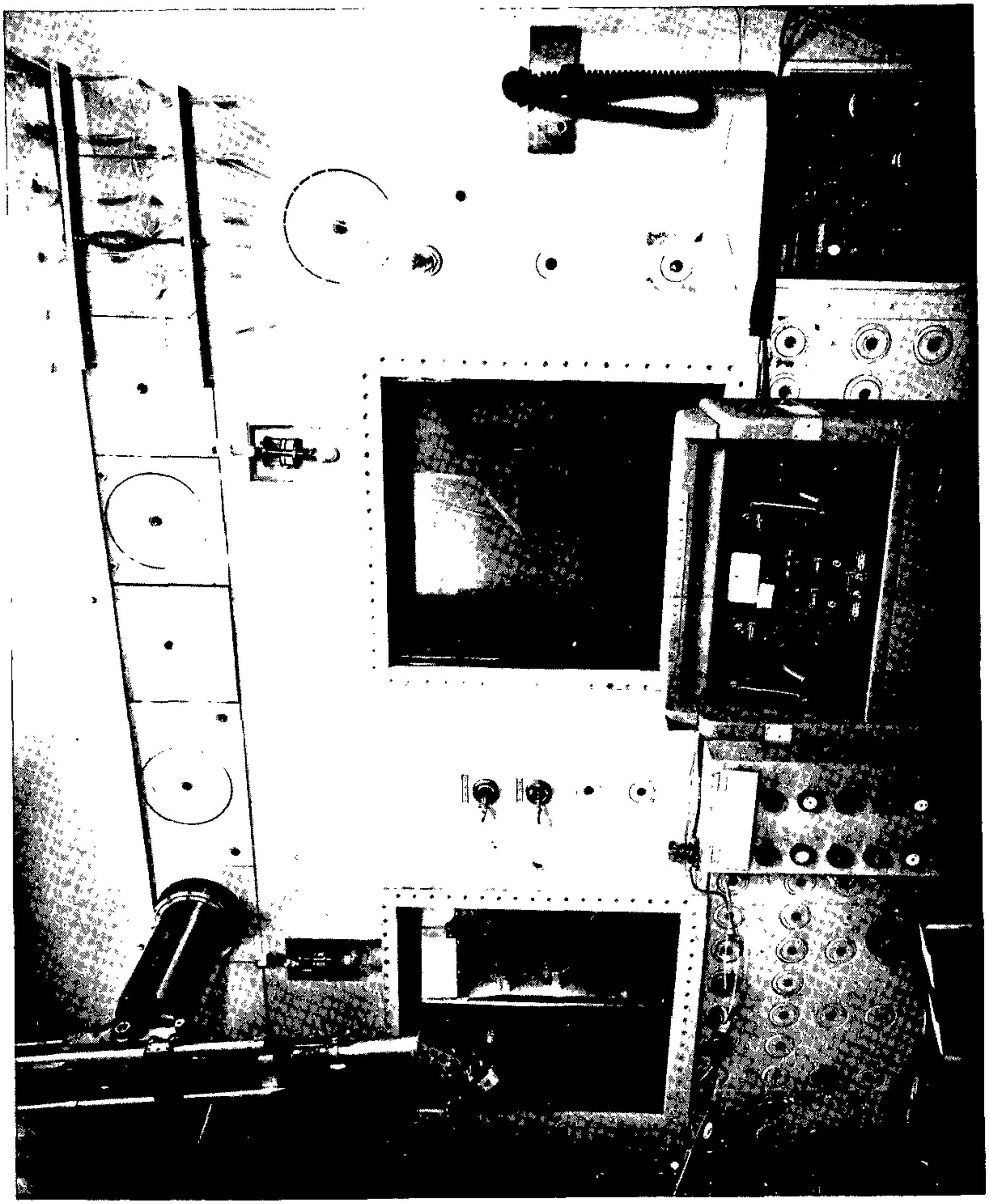


SOME ADAPTERS

FIGURE 11

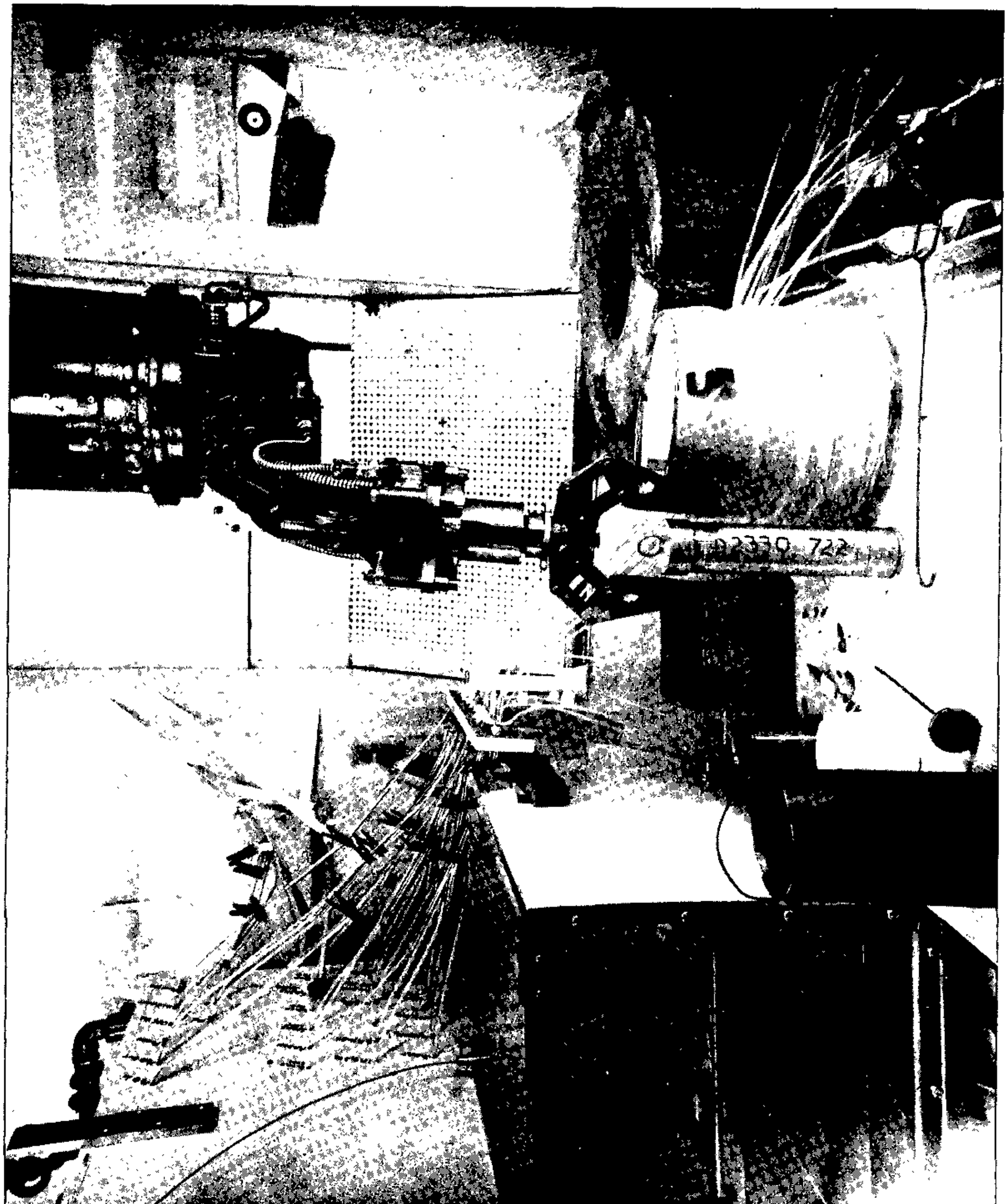
CLAMP DETAILS

FIGURE 12



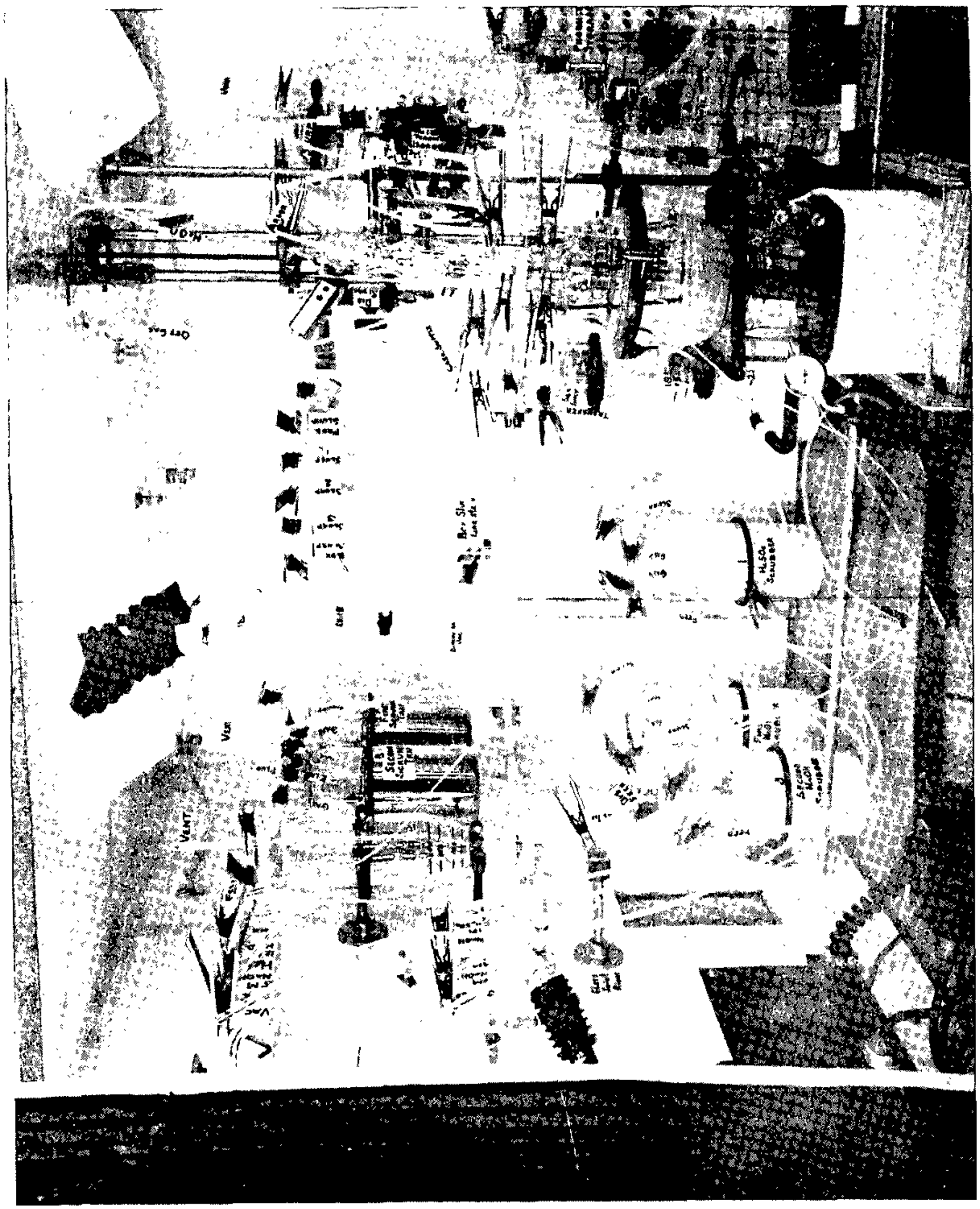
FRONT OF CELL

FIGURE 13



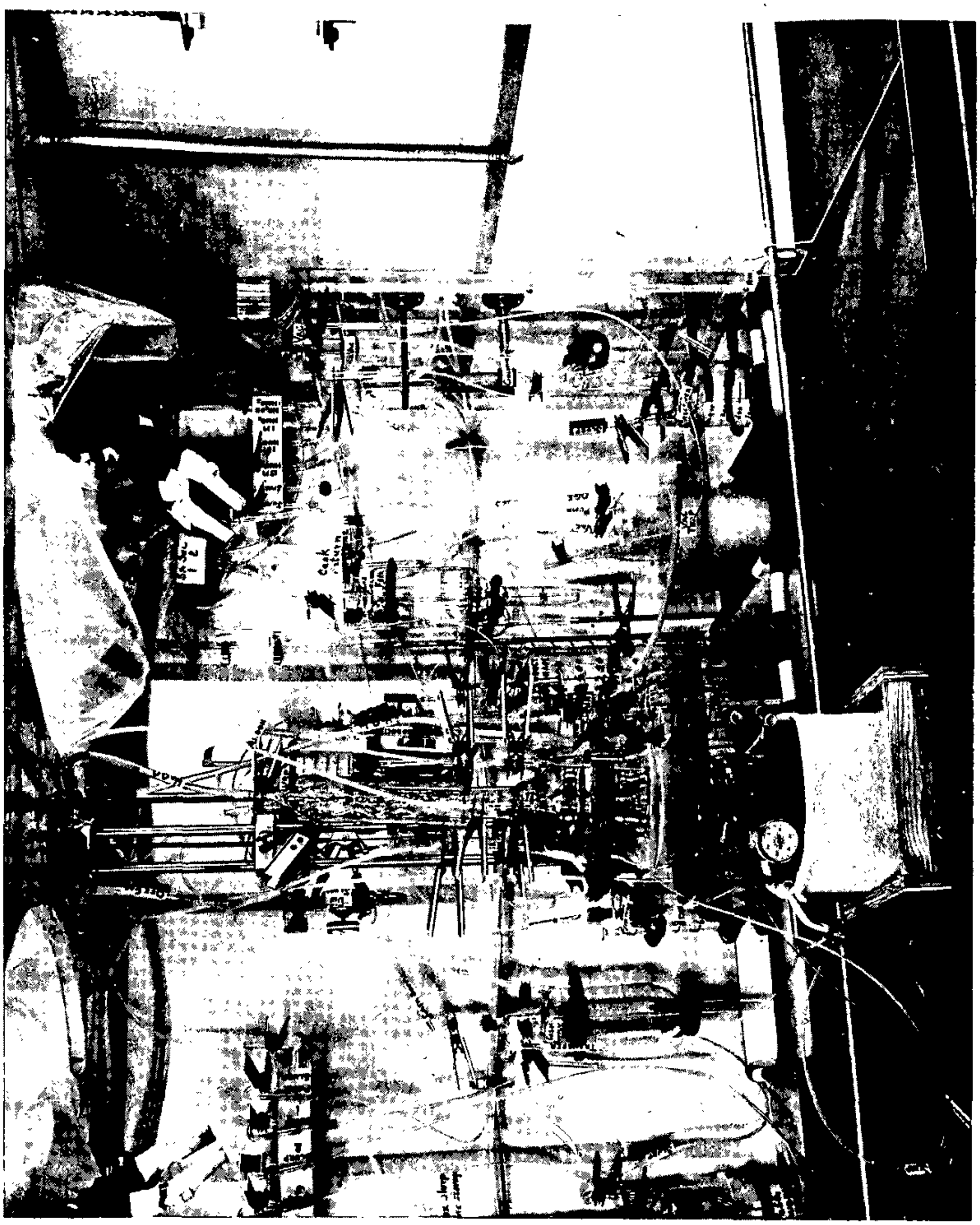
VIEW THROUGH RIGHT WINDOW

FIGURE 14



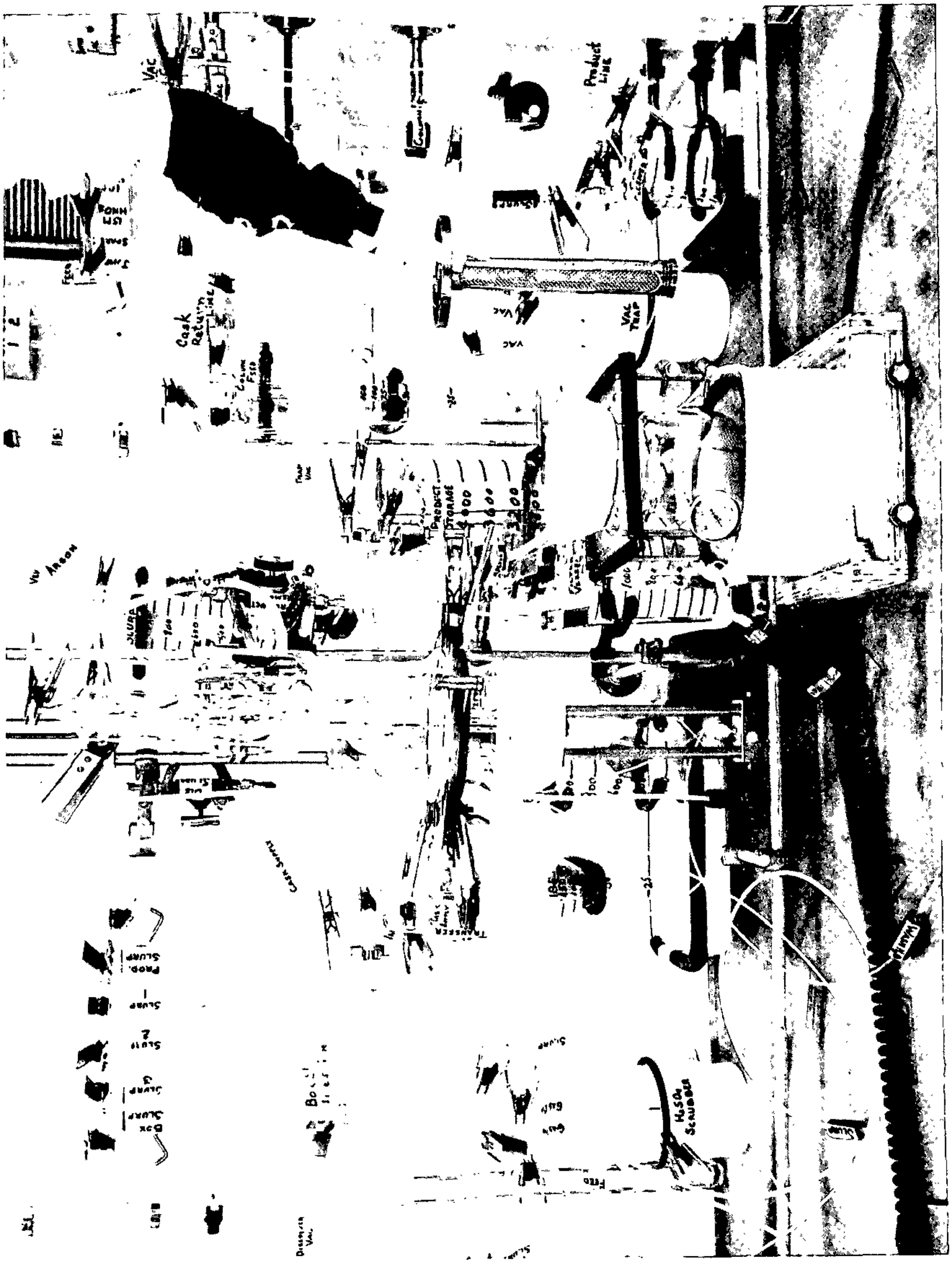
LEFT SIDE OF SYSTEM

FIGURE 15



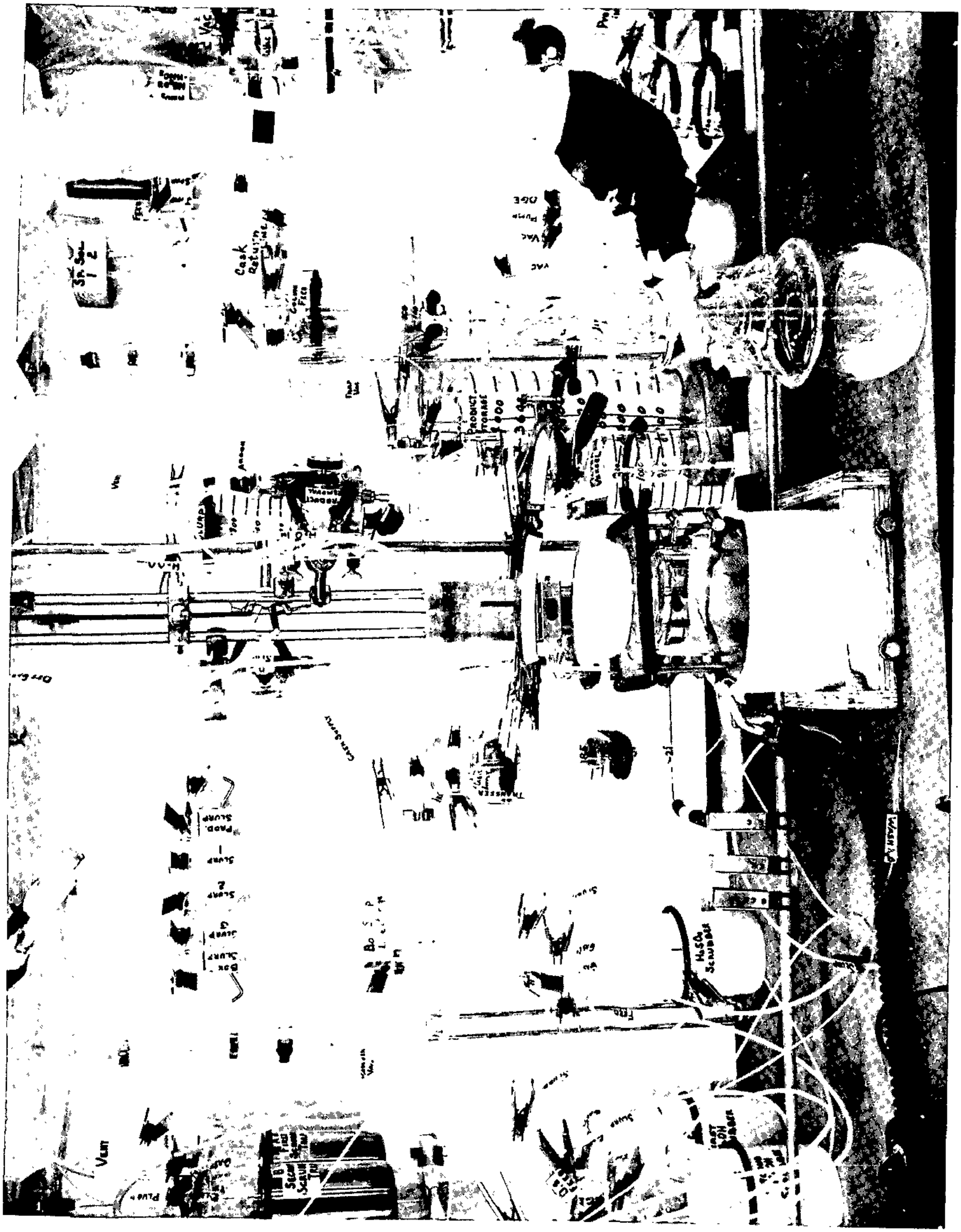
RIGHT SIDE OF SYSTEM

FIGURE 16



LOADING THE DISSOLVER

FIGURE 17



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