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DEVELOPMENTS IN CONTINUOUS ION EXCHANGE EQUIPMENT FOR AEC APPLICATIONS

J. T. Roberts

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ORNL-2504
Chemistry - Separation Processes for
Plutonium and Uranium

Contract No. W-7405-eng-26

CHEMICAL TECHNOLOGY DIVISION

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J. T. Roberts

DATE ISSUED

MAY 21 1958

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ABSTRACT*

Operational aspects of fixed-bed and continuous-countercurrent ion exchange are discussed and several types of continuous ion exchange contactors are described. The ORNL-Higgins contactor is described in detail.

*Revised version of paper presented at the annual meeting of the Engineering Liaison Program of the Union Carbide Corporation in New York City, October 14 through 16, 1957.

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INTRODUCTION

Ion exchange as a physical-chemical phenomenon has been recognized for over a century. As a chemical engineering unit operation, of the family of diffusional operations, mass transfer branch, it has been in use for a half-century but restricted almost exclusively to water softening until the late nineteen-thirties. Only in recent years has it achieved enough importance to receive more than passing mention in chemical engineering textbooks.

Since the ion exchange material is physically a solid (neglecting the recently developed liquid exchangers) it is almost always used in fixed beds. The operation then is a "percolation" operation, such as gas adsorption, chromatography, and regenerative heat exchange, in which mass or heat transfer is carried out between a flowing fluid and a stationary bed of granular solids. These are unsteady-state operations, with concentrations (or temperatures) in the two phases varying with time and with position in the bed, usually in a cyclic manner. Compared to continuous countercurrent steady-state operation, fixed-bed operation is more difficult to analyze mathematically and, in principle, more trouble to fit into continuous chemical processing flowsheets and less efficient in utilization of ion exchange material and chemical reagents. In practice, however, continuous countercurrent solid-liquid contactors have presented difficult mechanical problems associated with moving the solid phase, and most of the proposed designs, dating back to the nineteen-twenties, have seriously compromised either the high throughput capacity or the low theoretical stage height characteristics of the fixed bed. Technological breakthroughs in the last few years, however, indicate that continuous ion exchange equipment has reached engineering maturity. This paper covers briefly several types of continuous ion exchange contactors and discusses in some detail one developed at Oak Ridge National Laboratory.

OPERATIONAL ASPECTS OF ION EXCHANGE

Cyclic, fixed-bed operation involves alternating sorption and desorption (or "loading" and "elution") periods. Two or more beds can be operated on a staggered cycle to permit continuous feed and product withdrawal, but, even so, the composition of the exit streams will vary considerably during the cycle. Figure 1 is a cutaway view of a typical water-softening installation. Most ion exchange equipment used today is of this pressure percolation type, similar to sand pressure filters. Such equipment may be manually or automatically operated. Figure 2 shows fixed bed and continuous countercurrent methods of carrying out simple sorption-desorption processes by ion exchange. A fixed bed is fed with solution to be processed until its effective capacity for the ion being sorbed is exhausted, i.e., until the concentration of this ion in the effluent stream rises to an objectionable level. The ion is then desorbed, regenerating the resin, by eluting the bed with a suitable electrolyte. Common practice is to carry out both the sorption and desorption steps downflow. This procedure requires essentially complete desorption of the ion being processed in order to prevent excessive leakage during the succeeding sorption cycle. In cases where economy of

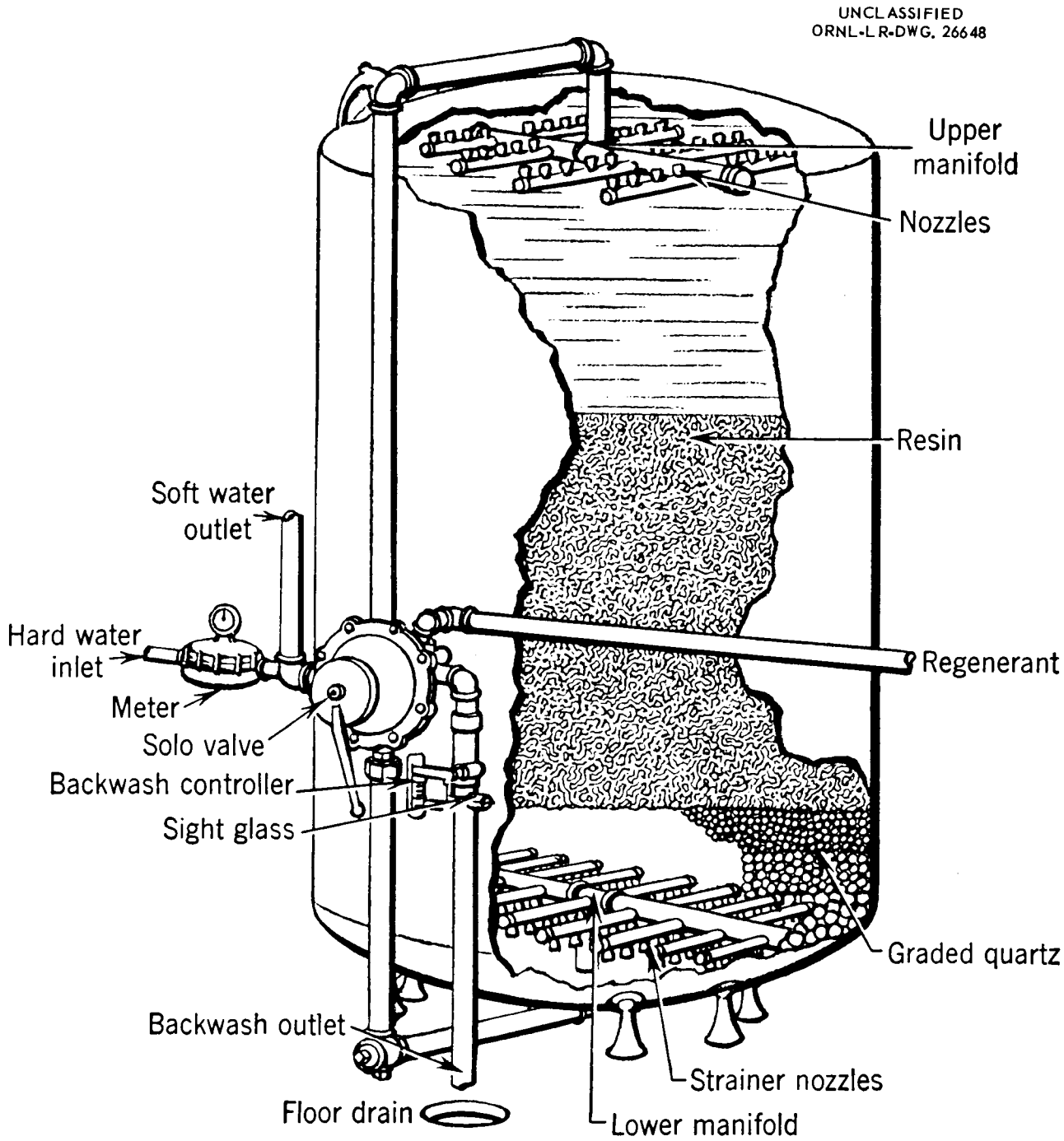

Fig. 1. Typical Water-softening Installation. (Courtesy Rohm and Haas Co.)

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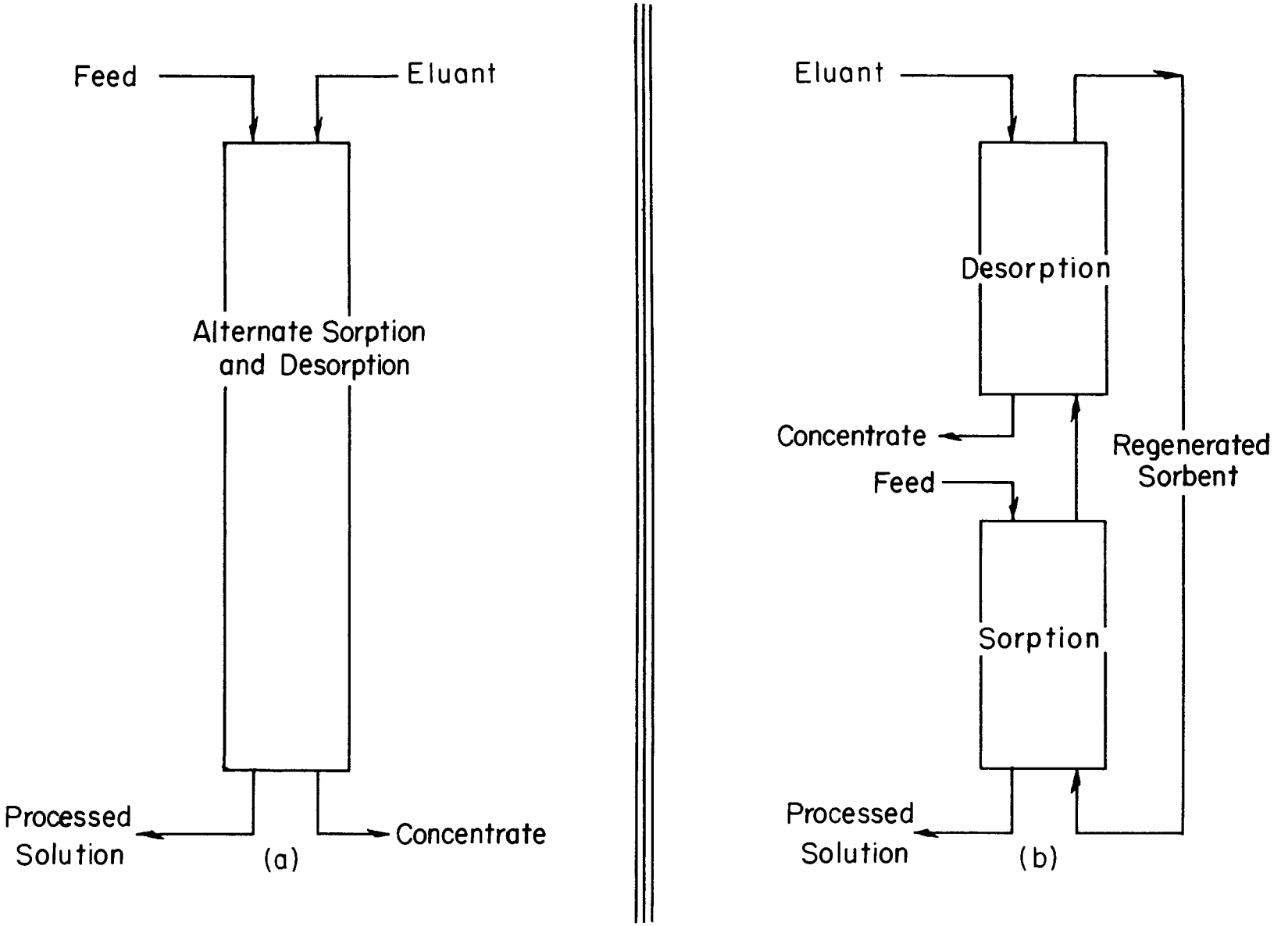


Fig. 2. (a) Fixed-bed and (b) Continuous-Countercurrent Ion Exchange.

eluting reagent is important, the tails from one desorption cycle can be used as the initial eluant in the next desorption cycle (this is called "split elution"). An even better method of operation, in principle, is counterflow, in which the sorption and desorption flows are in opposite directions, since complete regeneration is unnecessary. The advantage of downflow elution is that throughput rates are limited only by the allowable pressure drop while any upflow step requires precautions to prevent fluidization of the resin.

The same type of sorption-desorption process could be carried out continuously in a two-section countercurrent contactor. In the sorption section the regenerated resin would flow countercurrently to the process feed. In the desorption section the resin would flow counter to the regenerant solution. At steady state the processed stream and the concentrate stream would be of constant composition, in contrast with the analogous streams from the fixed bed, where concentrations vary from nearly zero to a peak value and back again. In Fig. 2 the resin is shown flowing up through the contactor, as it does in the ORNL-Higgins contactor. For a gravity-operated moving bed the direction of the flows would be reversed. For a mixer-settler type contactor the flows might well be horizontal rather than vertical.

Practical sorption-desorption processes may be more complicated than the simple case illustrated. Additional feed streams (e.g., washes) may be required between the two major steps. After each fixed bed cycle, or even after each step in the cycle, the bed may be backwashed and resettled in order to remove any solids which may have been filtered out of the solution by the bed and assure the uniform packing prerequisite to uniform flow distribution and hence efficient operation. In general, a continuous contactor will have at least as many sections as there are influent streams, while a fixed-bed system may still consist of a single bed.

Recovery of uranium from ore leach falls in this sorption-desorption category. Uranium in low concentrations in sulfate or carbonate liquors is readily sorbed as a complex ion on an ion exchange resin, with most of the objectionable components of the liquor passing through the bed, and is subsequently eluted as a comparatively pure and concentrated solution. Conventional fixed beds require careful filtration of the leach liquor feeds to avoid plugging.

A second general category of ion exchange problems, separations, is indicated in Fig. 3. A simple separation might be carried out by feeding a fixed bed alternately with the mixture of two ions to be separated and an eluting solution. The less strongly sorbed ion will move down the bed more rapidly than the other and the effluent may be collected in (at least) two fractions. The same separation would be carried out in a four-section continuous-countercurrent contactor. This figure shows the resin moving down through the contactor. The mixed feed enters at the middle, the eluting solution enters at the bottom, and the regenerated resin is recycled from bottom to top. The separation takes place in a manner closely analogous to that in a distillation column, the separation sections above and below the feed corresponding to the enriching and stripping sections. The top and bottom sections correspond to the condenser and reboiler, and the eluting solution, part of which is recovered and part of which comes out in the top and bottom product streams, corresponds to the heat input. Three or more ions may be separated on a single fixed bed, but, in general, continuous separation of N components requires N-1 contactors.

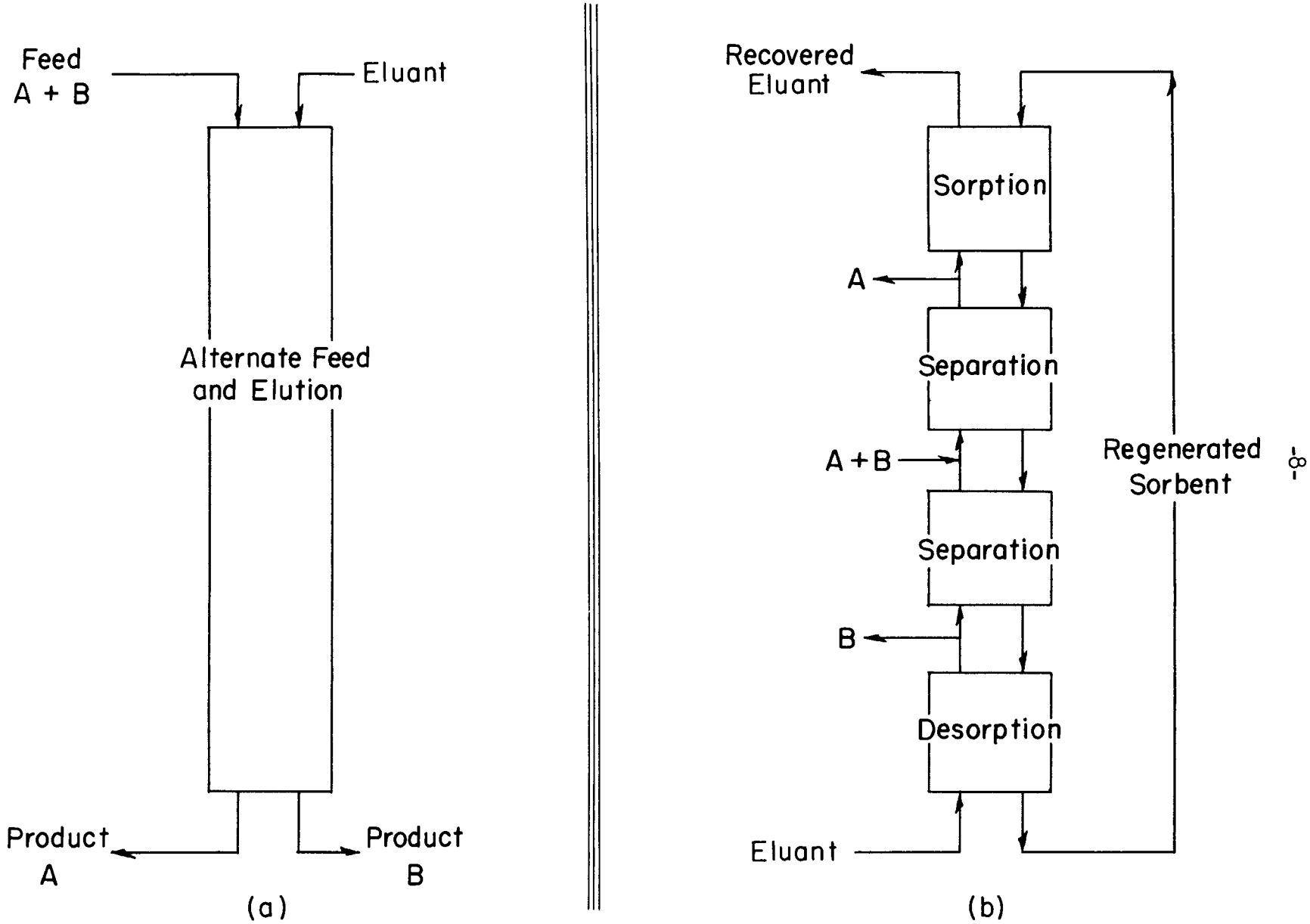


Fig. 3. Separation of Two Ions by (a) Fixed-bed and (b) Continuous-Countercurrent Ion Exchange.

CONTINUOUS ION EXCHANGE EQUIPMENT

A large number of continuous ion exchange contactors have been proposed in patent, commercial, and technical literature.[1] The earliest of these was probably in 1921, but the large majority are dated in the nineteen-fifties. Of these, this discussion will consider only five, three of which have been tested on recovery of uranium from ore leach liquors.

Dorr Contactor[2]

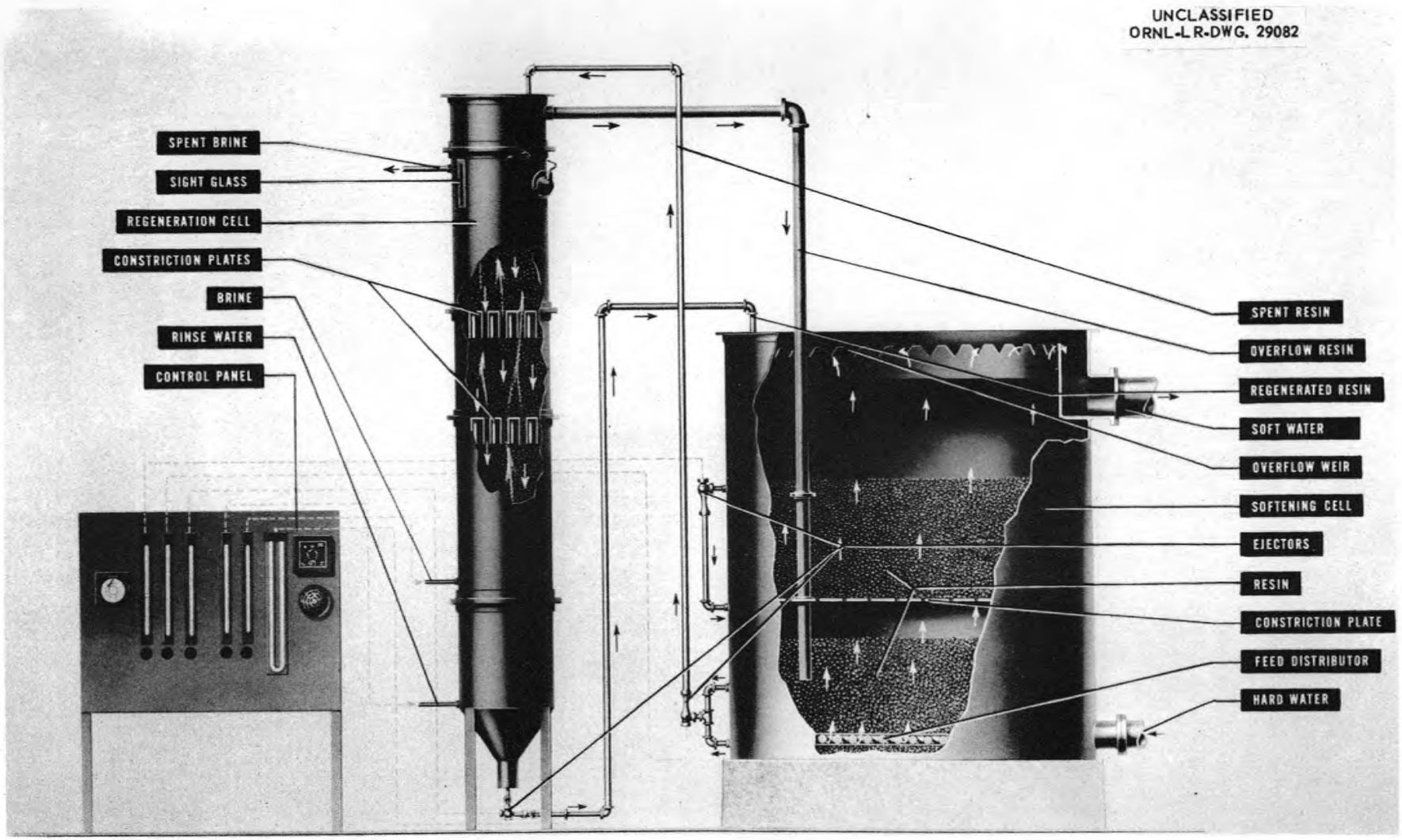
Figure 4 shows a continuous contactor put on the market by the Dorr Company a few years ago. It was reported that a 6-ft-dia water softening unit was installed at Baxter, Kansas, with a throughput capacity of 170 gal/min while reducing the hardness from 160 to 4 ppm.[1] It was also reported that only 10% of the ultimate capacity of the resin (84 cu ft) was utilized per pass. This contactor operates with solution upflow in both sections, with two fluidized beds in series in the softening (sorption) sections, and with the fluidized bed in the regeneration (desorption) section broken into several subsections by the so-called "constriction plates." The use of fluidized contact severely limits the number of stages available in a reasonable height; thus this contactor seems applicable only where equilibrium conditions permit the desired result to be accomplished with a few theoretical stages. Careful hydraulic balancing of this contactor is necessary for proper operation.

Resin-in-Basket Contactor[3,4]

Figure 5 shows a part of the Anaconda basket-contactor plant at Grants, N. M., which was developed by the AEC for resin-in pulp operation.

In the processing of western uranium ores, an oxidizing sulfuric acid leach converts the uranium to the hexavalent state, which is present in solution as an anionic sulfate complex. In this form the uranium can be removed from the leach liquor by sorption on an anion exchange resin. The uranium ores also contain 5-10% clay slimes, however, which hydrate during the leaching process and turn the leached pulp into a viscous mass. In order to avoid plugging conventional fixed ion exchange beds with these slimes, an expensive and time-consuming filtration step is necessary. The basket contactor eliminates this filtration step.

In the Anaconda plant pulp feeds to 14 series-connected, neoprene-lined steel troughs (50 x 6 x 6 ft), each trough fitted with 10 stainless steel baskets (4 x 4 x 4 ft) and each basket containing 15 cu ft of resin. The plant processes 1125 tons of ore feed per day. The baskets are dipped up and down in the trough through which the pulp flows. This vertical action keeps the resin fluidized and thus prevents the slimes from filtering out and plugging the bed. The pulp flows from trough to trough in series as the baskets oscillate at 5 cycles/min through a 15-in. stroke. Ten troughs are on sorption cycle and four on desorption at any one time. The resin does not flow with respect to the earth, but effectively continuous-countercurrent contact is achieved by moving the feed and take-off points from one trough to the next in a 14-trough time cycle.



THE DORRCO HYDRO-SOFTENER

Fig. 4. The Dorrco Continuous Hydro-Softener. (Courtesy The Dorr Co.)

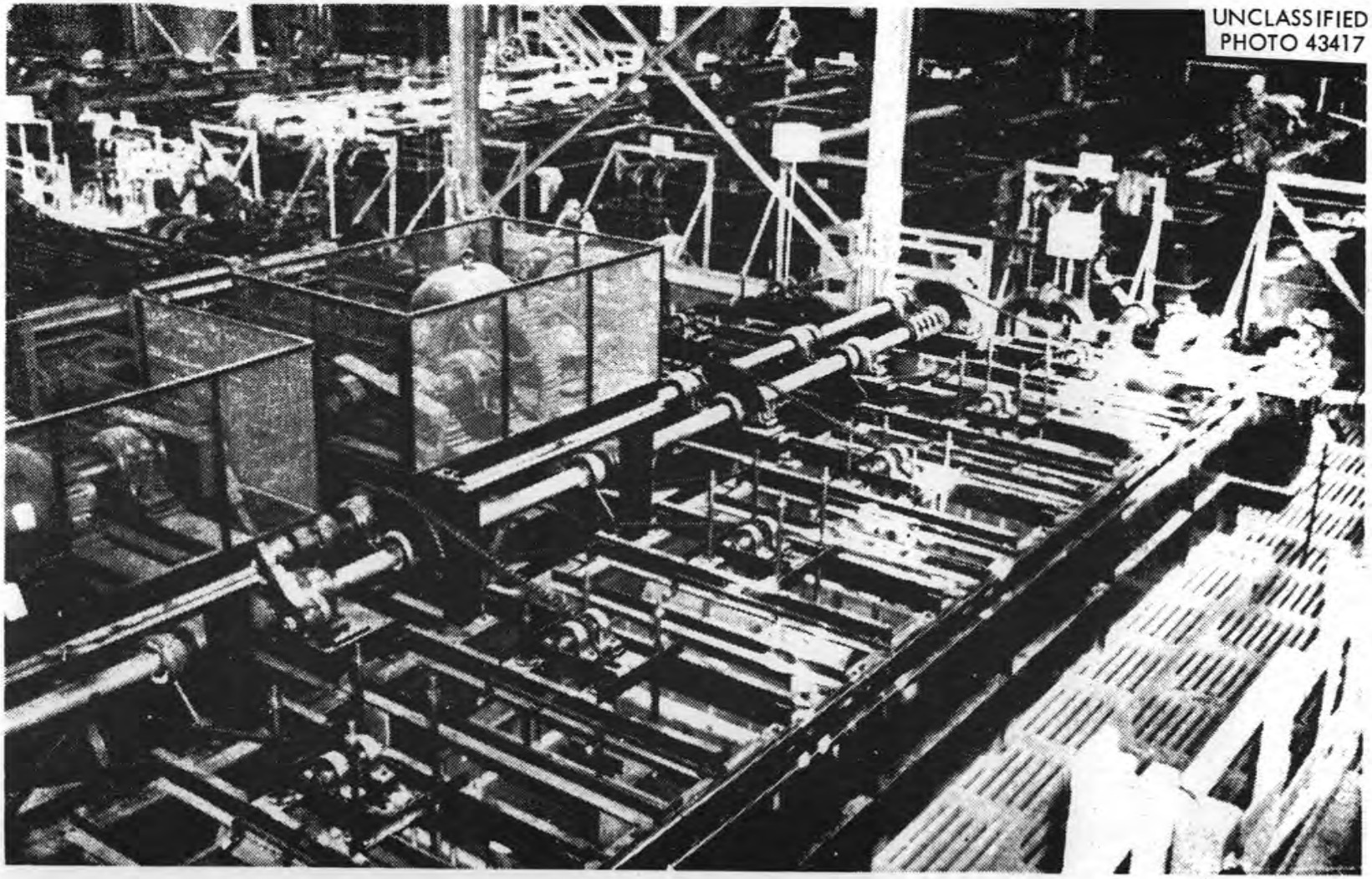


Fig. 5. Anaconda Basket-Contactor Plant, Grants, New Mexico. (Courtesy of the Anaconda Co.)

Basket resin-in-pulp is being used also in several other ore mills on the Colorado Plateau. While it has achieved its objective satisfactorily, it is basically an inefficient method of ion exchange contacting. It is also a high-capital-investment operation compared to some other systems.

Infilco Contactor[4]

Infilco, Incorporated, and Techmanix Corporation cooperated in designing and operating a resin-in-pulp uranium-recovery pilot plant using a mixer-settler contactor (Fig. 6). A modification of this contactor is now used in the Union Carbide mill at Maybelle, Colorado. This unit is based on adaptation of hydraulic techniques used in the Infilco Aero-Accelator machine for water and waste treatment, with modifications necessary for carrying out the resin-in-pulp operation without appreciable resin attrition. The pilot plant, with an ore capacity of 2 tons/day, includes six 30-in. exchange units, with a pulp capacity of 95 gal each. Four units are used for sorption of uranium and two for desorption. Resin flow between units is by air lift and gravity, pulp and eluant flow by gravity. Agitation is by air. Since the sorption and desorption stages are not dual purpose, each can be designed for its specific function. There are no moving parts. Standard 20-50 mesh resin can be used in this contactor, as opposed to the extra coarse, 10-20 mesh, grade used in the basket-type resin-in-pulp contactor. The Infilco contactor might be expected to have a contacting efficiency comparable to the Dorr contactor (better than the basket system), with perhaps some physical advantage from the horizontal arrangement of the stages. It appears to require careful balancing to control resin inventory per stage and resin flow between stages, though the pilot plant was reported to be "noncritical in adjustment."

ORNL-Jury Contactor[5-7]

Figure 7 shows schematically a dense-falling-bed contactor designed by S. H. Jury, University of Tennessee, an ORNL consultant. This contactor is similar to previously proposed gravity-operated moving-bed contactors except for the novel feature of a hydraulic ram.

Properly operated, the resin moves in the working column as a dense bed, i.e., with the particles maintaining fixed positions relative to each other. The resin is moved down the working column by gravity and by the force exerted by the hydraulic ram. It is removed from the bottom of the column and returned to the top by a jet. The principle of the hydraulic ram is that the downward frictional force exerted on the resin in the ram section by the downflowing liquid is transmitted to the working resin sections below, with fluidization thus prevented provided that the combined downward forces of the ram and of gravity exceed the upward forces exerted on the resin in the working sections by the upflowing solutions. Careful hydraulic balancing, i.e., zeroing of the indicated manometers, is necessary for proper operation of this contactor. Carefully designed low-solution-velocity take-offs are required to prevent the resin from jamming around the exits.

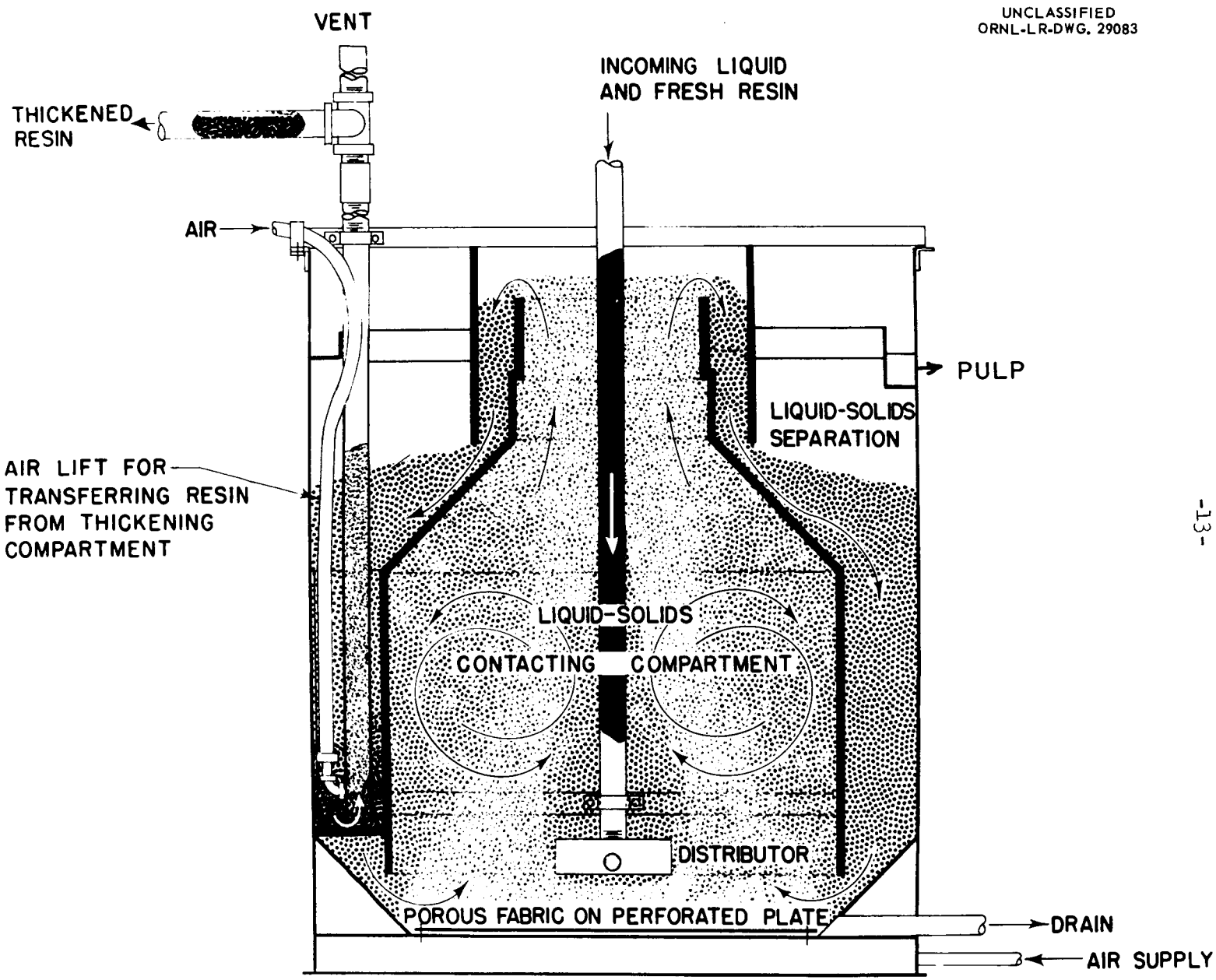


Fig. 6. Mixer-settler Contactor. (Courtesy Infilco, Inc.)

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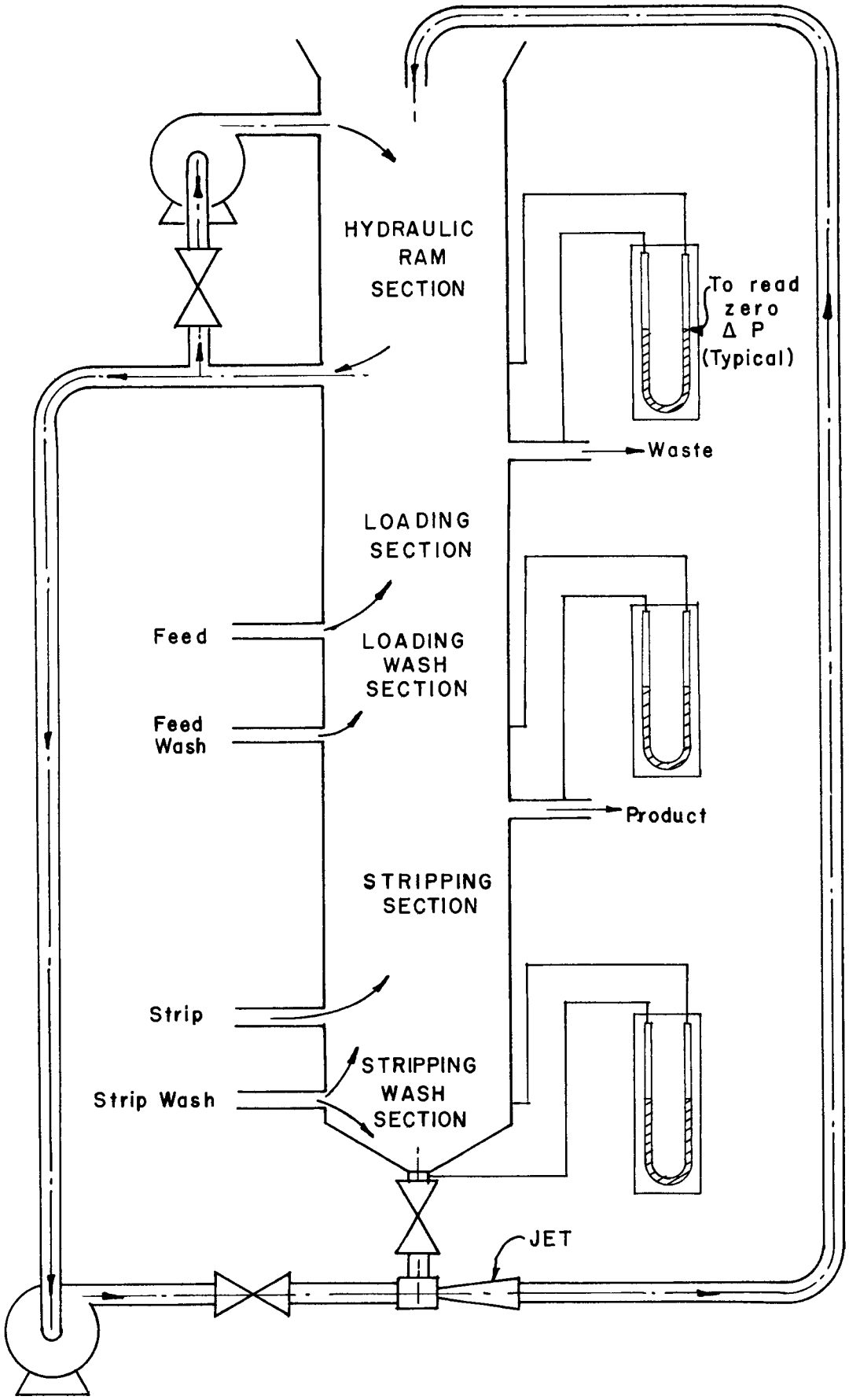

Fig. 7. Continuous Ion Exchange Contactor with Hydraulic Ram.

The Jury contactor has not yet reached the stage of development of the other contactors described in this paper, either in operating experience or in size. A 4-in.-dia experimental unit has been built at ORNL and transferred to the University of Tennessee for study, under subcontract. In principle, it is the only one of the contactors described in this paper which is both truly continuous and differentially countercurrent. As a dense-bed contactor it should, in principle, be able to operate with the low HETS usually associated with fixed-bed operation. In practice, it remains to be proved as a practical engineering device and at throughput rates competitive with fixed-bed operation. It has not been tried with resin-in-pulp operation.

THE ORNL-HIGGINS CONTACTOR

The contactor invented by I. R. Higgins[8] is described in more detail than the others since it is the one with which the author is most familiar and the one around which most of the applied ion exchange work at ORNL has centered. The Higgins contactor combines the high-throughput, low-HETS advantages of fixed-bed with the operational advantages of continuous-countercurrent flow. It has been tested in sizes up to 20 ft high and up to 3 ft dia.

Mode of Operation[9-11]

Figure 8 illustrates the mode of operation of the Higgins contactor. Strictly speaking, it is intermittent in operation rather than truly continuous, i.e., the resin and solution move alternately rather than at the same time, but in effect the distinction is unimportant. The solution flow is down against a packed bed, thus completely eliminating the danger of fluidization and providing highly efficient differential-countercurrent contact between the two phases. The solution flow period is typically several minutes long. Intermittently the bed is moved up (pulsed) a short distance relative to its length by interrupting the solution flow for several seconds and applying, hydraulically, a pressure drop across the bed. The column of resin slides as a piston in a cylinder, lubricated by its contained liquid, with the individual particles closely maintaining their positions relative to each other. The result is effectively continuous-countercurrent flow, with only small variations in stream composition, the high-throughput low-HETS features of the fixed bed being retained.

The two valves in the resin loop and the hydraulic pump are basic to the method of resin circulation. In Fig. 8 they are shown as plug valves and a hydraulic piston, which reverse their positions cyclically. Other types of valves and hydraulic actuators are discussed below. In effect, the valves act as check valves which permit the resin to move only in one direction. Thus the resin is moved around the loop with a remote pump in much the same manner as solutions are pumped by a check-valve assembly and a remote (Lapp-Pulsafeeder) diaphragm or (Milton-Roy) piston movement.

An additional valve in the middle of the working column is often a convenient means of isolating two sections without hydraulic balancing (in this contactor hydraulic balancing affects only solution flows, not the resin).

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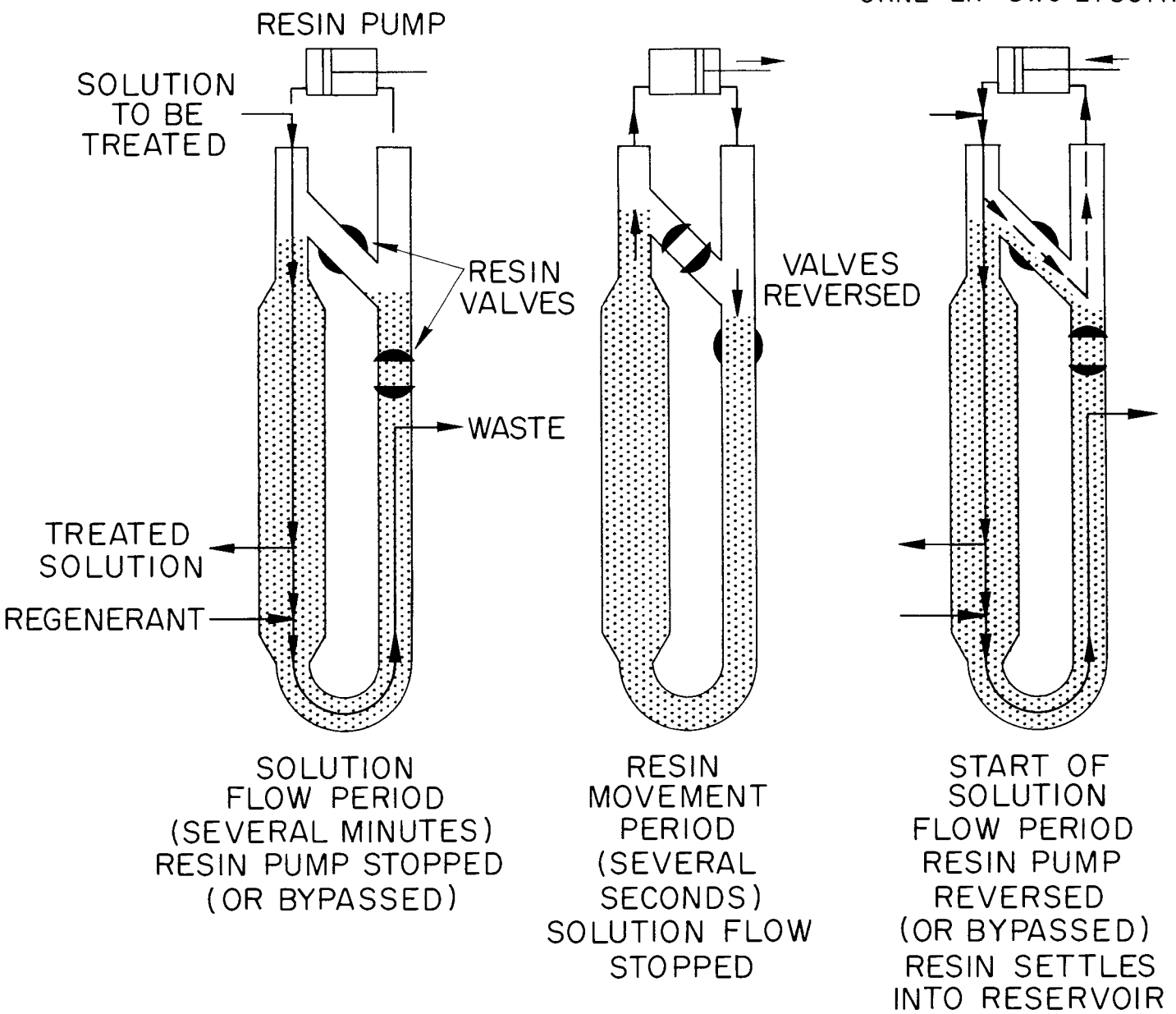

Fig. 8. Mode of Operation of the Higgins Countercurrent Solid-Liquid Contactor.

It permits lower feed pressures than would otherwise be necessary. Cutting off the feed and take-off streams during the resin movement period prevents these from bucking the resin-movement pump, and from causing resin fluidization above feed points and resin jamming around take-off points.

Principle of Resin Movement

The basic principle of resin movement in the Higgins contactor is that if a sufficiently high static pressure drop is built up across the resin bed sufficiently rapidly, the resin bed will be accelerated as a porous piston fast enough to move the desired amount before co-current liquid flow through the pores of the bed caused by the pressure drop causes excessive fluidization. The co-current flow of liquid through the resin bed during the movement may be thought of as a leakage or slippage by (through) the piston. It is a measure of the resistance to resin flow in the circuit. Since this slippage is in principle an undesirable, though temporary, reversal of the countercurrent contact of the two phases, it should be minimized.

The slippage may include liquid which flows through the bed (a) before the pressure drop has built up to the point at which resin movement is initiated, (b) during the period in which the resin is moving as a dense bed, and (c) after the resin bed begins to be fluidized. The last-mentioned slippage should be minimized by terminating the resin movement before fluidization goes too far. This requirement can be met by proper engineering design. The first-mentioned slippage is minimized by applying an adequate pressure drop as nearly instantaneously as practical. Because one is working with incompressible fluids, this requirement can be met practically by providing a hydraulic pressure system of ample reserve power and capacity. The slippage during the dense-bed movement period should be minimized by reducing resistance to resin flow to a practical minimum. Resin-to-wall friction is held down by using smooth walls of a material that does not have a high coefficient of friction for the resin and by avoiding unnecessarily large ratios of resin path length to diameter. Resin-to-resin friction occurs where resin mixing is caused by changes in diameter and direction of the resin path. These changes should be held to a practical minimum and should be accomplished in a streamlined manner.

Use of adequately powered hydraulic pressure sources and careful loop design makes it practical to operate engineering-scale equipment at slippage liquid volumes equal to 10-40% of the resin movement volume. Less attention to this aspect of contactor design may result in slippage values of 100-300%, though this may be acceptable in low-solution-concentration systems and can be compensated for even in high-concentration systems.

Hydraulic Actuators for Resin Movement[7,11]

For small contactors a double-acting hydraulic piston powered by an air-operated piston has been used successfully to move the resin. Figure 9 shows other hydraulic pressure sources which are recommended for larger contactors. Hydraulic gear pumps, turned on and off each cycle, have been used very successfully for contactor sizes to to 6 in. dia. Where the contactor solutions must

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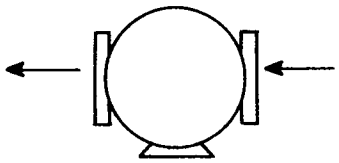
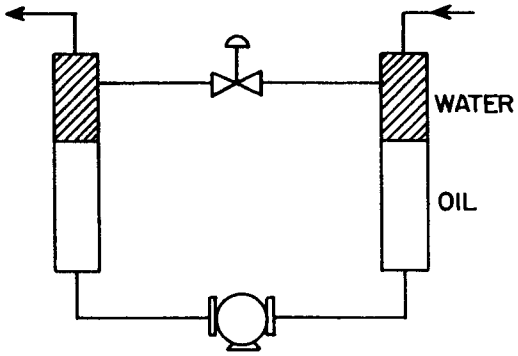
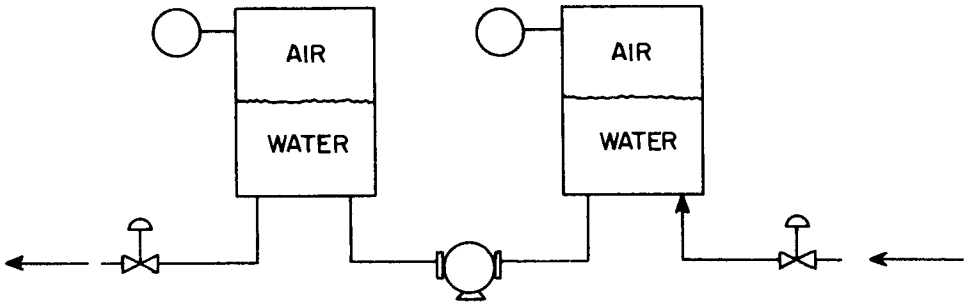


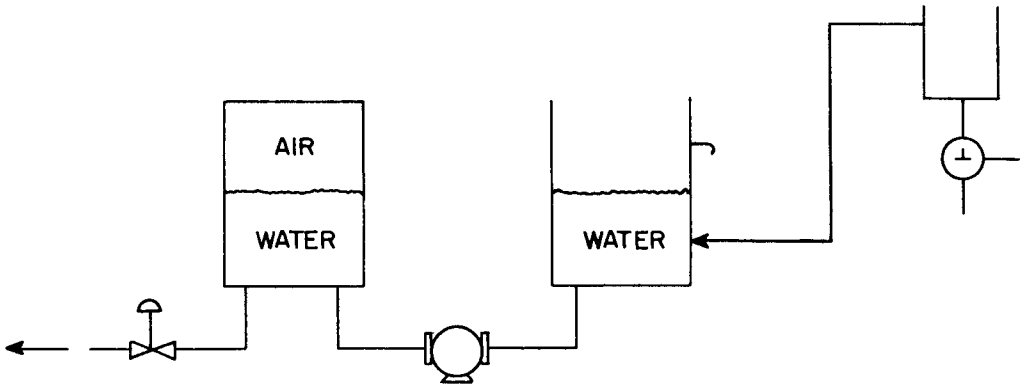
Postive Displacement Pump Using Contactor Fluid



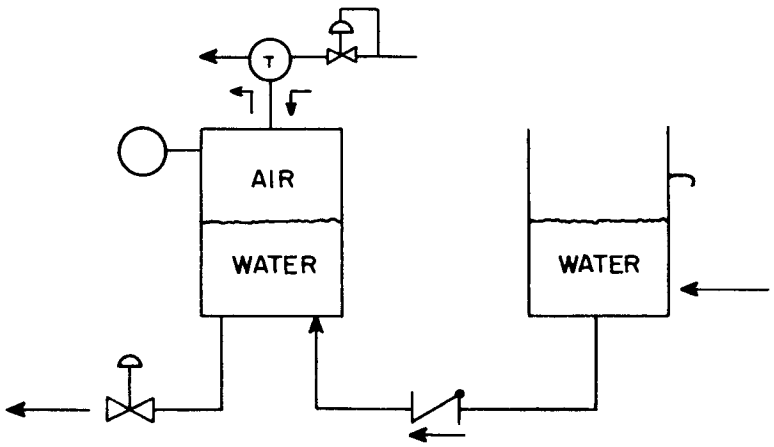
Postive Displacement Pump Using Oil Bath



Hydraulic Accumulator Using Vacuum Tank and Postive Displacement Pump



Hydraulic Accumulator Using Atmospheric Tank and Postive Displacement Pump



Hydraulic Accumulator Using Compressed Air and Check Valve

Fig. 9. Pulsing Mechanisms for Continuous Solid-Liquid Contactors.

be isolated from the pump, for corrosion or radioactivity reasons, the pump can be operated in oil. For large contactors, turning a large pump on and off each cycle presents operating problems. A continuously running pump with a bypass presumably could be used, but ORNL has used instead hydraulic accumulators. Two of these use a positive-displacement pump to accumulate resin-movement liquid and gas pressure in a reservoir tank during the whole contactor cycle, to be used during the resin-movement part of the cycle. Thus the pump can be smaller than it would have to be without the accumulator system. One system is double-acting, applying pressure behind the resin bed and vacuum at the front; the other is single-acting, applying pressure behind the resin bed while venting the top of the bed to atmosphere. The second requires a higher pressure to achieve the same pressure difference, but is somewhat simpler and reduces the danger of getting gas in the contactor. (Gas in the resin bed interferes with both resin and solution flow. The piston and pump methods of resin movement can also be used single-acting, of course.) The third accumulator system is also single-acting but is powered directly by compressed air; in effect, the pump is replaced by an air compressor. If the liquid is corrosive this may be an economically desirable substitution.

The use of gas pressure to move the resin requires adequate gas reservoir volume to avoid excessive loss of pressure during the resin movement period. Use of the regular water supply as a source of both pressure and liquid for moving the resin has been suggested but not yet tried.

Feed and Take-off Distributors[1,6,7,11]

Uniform liquid and resin flow over a column cross-section are important in achieving the low HETS values theoretically possible with ion exchange. Visual observations in glass columns indicate that in the Higgins contactor resin flow is quite uniform in straight sections and that end effects are fairly well restricted to the ends if changes in diameter and direction are made reasonably streamlined. Introducing and withdrawing liquid in a uniform manner is somewhat more difficult. Figures 10 and 11 indicate some of the types used at ORNL. The tee-type take-off works well if the screen is inserted in the column so that the resin flows over it. If it is recessed, the stagnant layer of resin against the screen tends to plug the screen. A tee-type feed works well at the top of a column. In the middle of a column a liquid-pocket type of feed gives uniform distribution, the pipe-type causing more resistance to resin flow than the screened-cage type. Other types of distributors, probably more elaborate, may be needed for large-diameter columns.

Laboratory-Scale Contactors[9-12]

Figure 12 shows a 1-in.-dia by 11-ft laboratory contactor of glass pipe construction. The resin is moved by a gear pump running in oil. The plug valves in the resin loop are actuated by air-operated, double-acting pistons. Figure 13 shows a similar 2-in.-dia by 20-ft-high contactor. The automatic instrumentation is mounted in the rack panel beside the column. One adjustable timer controls the length of the solution pumping period and the other the length of

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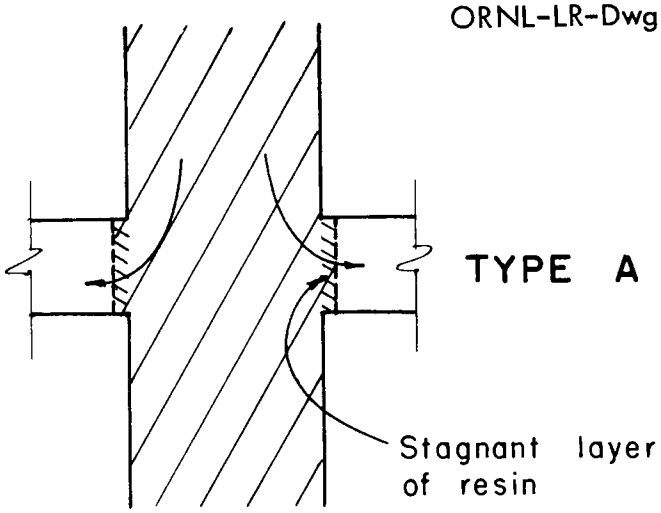

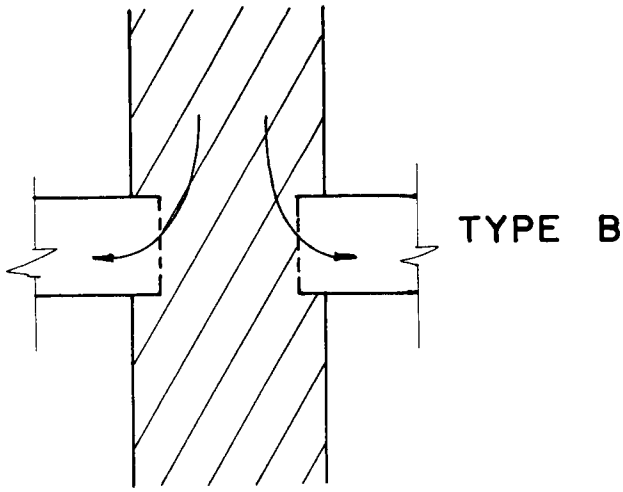

Fig. 10. Liquid Take-Offs for Continuous Ion Exchange Contactor.

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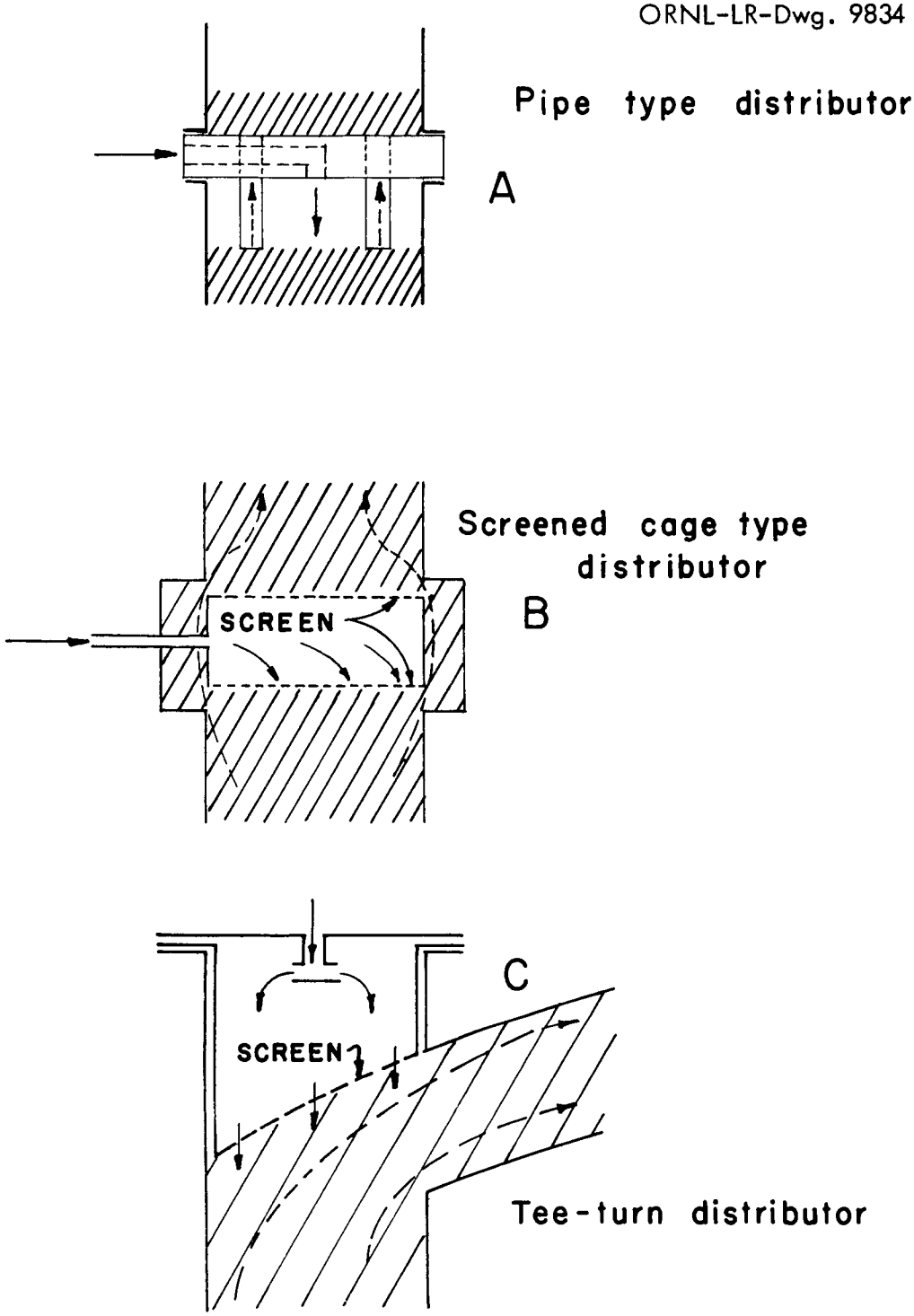

Fig. 11. Liquid Distributors for Continuous Ion Exchange Contactor.

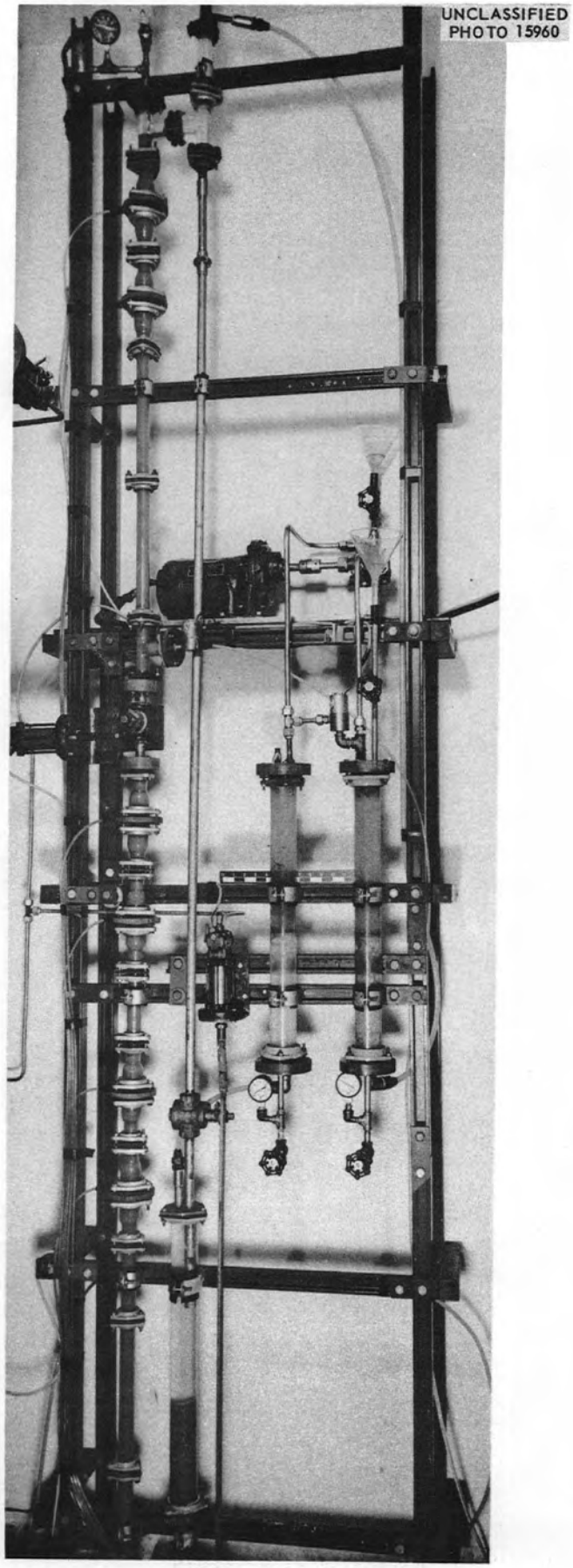

Fig. 12. 1-in.-dia by 11-ft High Glass-pipe Laboratory Contactor.

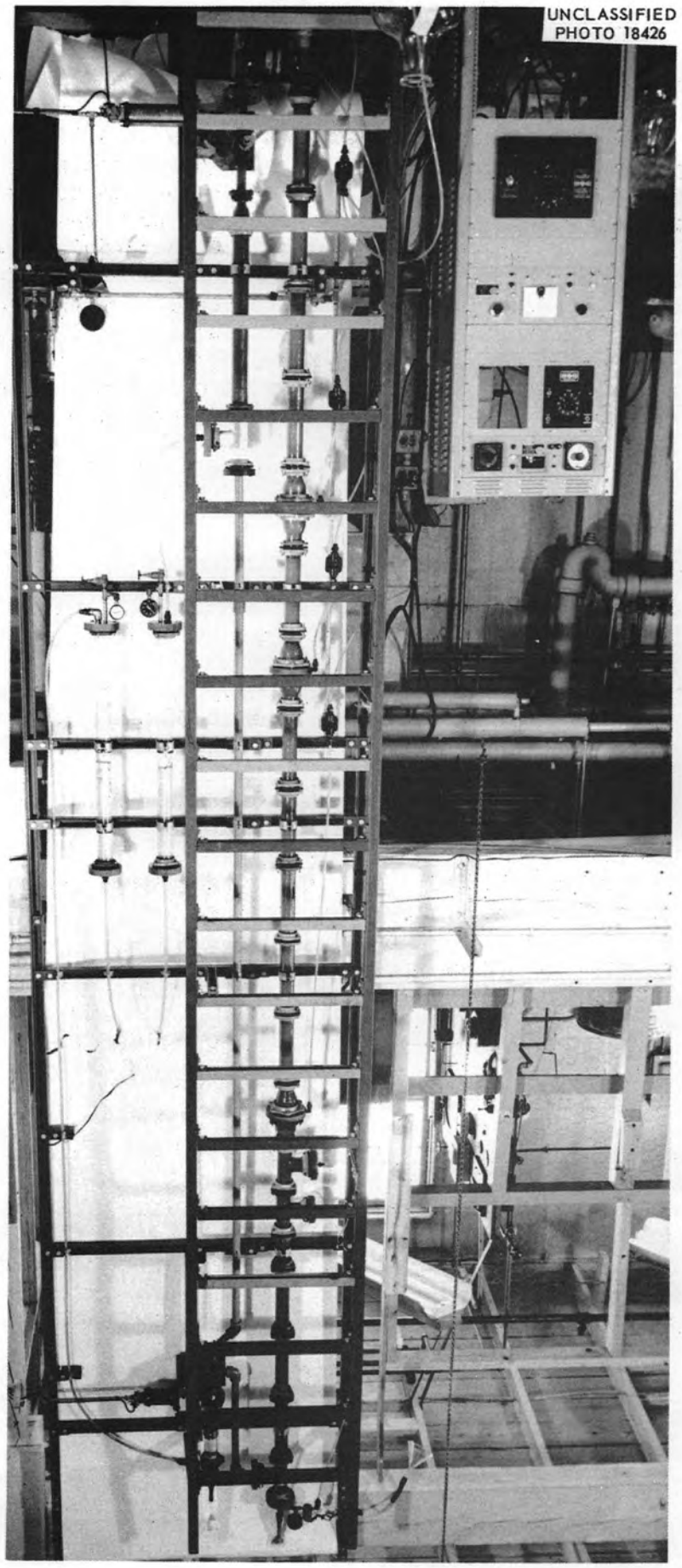

Fig. 13. 2-in.-dia by 20-ft High Contactor.

the resin movement period. Provisions are included for allowing part of the cycle to be self-controlled by the signal from a conductivity probe in the resin bed or in one of the exit streams instead of strictly on a time basis, if desired. (Other process variables might also be used for control purposes, e.g., pH, density, radioactivity, etc.) Figure 14 indicates the contactor arrangement used for resin-in-pulp studies. The uranium-bearing pulp passes through the extraction (sorption) section carrying most of the suspended matter with it. Most of the solids which follow the resin into the feed wash section are washed back into the extraction section. The small percentage of the solids which follow the resin through the feed wash section would gradually plug the bed if not removed, so removal is accomplished in the resin return leg. The resin settles in a fluidized fashion against a backwash stream and accumulates in the reservoir, ready to enter the elution (desorption) section, essentially free of solids. Figure 15 shows the 2-in.-dia contactor on which laboratory studies of this system were made. The column has been "folded" to reduce height requirements.

Pilot-Plant-Scale Contactors[6,7,13]

Figure 16 shows a 3-in.-dia contactor of stainless steel construction built for an ORNL radiochemical processing pilot plant. Figure 17 shows part of a 6-in.-dia contactor for scale-up studies of the 2-in. resin-in-pulp contactor mentioned above. A conductivity system is used to keep slippage water from diluting the product by pushing it out of the strip (desorption) section before product is withdrawn. Figure 18 illustrates the action of this slippage water eliminator. Figure 19 is a McCabe-Thiele analysis of a typical resin-in-pulp run on this contactor. The loading and stripping sections were each 4 ft long. Figure 20 indicates schematically the next stage of scale-up for resin-in-pulp studies. The 12-in.-dia loading and 8-in.-dia stripping sections were placed side by side to conserve height, but at the price of greatly increased resistance to resin flow, primarily in the long low-diameter connecting sections.

Figure 21 is a photograph of the completed contactor. This contactor was moved from ORNL to the resin-in-pulp pilot plant at Grand Junction, Colorado, and substituted on a test basis for the 24-basket system (1 cu ft of resin per basket) ordinarily used there to evaluate ores. The contactor was designed to process ore at approximately the 8 tons/day rate of the basket system, but it was found possible to operate at twice this rate and still make product of higher than normal concentration and purity. The contactor contained about 9 cu ft of resin in the working sections and 4 cu ft in the reservoir and connecting sections, a total of about half the resin inventory of the basket system.

The solution flow rate in the sorption section was 968 gal/hr/sq ft (feed plus feed wash). This is quite high as ion exchange flow rates go, about three times typical water softening flow rates. The pressure drop across the sorption section was 20 psig. The HETS was 1.5 ft, better than on the 6-in.-dia contactor at half the flow rate. The desorption section flow rate was 230 gal/hr/sq ft, the pressure drop 10 psig, and the HETS 4.7 ft. The corresponding HETS on the 6-in.-dia contactor, at half the solution flow rate and two-thirds the resin flow

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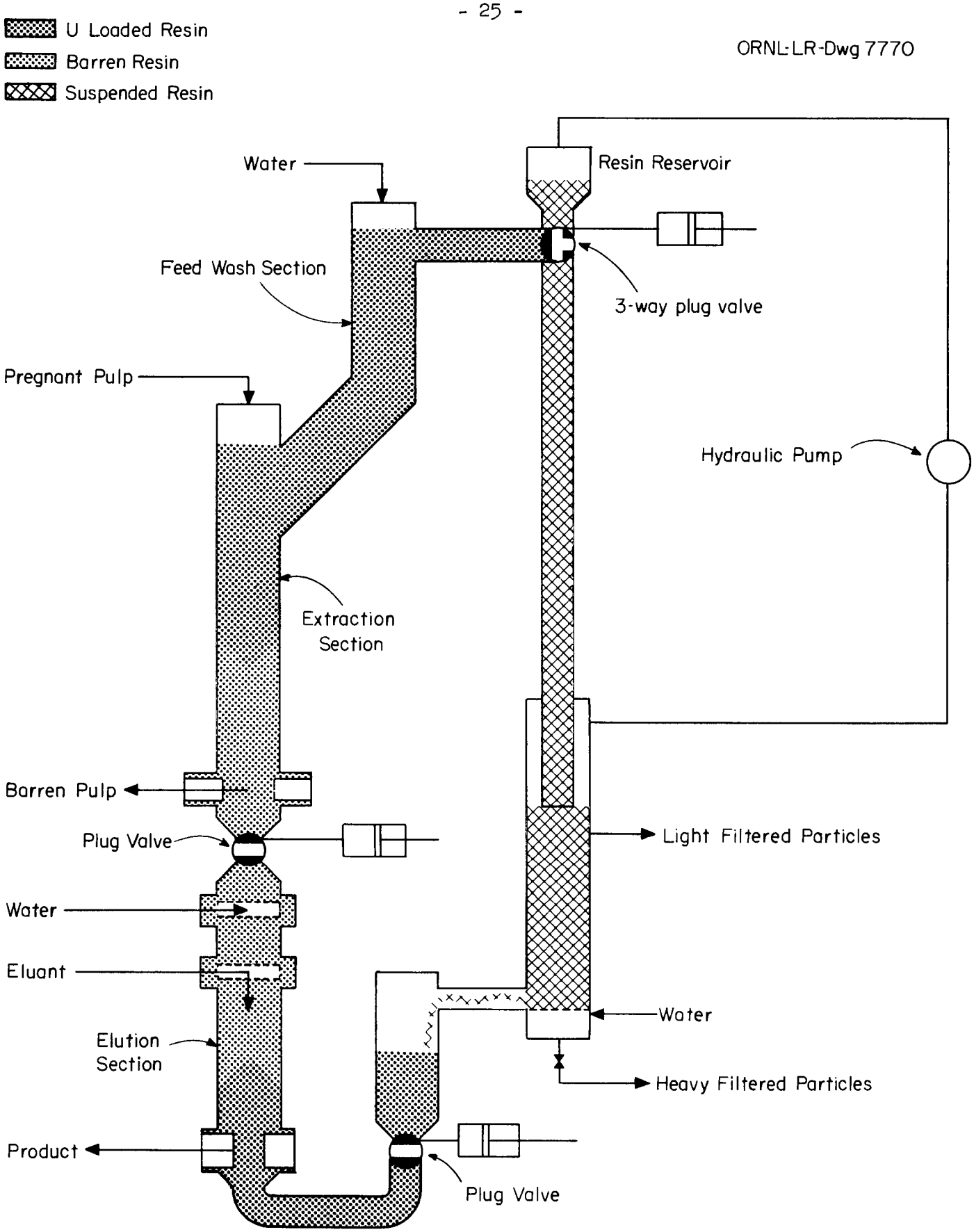

Fig. 14. Diagram of Contactor System for Use with Unfiltered Uranium Ore Leach Slurry.

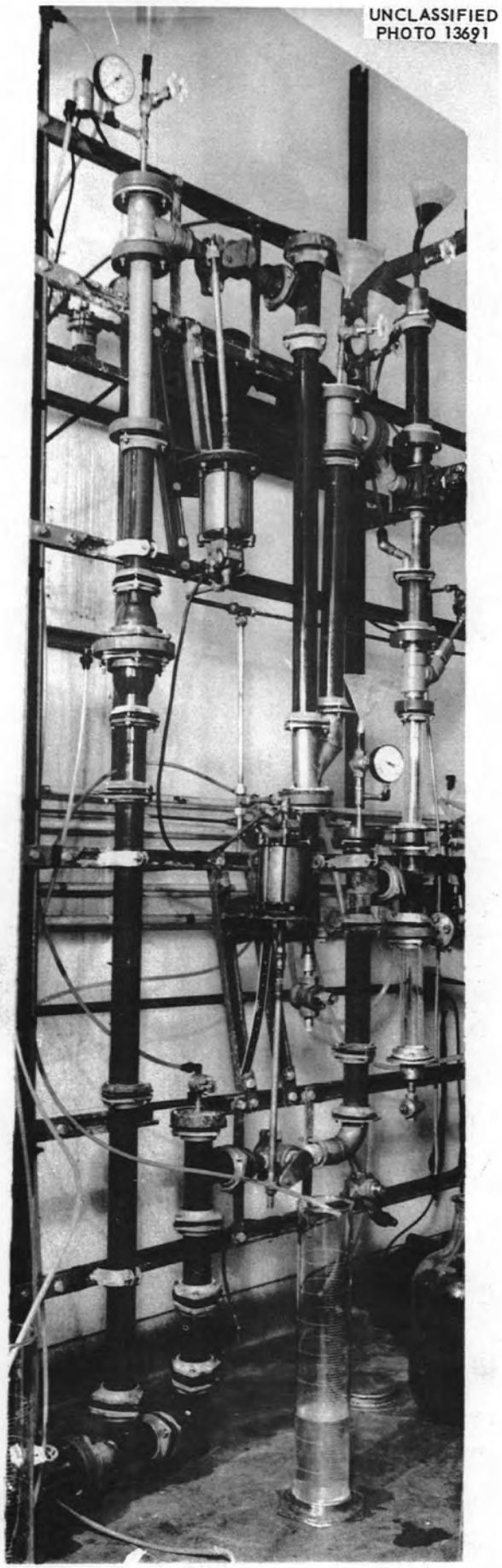

Fig. 15. 2-in.-dia Contactor used in Laboratory Studies.

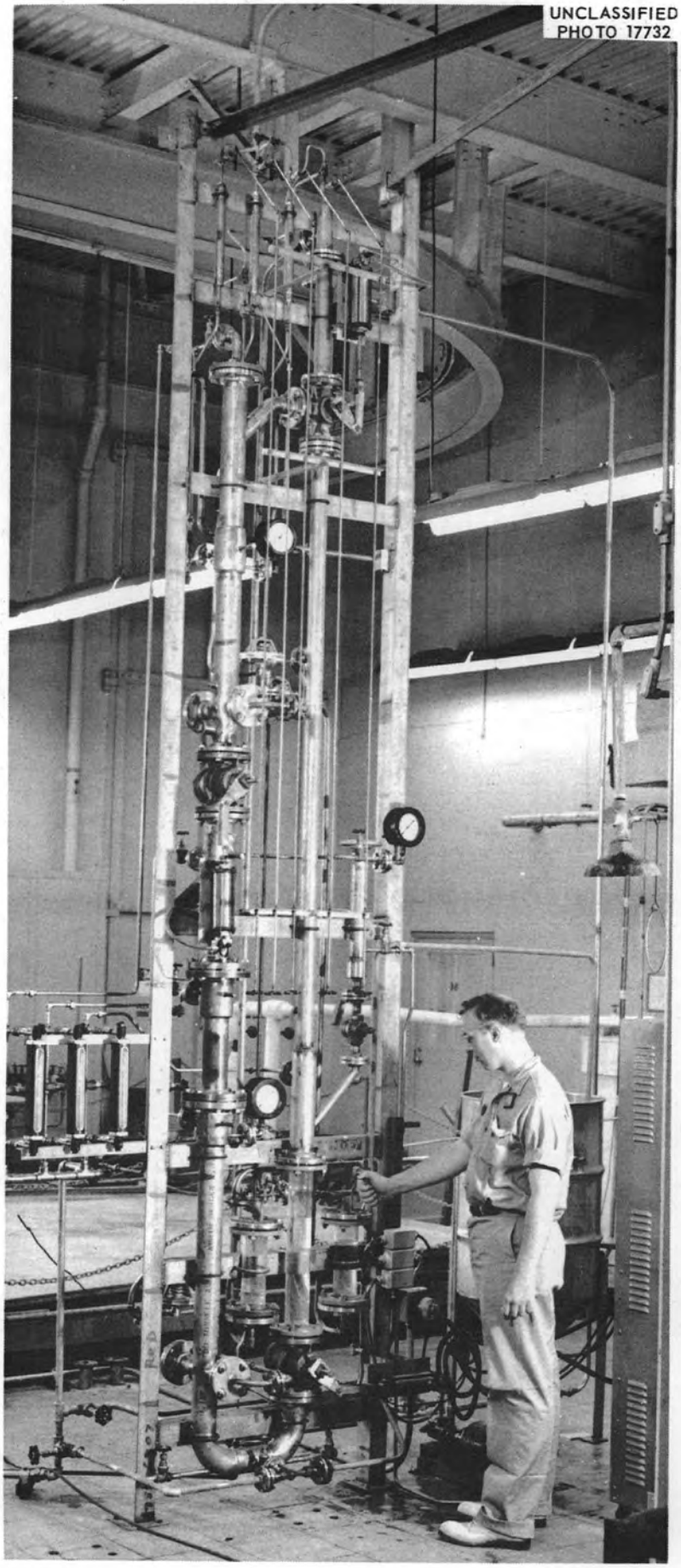

Fig. 16. 3-in.-dia Stainless Steel Contactor.

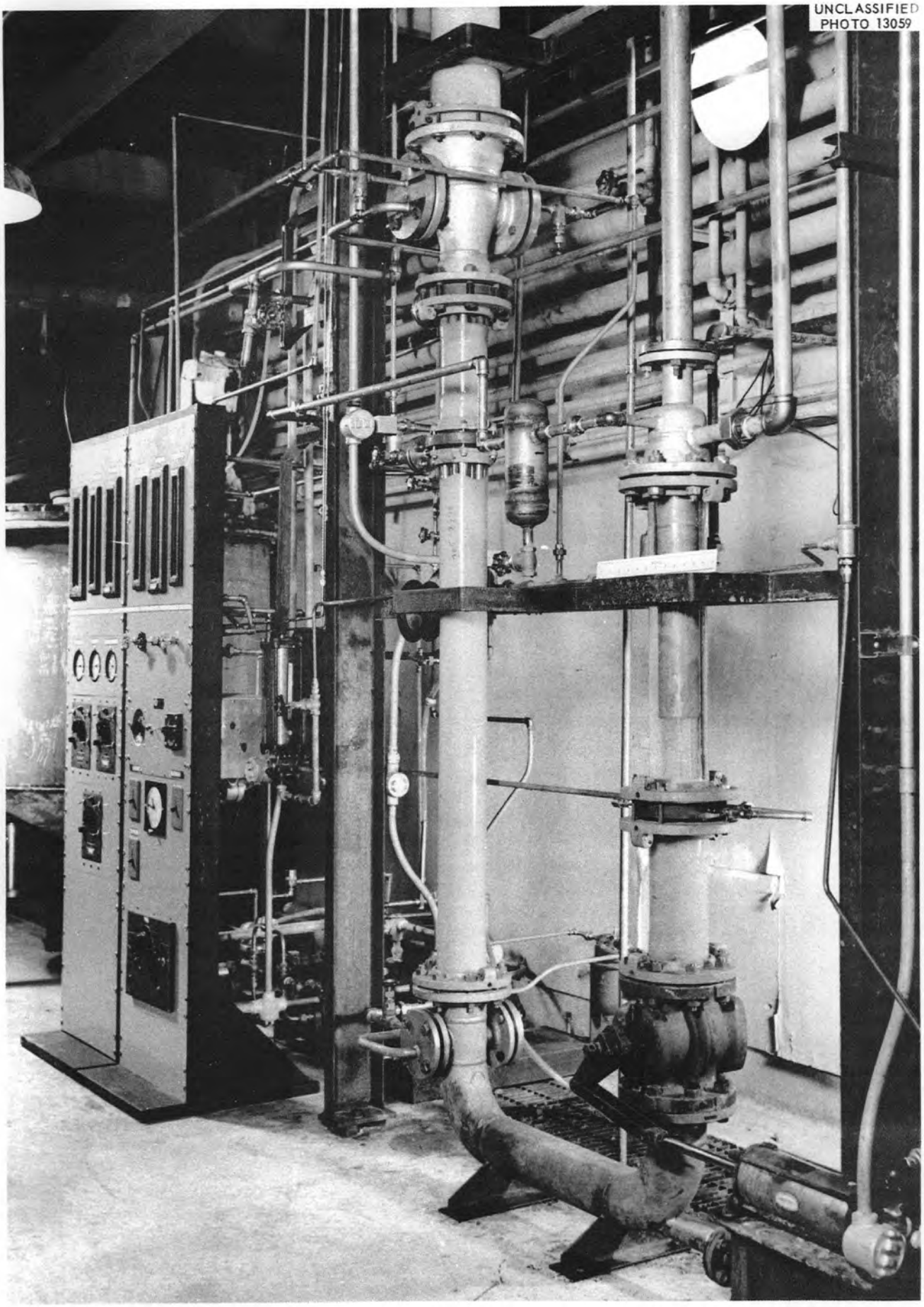

Fig. 17. Part of 6-in-dia Contactor used for Scale-up Studies.

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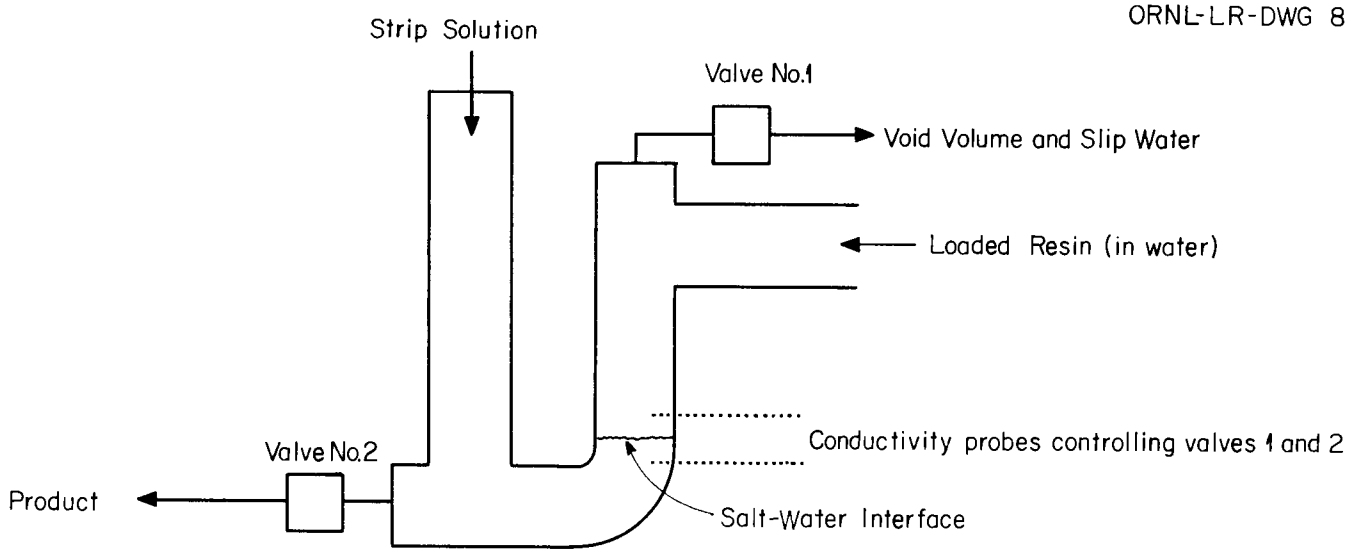
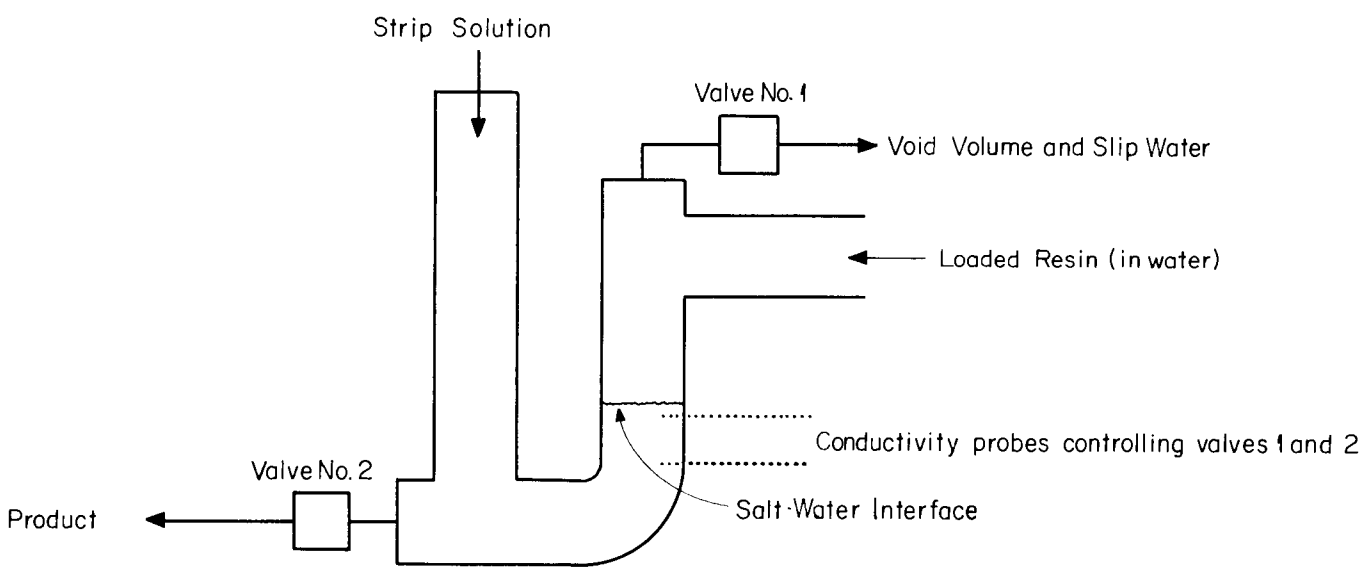


Just after resin movement; valve 1 open, valve 2 closed



Later, valve 1 closed, valve 2 open

Fig. 18. Conductivity Type Water Eliminator.

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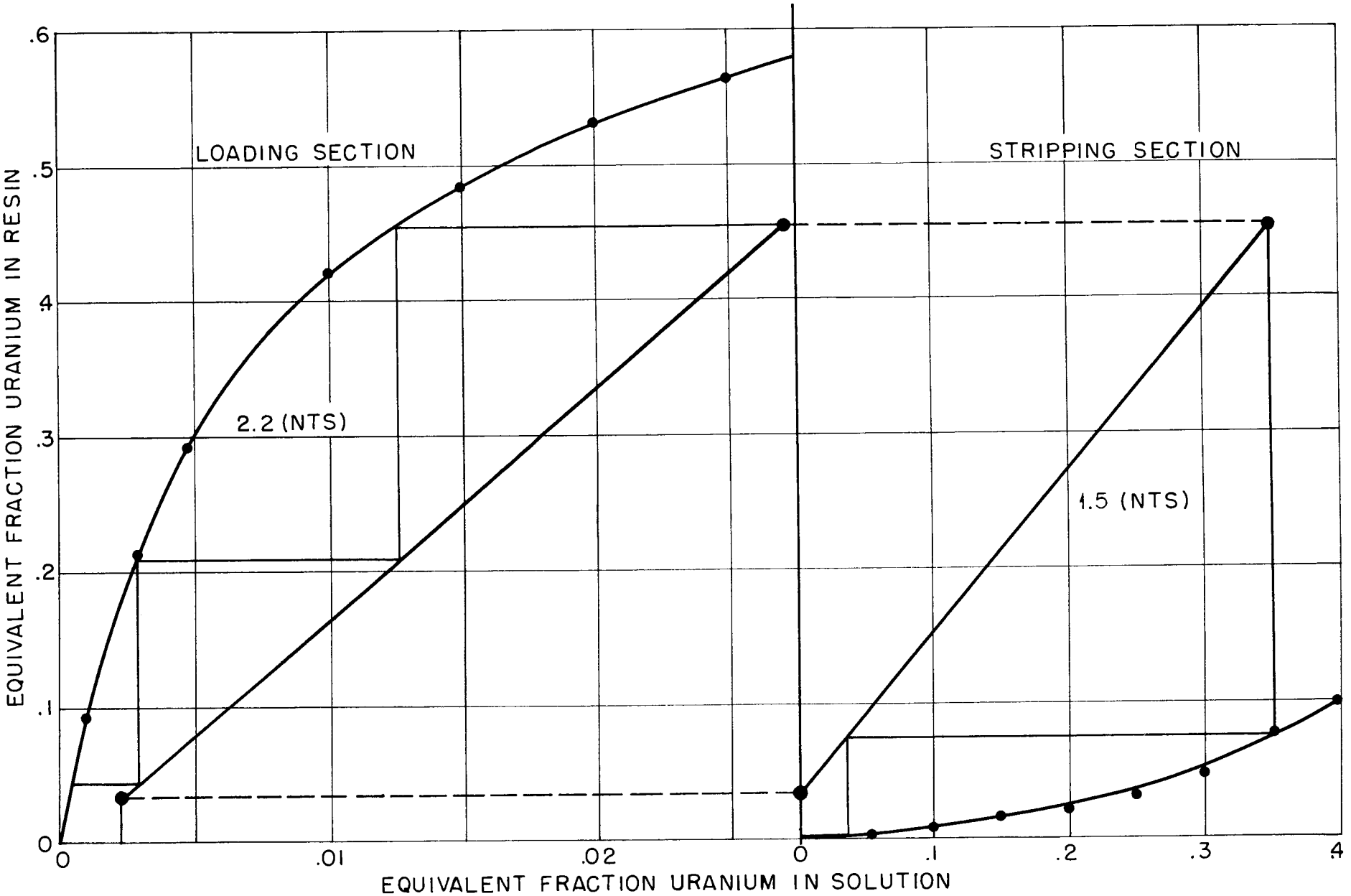

Fig. 19. McCabe-Thiele Diagram for Typical Resin-in-pulp Run in 6-in-dia Contactor.

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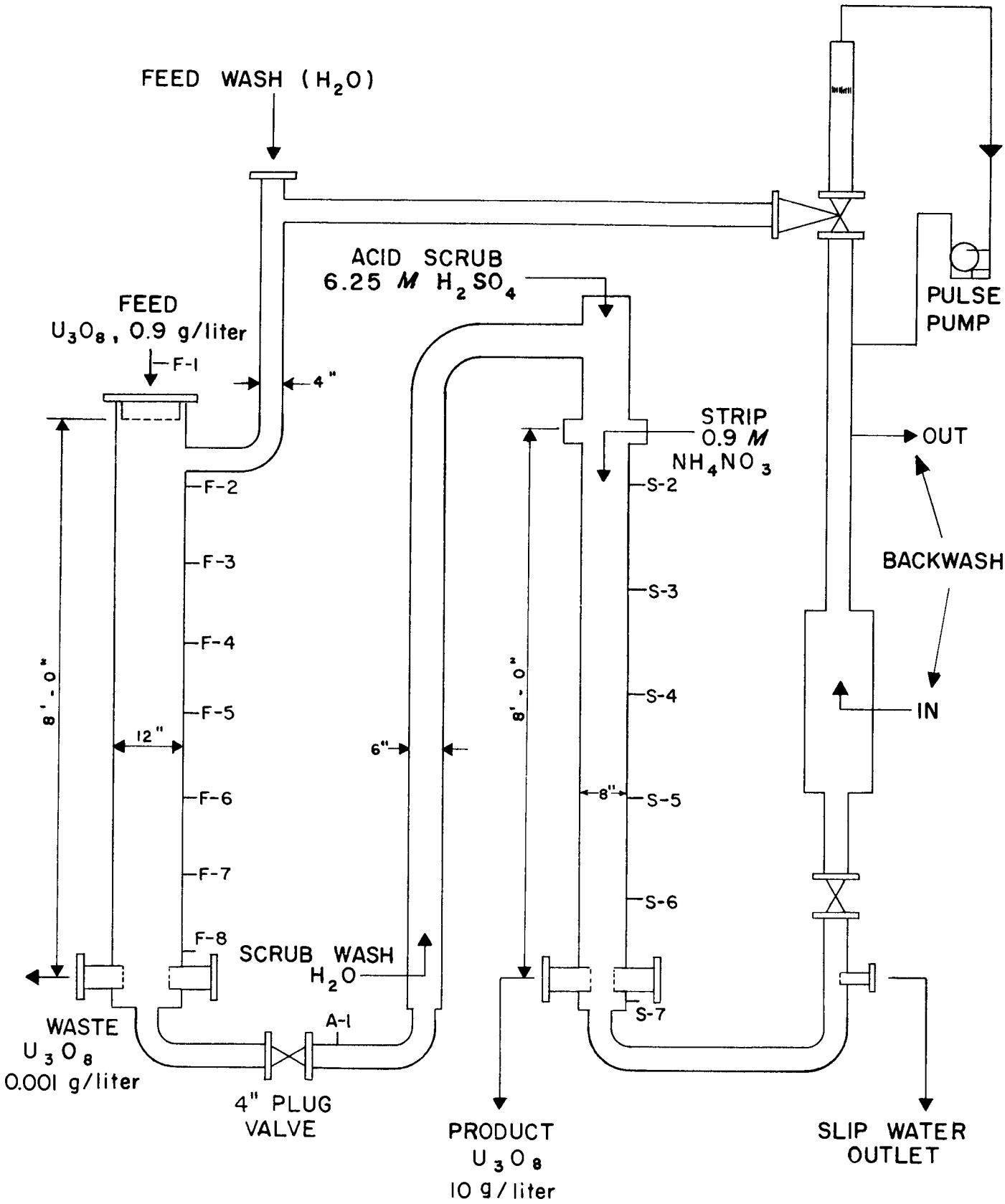

Fig. 20. Schematic Diagram of 12 in. dia. Higgins Ion Exchange Contactor Used in Grand Junction Tests. Resin Capacity ~ 13 cu ft.

Fig. 21. 12-in.-dia Higgins Ion Exchange Contactor used in Grand Junction Tests.

rate, was 2.7 ft. The 12-in.-dia contactor was pulsed on a 5-min cycle. The over-all pulse pressure was 45 psi and the slippage was 230% of the resin movement volume. This high slippage, to be compared with 70% for the 6-in.-dia contactor and 10-50% for the 36-in.-dia contactor described below, is blamed primarily on the high resistance to resin flow of the folded loop. It probably could be reduced considerably by increasing the pulsing pressure, either by installing a larger gear pump or switching to a hydraulic accumulator.

Thirty-Six-Inch-Diameter Hydraulic Test Loop[7,14]

After the Grand Junction test of the 12-in.-dia contactor, it was felt that the next step in scale-up for resin-in-pulp studies should be to 3 or 4 ft dia. Figure 22 indicates the conceptual design of such a contactor. To reduce slippage and pulse pressure requirements the proposed contactor is designed so that the sorption and desorption sections are pulsed alternately instead of at the same time. A contactor of this size should handle, nominally, about 200 tons/day of a typical ore. Since ORNL is not suitably located for actual ore testing at this rate, the scale-up studies have been limited to building only half of this large contactor and studying its resin flow and water flow characteristics and the performance of large components.

Figure 23 is a sketch of the 3-ft-dia loop constructed, and Figs. 24 and 25 are two photographic views. This loop holds about 150 cu ft of resin. A hydraulic accumulator (Fig. 26) was used to move the resin. It was built to permit either a single or double-acting pulse powered with a gear pump or a single-acting pulse powered from a compressed air line. Figure 27 shows volume of resin moved as a function of pulse time and initial pressure drop. The accumulator pressure dropped off during the pulse, about 25 to 33% during 10 seconds. Increasing the pressure drop, to about 40 psi for short pulses and up to about 70 psi for longer pulses, significantly increases resin movement rate. The wavy shape of the curves at pressures at and above 40 psi, indicating that the resin alternately accelerates and decelerates, is not adequately understood. Figure 28 shows slippage under the same conditions. It is significantly reduced, to 5-50%, by operating at 50 psi and above.

Three sets of liquid distribution probes (Fig. 24) were installed in the 36-in.-dia section to permit pressure difference measurements or liquid sampling for dye or concentration measurements. Preliminary pressure measurements indicated that the liquid flow over a cross-section is quite uniform, but more studies need to be made. A 12-in. butterfly valve (Fig. 29) was used in the bottom of the resin return leg and has performed very satisfactorily. This type of valve is cheaper than a plug valve of the same size.

Twelve-Inch Diameter, Sixty-Foot High Y-12 Contactor

Figure 30 shows the 12-in.-dia 60-ft-high contactor built by the Oak Ridge Y-12 plant for recovery of slightly enriched uranium from magnesium fluoride slag. This operation is quite similar to the unfiltered ore leach liquor application in that the feed contains finely divided solids. The, perhaps, extreme height was the result of the production rate specification and a criticality limitation on diameter.

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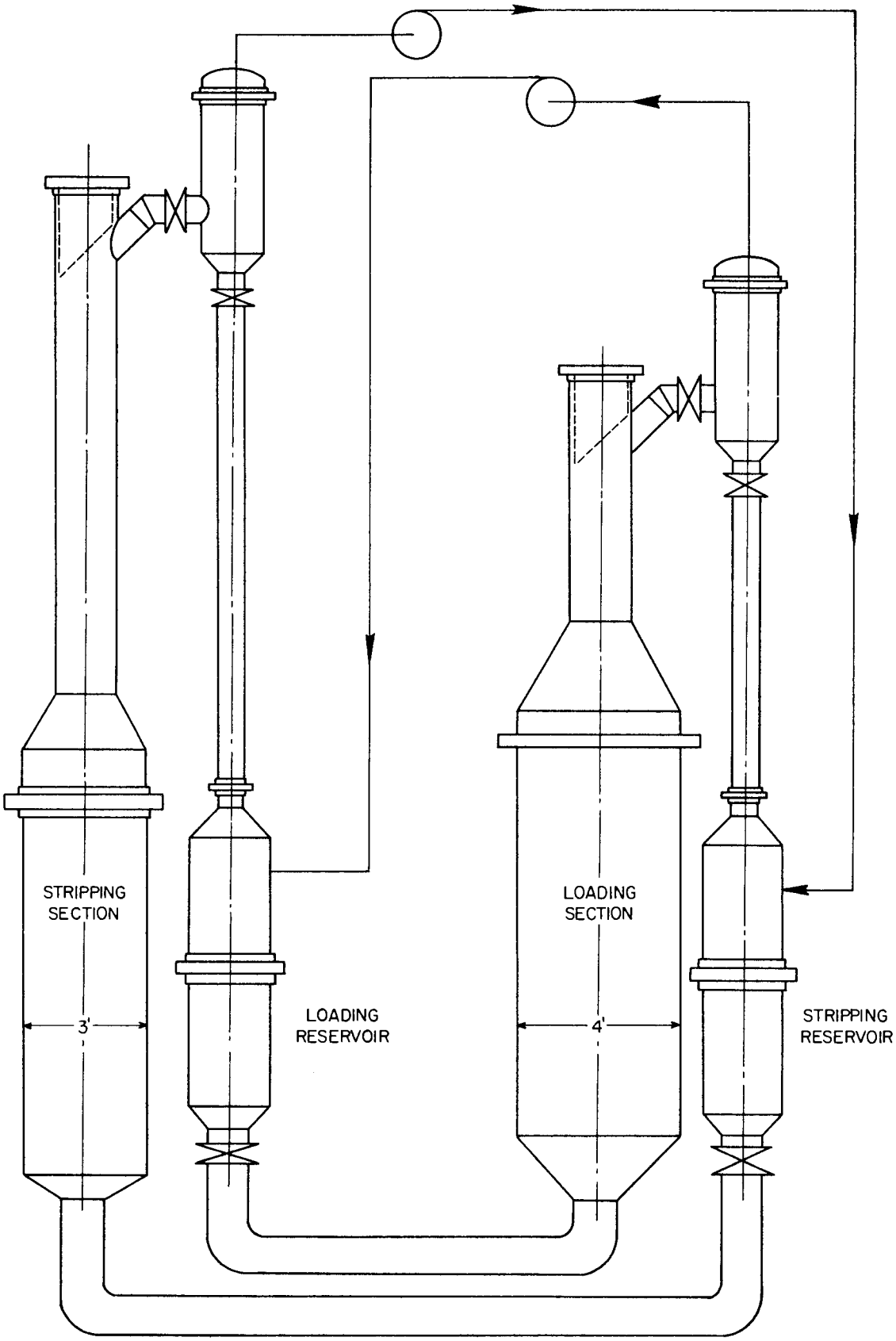

Fig. 22. Two-Section Continuous Countercurrent Solid-Liquid Contactor.

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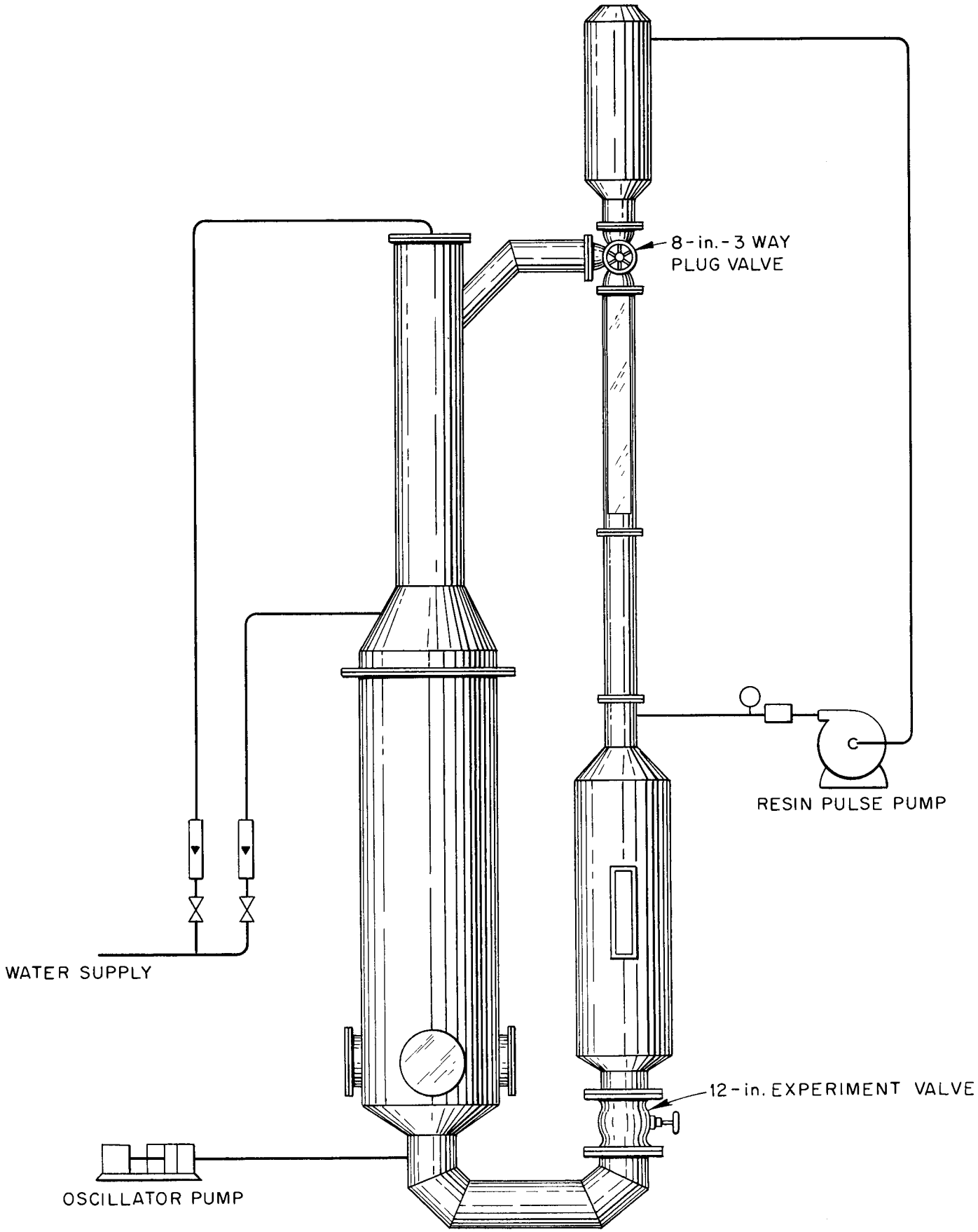

Fig. 23. 36-in. Resin Test Loop.

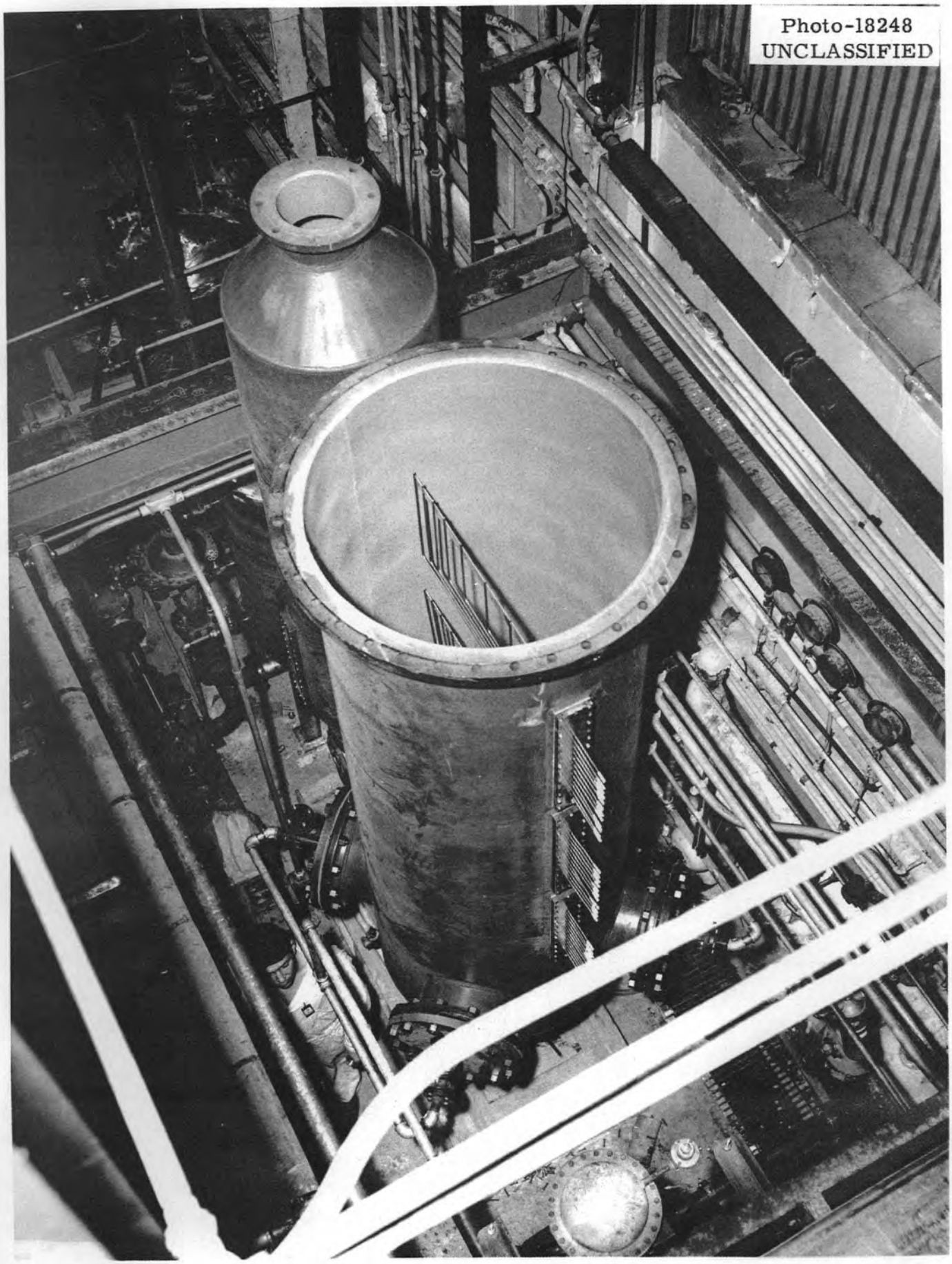

Fig. 24. Section of 36-in.-dia Test Loop.

Fig. 25. Section of 36-in.-dia Test Loop.

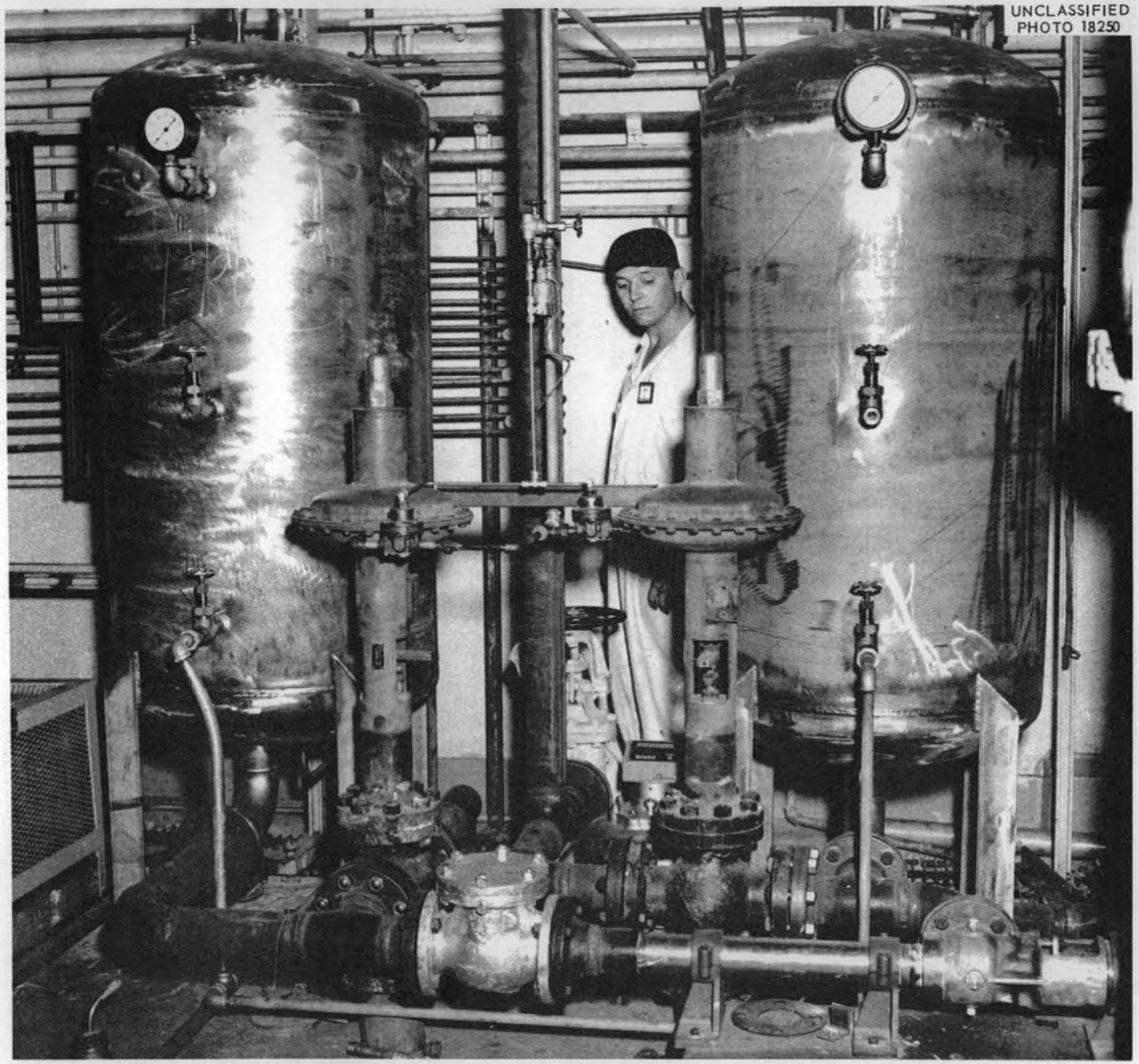

Fig. 26. Hydraulic Accumulator used with 36-in.-dia Test Loop.

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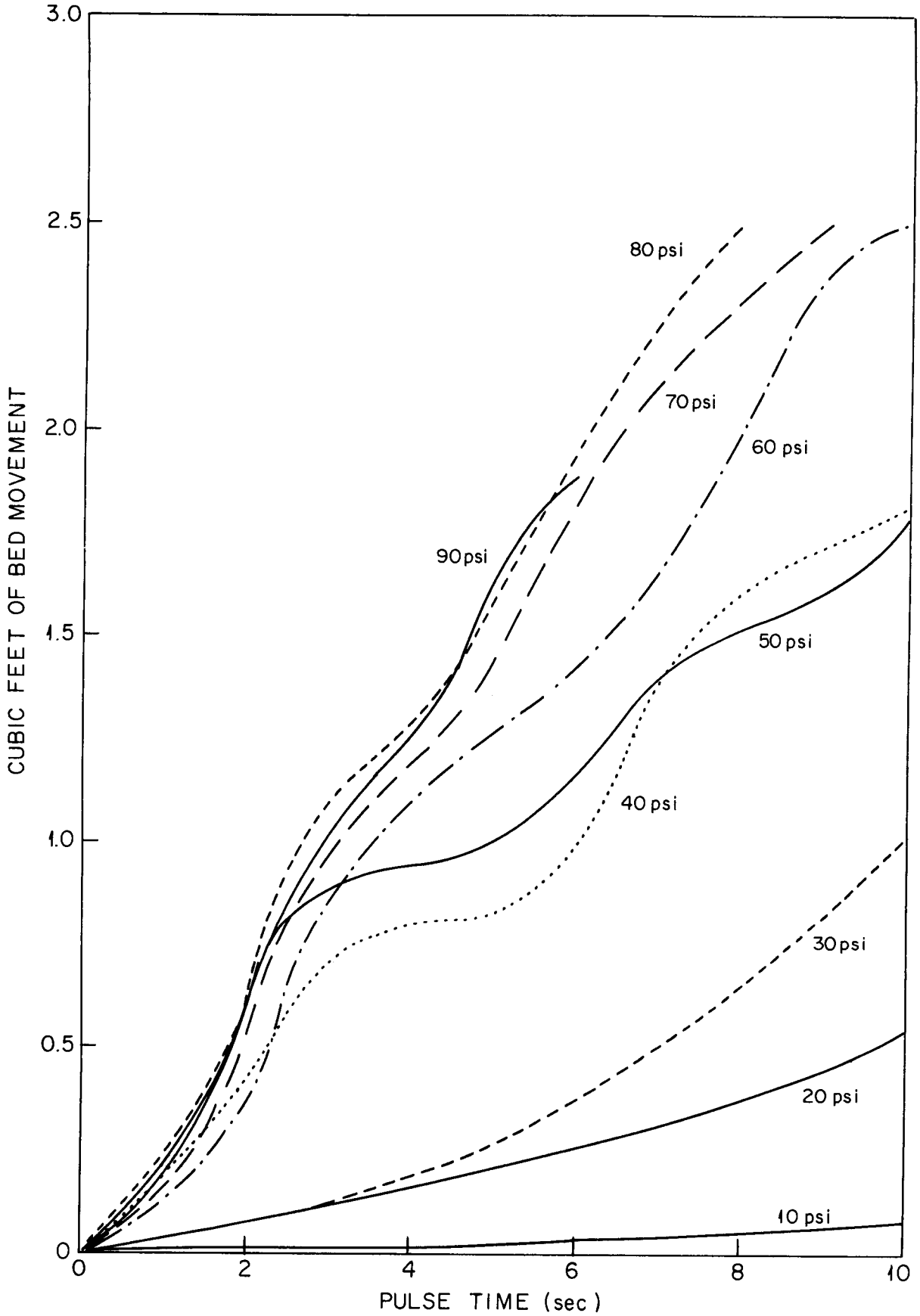

Fig. 27. Volume of Resin Moved as a Function of Pulse Time and Initial Pressure Drop.

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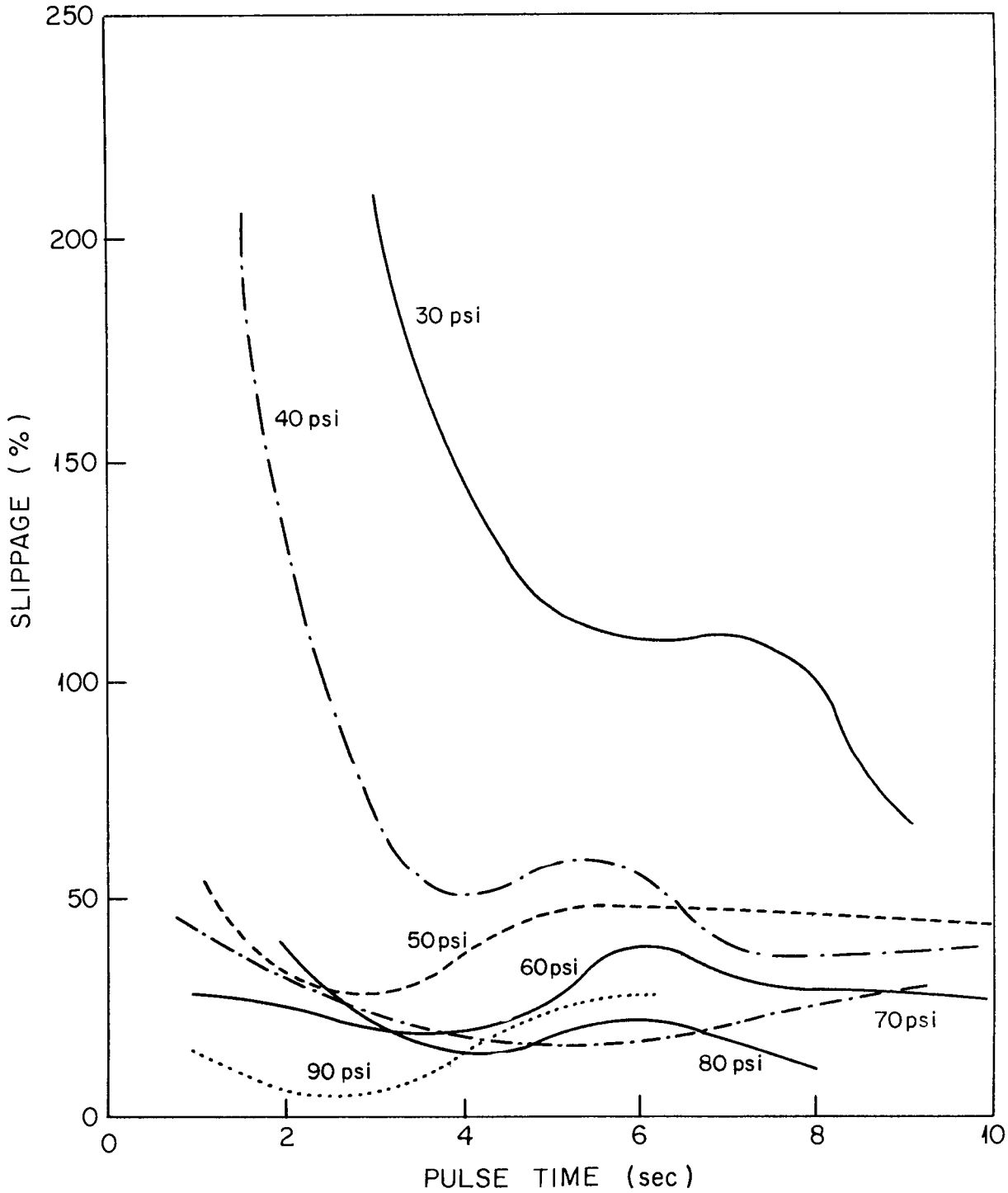

Fig. 28. Slippage as a Function of Pressure and Pulse Time.

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Fig. 29. Butterfly Resin Valve.

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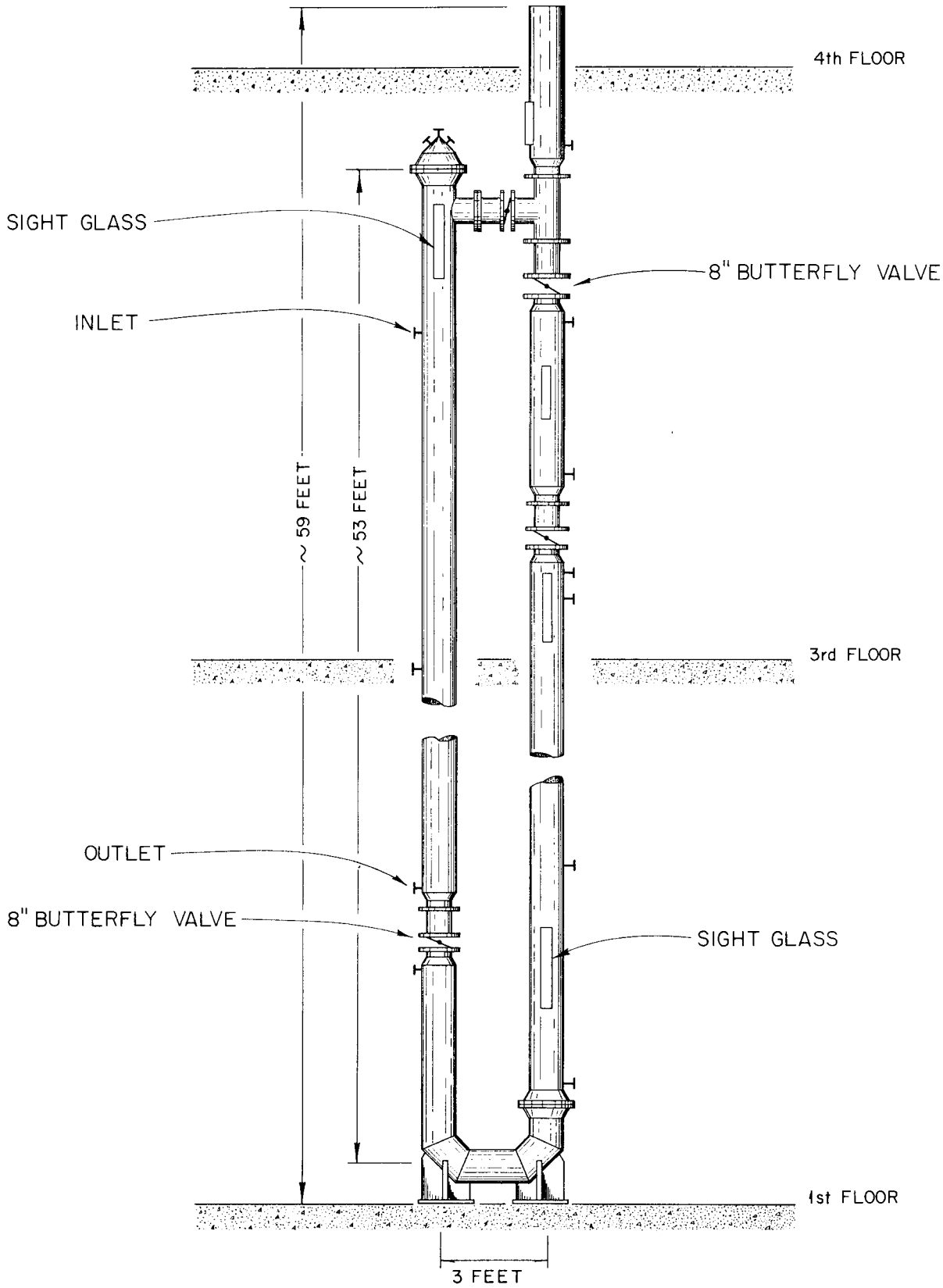

Fig. 30. 12-in.-dia Higgins Contactor

Non-AEC Developments[15]

The Higgins contactor has attracted some attention outside the U. S. AEC. The Dow Chemical Company has built and operated contactors up to 30 in. dia. Chemical Separations Corporation (Oak Ridge, Tennessee) and Illinois Water Treatment Company (Rockford, Illinois) have built several research and development scale contactors (1-3 in. dia) for sale, exhibition, and rent, and have answered queries on production scale contactors up to 7 ft dia.

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