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A PILOT PLANT FOR THE REDUCTION OF URANIUM HEXAFLUORIDE TO URANIUM TETRAFLUORIDE WITH TRICHLOROETHYLENE

J. E. Baker, H. V. Klaus,
R. A. Schmidt, and S. H. Smiley

Engineering Development Department
Technical Division
D. M. Lang, Superintendent

UNION CARBIDE NUCLEAR COMPANY
UNION CARBIDE AND CARBON CORPORATION
Oak Ridge Gaseous Diffusion Plant
Oak Ridge, Tennessee

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ABSTRACT

Pilot plant experiments are described in which trichloroethylene was used for the reduction of uranium hexafluoride to uranium tetrafluoride. After the preliminary results with liquid phase reduction had proven to be unsatisfactory, satisfactory results were obtained with a vapor phase reduction system.

It was found that vapor phase reduction at approximately 450°F., produced a low density product which contained only small quantities of uranium(VI); sintering the uranium tetrafluoride in a hydrogen fluoride atmosphere increased the product density to approximately 3 g./cc. The reduction was essentially complete, and the effluent gas contained less than 1 ppm. of uranium hexafluoride. The purity of the uranium tetrafluoride produced was equivalent to that of the uranium hexafluoride used as feed.

A complete discussion is given of the operation of the various parts of the system.

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A PILOT PLANT FOR THE REDUCTION OF URANIUM HEXAFLUORIDE TO URANIUM TETRAFLUORIDE WITH TRICHLOROETHYLENE

Early in 1947 the K-25 Laboratory Division showed that trichloroethylene was a promising reducing agent for the conversion of uranium hexafluoride to uranium tetrafluoride. Based on the Laboratory results the Design and Development Department started pilot plant work in October of 1947. The first experiments were performed with trichloroethylene in the liquid phase. The operation of a 16-foot trichloroethylene spray tower indicated that a satisfactory uranium tetrafluoride product could be obtained, but that the separation of solid uranium tetrafluoride from liquid trichloroethylene was not practicable. However, it was judged that the major operating difficulties encountered in spray tower operation would be eliminated if the reaction were carried out in the vapor phase.

Since the Laboratory Division had, independently, begun work on a small scale vapor phase reactor, further investigations by this Department were temporarily suspended. After subsequent laboratory experiments showed that uranium hexafluoride could be satisfactorily reduced to uranium tetrafluoride, and that the product could be purified and densified by sintering in an atmosphere of hydrogen fluoride[1][2], a pilot plant unit was built to obtain engineering data for the design of a system capable of operating under plant conditions. A process has been worked out for the continuous production of uranium tetrafluoride.

SUMMARY

The reduction of uranium hexafluoride to uranium tetrafluoride employing trichloroethylene as the reducing agent was studied at three different temperatures and corresponding phase conditions.

1. Liquid trichloroethylene at approximately 70°F., where the vapor pressure of trichloroethylene is relatively low (the "cold" liquid system).

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(1) Allen, A. L., Bernhardt, H. A., Mahoney, W. L., Massey, B. J., Schaffner, J. G., Wiggin, E. A., Continuous Method of Reducing UF_6 to UF_4 by Means of Trichloroethylene, Union Carbide Nuclear Company, Oak Ridge Gaseous Diffusion Plant, May 17, 1948 (K-215).

(2) Bernhardt, H. A., Gustison, R. A., Kirslis, S. S., Posey, J. C., The Batch Reduction of UF_6 to UF_4 by Trichloroethylene, Union Carbide Nuclear Company, Oak Ridge Gaseous Diffusion Plant, February 9, 1949 (K-348).

2. Trichloroethylene at 180°F., where the vapor pressure of trichloroethylene is almost one atmosphere (the "hot" liquid system).

3. Reaction at 450°F., where the reaction is completely in the vapor phase.

Liquid Trichloroethylene System

Cold Trichloroethylene System. Figure 1 is a schematic flow sheet of the cold liquid trichloroethylene system.

A mixture of uranium hexafluoride in nitrogen (0.6 to 2.5% uranium hexafluoride by volume) was fed to a 16-foot spray column in which liquid trichloroethylene was circulated at room temperature. Nitrogen-uranium hexafluoride gas flows of 750 and 1500 scfd* were employed. Under these conditions, the concentration of uranium hexafluoride in effluent gas was reduced to 0.001 to 0.1 mol % (10 to 100 ppm.). No significant difference in effluent gas composition was observed when the effective height of the column was reduced to 8 feet, indicating that the initial rate of reaction is probably very rapid.

After one to two hours of continuous operation, plugs developed in the uranium hexafluoride feed nozzles even though protecting umbrellas above the nozzles tended to prevent direct contact of the process gas with the liquid spray.

Hot Trichloroethylene System. Figure 2 is a schematic flow sheet of the hot liquid trichloroethylene system.

Heating the trichloroethylene to 180°F. improved the efficiency of the reactions occurring in the 16-foot spray tower. At a flow of 1500 scfd. with a feed gas containing 1.6 mol % uranium hexafluoride, the effluent gas contained only 1 ppm. of uranium hexafluoride.

The solid uranium tetrafluoride from the hot liquid system contained 54% uranium(VI) in the earliest runs. This large amount of hexavalent uranium in the form of uranyl fluoride resulted from the hydrolysis of uranium hexafluoride by the abnormally high concentration of water in the reactor. It was found that by carefully flushing out the system with trichloroethylene to remove the water and by distilling the trichloroethylene before use, the uranium(VI) content of the product removed from the system was reduced to less than 4%.

Some improvement in gas feed nozzle performance was observed when the nozzle was heated electrically and the opening restricted to produce an exit velocity of 30 fps. With this arrangement, the nozzle could function several hours without plugging. A Sharples Super Centrifuge would completely separate the solid product from the liquid, but the removal of solids from the centrifuge bowl introduced a serious handling problem.

*standard cubic feet per day.

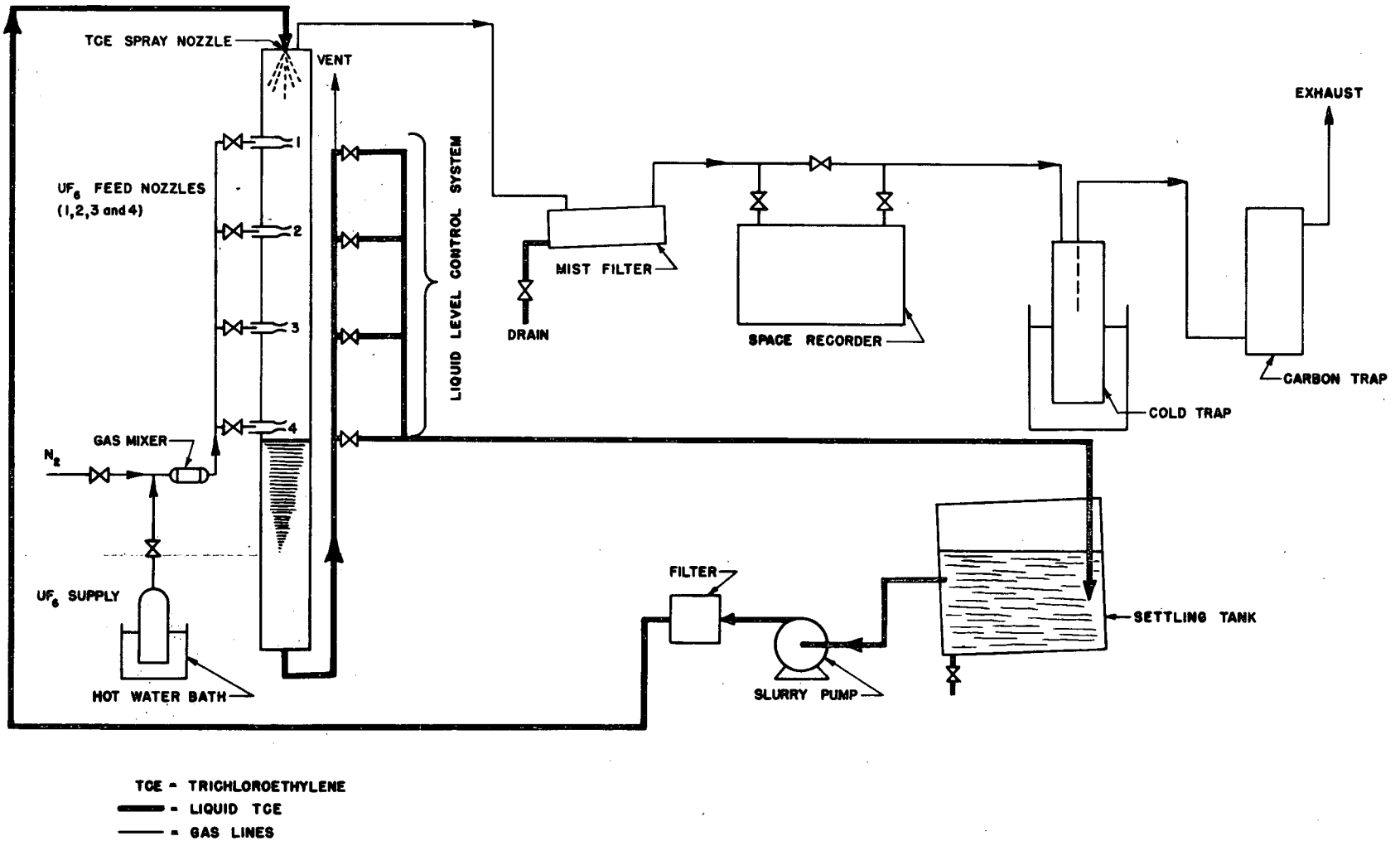

Figure 1
SYSTEM FOR THE REDUCTION OF URANIUM HEXAFLUORIDE
WITH COLD LIQUID TRICHLOROETHYLENE

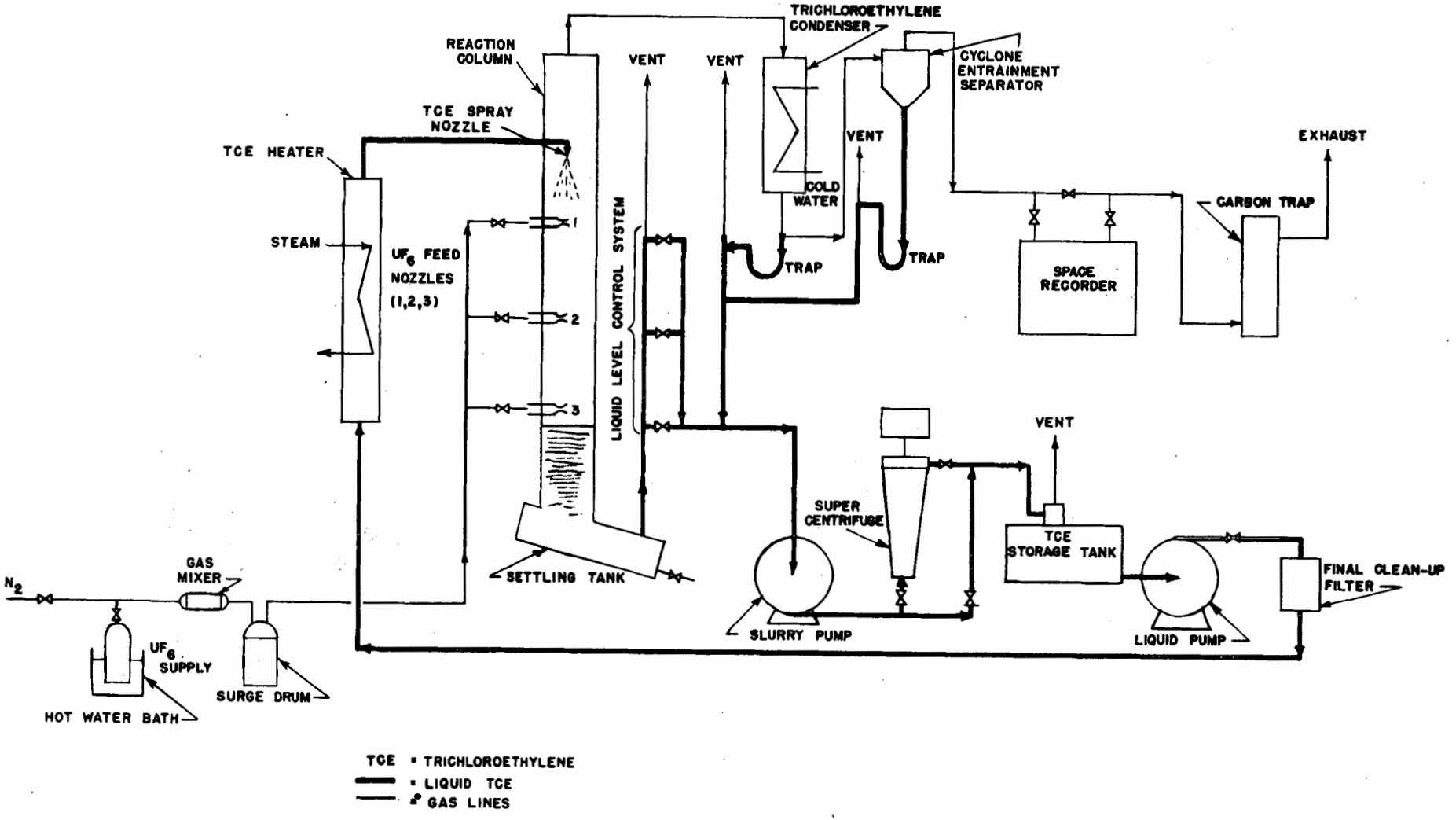

Figure 2
SYSTEM FOR THE REDUCTION OF URANIUM HEXAFLUORIDE
WITH HOT LIQUID TRICHLOROETHYLENE

Vapor Trichloroethylene System

Figure 3 presents a flow sheet of the vapor phase reactor pilot plant which was used in most of the runs. This arrangement allowed better study of each of the components than was possible with the vertical, tandem arrangement of the apparatus used in the final runs (Figure 18) where safety, ease of handling, and general practicability were governing factors.

The first runs were made at atmospheric pressure in a 4-foot Monel reactor, 5 inches inside diameter. Performance at sub-atmospheric pressures was studied in a 7-foot reactor, which was equipped with a funnel to increase the gas path to 11 feet. The preheated uranium hexafluoride-nitrogen and trichloroethylene gas streams were introduced separately to the reactor through two nozzles. No difficulty was encountered with nozzles plugging during operation in the vapor phase. The minimum gas velocity from the uranium hexafluoride-nitrogen nozzle in these runs was approximately 200 fps. at operating temperatures and pressures. Employing feed gas flows of 3200 scfd. with a uranium hexafluoride concentration of 2.0 mol % and a pressure of 3 psia., the effluent gas contained less than 1 ppm. uranium hexafluoride. Other runs with flows from 750 to 2100 scfd. and concentrations in excess of 3 mol % resulted in less than 1 ppm. of uranium hexafluoride in the exit gas at pressures from 2 to 4 psia.

Operating the reactor at 450°F. yielded a low density product with only small quantities of uranium(VI). Sintering the uranium tetrafluoride in a hydrogen fluoride atmosphere increased the product density to an acceptable value (approximately 3 g./cc.).

The ultimate particle size of the uranium tetrafluoride product was determined to be 50 to 90% less than 1 micron and 95 to 99% less than 5 microns; however, much of the product agglomerated to form larger aggregates.

It was observed that 50 to 75% of the solid product settled out in the reactor. Cyclone separators placed in series with the reactor reduced the solids content of the gas to 5 to 10% of its original value. A sintered metal filter placed directly after the reactor removed all of the solids but plugged rapidly because of the large solid load to which it was subjected. The combination of an electrostatic precipitator and a sintered metal filter [redacted] however, was

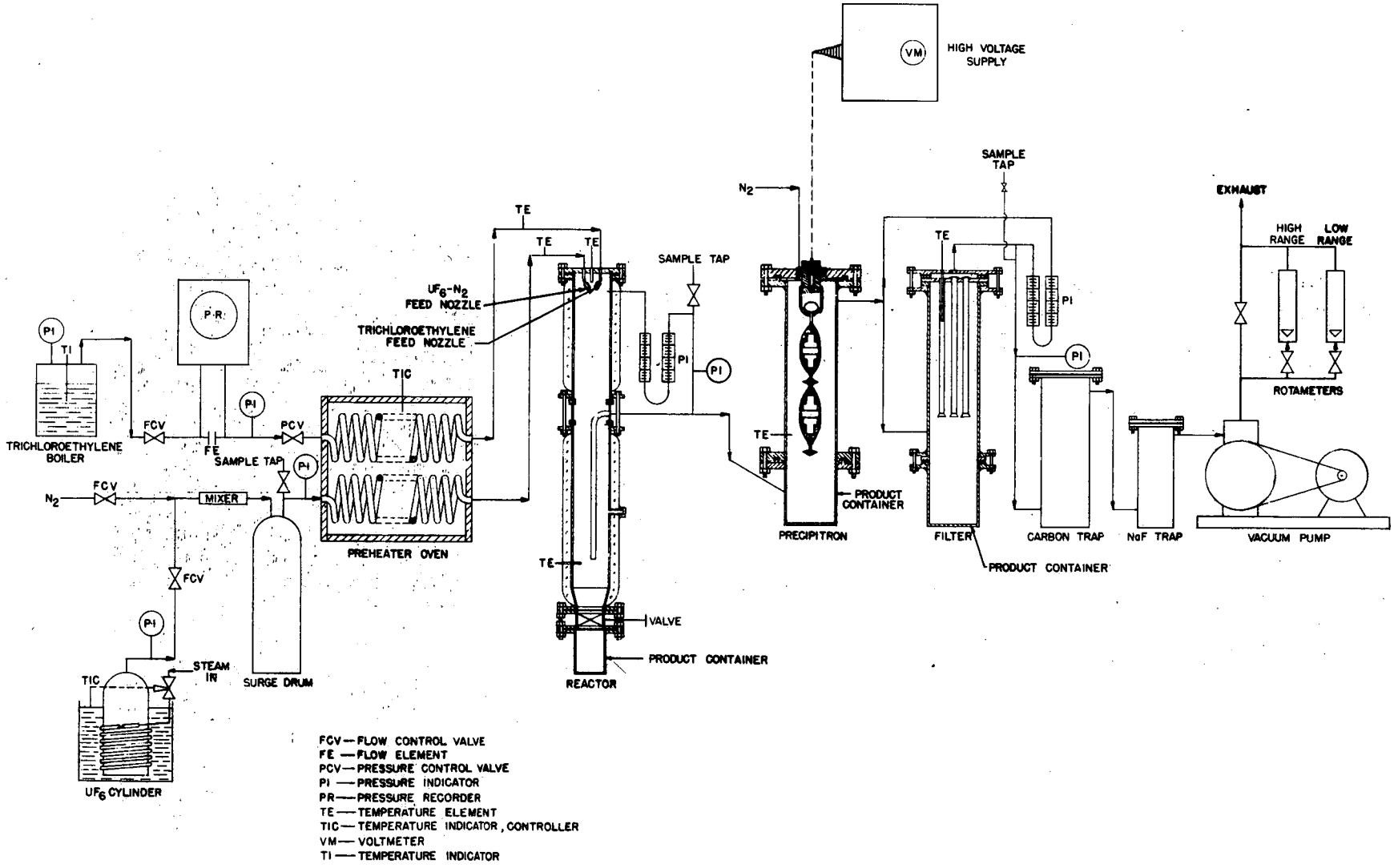

Figure 3
SYSTEM FOR THE REDUCTION OF URANIUM HEXAFLUORIDE WITH TRICHLOROETHYLENE VAPOR

efficiency, and produced a uranium tetrafluoride product which was of the same purity as the uranium hexafluoride feed to the system.

LIQUID TRICHLOROETHYLENE REDUCTION

Cold Liquid System - Equipment

The original pilot plant (Figure 1) was designed to recirculate trichloroethylene through the system at room temperature. The liquid was introduced through a spray nozzle at the top of the column where it fell countercurrent to the rising gas stream containing uranium hexafluoride. The suspension of uranium tetrafluoride in trichloroethylene drained into a settling tank 5 feet in diameter and 4 feet high, and a pump recirculated the liquid from the tank, through a filter and a metering orifice, to the spray nozzle.

The column was built of 4-inch Monel pipe with the liquid spray nozzle at the top and a liquid outlet at the bottom. Flanges were provided at four positions to install gas feed nozzles, 4, 8, 12, and 16 feet from the top of the column. The liquid level was observed through windows placed slightly below the uranium hexafluoride feed nozzles. Overflow valves placed at four levels in the liquid outlet line controlled the effective column height.

Trichloroethylene lines were made of iron pipe and copper tubing, and the settling tank and liquid valves were made of steel. All gas stream lines and cold traps were copper with brass Kerotest valves.

Uranium hexafluoride, vaporized from a cylinder which was heated in a water bath, was introduced into a metered nitrogen stream through a needle valve. The uranium hexafluoride-nitrogen stream passed through a mixing orifice chamber to insure homogeneity. Analytical samples were withdrawn from a sample tap near the gas feed nozzles. The nozzles were made of copper tubing bent downward with a bell shaped umbrellas surrounding the discharge ends.

Leaving the top of the column, the exit gas flowed through a Westinghouse mist filter which removed considerable entrained trichloroethylene and to a cold trap and carbon trap to remove traces of uranium hexafluoride.

A recording ionization chamber was installed to provide continuous analytical data on the uranium hexafluoride concentrations in the feed and exhaust streams but was found unsatisfactory due to relatively large amounts of trichloroethylene in the system.

Cold Liquid System - Results

In all runs, the maximum trichloroethylene flow of 85 pounds per minute was maintained. Uranium hexafluoride-nitrogen gas flows were adjusted at 750 to 1500 scfd.

Uranium hexafluoride concentrations in nitrogen were varied from 0.6 to 2.5 mol %. Column heights of 8 and 16 feet were employed.

Analysis of the effluent gas indicated that it contained from several to approximately 100 ppm. of unreacted uranium hexafluoride. Since no significant difference in the uranium hexafluoride concentration of the effluent gas was observed when either the 8 or 16 feet of column were employed, it was judged that at the lower temperature of operation, the reaction had attained equilibrium for all practical purposes.

Plugging of the gas feed nozzles occurred after one to two hours of operation. Apparently, a fine spray produced by trichloroethylene droplets striking the nozzle umbrellas was chiefly responsible for the plugging, since uranium tetrafluoride was produced inside the gas nozzle opening.

Appreciable quantities of uranium tetrafluoride solid were detected in the filter placed in the trichloroethylene feed line, indicating that the settling tank was only partly effective.

Hot Liquid System - Equipment

The use of hot trichloroethylene necessitated some changes in design to condense the vapors and to separate any liquid entrainment which might result.

A Sharples Super Centrifuge was employed to determine whether complete separation of uranium tetrafluoride product from the trichloroethylene liquid was practicable.

The trichloroethylene spray nozzle in the reaction tower was mounted in the flange located four feet from the top to provide free space for some of the trichloroethylene mist to settle.

The slurry leaving the tower drained to a pump which discharged into a size 16 Sharples Super Centrifuge. The supernatant was stored in a 30-gallon stainless steel drum from which it was pumped through the metering orifice and a copper tube heat exchanger where it was heated to 180°F. Since the hot liquid was the only source of heat for the reaction, the column was well insulated.

The gas feed system was modified by adding a surge tank to reduce pressure fluctuations while gas samples were withdrawn.

A trace indicator sampler was used to detect small quantities of uranium(VI) in the effluent gas. The exhaust gas passed through a condenser to remove trichloroethylene vapor and then through a cyclone to reduce mist entrainment. Traps similar to those in the cold liquid system were used to pick up the last traces of uranium hexafluoride in exit gas.

A new nozzle design was employed which incorporated electrical heating and used a higher velocity jet (30 fps.) together with a spray shield.

Hot Liquid System - Results

The results of the experiments with the hot trichloroethylene system are shown in Table 1. An investigation to determine the cause of the high uranium(VI) concentrations in the solid product from Runs L-1, L-2, L-3 showed that the trichloroethylene recirculating system contained relatively large amounts of water. Although the trichloroethylene was not analyzed, it probably contained some dissolved hexavalent uranium. It is suggested that the high uranium(VI) concentrations in the effluent gas of these early runs was partly caused by entrainment due to formation of trichloroethylene mist. Runs L-4 and L-5 were made after the system was carefully flushed with commercial trichloroethylene. Runs L-6, L-7, and L-8 were performed with trichloroethylene which had been partially purified* by distillation.

It will be noted that reasonably low uranium(VI) concentrations in the solid product were obtained in Runs L-6, L-7, and L-8. The uranium(VI) in the effluent gas was also low even at relatively high gas flows. Reducing the trichloroethylene flow from 55 pounds per minute to 18 pounds per minute had no significant effect on the column stripping efficiency.

The gas feed nozzle, although an improvement over the one used for the cold trichloroethylene experiments, plugged after several hours of operation.

The liquid leaving the Super Centrifuge contained less than 0.1 ppm. of uranium.

Upon completion of these tests, work on the hot liquid spray system was suspended in favor of a pilot plant investigation of a vapor phase reactor, since Laboratory studies indicated greater feasibility for such a system.

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*The distilled trichloroethylene contained small concentrations of pyridine, a corrosion inhibitor.

TABLE I

TRICHLOROETHYLENE AT 180°F.

Run No.	Gas Feed Rate, scfd.	Gas Feed Composition, Mol % UF_6	Effective Column Length, ft.	Trichloroethylene Flow Rate, lbs./min.	Effluent Gas Analysis, ppm. UF_6	UF_4 Product Analysis, % Uranium(VI) not reduced
*L-1	800	1.8	12	53	30	--
*L-2	750	1.7	16	53	5	54
*L-3	750	1.3	8	53	21	--
L-4	750	1.9	8	55	7	13
L-5	750	1.9	8	18	10	--
L-6	1400	1.4	8	55	6	6.8
L-7	1800	1.7	12	55	2	8.8
L-8	1500	1.6	16	55	1	3.4

*In Runs L-1, L-2, L-3, relatively large amounts of water were present in the system.

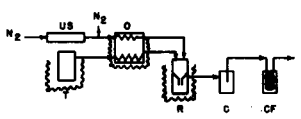
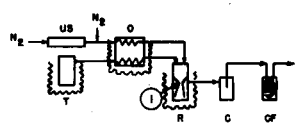
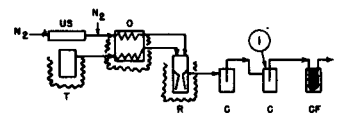
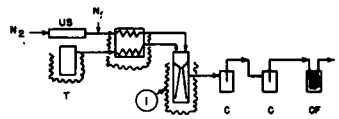
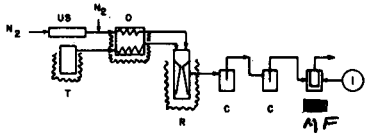
VAPOR TRICHLOROETHYLENE REDUCTION

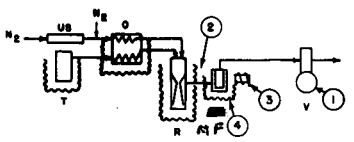
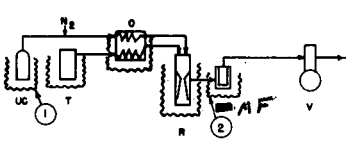
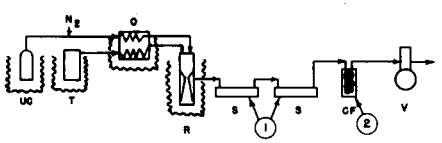
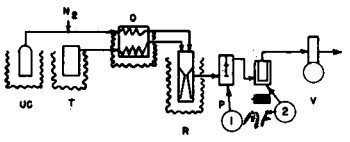
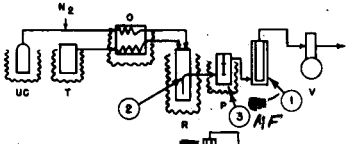
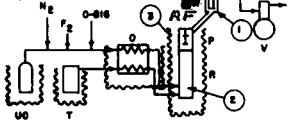
Figure 4 is a schematic representation of changes made in the pilot plant set-up as dictated by operating experience.

Equipment

Uranium Hexafluoride Feed System. The original uranium hexafluoride feed system involved the use of a gas saturator to introduce uranium hexafluoride in the nitrogen stream. A small metered flow of nitrogen was passed through a heated horizontal tube 3 inches by 3 feet long, containing solid uranium hexafluoride; the uranium hexafluoride sublimed and was carried out by the gas stream. A second metered nitrogen stream diluted the effluent from the uranium hexafluoride saturator to the desired feed concentration.

The saturator method proved unsatisfactory when the reactor was operated below atmospheric pressure. The saturator tube was replaced by a uranium hexafluoride shipping cylinder (50 lbs. capacity), which was heated in a temperature controlled water bath. The vapor pressure of the uranium hexafluoride was indicated by a pressure gauge. The uranium hexafluoride and nitrogen flows were controlled by means of Hoke needle valves, and the gas streams were mixed in a chamber to insure homogeneous feed. The uranium hexafluoride concentration was determined by laboratory analysis, and the inert gas flow was measured by a rotameter on the vacuum pump discharge. A surge drum was installed to provide capacity so that feed samples could be withdrawn without affecting the system. Before entering the reactor, the gas mixture passed through a preheater (a Monel coil in an oven maintained at 450°F.).

ARRANGEMENT	RUN NO.	REMARKS
(1) 	1, 2, 3	
(2) 	4	(1) REPLACED SHORT FUNNEL WITH LONGER ONE
(3) 	5	(1) INSTALLED SECOND CYCLONE
(4) 	6	(1) INSTALLED 7' REACTOR & LONG FUNNEL
(5) 	7	(1) REPLACED COTTON FILTER WITH  Sintered Metal

ARRANGEMENT	RUN NO.	REMARKS
(6) 	8	(1) INSTALLED VACUUM PUMP (2) REMOVED CYCLONES (3) PLACED VIBRATOR ON FILTER (4) REMOVED VIBRATOR, HEATED FILTER
(7) 	9, 10 11, 12, 13, 14	(1) REPLACED UF_6 SATURATOR WITH CYLINDER (2) HEATED FILTER
(8) 	15, 16	(1) INSTALLED SETTLERS (2) INSTALLED HIGH FLOW COTTON FILTER
(9) 	17	(1) INSTALLED PRECIPITRON Sintered metal FILTER, (2) INSTALLED Sintered metal FILTER
	18	(1) FILTER (2) FUNNEL REPLACED WITH 3/4" TUBE
(10) 	19, 20	(3) HEATER ON PRECIPITRON
	A-1, A-2, A-3 A-4, A-5	Sintered metal (1) (2) 3/4" TUBE REMOVED. NO FUNNELS, TUBES OR BAFFLES IN REACTOR. (3) PRECIPITRON EQUIPPED WITH SCRAPER
(11) 		

KEY

R — REACTOR
O — PREHEAT OVEN
C — CYCLONE
CF — COTTON FILTER
MF — METAL FILTER
P — PRECIPITRON
C-816 PERFLURO DIMETHYL-HEXANE (COOLANT)
V — VACUUM PUMP
T — TRICHLORDETHYLENE BOILER
US — UF_6 SATURATOR
UC — UF_6 CYLINDER
S — SETTLER
— HEATER

Sintered Metal

Figure 4
SCHEMATIC REPRESENTATION OF CHANGES TO VAPOR PHASE REDUCTION SYSTEM

Trichloroethylene Feed System. The trichloroethylene system consisted of a boiler, a flow control valve, an orifice type flow meter, and a pressure control valve located downstream from the orifice. All of these units were manually operated.

The boiler was a stainless steel container having a Calrod heater with a Variac to control the temperature. A thermo-well with a thermometer and a pressure gauge indicated the temperature and pressure, while a Hoke needle valve controlled the flow. The ΔP across the 0.0135-inch orifice was measured and recorded by a DBM and a pressure recorder. For convenience, the downstream pressure at the orifice was maintained at atmospheric pressure for all runs.

The trichloroethylene vapor passed through the preheater where the temperature was raised to 450°F. before entering the reactor. The trichloroethylene used was treated with sulfuric acid to precipitate the pyridine inhibitor and was then doubly distilled. Results of a spectrographic analysis on a sample of trichloroethylene, as used in the system, are presented in Table 2.

TABLE 2

RESULTS OF SPECTROGRAPHIC ANALYSIS FOR METAL IMPURITIES IN DOUBLE-DISTILLED TRICHLOROETHYLENE

Cu	0.1 ppm.
Fe	0.5 ppm.
Mg	1.0 ppm.
Mn	0.1 ppm.
Ni	1.0 ppm.
Si	1.0 ppm.

Elements not found:

Ag	In
Al	Li
As	Mo
Be	Na
Bi	P
Cd	Sb
Co	Ti
Cr	Tl
Ga	V
Hg	W
	Zn

Fluorine Feed System. Fluorine was supplied to the unit from a building header. Flow was controlled to the system by a Hoke needle valve, was

measured by an orifice, and transmitted through a DBM to a pen pressure recorder. The fluorine was fed into the nitrogen-uranium hexafluoride gas stream at a point before the gas preheat oven and reached the reactor through the nitrogen-uranium hexafluoride nozzle.

Coolant Feed System. The coolant (perfluorodimethylcyclohexane) was fed into the nitrogen-uranium hexafluoride gas stream from a burette at a point before the gas preheat oven. Flow from the burette was controlled by a Hoke needle valve. The coolant vapor was introduced into the reactor through the nitrogen-uranium hexafluoride nozzle.

Sample Taps. Figure 2 and Figure 18 show the location of sample taps in the unit. The concentration of uranium hexafluoride in the gas stream was measured before entering the reactor, after leaving the reactor, and after leaving the filter.

Reactor. The reactor consisted of a 5-inch inside diameter reaction chamber, a set of feed nozzles, a settler, and a gas outlet. The entire reactor was heated electrically. In Runs 1 through 20, the nozzles entered the top of the reactor and were directed toward each other so the gas streams met several inches below the nozzles. The reactants passed into the reaction chamber where the uranium tetrafluoride powder was produced. The dust-laden gas continued down through the funnel (except Runs following Run 17 - Figure 4 - Arrangement 10) and an abrupt change in direction of flow occurred as the stream turned up around the funnel spout toward the outlet. The powder fell through a steel gate valve and was removed in a nickel receiver which was bolted to the bottom of the reactor.

The first reactor was a 4-foot Monel tube with a funnel suspended halfway down. The outlet was located directly below the funnel cone. At a flow of 1500 scfd. the gas passed through the reactor in about 15 seconds. Two 1,000-watt heating coils maintained the reactor at operating temperatures and were controlled manually by Variacs.

The reactor height was later increased by adding a 3-foot section. The funnel and take-off points were raised and installed at the point where the two sections joined (Figure 5). The new section was heated by a 1000-watt heating coil. Using the larger reactor, the average retention time of the reacting gases was nearly 30 seconds at atmospheric pressure with a flow of 1500 scfd. When the system operated at 2 psia., the contact time was reduced to 4 to 5 second. For Runs 18, 19, and 20, the funnel in the reactor was replaced by a 3/4-inch pipe which extended to the bottom of the reactor* to prevent short circuiting and to carry the effluent gases out of the system.

*Solid uranium tetrafluoride, formed in the reactor, bridged the funnel opening and created a prohibitive pressure drop across the reactor.

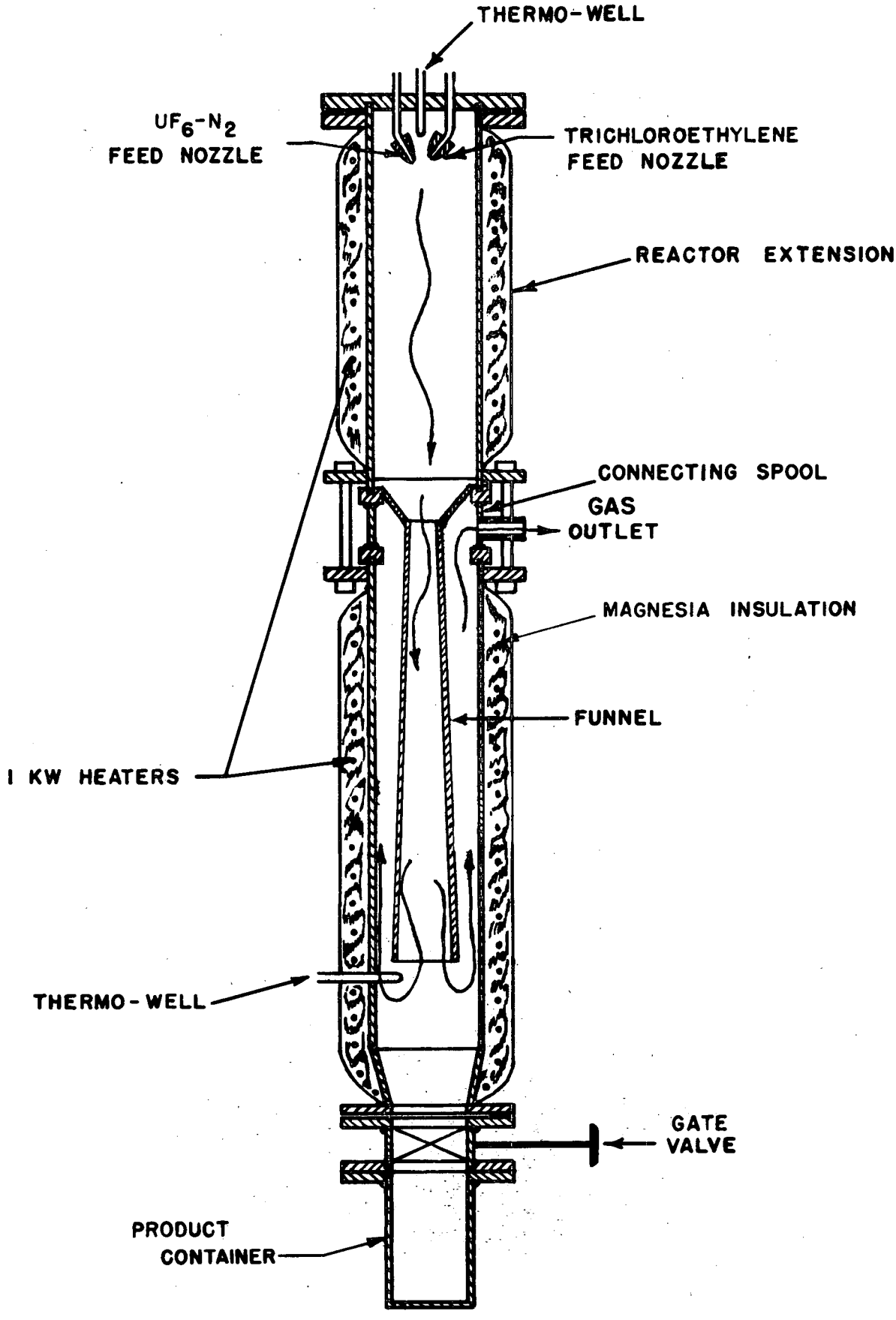


Figure 5
VAPOR PHASE REDUCTION REACTOR

The trichloroethylene and uranium hexafluoride-nitrogen nozzles were made of brass and sized to provide a minimum velocity of 200 fps. at 450°F., 750 scfd. and 2 psia.

Upon completion of the first 20 runs, enough data were collected to indicate that a vertical, tandem arrangement of components was desirable and feasible. For Runs A-1 through A-5 the equipment was rearranged as shown in Figure 18 and Figure 9. The 7-foot reactor employed for the preceeding runs was also used in this series of runs. However, the 3/4-inch pipe was removed and no other funnel or baffle was substituted. The gas feed lines were introduced into the reactor at a point about 3 feet from the bottom of the chamber and continued down the inside wall of the reactor to a point about 1-1/2 feet from the bottom where they terminated in the gas feed nozzles.

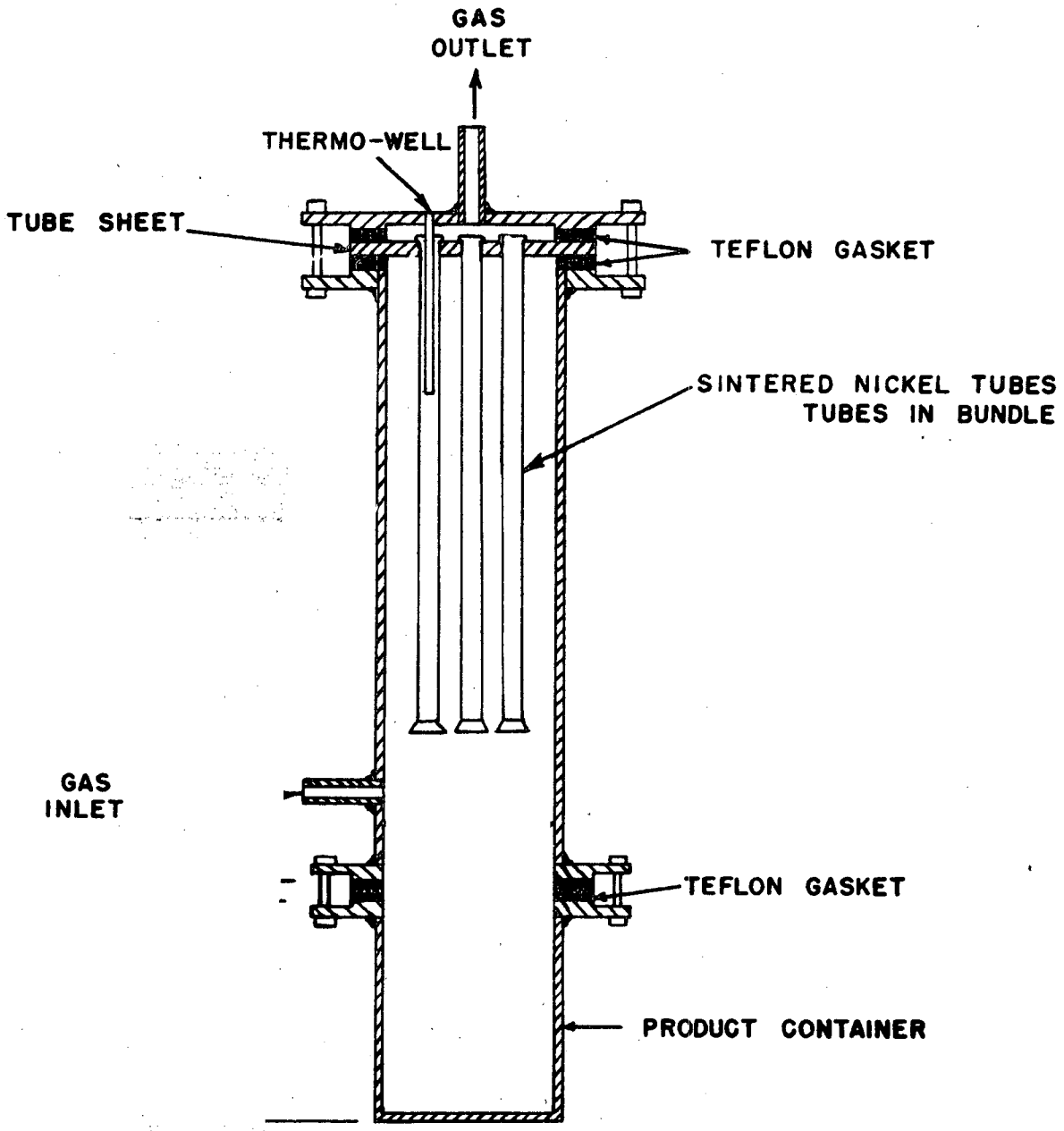
The trichloroethylene and uranium hexafluoride-nitrogen nozzles were made of nickel and sized to provide a minimum velocity of 100 fps. at 450°F., 750 scfd. and 2 psia.

Cyclone Separators. Since the uranium tetrafluoride produced was a light powder of small particle size, a large percentage of it was carried out of the reactor with the exit gas.

The first device employed to collect this dust was a cyclone separator followed by a Ful-Flo cotton filter. At the time the cyclone was designed, very little was known about the size or other characteristics of the uranium tetrafluoride powder. The cyclone, however, was designed to handle a gas flow of 1500 scfd. and to remove particles as small as 10 microns in diameter. Four runs were made using the reactor, cyclone and a Ful-Flo cotton filter arranged in series, and three experiments were conducted with two cyclones in series before the cotton filter. Monel receivers were bolted to flanges on the bottom of the cyclones for removal of the product.

Settling Drums. To study the settling rates of the uranium tetrafluoride product, the cyclones were removed and two settling chambers in series were used directly after the reactor. The settlers were cylinders, each with a cross sectional area of about one square foot and about four feet in length. Two runs were made using this arrangement.

Sintered Metal Filters. The performance of a sintered metal filter was next determined. The first filter unit consisted of a bundle containing four [redacted] tubes of sintered metal. The bottom end of each tube was sealed with a nickel plug welded in place, and the top ends were rolled into a nickel sheet. The tube bundle was mounted in a shell so that gas entering the filter flowed from the outside of the tubes to the inside through the tube walls. It was necessary to change the size of the bundle twice in subsequent runs to obtain filters with greater life expectancies. Thus, the second filter was a bundle of four [redacted] tubes, and finally the filter contained a bundle of four [redacted] Figure 6 presents the structural details of [redacted] filter.



NOTE:
CONSTRUCTED SO THAT THE ONLY METAL SURFACE CONTACTING GAS IS NICKEL.

Figure 6
VAPOR PHASE SYSTEM FILTER

Precipitron. After testing the cyclone as a possible solids remover, a Precipitron was installed in its place. The Precipitron, which was designed and built by the Instrument Department, is shown in Figure 7.

The Precipitron, constructed of nickel, consisted of a 4-foot vertical section of 4-inch pipe with a 1.5-inch diameter electrode suspended from the cover. The electrode was insulated from the shell by a fluorothene gasket. The unit was equipped with a nitrogen bleed to keep the uranium tetrafluoride dust from gathering on the insulator. A nickel receiver was mounted on the bottom flange of the Precipitron for ease of removal of product. The Precipitron was designed to operate with a maximum potential of 15 kv.

For the series of runs A-1 to A-5 a new Precipitron was used. This precipitator retained all the features of the first Precipitron and also included a manually operated scraper device for keeping the product from accumulating on the electrode and inside wall of the Precipitron. The Precipitron was constructed so that it could be mounted on top of the reactor. Gases exit from the Precipitron through a 2-inch pipe arranged at a 22° angle to the Precipitron wall and leaving the Precipitron about 1 foot from the top (see Figure 15).

Results - Runs 1 - 20 (Components of the Pilot Plant Arranged to Test Individual Unit Efficiencies)

In all of the tests with the vapor phase reactor, essentially complete conversion of the uranium hexafluoride to uranium tetrafluoride was realized. The concentration of uranium hexafluoride in the effluent gas was less than 1 ppm., and as can be seen in table 3 the uranium(VI) content of the product was low.

Product Density. The apparent density of the uranium tetrafluoride produced by this reaction was found to vary from 0.22 to 1.62 g./cc.

The uranium tetrafluoride was sintered in a platinum lined bomb in half an atmosphere of hydrogen fluoride at 1650°F. for about 2 hours[3]. The bomb was then cooled and opened, and the product was removed and ground. Sintering the product in a hydrogen fluoride atmosphere increased its density to an approximate value of 3 g./cc. (Table 4).

---

(3) Bernhardt, H. A., Gustison, R. A., Kirslis, S. S., Posey, J. C., The Batch Reduction of UF_6 to UF_4 by Trichloroethylene, Union Carbide Nuclear Company, Oak Ridge Gaseous Diffusion Plant, February 9, 1949 (K-348).

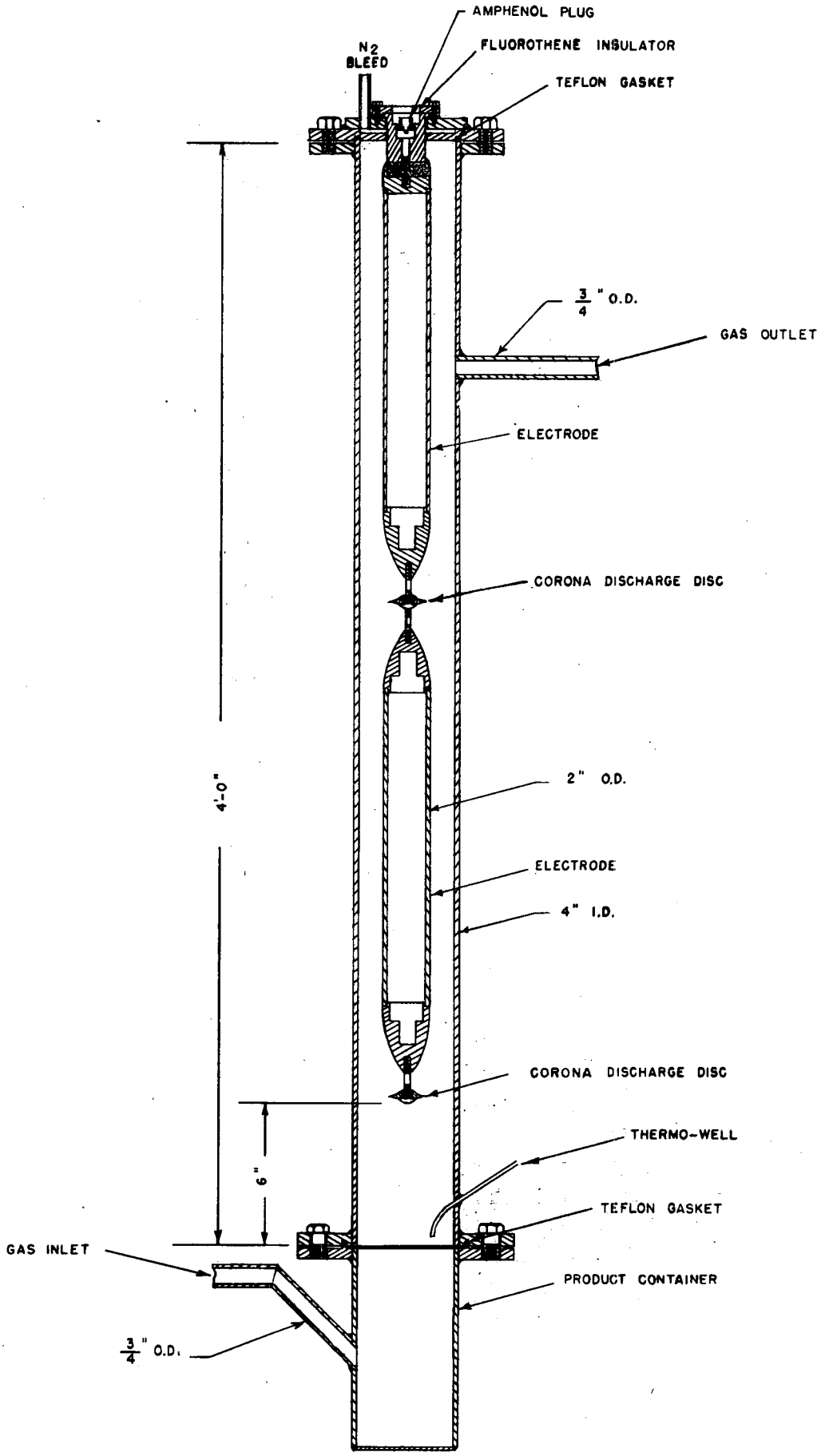


Figure 7
VAPOR PHASE SYSTEM PRECIPITRON

TABLE 3

URANIUM(VI) CONTENT OF PRODUCT (VAPOR PHASE REACTOR)

	Before Sintering		After Sintering	
Run No.	Total Uranium, %	Uranium(VI), %*	Total Uranium, %	Uranium(VI), %*
5			75.7	0.3
6	75.5	3.8		
7	75.1	5.0	75.8	0.0
10 and 11	76.1	0.15	76.1	0.15
14			75.7	0.3
17	75.6	2.0	75.8	0.1
18	77.0	7.1	75.7	1.6
20	76.0	5.9	75.9	2.7

*The analytical procedure employed to determine the uranium(VI) content was later found to be inaccurate (biased high in most cases) and is probably responsible for the high values obtained before sintering.

TABLE 4

APPARENT DENSITY OF URANIUM TETRAFLUORIDE PRODUCT
(VAPOR PHASE REACTOR)

Run No.	Apparent Density Before Sintering, g./cc.	Apparent Density After Sintering, g./cc.
5	0.22	2.87
6	0.50	1.94*
7	0.40	3.00
14	0.71	2.39
17	0.68	3.01
18	1.25	3.28
20	1.62	3.76

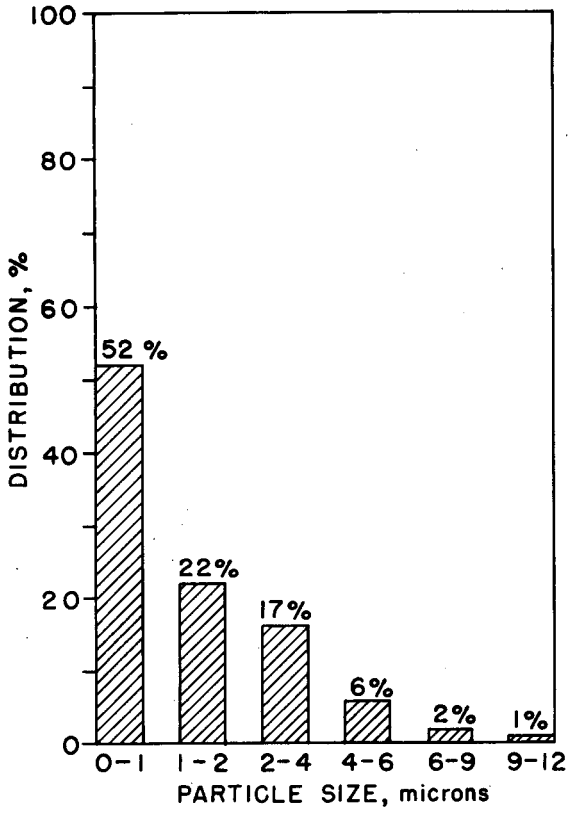
*First sample sintered - Procedure changed so that sintering and grinding times increased.

Particle Size of Product. Particle size measurements were made on the unsintered uranium tetrafluoride (Table 5), by making photomicrographs and measuring the sizes of the magnified particles. It will be noted that, on the average, 50 to 90% of the product was less than 1 micron and 95 to 99% was less than 5 microns. Particle size distribution for runs 18 and 20 is shown in Figures 8 and 9.

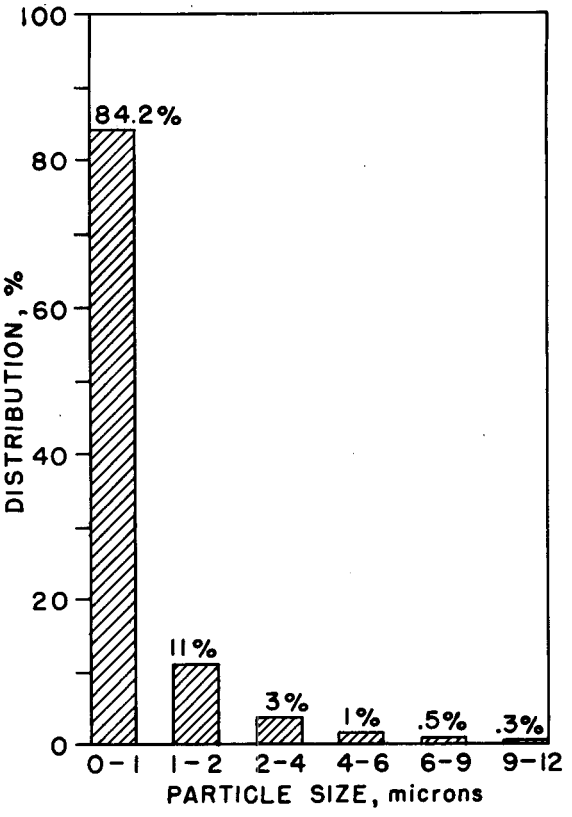
TABLE 5

PARTICLE SIZE OF PRODUCT (VAPOR PHASE REACTOR)

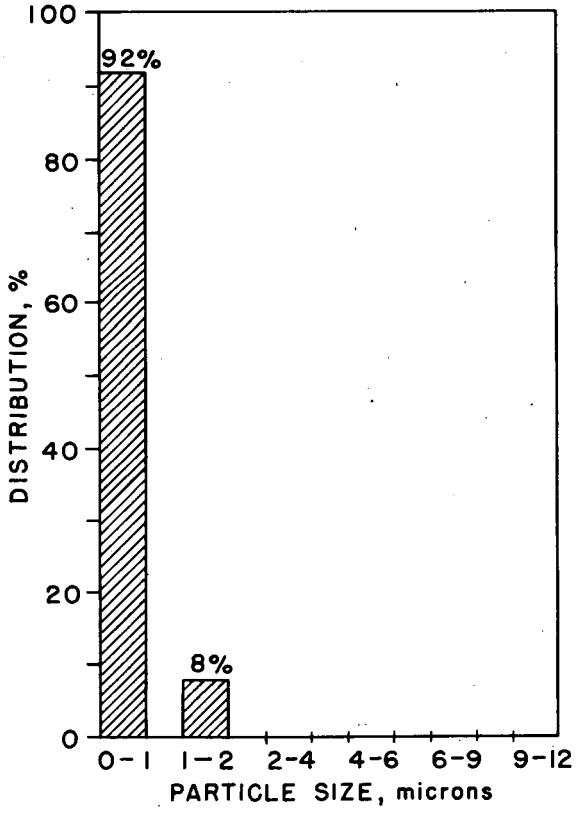
Run No.	Equipment	Particle Size Distribution, %											
		<0.6	<1.0	0.6-1.5	1.0-3.0	1.5-3	3-5	5-10	>5	>10 microns			
7	Reactor		46.8		46.4		5.2		1.6				
	Cyclone No. 1		52.7		42.1		4.3		0.9				
	Cyclone No. 2 Filter		47.1		49.5		2.8		0.6				
8	Reactor		35.7		51.8		9.2		3.3				
	Filter		47.8		47.4		2.9		1.8				
10 and 11	Reactor	74.9		19.5		4.4	0.7	0.5		0.0			
	Filter	41.3		44.8		11.1	2.1	0.7		0.0			
14	Reactor + Settler	60.6		32.7		4.5	2.1	0.7		0.0			
15 and 16	Reactor + Settler No. 1	60.8		31.7		5.4	1.5	0.5		0.0			
17	Reactor		71.8		22.9		4.0		1.3				
	Precipitron		71.5		25.2		2.8		0.5				
	Filter		84.4		15.3		0.3		---				
		<1	1-2	2-4	4-6	6-9	9-12	12-18	18-24	24-36	36-48	>48 microns	
18	Reactor	53.4	21.4	16.3	5.1	2.2	0.8	0.3	0.3	0.2	0.1	0.0	
	Precipitron	84.8	10.9	3.4	0.7	0.2	0.1						
		<0.5	0.5-1	1-1.5	1.5-2	2-3	3-5 microns						
20	Filter	81.6	13.9	1.6	2.3	0.4	0.3						
	Precipitron	87.2	11.5	1.1	0.3								
	Reactor	68.8	23.8	4.8	2.5								



RETAINED IN REACTOR

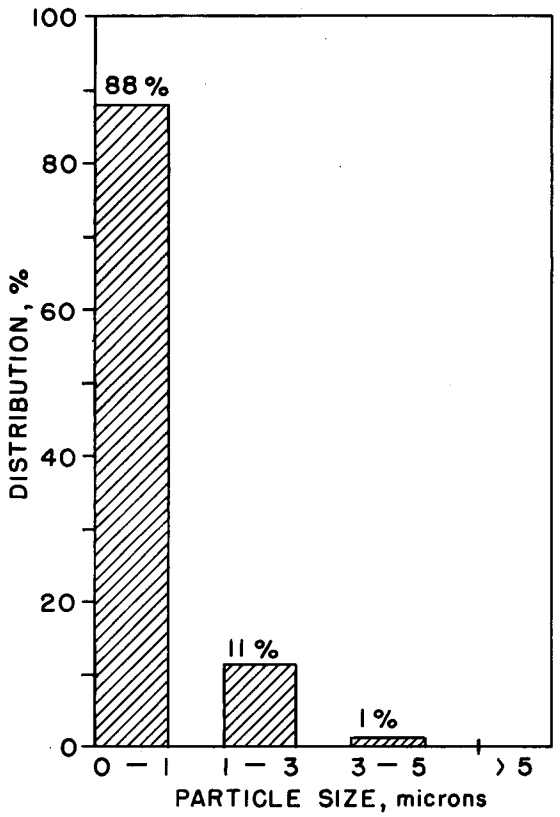


RETAINED BY PRECIPITRON

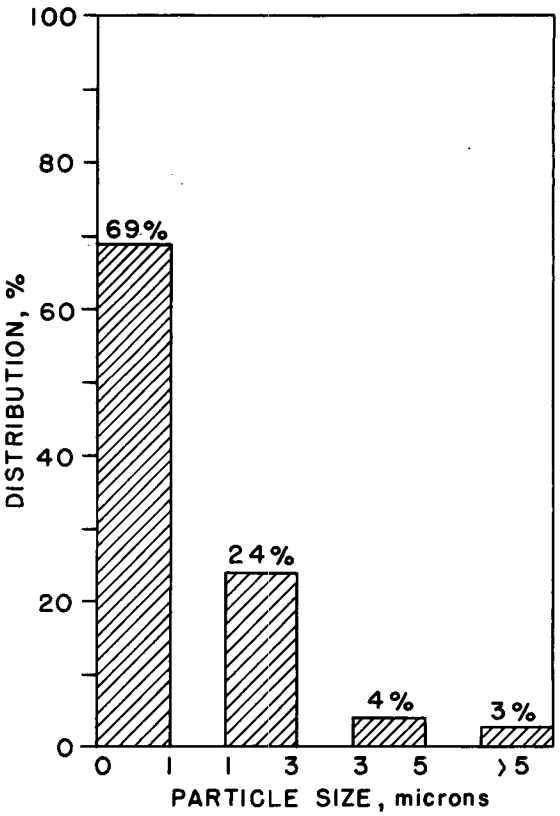


RETAINED IN FILTER

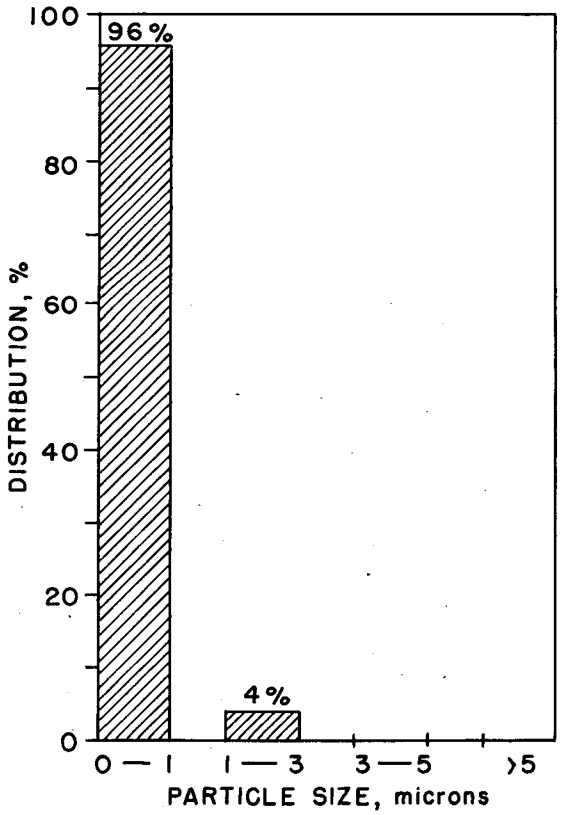
Figure 9
PARTICLE SIZE DISTRIBUTION - RUN 20



RETAINED IN PRECIPITRON



RETAINED IN REACTOR



RETAINED IN FILTER

Figure 8

PARTICLE SIZE DISTRIBUTION - RUN 18

Vapor Phase Reactor Performance. In all runs, where the components of the unit were mounted independently, the uranium tetrafluoride produced in the reaction formed a coat on the walls of the reactor, but excessive build-up was prevented by occasionally rapping the outside of the reactor. A small, and more or less, constant amount of uranium tetrafluoride was held in the reactor in the form of a hard thin crust which adhered tenaciously to the walls, but this material could be readily removed by scraping.

Some difficulties were experienced with plugging of the funnel when uranium tetrafluoride product bridged the opening and created a prohibitive pressure drop in the system. The funnel was removed and a 3/4-inch pipe was installed, which extended to the bottom of the reactor and served as the effluent gas line. Although this line, too, plugged eventually, it was thus possible to collect performance data on the other parts of the withdrawal system. No difficulties were experienced with nozzle operation when a minimum escape velocity of 200 fps. was maintained*.

The pressure drop across the nozzle at a gas velocity of 200 fps. was approximately 0.1 psi.

Approximately 50 to 75% of the uranium tetrafluoride produced settled out in the reactor (Table 6).

Cyclone Separators. The distribution of product for runs 5, 6, and 7 is shown in Table 7.

An empirical relationship involving gas flow, solid "loading" (g. uranium tetrafluoride/cu. ft.) and the efficiency of solids removal can be derived if the reactor and cyclones are considered as parts of a single unit.

The over-all efficiency, E, at the exit of each portion of the unit is related to the "loading", l, and the gas flow rate, V, according to the following empirical equations.

For the reactor:

$$100 - E = 45.5\ V\sqrt{l} \qquad (1)^{**}$$

For cyclone No. 1:

$$100 - E = 12.7\ V\sqrt{l} \qquad (2)^{**}$$

*Although Laboratory studies indicate that plugging will not occur if the velocity of the gas at the nozzle is as low as 30 fps. the higher velocities were studied to determine pressure effects in the system.

**Notes G. H. Montillon to S. H. Smiley.

For cyclone No. 2:

$$100 - E = 8.0\ V\sqrt{1} \qquad (3)*$$

The curves of equation (1), (2), and (3) are plotted in Figure 10 and the agreement with the experimental data is indicated. It should be recognized that equations (1), (2), and (3) are empirical and, therefore, applied only to the specific equipment employed for the study. However, it should be realized that similar equipment should produce comparable but not identical curves.

TABLE 6

REACTOR PRODUCT REMOVAL EFFICIENCY (VAPOR PHASE REACTOR)

Run No.	Grams of UF_4 — In Reactor	Grams of UF_4 — Total	% UF_4 In Reactor	Flow, Scfd.	Pressure, Psia.	Length of Run, Hrs.	Mol % UF_6 in Feed
5	380	460	82.8	560	Atmospheric	2.25	1.1
6	1013	1516	67.0	680	Atmospheric	3.08	3.3
7	1157	2211	52.3	1040	Atmospheric	10.16	1.5
8	1457	2047	70.8	1130	3 - 7	9.85	0.9
9	594	796	74.6	750	2-1/2 - 4	10.16	0.53
10	1016	1457	69.5	750	5	4.5	2.4
12	426	768	55.5	750**	3 - 5**		
				2000**	10 - 12**	6.4	0.46
13	1980	2847	69.5	750**	3 - 5**		
				2000 to 2400	6 - 9	3.65	2.6
14	1762	2324	76.7	1800	7 - 10	2.0	3.45
15	1599	2139	74.7	1750 to 3100	2 - 3	2.0	2.6
17	3266	5200	62.8	1000	2-1/2	8.4	3.3
18	8714	17031	51.8	1800	2-1/2	33.33	1.5
19	1650	2724	60.5	1800	2-1/2	13.83	0.57
20	3112	4751	65.5	1830	2-1/2	10.66	1.3

NOTE: Runs 1, 2, 3, 4, 11, and 16 are not included in this table since no material balances were made for these runs.

**Pressure and flow conditions were changed during run.

*Notes G. H. Montillon to S. H. Smiley.

TABLE 7

DISTRIBUTION OF PRODUCT - EFFICIENCY OF COMPONENTS
(VAPOR PHASE REACTOR)

	Weight Product, g.	Total Product Removed, %	Efficiency of Unit*, %
Run - 5			
Reactor	380	82.8	83
Cyclone No. 1	53	11.5	67
Cyclone No. 2	7	1.5	27
Filter	19.1	4.2	100
Run - 6			
Reactor	1013	67.0	67
Cyclone No. 1	367	24.1	73
Cyclone No. 2	62	4.1	46
Filter	74	4.8	100
Run - 7			
Reactor	1157	52.3	52
Cyclone No. 1	764	34.4	72
Cyclone No. 2	109	4.9	37
Filter	189	8.4	100

*Based on $\frac{\text{amount removed}}{\text{amount received}}$ for each unit.

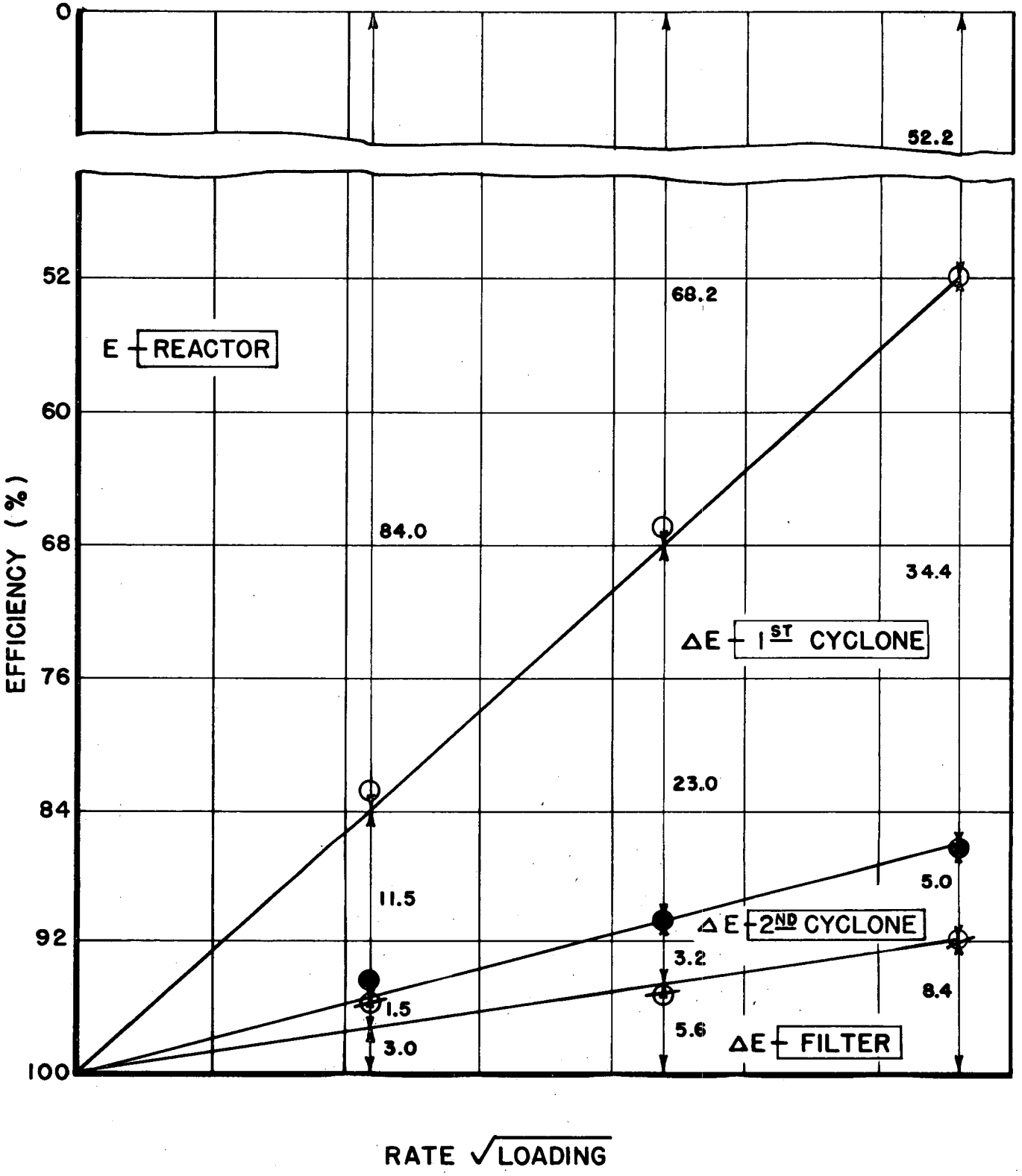


Figure 10
EFFICIENCY OF SOLIDS REMOVAL AS A FUNCTION
OF GAS FLOW RATE AND SOLID LOADING -
VAPOR PHASE SYSTEM

Settling Drums. At an absolute pressure of 2 psia. and a flow of 2200 scfd., the retention time of the gas, which was traveling at slightly over 0.2 feet per second, was 20 seconds in each cylinder.

Most of the product that escaped the reactor was found in the first settler with very little in the second one or in the filter located after the settlers. Except for a small pile-up of uranium tetrafluoride, caused by impingement from the inlet pipe, the material was distributed evenly around the periphery over the entire length of the settler. No true gravity settling was noted.

Precipitron. Table 8 shows the efficiency of solids removal by the Precipitron under varying uranium tetrafluoride loadings and gas flows. Figure 11 is a plot of Precipitron efficiency versus uranium tetrafluoride loads for constant flow conditions, which demonstrates that the efficiency is a direct function of the amount of material fed.

In order to calculate Precipitron efficiencies at any uranium tetrafluoride loading and gas flow, use was made of the following equation:

$$\log (1 - E) = t \log K \qquad (4)*$$

That is, for any given dispersoid of uniform size and character in a given precipitator, the Precipitron efficiency, E, is related to the time that the gas remains in the active field of the precipitator, and K, the so-called precipitation constant for that type of precipitator is always less than 1.

In order to fit the experimental conditions, equation (4) was modified to include the effect of the uranium tetrafluoride load on the Precipitron. It was found that the following empirical relationship correlated the data and allowed prediction of Precipitron efficiency under plant operating conditions.

$$\log (1 - E) = \frac{\log K}{V \times \sqrt{l}} \qquad (5)**$$

where, E = efficiency (a fraction of 1)
V = rate calculated as $ft.^3/min.$
l = loading in grams/$ft.^3$
log K = a negative constant

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*Perry, J. H., Chemical Engineering Handbook, 2nd ed. New York, McGraw-Hill, 1941, p. 1869.

**Notes G. H. Montillon to S. H. Smiley.

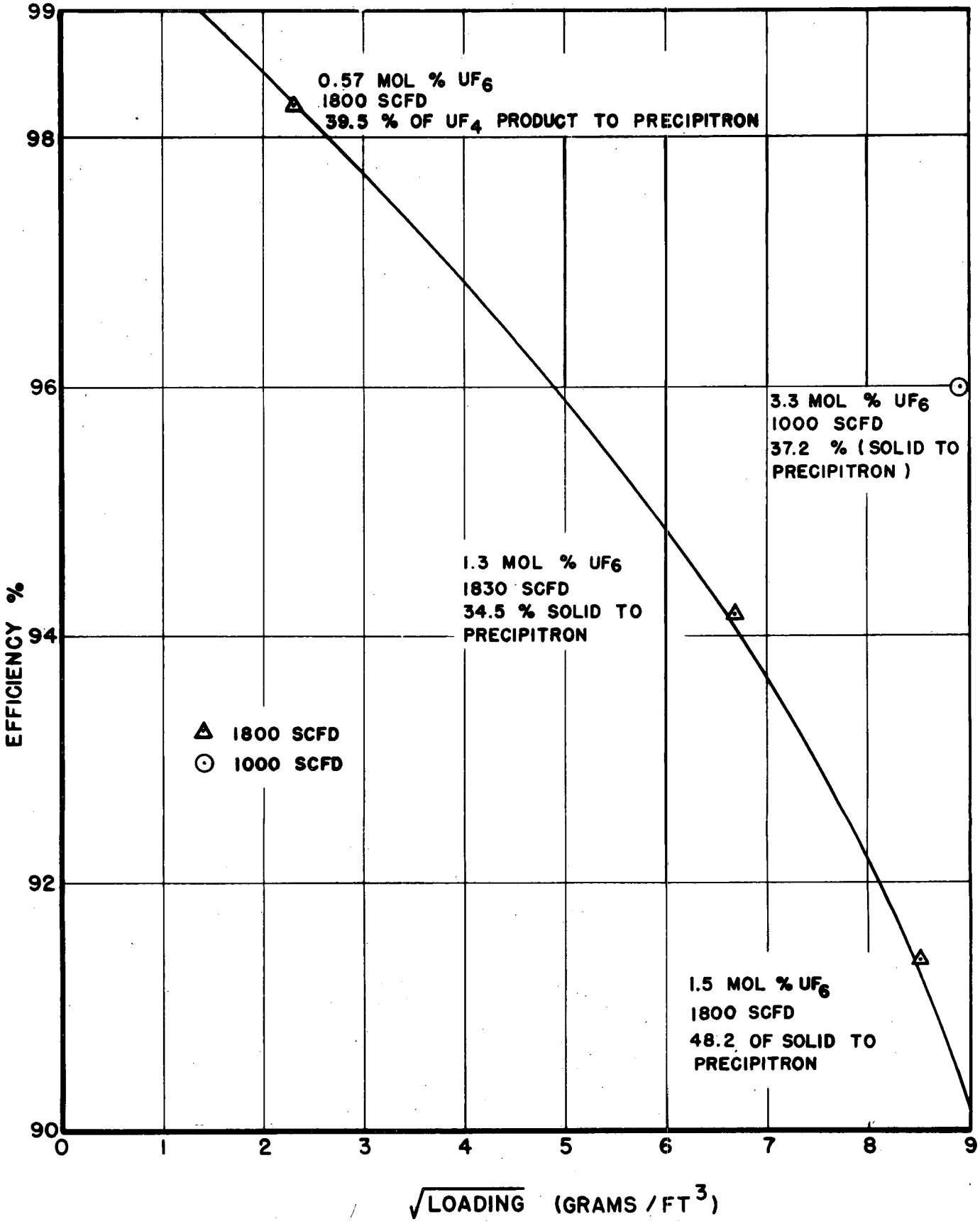

Figure 11
PRECIPITRON EFFICIENCY AS A FUNCTION
OF SOLID LOADING

TABLE 8

EFFICIENCY OF PRECIPITRON

Run No.	Length of Run	Efficiency of Precipitron	% Total Solids Removed		Gas Flow scfd.	UF_6 Conc. mol %	Remarks
			Reactor	Precipitron			
17	8 hrs. 25 min.	96.0%	62.8	35.7	1000	3.3	
18	33 hrs. 20 min.	91.4	51.8	44.0	1800	1.5	
19	13 hrs. 50 min.	98.3	60.5	38.8	1800	0.57	Pptron. heated to 400°F.
20	10 hrs. 40 min.	94.2	65.5	32.5	1830	1.3	

Figure 12 plots the experimental data in relation to the curve calculated by equation (5). Point A on the curve denotes plant operating conditions and is related to a Precipitron efficiency of 98.5%. Runs 17 and 18 were made with the Precipitron at room temperature.

Most of the uranium tetrafluoride product which was built up on the walls of the Precipitron and on the electrode could be removed by rapping the outside of the unit, particularly when the Precipitron was operated at an elevated temperature (400°F.) (Runs 19 and 20). For continuous operation, it was later found desirable to fit the Precipitron with a "scraper" device which was used to prevent an excessive accumulation of material on the walls. The input voltage to the Precipitron was approximately constant at about 9000 volts for all runs.

Filters. In order to obtain data on the plugging characteristics of the filter, Run No. 8 was made with a small filter on the effluent gas from the reactor. The filter downstream pressure was maintained at 1.5 psia. with an average gas flow of 1300 scfd. and a uranium hexafluoride concentration of approximately one mol %. Figure 13 shows a uniform increase in ΔP across the filter during the 590 minute run. The average increase in pressure was 0.244 psi. per hour.

With the Precipitron installed after the reactor, Run No. 17 was made using a filter with a larger filtering area. The plugging rate of the filter during this run appeared to level off with time (figure 14). From this figure, it can be seen that the filter plugged from an initial ΔP of 0.57 psi. to 1.42 psi., an increase of 0.85 psi. in the ΔP over a period of eight hours and 25

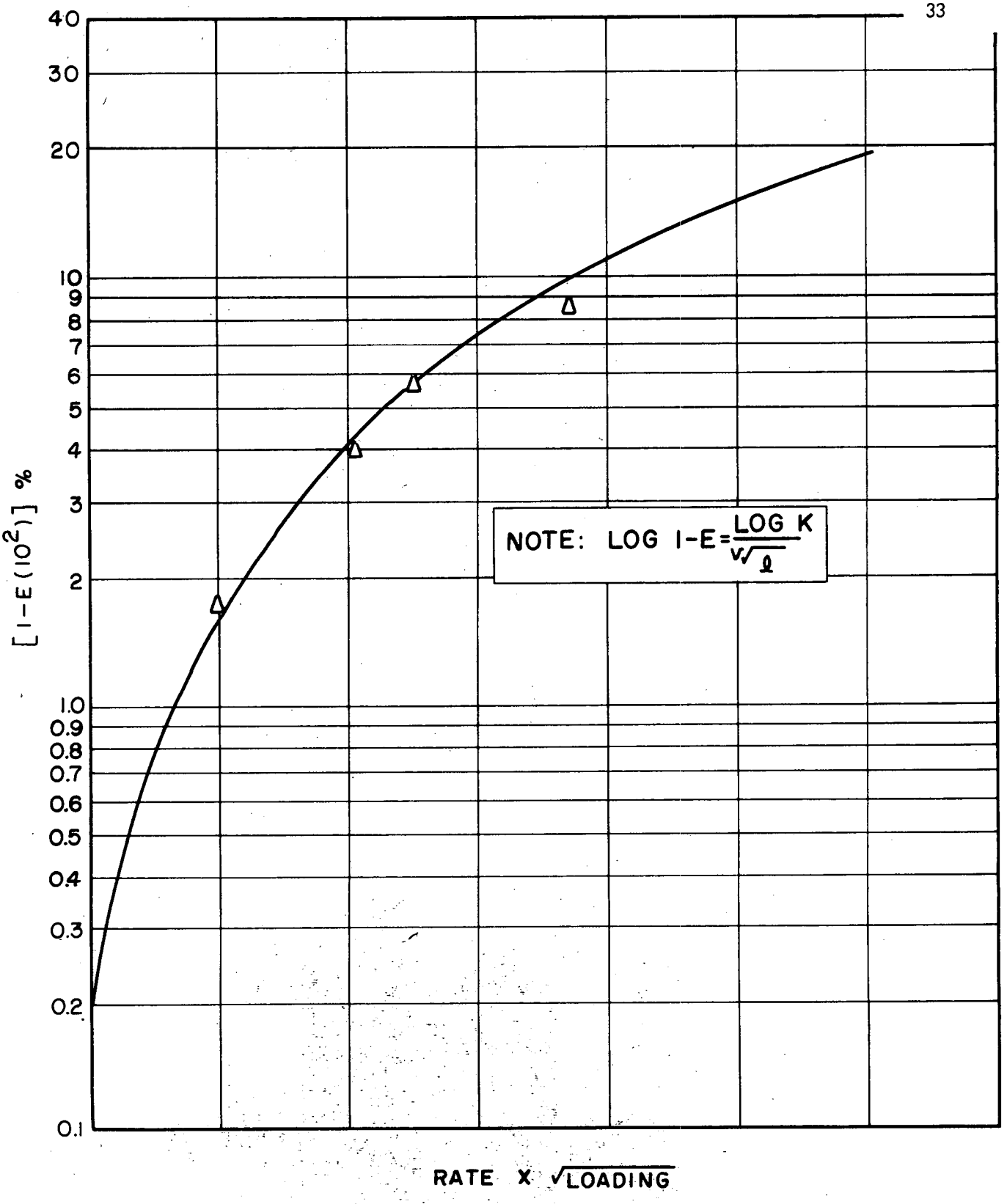

Figure 12
PRECIPITRON EFFICIENCY AS A FUNCTION
OF GAS FLOW RATE AND SOLID LOADING

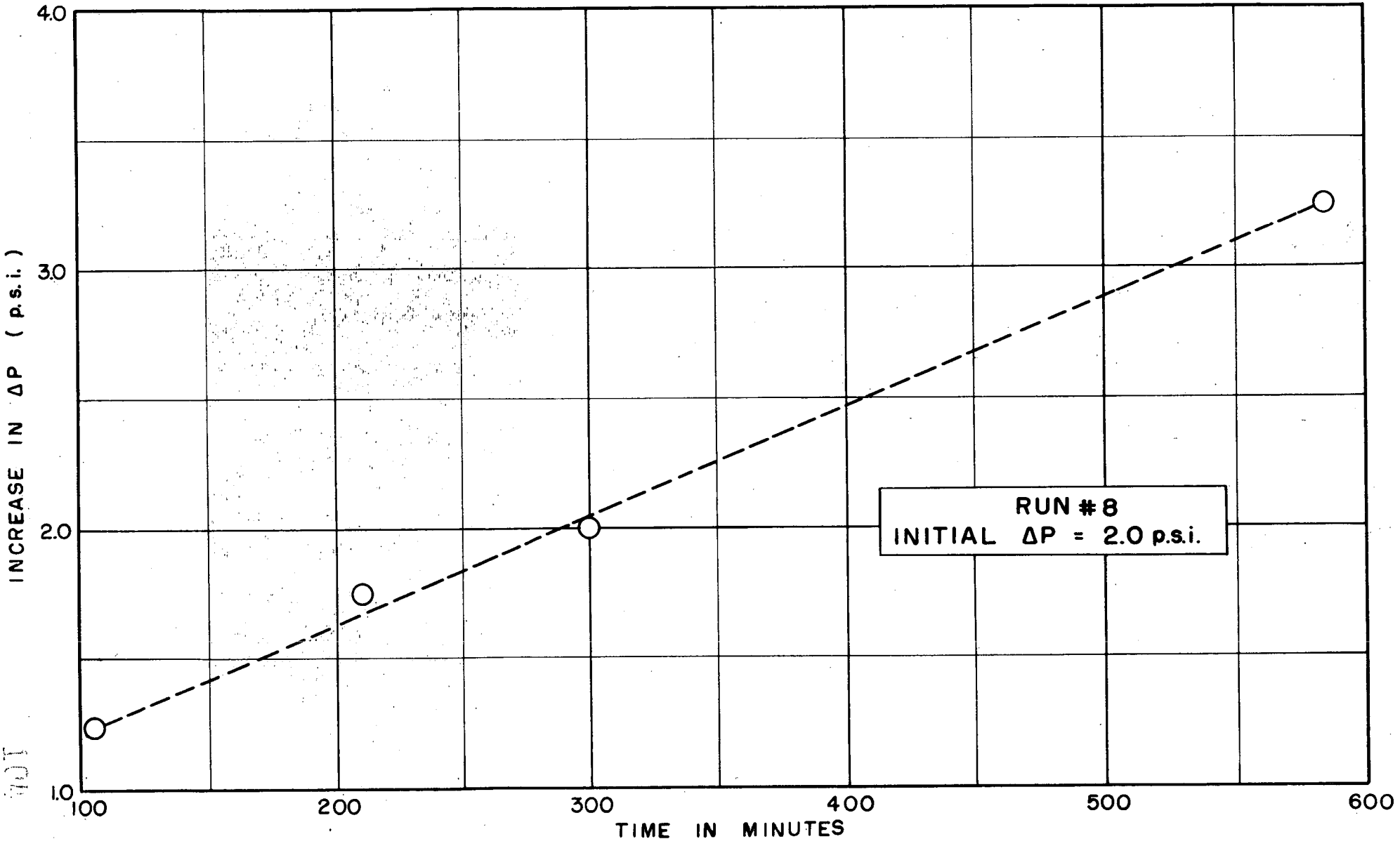

Figure 13
FILTER PERFORMANCE INCREASE IN
ΔP ACROSS FILTER AGAINST TIME

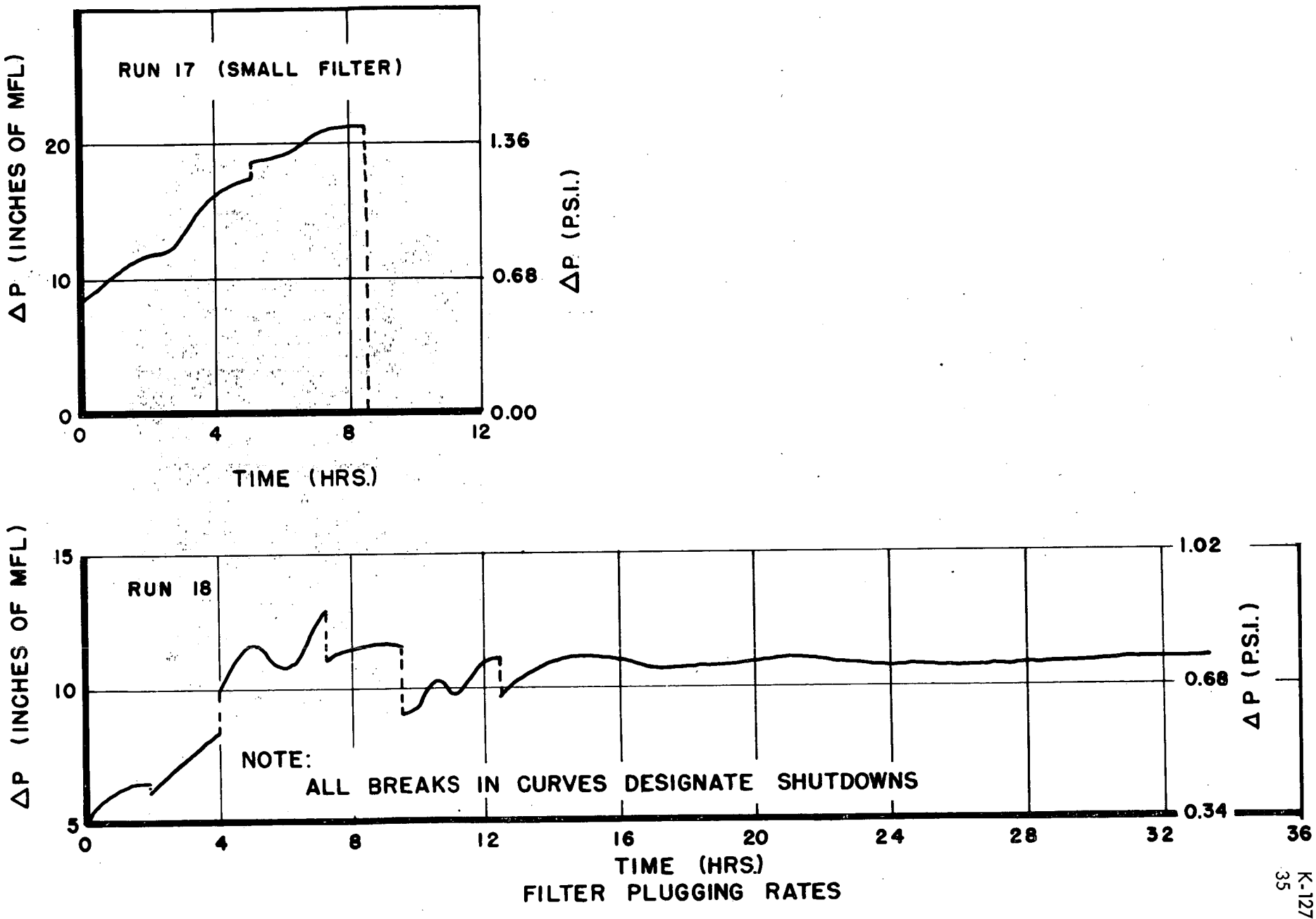

Figure 14
FILTER PERFORMANCE CURVES

minutes. Flows during this run were maintained at 1000 scfd. with an average uranium hexafluoride concentration of 3.6 mol %. Upon completion of the run, 77 grams of uranium tetrafluoride were removed from the filter (1.35% of the total solids processed).

In Run No. 18, (Figure 14), a filter with an even larger surface was used. Flows were maintained at 1800 scfd. and the uranium hexafluoride concentration was 1.5 mol %. Of the uranium tetrafluoride produced, 715 grams or 4.2% of the total was collected by the filter. The ΔP across the filter increased by 0.42 psi. over the 33-hour period, and the plugging rate decreased after the fourteenth hour of operation and remained nearly constant for the rest of the run. In Run No. 17 where flows were maintained at 1000 scfd., 1.35% of the total product reached the filter, and in Run No. 18, with flows held at 1800 scfd., approximately 4% of the solid reached the filter.

It is felt that the rapid initial plug of the tubes was caused by the filling up of a large percentage of the pores with uranium tetrafluoride. However, it is possible that the plug was produced by some of the reaction products.

It was not possible in any of the runs to remove all of the product adhering to the filter tubes without carefully removing the tube bundle and manually scraping the material off the tubes.

Results - Runs A-1 to A-5 (Components of Unit Arranged Vertically in Tandem)

Data on the performance of the individual components of the product withdrawal system indicated an arrangement of parts as shown in Figure 18. Since a more compact and practical arrangement is expedient for operation in the plant to meet health and critical hazard requirements, the following modifications were made.

1. In order to have only one product withdrawal point and to eliminate the necessity of having small lines carry relatively large amounts of solids, the Precipitron was mounted directly above the seven-foot reactor. The length of the Precipitron was increased to six feet to compensate for any additional load that might be placed upon it by the new equipment arrangement, and it was equipped with a scraper (Figure 15) for the reasons described earlier.

2. Although it is desirable to have the filter mounted directly above the Precipitron, it is also necessary to have two filters on the unit to insure continuous operation in the plant (i.e., when the pressure drop across one filter becomes excessive, it is valved off and operation is continued with the second filter). Although this operation prohibits a

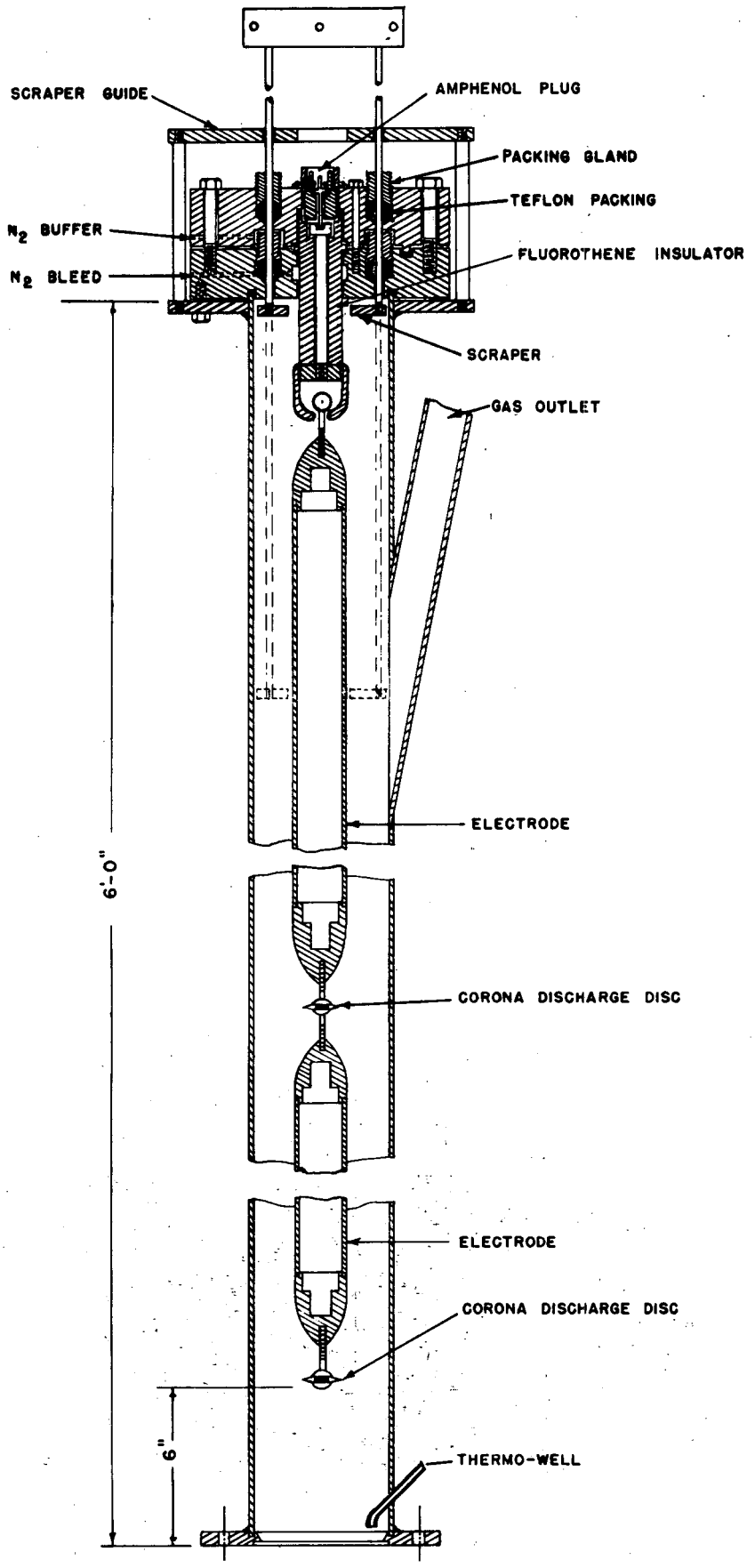

Figure 15
PRECIPITRON WITH SCRAPER ARRANGEMENT, VAPOR PHASE SYSTEM

vertical tandem arrangement of components, the filters can be mounted higher than the Precipitron, but not directly above it. The experimental unit was built with only one filter.

3. All baffles in the reactor were eliminated. The feed gas and trichloroethylene were introduced approximately 1.5 feet from the bottom of the reactor (Figure 16 shows the new reactor).

4. The uranium hexafluoride-nitrogen nozzle was sized to produce a jet velocity of 100 fps. at standard plant operating conditions.

5. The reactor was built to accommodate a product withdrawal valve (Figure 17) which would permit periodic removal of uranium tetrafluoride under "always safe" conditions.

6. The reactor was fitted with an electromagnetic rapper which automatically rapped the reactor shell once every three seconds during operation.

The new equipment assembly is shown in Figures 18 and 19.

A shakedown run and four long runs were made with the newly arranged unit. In the last run amounts of fluorine and perfluorodimethylcyclohexane (C_8F_4) were introduced into the reactor along with the uranium hexafluoride-nitrogen stream. An excess of trichloroethylene over that required to reduce all uranium hexafluoride and fluorine present was added. (See Table 9.)

The unit was operated with the system pressure varying from 1.75 to 4.0 psia.

The gas flow was varied from 500 to 3000 scfd., while the uranium hexafluoride mass flow varied from a minimum of 0.41 mol % at a gas flow of 800 scfd. and a system pressure of 2 psia. to a maximum of 1.50 mol % for a gas flow of 690 scfd. and a system pressure of 2.25 psia. One high flow run was made where the uranium hexafluoride concentration was maintained at 0.36 mol % with a gas flow of 3000 scfd., and a system pressure of 4.0 psia.

A run was made in which fluorine and perfluorodimethylcyclohexane were introduced into the reactor along with the other reactants. In this run the uranium hexafluoride concentration was maintained at 1.50 mol % at a gas flow of 690 scfd. and a system pressure of 2.25 psia. Fluorine was added in the amount of 2 mol per cent and perfluorodimethylcyclohexane in the amount of 0.14 mol per cent (0.37 pound/day). This run was continued for 72.5 hours.

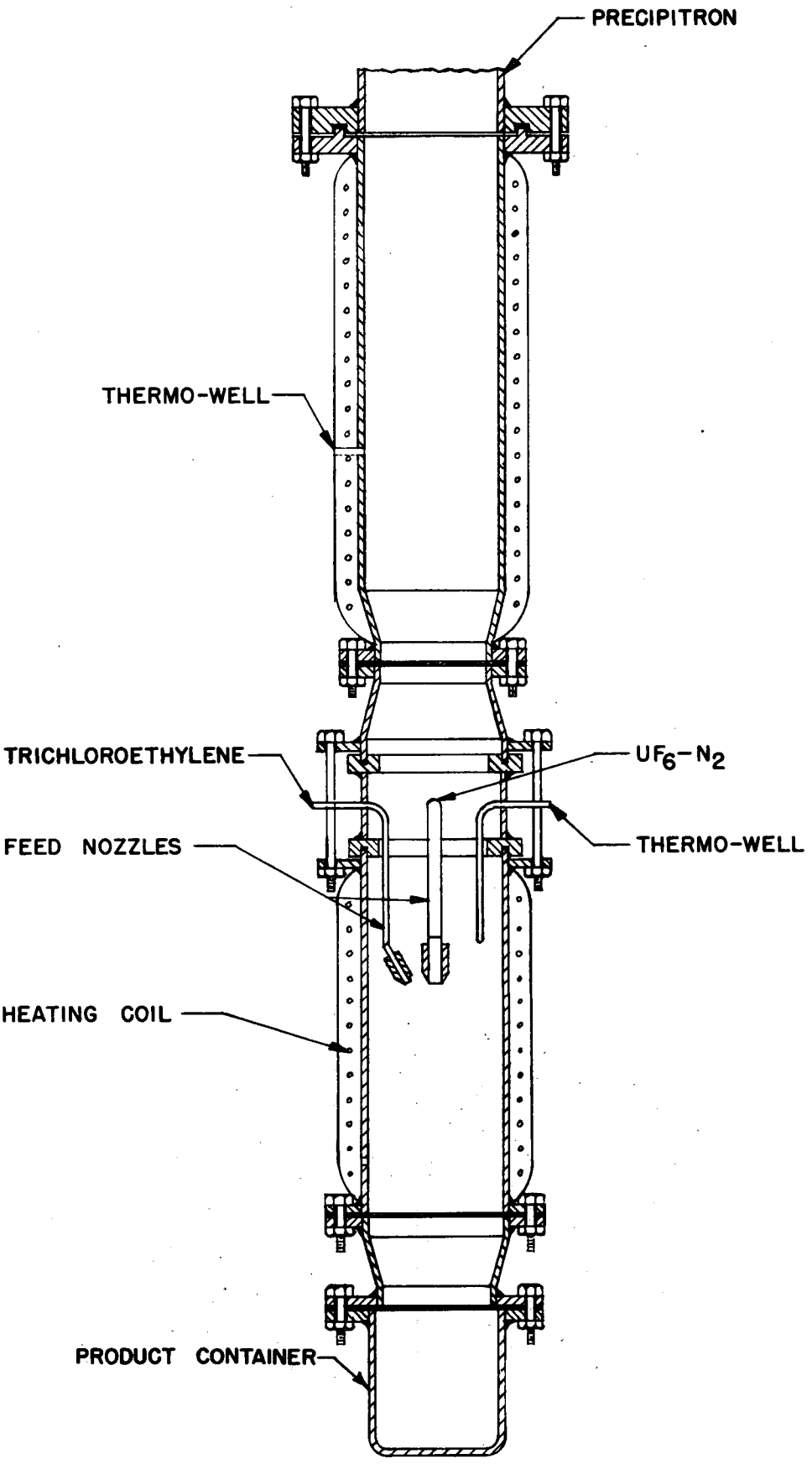

Figure 16
NEW PILOT PLANT REACTOR -
VAPOR PHASE SYSTEM

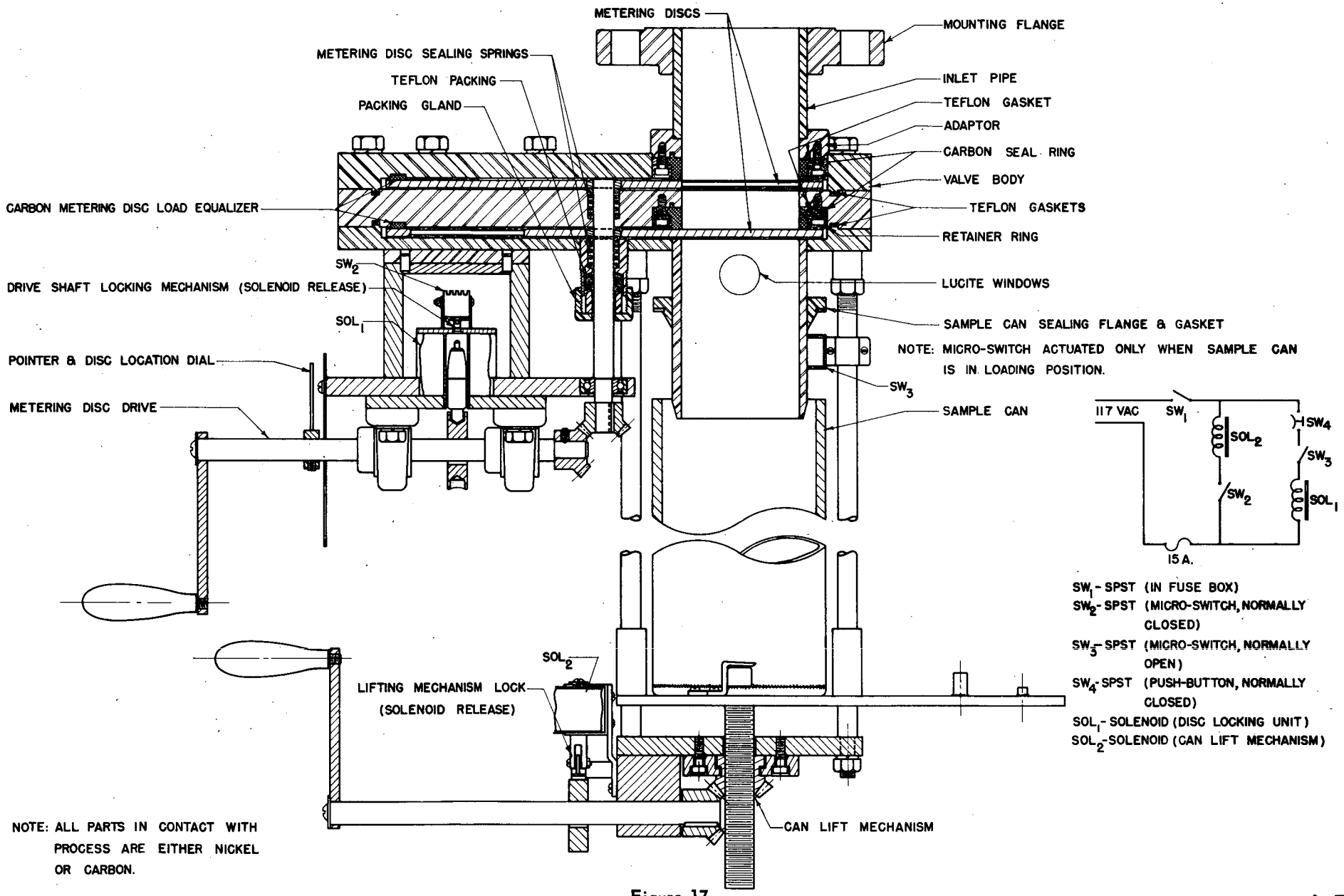

Figure 17
PRODUCT WITHDRAWAL VALVE -
VAPOR PHASE SYSTEM

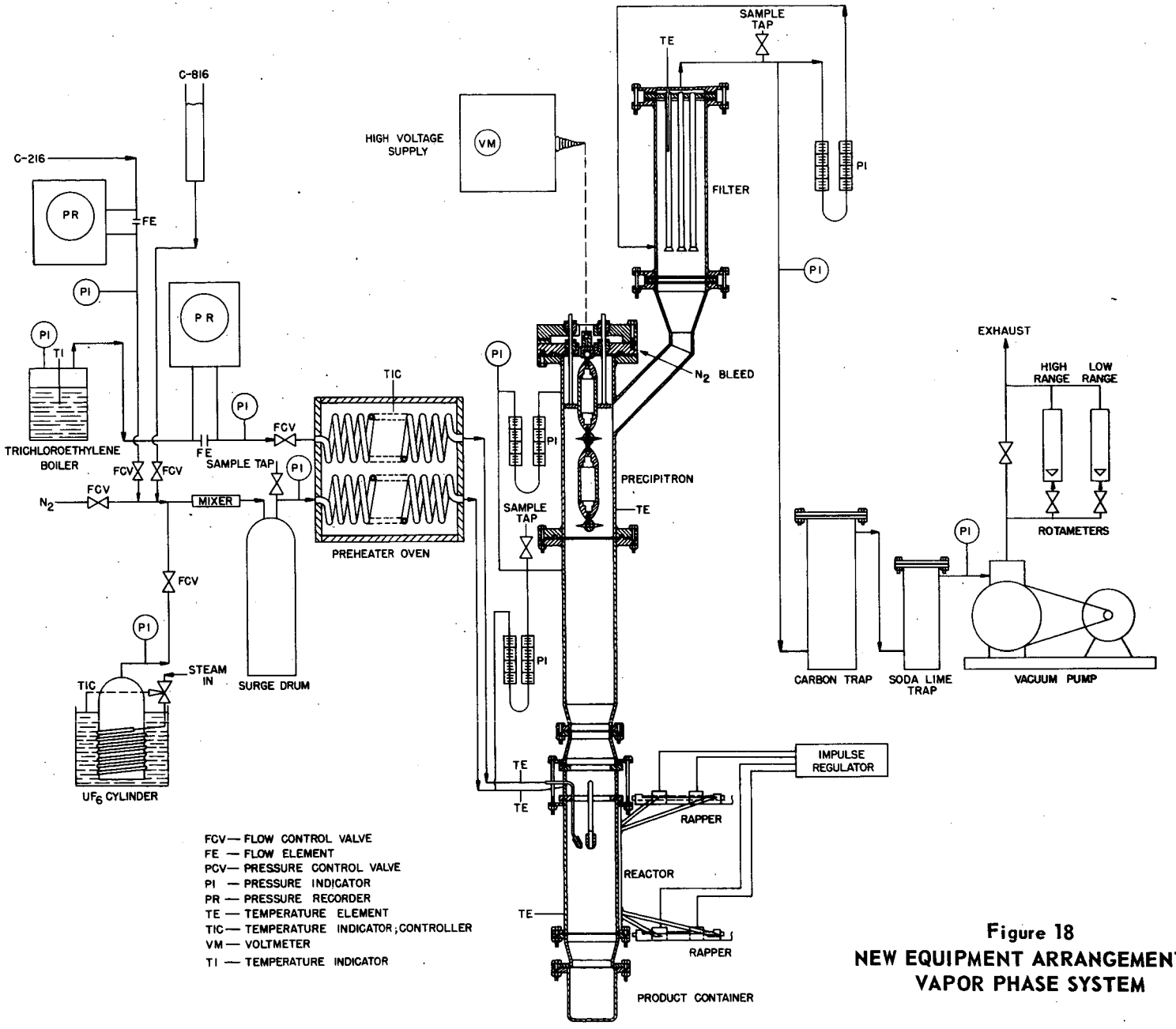

Figure 18
NEW EQUIPMENT ARRANGEMENT - VAPOR PHASE SYSTEM

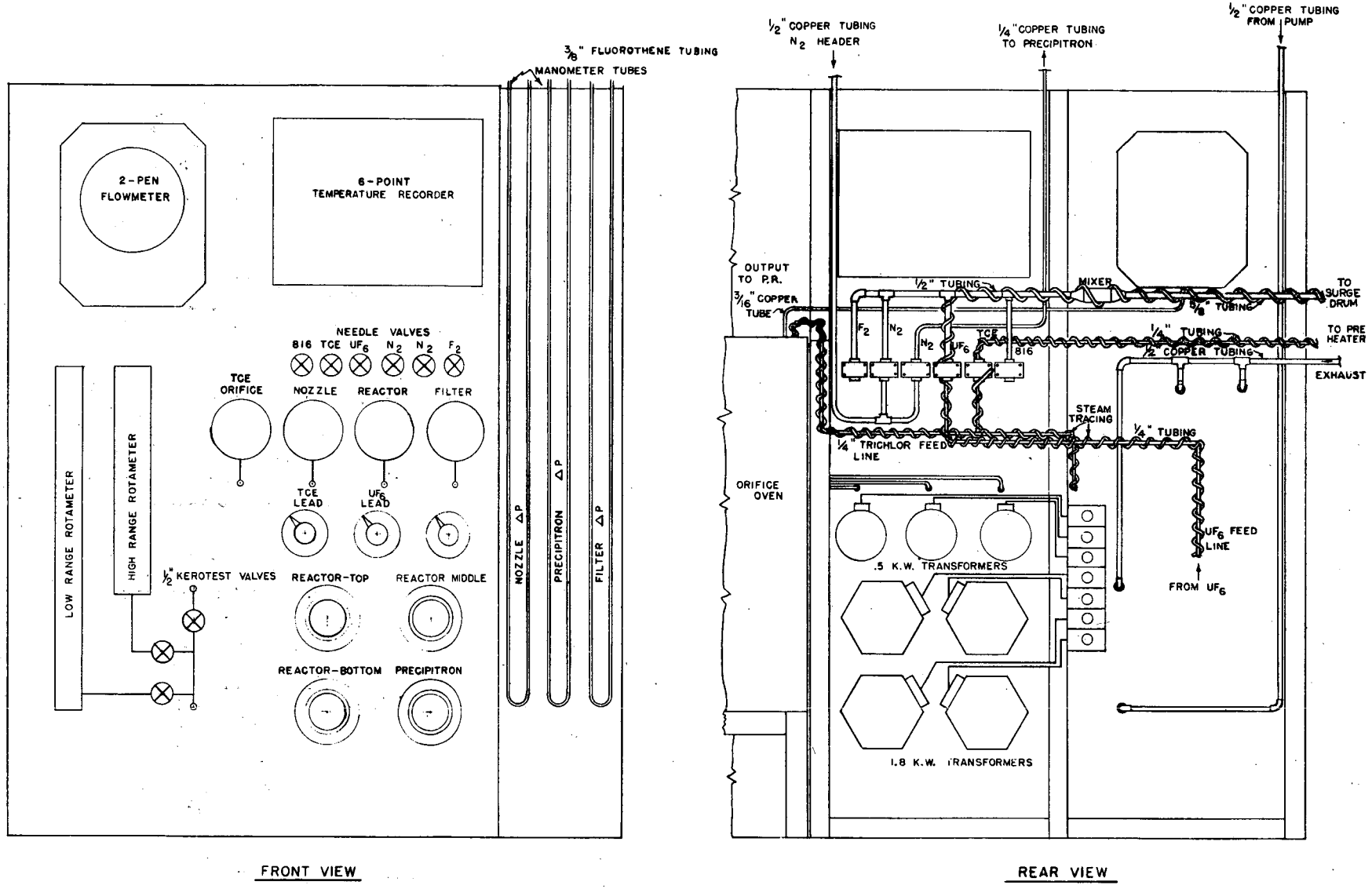

Figure 19
INSTRUMENT PANEL BOARD -
VAPOR PHASE SYSTEM

Reactor and Precipitron. During the series of runs A-1 to A-5 the reactor and Precipitron performed very well. The mechanical scraper in the Precipitron and the electromagnetic rapper on the lower part of the reaction chamber functioned to prevent a large holdup of product in the system. The combined reactor and Precipitron efficiencies (Table 9) varied from 97 to better than 99%. No mechanical difficulties were encountered in this series of runs.

Filter Performance. During the 72.5 hour run, (Run A-5) in which five gases were introduced into the reactor, the pressure drop across the sintered metal filter increased 0.05 psi. while removing 33 grams of uranium tetrafluoride.

Less than 1 ppm. of uranium hexafluoride was found in the gas stream at the exit of the filter. In instances where analyses were made on gas samples of 10 liters volume, or greater, the uranium hexafluoride in the effluent was found in every case to be less than 0.1 ppm. For run A-3, the filter plugging curve followed that of Figure 14 from earlier work (i.e., 0.6 psi. plug occurred in the first ten hours of operation). Some difficulty with the MFL manometers made it impossible to get measured values for the increase in ΔP across the filter for Run A-4. However, other system pressure measurements showed that the plug during the 88-hour run was of the same order of magnitude as that measured for Run A-5.

Product Purity. Consideration was given to product purity in the last runs with the vertical tandem unit. The analyses of three batches of uranium tetrafluoride product from the last run are presented in Table 11. Analyses performed on the uranium hexafluoride feed material used in the same run are presented in Table 10.

The total impurities average 24 ppm. in the feed material. In the unsintered product the total impurities average 68.7 ppm., 79% of which is iron and nickel, and in the sintered product the total impurities average 38.0 ppm., 89% of which is iron and nickel.

TABLE 9

TEST DATA - VAPOR PHASE REACTOR (RUNS A-1 THROUGH A-5)

Run No.	Flow, Scfd.	UF_6 Conc., Mol %	Fluorine Conc., Mol %	Coolant Conc., Mol %	Mol ratio $TCE/F_2 + UF_6$	UF_6 In Effluent, ppm.	System Pressure, Psia.	Duration of Run
A-1	800	0.41	---	---	7.3	<1*	2.0	12 hrs. 37 min.
A-2	500	1.30	---	---	4.4	<1*	1.75	24 hrs. 33 min.
A-3	3000	0.36	---	---	2.6	<1*	4.0	23 hrs. 0 min.
A-4	675	1.05	---	---	2.5	<1*	2.0	88 hrs. 42 min.
A-5	690	1.50	2.03	0.14 (0.37 lb./day)	1.3	<1*	2.25	72 hrs. 42 min.

Run No.	Filter ΔP Increase, psi.	Weight of UF_4 Produced, g.	Weight UF_4 Removed By Filter Bundle, g.	UF_4 Removed By Filter Bundle, %
A-1	0.18	680	---	---
A-2	---	2,656	54	2.0
A-3	0.79	4,657	111	2.3
A-4	---	10,542	34	0.32
A-5	0.05	14,500	33	0.23

*For each run several large gas samples were measured to contain less than 0.1 ppm. of uranium(VI). In no case was the value greater than 0.1 ppm. However, the number of large samples taken cannot justify (at this time) claiming <1 ppm. of uranium(VI) in the effluent gas.

TABLE 10

RESULTS OF SPECTROGRAPHIC ANALYSES PERFORMED ON UF_6 USED AS FEED MATERIAL IN VAPOR PHASE REACTOR

Sample No.	Ag	Cd	Cr	Cu	Fe	Mg	Ni	Si	Total
1	NF	NF	10	2	5	2	NF	1	20
2	0.2	NF	1	1	5	2	NF	1	10
3	0.2	NF	20	2	5	2	NF	1	30
4	0.5	NF	20	1	5	2	NF	1	29
5	0.2	NF	20	4	5	2	NF	1	32
Average	0.2	NF	14	2	5	2	NF	1	24.2

TABLE 11

RESULTS OF SPECTROGRAPHIC ANALYSES OF PRODUCT, BEFORE AND AFTER SINTERING

Batch No. 1 Before Sintering	Ag	Cd	Cr	Cu	Fe	Mg	Ni	Si	Total Impurities
Sample No. 1	N.F.	0.2	N.F.	5	40	9	30	4	88.2 ppm.
Sample No. 2	N.F.	0.1	N.F.	5	50	9	30	9	103.1 ppm.
Sample No. 3	1.0	0.1	N.F.	5	20	9	40	2	77.1 ppm.
Sample No. 4	0.2	0.2	N.F.	5	30	9	30	6	80.4 ppm.
Batch No. 2 Before Sintering	N.F.	N.F.	N.F.	5	8	1	20	1	35 ppm.
Batch No. 3 Before Sintering	N.F.	N.F.	N.F.	2	10	1	15	N.F.	28 ppm.
Average	0.2	0.1	N.F.	4.5	26.3	6.3	27.5	3.7	68.7 ppm.
Batch No. 1 After Sintering									
Sample No. 1	0.1	N.F.	N.F.	0.1	30	1	10	1	42.2 ppm.
Sample No. 2	0.1	N.F.	N.F.	0.1	20	1	5	N.F.	26.2 ppm.
Batch No. 2 After Sintering									
Sample No. 1	0.1	N.F.	N.F.	0.5	20	1	20	N.F.	41.6 ppm.
Sample No. 2	0.1	N.F.	N.F.	0.5	10	1	10	N.F.	21.6 ppm.
Batch No. 3 After Sintering									
Sample No. 1	0.2	N.F.	N.F.	2	25	5	20	2	54.2 ppm.
Sample No. 2	0.2	N.F.	N.F.	2	20	5	15	1	43.2 ppm.
Average	0.13	N.F.	N.F.	0.87	20.8	2.3	13.3	0.67	38.0 ppm.

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