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234-5 FLUORINATOR FILTER AND POWDER RECYCLE STUDIES

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Chemical Research and Development
HANFORD LABORATORIES OPERATION

December 15, 1959

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234-5 FLUORINATOR FILTER AND POWDER RECYCLE STUDIES

Introduction

As requested by Facilities Engineering, studies have been conducted on a fluorinator off-gas filter system and a pneumatic powder transfer device for the 234-5 Building new fluoride line currently being designed.

Summary and Conclusions

A filter assembly consisting of three 1-1/2" O.D. x 6" long (84 sq in. total area), porous carbon filter tubes (grade 40) was designed and tested, using cerium fluoride powder and air, to determine the characteristics of the assembly when installed as an integral part of a vibrating fluorinator. The assembly was tested with an off-gas rate equivalent to the existing fluorinator off-gas rate. A pneumatic powder transfer device was developed for recycling unreacted powder back to the fluorinator head end.

The filter assembly tested proved that regeneration of the filter assembly can be achieved simply by allowing the pressure across the filters to equalize while vibrating the assembly. The accumulated filter cake readily crumbles off in a short time (~1 min). The useful filtering life of the assembly was apparently not affected by the continuous or semi-continuous vibration of the assembly. Sufficient filter area (84 sq in.) can be adequately assembled in a critically safe (4 in. diameter) fluorinator off-gas tee (see Figure 1b and 2) to give filter cycles of 40 hours duration between regenerations for an expected off-gas dust rate of 15 grams/hr (present average dust rate of 234-5 fluorinators). Exceptionally high dusting rates of 60 grams/hr (maximum attained in 234-5 operation) would decrease the cycle time to 10 hours. Over-all filter life is indefinite, but should exceed several months of continuous service under normal operating conditions.

A pneumatic transfer device was developed. The device is basically a modified 2-1/2 inch cyclone which operates in reverse (i.e., as a powder fluidizing device, see Figure 5). Feeding cerium fluoride powder to the unit at a rate of 200 to 300 grams/min, 24 one-kilogram batches of powder were successfully recycled through 1/2 inch I.D. tubing over a distance of 10 feet and a lift of 3 feet. The air rate requirements for the transfer (4SCFM) was equal to the gas feed rate contemplated for the fluorinator, i.e., by simply diverting the gas feed from the fluorinator, through the device, the powder can be recycled with no additional increase in off-gas load to the filter system.

Fluorinator System

A schematic drawing of the existing plutonium fluorinator system used in the 234-5 Building production line is shown in Figure 1a. The plutonium oxide powder from a calciner is fed continuously to an inclined fluorinating tube which is intermittently vibrated to keep the powder moving through the tube. Fluorine and oxygen are fed continuously and countercurrent to the moving powder bed. The gases react with the oxide to give plutonium fluoride. The off-gas from this reaction is passed through a porous carbon (grade 40) filter tube (144 sq in. filtering area) to recover entrained plutonium powder. Periodically the filter chamber must be manually emptied and the filter manually regenerated. The product, plutonium fluoride, is discharged continuously out the low end of the tube until enough has been collected to make a batch for further processing. Occasionally, the product is not completely fluorinated and the accumulated batch must be recycled manually into the feed end of the tube. It is evident that manual contact by operating and maintenance personnel are required for both the filter clean-out and the recycle operation. These contacts result in radiation exposure to the personnel involved and in addition provide an undesirable contamination hazard.

Presently Facilities Engineering is designing a new line which involved a new concept for controlling plutonium contamination and reducing radiation exposure of operating and maintenance personnel. From this concept evolves two equipment changes which are desirable for the fluroinator. Figure 1b shows the contemplated changes which, briefly, involve the integration of the filter chamber with the fluorinator, and a recycle device requiring no manual contact. Regeneration of the filters, in place, by some remote means would eliminate manual contact currently required and reduce the filter chamber disassembly operations solely to one of replacing the filters when permanently plugged.

Filter Tests

Filter Assembly - An assembly of three filters was made for testing as shown in Figure 2. The filter tubes were porous carbon grade 40 (presently used in the existing fluorinator off-gas filter). Tube diameter and length were chosen to provide sufficient filtering area in a critically safe chamber 4 inches inside diameter by 6 inches long. The filters were mounted to obtain maximum structural strength by subjecting them to a compressive load. A good seal was obtained with hard rubber gaskets at the end of the tubes. Since the tests, carbon cloth has been purchased but not tested for use as a gasket material. However, several donut wafers of carbon cloth should provide as good a seal as the rubber, and be corrosion resistant to the hot fluorine off-gas.

Equipment Set-Up - Figure 3 shows the equipment used to test the filter assembly. Raw air was cleaned and dried by passing it through a silica gel bed. The air was then bubbled up through a bed of oven dried cerium fluoride powder, in a 3 inch glass tee, and discharged into the filter assembly chamber. The bed of fluoride powder was continuously replenished by the constant addition of powder to the flidizer from a hopper. In the filter chamber the dust laden air was filtered through the porous carbon tubes and passed through a rotameter into a vacuum system. In order to simulate the effect of intermittent vibrating action the filters would experience in the new fluorinator, the entire assembly was vibrated continuously (in lieu of intermittent), on the assumption that continuous vibration would be a "worse" case as far as filter pluggage was concerned. Since previous studies (HW-59381) had indicated the filters could be regenerated simply by vibrating the cake off while equalizing the pressure across the filters, this method of filter regeneration was used after each run. Each run was composed of an accelerated (high dust load) filter cycle, using a dirty gas rate of 4 SCFM with a temporary increase in the gas rate to 5 SCFM during a simulated powder transfer into the fluidizing tee from a powder recycle station. The pressure drop history of each run was continuously recorded. Average dust loadings for each run were calculated from the measured quantity of powder collected on the filters during each run. A total of 15 runs were made on the same filter elements.

Results - The data obtained is shown in Table I. The complete pressure drop history of a few runs is shown in Figure 4. It can be seen from Table I that average run dust loadings ranged from 65 to 120 grams/hr. The 234-5 fluorinators produce dust in the off-gas system at a rate of 10 to 50 grams/hr with the normal running about 15 grams/hr. Estimates indicate that the filter assembly tested would need to be regenerated once every 40 hours or so under normal dusting loads. The data also indicates a slow rise of the initial pressure drop after each regeneration. Estimates indicate that the total number of regenerations possible may exceed 100 or more under ideal conditions.

Powder Recycle

A crude powder recycle station was used during the testing of the filter assembly. This was to determine the effect of transferring powder pneumatically into the filter system. Since no unusual effects upon the filter system were noted, a detailed study on pneumatic transfer devices was conducted.

Cyclonic Powder Transfer Device - Several pneumatic powder transfer devices were tested in the laboratory for possible use. The resulting device is shown in Figure 5. The operating principle is simple. Air at high velocity (approximately 900 fps) is introduced through a 7/64 inch orifice tangentially into the bottom end of a tapered cone. The air stream is contoured in a helical path upwards to a 1/2 inch diameter tangential outlet at the top of the 2 1/2 inch diameter cone. The exit gas velocity is approximately 50 fps through a 1/2 inch I.D. tube. High-gas velocities are thus produced along the cone walls and are of sufficient magnitude to keep heavy (1/4 in. diameter SS balls) particles rotating along the cone wall. The taper of the cone wall, in combination with the high rotational speeds the solids attain, act on the solids very much like a centrifugal governor; thus the solids will climb upwards and outwards against the wall, until they reach the tangential outlet. A reverse wall taper at this point prevents the solids from rising further. The powder to be transferred is fed into the cone through a 2-inch diameter opening in the top. It was found necessary to control the rate of dumping into the cyclone to prevent swamping it. A rotary star valve was found suitable for feeding the powder at a controlled rate.

Test Equipment - Figure 6 shows the assembly of equipment used to test the powder transfer device. Filtered air was passed through a silica gel bed into the cyclone. After the air flow was started a mixture of fine and course cerium fluoride powder was fed continuously to the cyclone by a rotary star valve feeder until a batch (1 kg) of powder had been transferred. The powder, as it falls into the cyclone is flung to the walls and fluidized out through the 1/2 inch tangential outlet. The outlet tubing was 1/2 inch I.D. saran tubing through which the fluidized powder flowed a horizontal distance of 10 feet with a vertical rise of 3 feet, and around a long radius 90 degree bend into a 4-inch glass tee. The glass tee was the powder receiver and filter chamber. The air in the chamber passed through the filter assembly (previously tested) through a rotameter, into the vacuum system. After every four batches, the cyclone was disassembled, inspected, and reassembled without cleaning it out.

Results - The cyclone was found to successfully transfer 1 kg batches of cerium fluoride powder through the 1/2 inch I.D. transfer line. No pluggage or powder buildup in the transfer line was observed after 24 batch transfers. Also no pluggage of the 7/64 inch inlet orifice was observed. Slight scaling of the cyclone walls was observed. This, however, was less than 1/16 inch thick and did not build up with continued use or hamper the operation of the cyclone. Pressure observations show that it is possible to maintain the cyclone and transfer line under negative pressure providing approximately two inches of mercury vacuum could be applied at the powder discharge point (filter chamber or fluorinator). The only positive pressure point in the system can be restricted to the upstream side of the inlet orifice which would require sufficient pressure (<10 psi) to give the entering gas a high velocity.

Discussion

The only difficulty encountered in filter testing was due to an off-standard condition. When raw air (highly contaminated with compressor oil and moisture) was used, a hard scaly filter cake was formed on parts of the filters. This hard scale did not fall off during regeneration of the filters. However, the scale did not plug the filters anymore than an equivalent thickness of dry cake and consequently would not make the filters inoperative unless the scale completely covers each filter to a thickness of 1/8 or 1/4 inch, which does not appear likely under the fluorinator conditions (i.e., off-gas temp. 300-450 C). In the event excessive scale does form, remote methods (vibrating, blow back, etc.) of regeneration do not appear to be sufficient to restore the filters to operation, but rather a manually clean out would be required.

Previous tests made on a preliminary lucite powder transfer cyclone with an assortment of steel balls (up to 1/4 inch diameter), paper fastener heads, course and fine fluoride powder, and raw air for transferring, indicated the cyclone had sufficient power to transfer these types of materials, but such off-standard conditions led to severe internal scaling of the cyclone walls and pluggage of the air inlet orifice. Pluggage of the inlet orifice can be tolerated since it produces little danger of spreading contamination, due to pressurization, since any pressure produced is at the upstream ("cold") side of the inlet orifice. In the event of orifice pluggage a built-in needle plunger could be used to unplug it or the unit could be designed for easy replacement (i.e., flanged to powder feeding chamber). The advantages of this system over others considered for this particular application are: (1) high transfer capacity (300 gram/min) with large heavy particles (up to 1/4 in. steel balls), (2) low pressure drop (2 inches Hg) from the orifice discharge to point of powder discharge, (3) low gas requirements (4 SCFM of air), (4) simplicity, and (5) least hazardous for handling plutonium powder with gas since the most vunerable pluggage point is the inlet orifice (mentioned above).

TABLE I

RUN DATA SUMMARY

Run No.	Powder Collected, (grams)	Dust Load, grams/hr	Run Time, (minutes)	ΔP, Initial, (In. WG)	ΔP, End, (In. WG)	Air Rate, l/min	Remarks Vibrating f = 8650 c/min, a = 1/32 inch
A	Air Only	0	16 hrs	6	6	90	Some initial flaking of carbon upon installation - no effects from vibrating
1	↑	↑	220	8	48	90→120	Started at 90 l/min and raised to 120 l/min
2	438	76.4	78	16	44	120	
3	↓	↓	34	15.5	73	120	
4	↓	↓	12	17.5	28	120	
5	440	110	240	12	150	120	Made a 300 gram transfer at end of 90 min using 150 l/min air
6	349	65.4	320	16	160	120	Made a 300 gram transfer at end of 200 min using 150 l/min air
7	326	65.2	300	17.5	145	120	Made a 300 gram transfer at end of 180 min using 150 l/min air
8	454	91	300	14	185	120	Ditto Run 7
9	585	117	300	16	185	120	Made a 300 gram transfer at end of 210 min using 150 l/min air
10	355	66.6	300	12	120	120	No transfer made
11	542	120	270	24	170	120	Made a 300 gram transfer at end of 60 min using 150 l/min air
12	539	92.5	350	17	190	120	Made a 300 gram transfer at end of 90 min using 130 l/min air
13	524	82.6	380	18	190	120	Made a 300 gram transfer at end of 110 min using 130 l/min air
14	509	102	300	20	192	120	Made a 300 gram transfer at end of 150 min using 140 l/min air
15	562	93.7	360	26	210	120	Made a 300 gram transfer at end of 300 min using 140 l/min air

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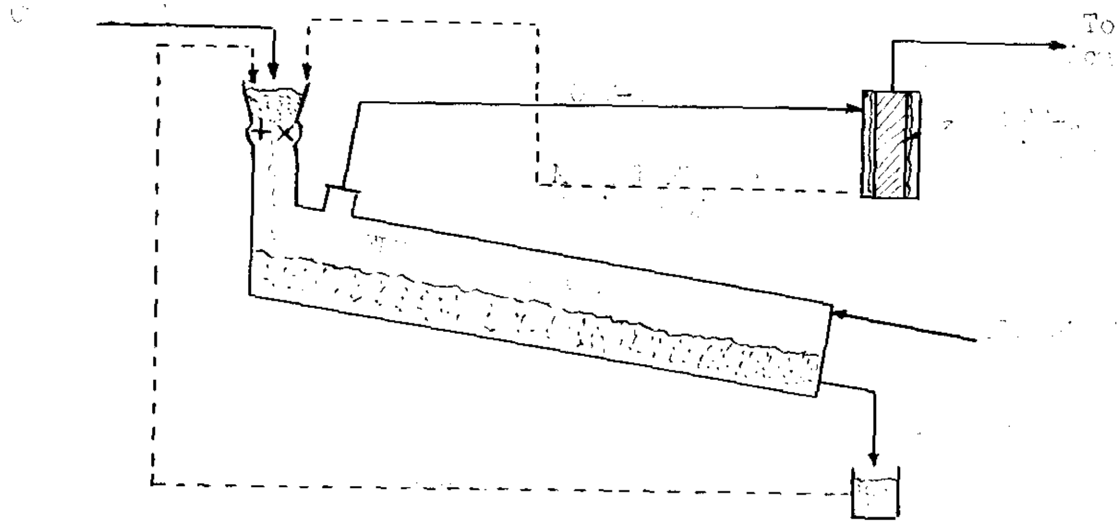

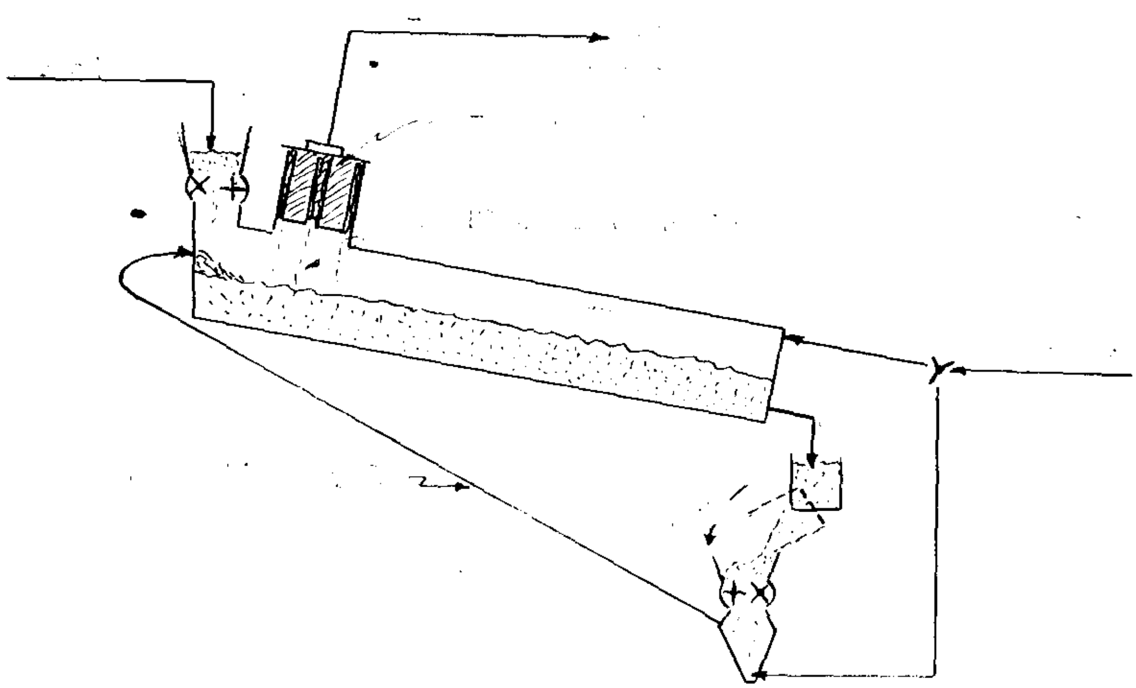

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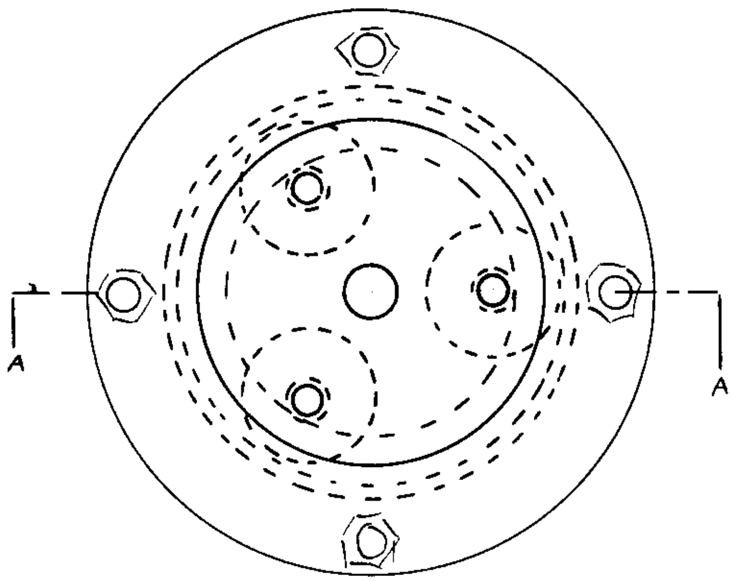

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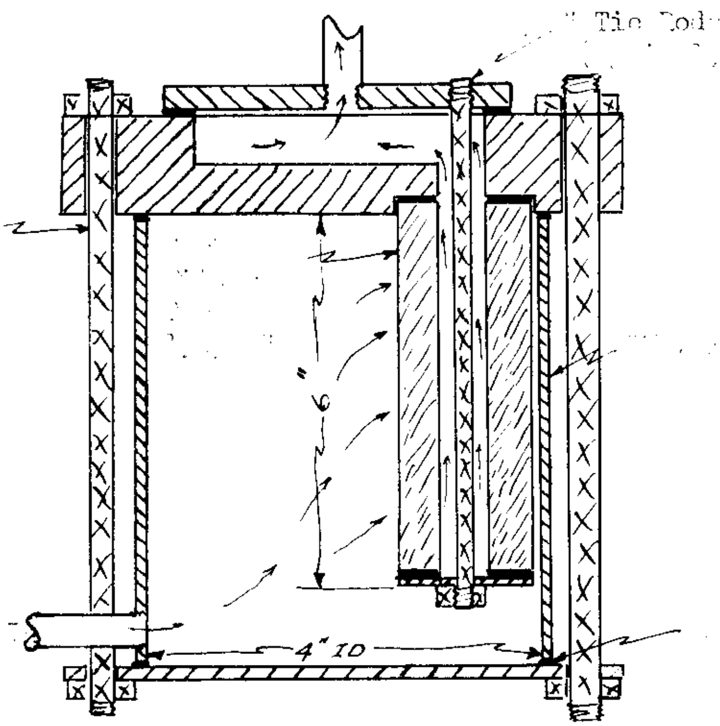

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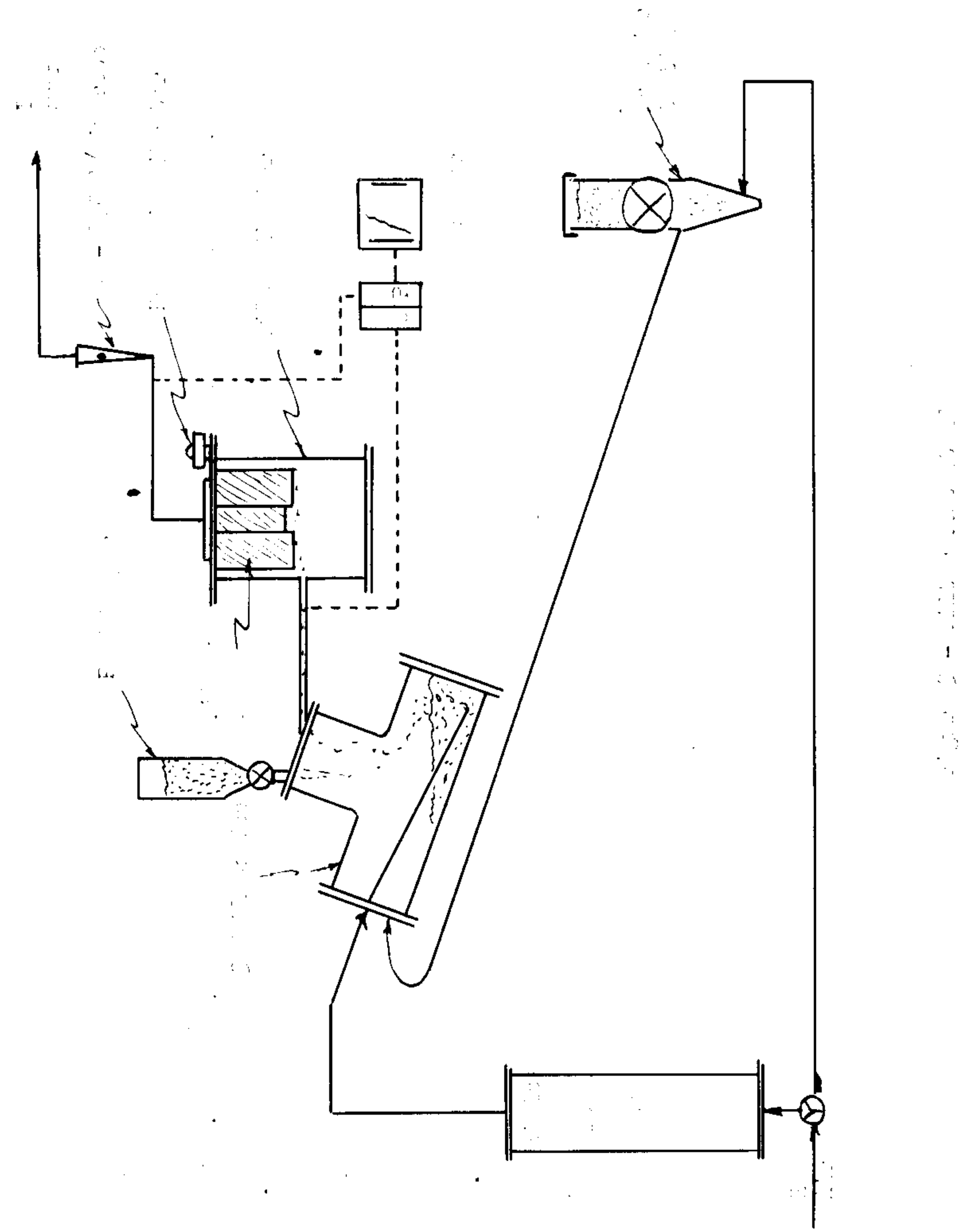


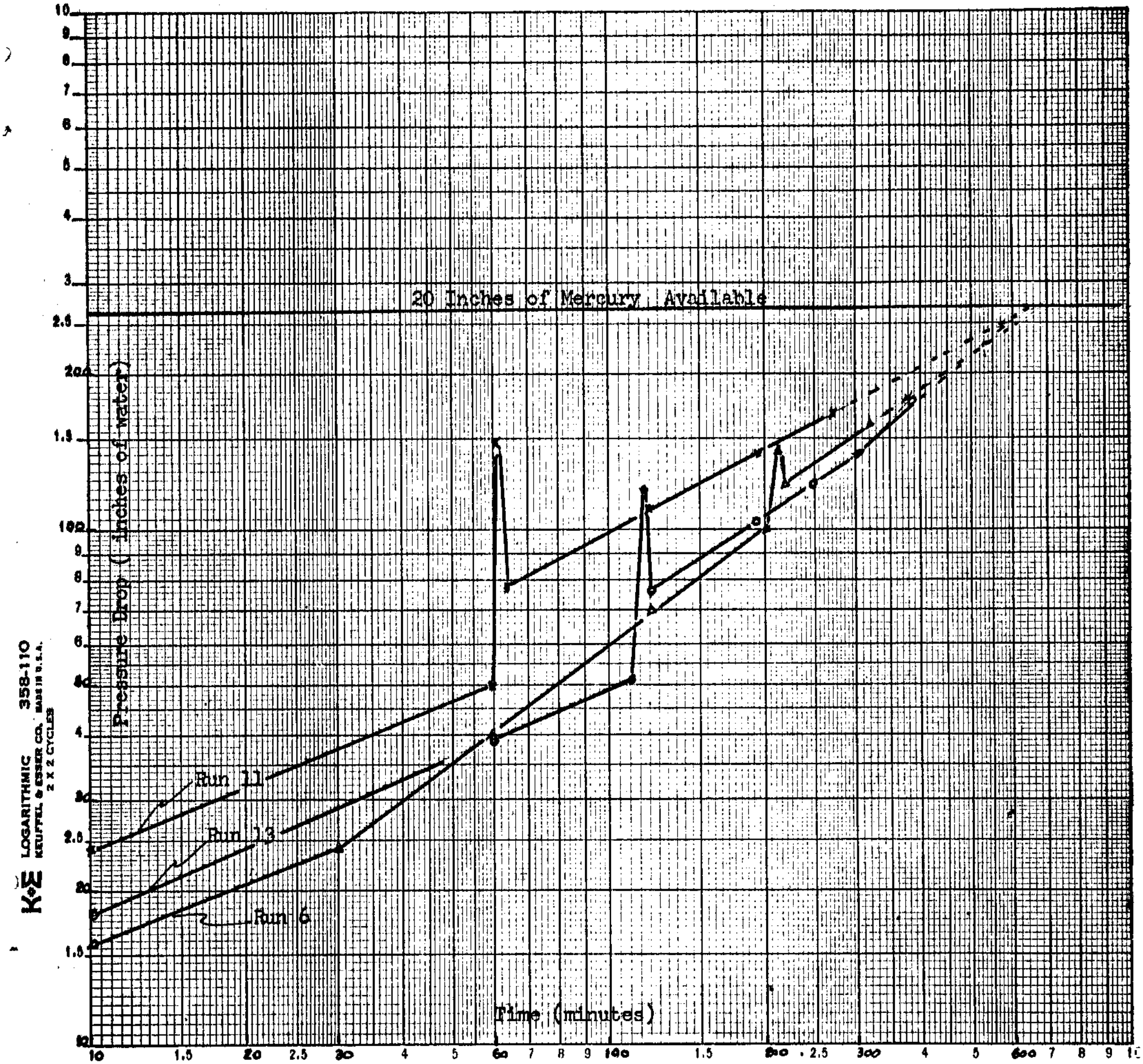
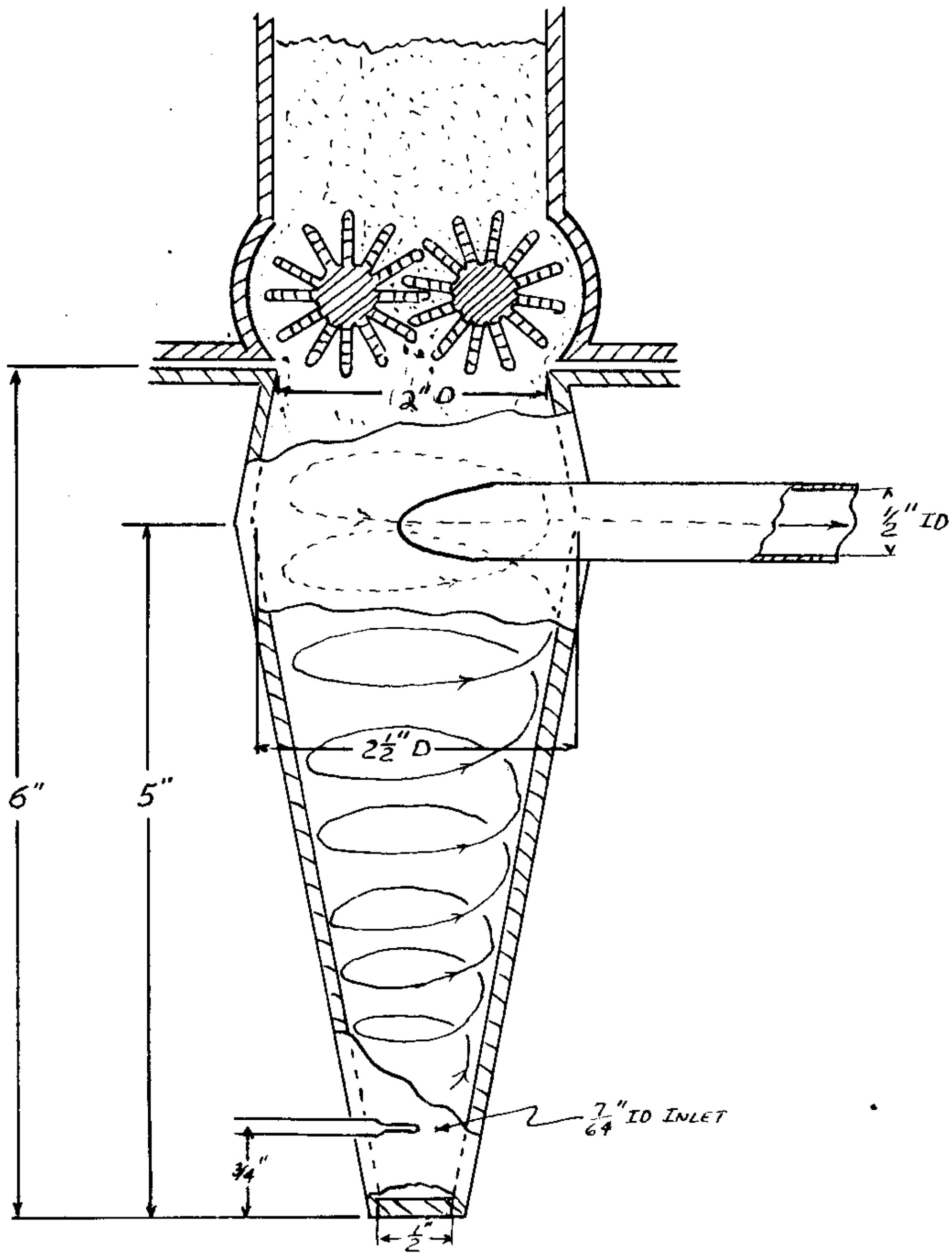


Figure 4 - Filter Assembly Pressure Drop History


2" D
1/2" ID
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