K-1379(Del.)

Date of Issue: June 27, 1958

Photostat Price $ 3.30
Microfilm Price $ 2.40

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

DEVELOPMENT OF THE CONTINUOUS METHOD FOR THE REDUCTION OF URANIUM HEXAFLUORIDE WITH HYDROGEN - PROCESS DEVELOPMENT. HOT WALL REACTOR

S. H. Smiley
D. C. Brater

Technical Division
D. M. Lang, Superintendent

UNION CARBIDE NUCLEAR COMPANY
DIVISION OF UNION CARBIDE CORPORATION
Oak Ridge Gaseous Diffusion Plant
Oak Ridge, Tennessee

Declassified With Deletions: August 21, 1959.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

DECLASSIFIED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Report Number: K-1379

Title: DEVELOPMENT OF THE CONTINUOUS METHOD FOR THE REDUCTION OF URANIUM HEXAFLUORIDE WITH HYDROGEN - PROCESS DEVELOPMENT. HOT WALL REACTOR

Authors: S. H. Smiley
D. C. Brater

A B S T R A C T

A continuous process for the reduction of uranium hexafluoride to uranium tetrafluoride has been developed and proved on a pilot-plant scale. Complete conversion to uranium tetrafluoride was realized by contacting gaseous uranium hexafluoride with hydrogen in a heated, vertical, open-tube reactor. The purity and density of the solid product met metal grade uranium tetrafluoride specifications. Some difficulty with the accumulation of fused uranium fluorides in the tower was encountered, however, and it was necessary to stop and deslag the unit about every 8 to 24 hours.

The reaction of uranium hexafluoride with gaseous trichloroethylene was studied before the tests with hydrogen were made. Although the reduction to uranium tetrafluoride was complete, the solid product was highly contaminated with the organic by-products of the reaction and was quite low in density. Tests of this method were discontinued when promising results were obtained with hydrogen as the reductant.

UNCLASSIFIED

DEVELOPMENT OF THE CONTINUOUS METHOD FOR THE REDUCTION OF URANIUM HEXAFLUORIDE WITH HYDROGEN - PROCESS DEVELOPMENT. HOT WALL REACTOR

The need for a large-scale facility for the reduction of normal and near normal assay uranium hexafluoride to uranium tetrafluoride was recognized in early 1951. A development program was initiated, therefore, to determine if the previously studied method of reducing very dilute uranium hexafluoride with gaseous trichloroethylene[1] could be adapted to process large quantities of material. It was soon shown, however, that although the reduction to uranium tetrafluoride was complete, the powder was highly contaminated with the organic by-products of the reaction, in particular, hexachlorobenzene. In addition, high temperature sintering of the low density product (packed density of 0.5 to 1.7 g./cc.) would be necessary to meet the specifications for metal grade uranium tetrafluoride.

Studies were then made in the pilot-plant equipment with hydrogen as the reductant, since use of this gas would eliminate the serious purity problem encountered with the trichloroethylene. Although laboratory-scale tests of this thermodynamically feasible reaction had been unsuccessful[2,3], it seemed possible that the reduction would be self-sustaining if relatively large quantities of hot uranium hexafluoride and hydrogen were introduced through nozzles at the top of the tower. It was soon shown that complete reduction to uranium tetrafluoride is possible with gas and reactor wall temperatures in the range of 500 to 600°F. and hydrogen excesses as low as 30%. The packed density of the uranium tetrafluoride was dependent on the operating conditions and could be held at about 3.5 g./cc. The product contained less than 50 ppm. metallic impurities and approximately 1-1/2% uranyl fluoride.

During operation, the 6-inch diameter pilot-plant reactor plugged gradually with fused uranium fluorides, probably UF_5, U_2F_9, and U_4F_{17}, and it was necessary to scrape the walls of the tower periodically. Diluting the uranium hexafluoride with nitrogen, increasing the hydrogen excess, and grading the temperature down the reactor decreased but did not eliminate the fouling. In general, it was necessary to clean the reactor every 8 to 24 hours.

The experimental program was terminated at this point, and at the request of the Atomic Energy Commission, approximately 45,000 pounds of uranium tetrafluoride was prepared in the pilot plant. This process was then studied further by the National Lead Company of Ohio, and a production facility was built and operated successfully.

Descriptions of the processes, the pilot-plant facilities, and the results of the experimental tests with both trichloroethylene and hydrogen follow.

REDUCTION WITH TRICHLOROETHYLENE

Process Technology

Uranium hexafluoride reacts with trichloroethylene to form uranium tetrafluoride according to the following equation:

$$UF_6 + CHCl = CCl_2 \longrightarrow UF_4 + \text{Chlorofluorocarbons} + HF$$

A mixture of chlorofluorocarbon compounds is formed by halogen addition and substitution, as well as by splitting of the trichloroethylene and fluorination of the methane fragments.

Process Description

The pilot plant was designed to reduce 150 pounds of uranium hexafluoride per hour to uranium tetrafluoride. The facility consisted essentially of (1) uranium hexafluoride and trichloroethylene vaporizers, (2) a vertical pipe reactor, (3) a dust settling and product collection system, and (4) an outlet gas sampling and disposal system. In operation, the uranium hexafluoride and trichloroethylene were vaporized separately from weighed containers, metered, preheated to the desired temperature, and introduced into the top of the reactor. Most of the solid uranium tetrafluoride product was separated from the by-product gases in the reactor settling chamber, and the remainder was collected by an electrostatic precipitator and a sintered metal filter.

A flow and instrumentation diagram of the pilot plant is shown as figure 1. A detailed description of the equipment follows.

Uranium Hexafluoride Feed System. The uranium hexafluoride feed was supplied in steel acetylene cylinders which contained about 350 pounds of material. The cylinders were placed in 30-gallon drums and were heated with hot water to vaporize the uranium hexafluoride. The gaseous uranium hexafluoride then passed through a metering station which included, in sequence, a pressure control valve, a surge drum, an orifice, and a flow control valve. With this system, both the density and the volume flow of the gas were controlled. All lines containing uranium hexafluoride were traced with copper tubing and heated with steam.

Trichloroethylene Feed System. Gaseous trichloroethylene was supplied by either of two electrically-heated vaporizers, which were mounted on scales. Metering of the gas was accomplished with a control valve system similar to that used for the uranium hexafluoride.

Preheaters. Electrically-heated, two-pass tube bundles were used as gas preheaters. The heat input was controlled by the gas temperatures, and for safety, thermocouples tacked to the tube wall actuated an alarm if excessive temperatures were reached.

Reactor Section. The reactor, figure 2, consisted essentially of a vertical, 6-inch diameter, 10-foot long pipe and a 12-inch diameter solids settling section mounted concentrically around the lower 4-1/2 feet of the tower. The entire unit was wrapped with beaded Nichrome wire, and independent temperature controls were provided for three heating zones along the length of the reactor. Two air hammers were attached to the reactor to furnish the vibration necessary to prevent the accumulation of solids on the tower walls.

The uranium hexafluoride and trichloroethylene feed gases were charged to the top of the tower through the assembly shown in figure 2. The uranium hexafluoride entered through a vertical pipe centered in the top flange, and the trichloroethylene was introduced through four pipe nozzles angled to provide a swirling action around the uranium hexafluoride.

The uranium tetrafluoride product was collected in a hopper at the bottom of the settling section and was discharged by a screw conveyor into a receiver. A Gemco valve was installed after the screw to prevent gas leakage when the receiver was removed.

Electrostatic Precipitator.[4] The precipitator, 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 precipitator for ease of removal of product. The precipitator was designed to operate with a maximum potential of 15 kv.

Filter. A vertical, 6-inch diameter pipe containing several sintered nickel filters was employed for final stripping of entrained solids from the outlet gases.

Outlet Gas Sampling System. Dry ice and liquid nitrogen condensers were provided to collect samples of the exit gas. A portion of the gas was passed through (1) a metering orifice, (2) the condensers which were arranged in series, and (3) a second orifice.

Gas Disposal System. The outlet gases were passed through a water-cooled condenser and a dry ice-trichloroethylene cooled condenser before being discharged through a 60-foot high stack to the atmosphere.

Data and Discussion

The results of the studies with trichloroethylene and analyses of outlet gases from some of the runs are presented in tables I and II. Complete conversion of the uranium hexafluoride to uranium tetrafluoride was obtained in all of the tests, but the packed density of the product was low, less than 1.7 g./cc., and the carbon content of the uranium tetrafluoride was high. Attempts were made to increase the purity of the product by diluting the

DECLASSIFIED

inlet gases with hydrogen, hydrogen fluoride, and nitrogen, but no significant improvements were noted.

In addition to the difficulty with carbon contamination of the uranium tetrafluoride, condensation of some of the organic reaction products, primarily hexachlorobenzene, presented a serious problem. Large deposits of these materials were found in the product collection system and the outlet lines.

Experimental studies with the trichloroethylene-uranium hexafluoride system were discontinued after favorable results were obtained with hydrogen as the reductant.

In connection with the above program, methods of removing the carbon from uranium tetrafluoride were investigated. Small-scale tests were made in which the product from the pilot-plant runs was charged into pans and heated under atmospheres of nitrogen, hydrogen fluoride, hydrogen, oxygen, and a mixture of hydrogen fluoride and oxygen. The results of these studies, table III, showed that only the treatment with oxygen reduced the carbon content significantly and that the addition of hydrogen fluoride was necessary to prevent the formation of uranyl fluoride.

REDUCTION WITH HYDROGEN

Process Technology

The reaction of uranium hexafluoride with hydrogen can be represented by the following equation:

$$UF_6\ (g) + H_2\ (g) \longrightarrow UF_4\ (s) + 2\ HF + 124{,}000\ \text{Btu./lb. mol}$$

The thermodynamic equilibrium, even at room temperature, is considerably toward the formation of uranium tetrafluoride, but a high activation energy is required to initiate the reduction reaction.

Equipment Description

The equipment used in the original runs was that described above for the trichloroethylene reduction. For clarity, changes made in the equipment during the course of the study are presented along with the results of the experimental tests.

Data and Discussion

The results of the experimental tests are presented in table IV. The initial studies, runs 12 through 26, were made in the pilot plant which had been employed for the trichloroethylene-uranium hexafluoride experiments. The first two runs, 12 and 13, were very short, about 10 minutes each, and were made only to determine if the reduction reaction could be initiated. In these tests, a rapid increase in the reactor wall temperatures and the absence of uranium hexafluoride in the outlet gases indicated that complete reduction was taking place. The small amount of solid product

was green in color and appeared to be good uranium tetrafluoride.

A series of runs, 14 through 26, was then made to determine the effect of operating variables on the performance of the reactor and the quality of the product. It was shown that (1) the reduction can be initiated with inlet gas and reactor wall temperatures as low as 550 and 420°F., respectively; (2) no reaction occurs with gas and wall temperatures of 250°F.; (3) complete reduction is possible in the pilot-plant reactor with feed rates as high as 286 pounds of uranium hexafluoride per hour; (4) complete reduction is possible with the uranium hexafluoride feed diluted with an equal molar quantity of nitrogen; (5) uranium hexafluoride packed densities as high as 3.6 g./cc. can be obtained; and (6) decreasing the hydrogen excess increases the density of the product.

In the above tests, the temperature of the reactor increased rapidly after the gas flows were started, and it was necessary to stop several runs to prevent overheating of the tower. The upper 6-inch reactor section was wrapped with copper tubing and cooled with water after run 22, but adequate temperature control was still lacking.

In most of the runs, solid reaction products fused on the walls of the 6-inch diameter reaction section. Although none of the tests were stopped by complete plugging of the tower, it appeared that this would have occurred had the runs been longer.

In an attempt to eliminate the fouling, the reactor was altered to remove the portion of the 6-inch pipe that extended into the settling section and to lengthen this 12-inch diameter section, figure 3. It was hoped that the heat required to initiate the reaction could be supplied by the walls of the upper 6-inch pipe and that most of the reduction would occur in the larger portion of the reactor.

As can be seen from the results of runs 27 through 41, no fouling was encountered with this reactor unless the reduction occurred in the 6-inch pipe section. The density of the product was not so high as desired in most cases, however, and some uranium hexafluoride was noted in the outlet gas during four of the runs. Higher densities were obtained without reactor fouling in runs 40 and 41, in which inlet gas temperatures of at least 750°F. were employed. Operation in this manner was satisfactory for the short experimental tests, but periodic cleaning of crystalline solids from the uranium hexafluoride inlet line was necessary. These deposits were later found to be due to the reaction of uranium hexafluoride with the heated Monel line.

The reactor was then replaced with a longer column made entirely of 6-inch diameter pipe, figure 4, and the number of heating zones was increased to four. The lengths of these zones, starting from the top of the reactor, were 30, 36, 60, and 24 inches. Cooling coils similar to those described above were furnished for each zone. After run 53, the temperature control was improved further by increasing the number of zones to five, which had lengths of 24, 21, 21, 60, and 24 inches, starting from the top of the reactor. Automatic on-off control was provided for the heaters after run 59.

With the increased length of reactor and number of heating (or cooling) zones, it was possible to effect a more gradual increase in temperature down the tower. Some improvement in reactor performance was noted under these conditions; however, success was sporadic. Results were sometimes contradictory, and it was not always possible to repeat good runs. Frequently, the point at which most of the reaction was occurring would drop gradually, and in some cases, temperature measurements indicated that most of the reaction was taking place in the electrostatic precipitator and outlet lines. When the tower temperatures were increased to prevent this, the reactor often fouled. Thus, it appeared that a longer reactor would be required to effect complete conversion of the uranium hexafluoride to uranium tetrafluoride with little or no build-up of solids in the reactor.

It is of interest that the density of the product from the studies in the 6-inch diameter reactor was usually higher than that obtained in previous runs under nearly comparable conditions. Density specifications for metal grade uranium tetrafluoride were met in many of the tests.

During the experimental studies, operation of all of the auxiliary equipment but the electrostatic precipitator was satisfactory. The electrical insulator in the head of this unit burned out, and most of the tests were made with only the filter. The solids entrained in the outlet gases were removed completely by the metal filter elements, however, and it appeared that the rough stripping by the precipitator was not necessary.

Frequent vibration of the reactor was required to minimize fouling and to dislodge the powder collected in the electrostatic precipitator. This was accomplished with air hammers mounted at three points along the reactor.

The tests were discontinued after run 73, and at the request of the Atomic Energy Commission, approximately 45,000 pounds of uranium tetrafluoride was prepared in the pilot-plant reactor. High wall temperatures, graded from 800 to 1000°F., a uranium hexafluoride feed rate of 100 pounds per hour, and a hydrogen excess of 100% were employed for this production. The average packed density of the product was 3.6 g./cc. and ranged from 3.4 to 4.0 g./cc. The total metallic impurities ranged from 2 to 130 ppm. and averaged about 40 ppm. Continuous operation for extended periods of time was prevented by the expected reactor fouling, and it was necessary to shut down periodically, usually after about 8 hours of operation, to remove the solids from the reactor walls. No difficulties other than this were encountered, and complete reduction to uranium tetrafluoride was realized during the entire period.

Although the experimental program was not entirely successful in developing a reduction reactor which could be operated continuously, sufficient tests were made to indicate that the reduction of uranium hexafluoride with hydrogen was feasible for production use. It was indicated that increasing either the length or the diameter of the reactor would decrease the amount of fouling. With respect to product quality, both the density and purity specifications for metal grade uranium tetrafluoride could be met easily.

The study of the hydrogen reduction system was continued by the National Lead Company of Ohio, and a production facility was built and operated successfully. At a later date, a plant of similar design was installed at the Union Carbide Nuclear Company Paducah facility. Slagging has not been eliminated in the equipment, but reactor clean-out is performed on a daily basis by cooling the walls slightly and vibrating the reactor. In view of the very high processing rates possible in this system, the time lost for deslagging, about 2 hours, can be tolerated.

BIBLIOGRAPHY

(1) Baker, J. E., Klaus, H. F., Schmidt, R. A., and Smiley, S. H., A Pilot Plant for the Reduction of Uranium Hexafluoride to Uranium Tetrafluoride With Trichloroethylene, Union Carbide Nuclear Company, Oak Ridge Gaseous Diffusion Plant, May 31, 1956 (K-1271).

(2) Bernhardt, H. A., Gustison, R. A., and Kirslis, S. S., Reduction of Uranium Hexafluoride to Uranium Tetrafluoride Using Hydrogen, Union Carbide Nuclear Company, Oak Ridge Gaseous Diffusion Plant, October 11, 1948 (K-279).

(3) Katz, J. J., and Rabinowitch, E., The Chemistry of Uranium, Part I, McGraw-Hill Book Company, Inc., New York (1951).

(4) Smith, R. C., An Electrostatic Precipitator for the Collection of Uranium Tetrafluoride Dust, Union Carbide Nuclear Company, Oak Ridge Gaseous Diffusion Plant, April 30, 1956 (K-1229).

TABLE I

URANIUM HEXAFLUORIDE REDUCTION WITH TRICHLOROETHYLENE

Run Number	UF_6 Rate, lb./hr.	TCE Rate, lb./hr.	TCE/UF_6, Molar Ratio	UF_6 Temp., °F.	TCE Temp., °F.	Reactor Temp., °F.	UF_6 Conc., %	TCE Conc., %	Product Analyses			
									F, %	U, %	C, %	Density, g./cc.
1	125	152	3.26	555	580	900	100	100	22.0	68.0	5.19	0.92
2	74	32	1.15	360	360	620	100	100	22.6	70.9	0.95	1.13
3	83	39	1.25	330	325	470	20(1)	100	24.4	75.3	0.28	0.60
4	86	24	0.75	265	265	310	43(1)	100	23.6	74.1	0.44	0.76
5	56	41	1.95	250	250	225	10(1)	100	24.0	75.4	0.36	1.00
6	126	33	0.70	200	185	300	25(1)	100	23.9	75.2	0.16	0.97
7	30	9	0.80	205	180	165	100	100	22.4	73.9	0.12	1.66
8	93	31	0.89	335	335	300	100	32(1)	23.9	74.7	0.33	0.90
9(2)	78	32	1.10	410	415	360	100	100	24.1	75.4	0.32	0.98
10(3)	76	36	1.27	290	300	375	100	100	22.4	70.2	5.20	1.20
11(4)	86	26	0.81	325	310	285	100	100	24.0	75.7	0.63	0.90

(1) Diluted with nitrogen.

(2) Two pounds of hydrogen fluoride per hour added to reactor.

(3) Eighty cubic feet of hydrogen per hour added to reactor.

(4) Four pounds of hydrogen fluoride per hour added to reactor.

CONFIDENTIAL

CONFIDENTIAL

TABLE II

URANIUM HEXAFLUORIDE REDUCTION WITH TRICHLOROETHYLENE
COMPOSITION OF OUTLET GAS IN WEIGHT PERCENT

Run Number	1	2	3	4	6	9	10	11
Nitrogen	--	--	18.5	34.7	35.9	--	29.7	41.2
Freon-12 (CCl_2F_2)	6	23	4.8	5.7	5.1	22.2	1.8	10.8
Freon-13 ($CClF_3$)	39	43	0.2	15.4	6.3	17.4	12.0	43.6
Freon-102 (CClF = CClF)	--	--	1.6	--	--	--	--	--
Freon-113 (CCl_2FCClF_2)	--	--	10.1	2.9	11.1	2.6	--	--
Freon-114 ($CClF_2CClF_2$)	--	4	4.1	6.3	9.4	20.0	0.9	4.4
Freon-115 ($CClF_2CF_3$)	--	--	--	2.7	2.7	4.2	--	--
Freon-222($CHClFCCl_2F$)	--	--	8.6	2.1	--	1.9	--	--
Trichloroethylene	3	--	20.1	7.4	13.6	3.5	13.9	--
Tetrachloroethylene	--	--	18.9	4.9	--	--	2.4	--
Hydrochloric Acid	1	3	--	4.9	1.1	2.3	--	--
Hydrofluoric Acid	5	3	--	--	5.1	17.4	8.1	--
Pentachloroethene	1	--	--	--	--	--	--	--
Residue (Water, HCl, HF, Salts)	45	24	--	3.5	--	--	24.7	--
High Boilers	--	--	13.1	9.5	9.7	8.5	6.5	--

CONFIDENTIAL

TABLE III

URANIUM HEXAFLUORIDE REDUCTION WITH TRICHLOROETHYLENE
REMOVAL OF CARBON

Run Number	Reactor Temp., °F.	Weight Loss, %	Fluorine, %		Uranium, %		Carbon, %		U^{+6}, %	Length Of Run, hours
			Before	After	Before	After	Before	After		
Oxygen Purification Treatment										
S-8-1	810	1.3	24.4	24.0	75.3	75.8	0.28	0.04	4.2	3.0
S-8-2	750	5.8	23.6	23.8	70.9	75.9	0.95	0.06	4.2	3.0
S-9-1	1000	2.3	24.4	23.7	75.3	75.9	0.28	--	4.1	3.0
S-9-2	900	5.8	23.6	22.7	70.9	75.7	0.95	0.16	10.5	3.0
S-11-1	950	2.6	24.1	23.3	75.4	76.0	0.32	0.07	5.8	3.0
S-11-2	700	8.4	22.0	19.1	68.0	77.5	5.19	0.04	23.9	3.0
S-13-1	850	14.8	23.9	22.3	75.2	75.5	0.16	--	9.7	2.8
S-13-2	550	20.3	22.0	22.1	68.0	75.6	5.19	0.03	12.4	2.8
S-14-1	850	1.4	24.4	24.2	75.3	75.8	0.28	0.03	1.1	5.0
S-14-2	500	1.1	23.1	22.4	70.2	71.8	5.20	4.19	--	5.0
S-15-1	700	--	23.9	23.7	75.2	75.8	0.16	0.02	3.6	5.0
S-15-2	450	--	23.1	25.3	70.2	66.4	5.20	4.97	--	5.0
S-19-1	1200	7.6	23.9	23.6	75.2	75.3	0.16	0.02	1.7	6.0
S-19-2	900	8.8	24.1	23.9	75.4	75.4	0.32	0.01	1.8	6.0
S-20-1	800	0.7	23.9	23.6	75.2	75.6	0.16	0.02	3.4	6.0
S-20-2	670	0	24.1	23.9	75.4	75.3	0.32	0.27	0	6.0
Oxygen-Hydrofluoric Acid Purification Treatment										
S-17-1	1050	2.5	24.1	24.0	75.7	75.8	0.63	0.05	0.7	4.0
S-17-2	800	2.0	23.9	23.9	75.2	75.5	0.16	--	0.4	4.0
S-18-1	775	2.7	24.1	24.0	75.7	75.8	0.63	--	0	4.0
S-18-2	630	1.4	23.9	23.9	75.2	75.3	0.16	0.11	0.9	4.0
S-21-1	1030	--	23.9	23.8	75.2	75.6	0.16	0.02	1.2	6.0
S-21-2	800	0	24.1	22.2	75.4	75.4	0.32	0.23	--	6.0

TABLE IV

URANIUM HEXAFLUORIDE REDUCTION WITH HYDROGEN

Run No.	Feed Rate, Pounds UF_6/hr.	H_2 Excess, %	UF_6 Temp., °F.	H_2 Temp., °F.	Reactor Temp.*, °F. Zone 1	Zone 2	Length Of Run, Hours	UF_6 In Outlet Gas	Product Color	Product Analyses % F	% U	% U^{+4}	Packed Density, g./cc.	Remarks
14	106	130	830	700	730		0.50	None	Green	24.0	75.5	75.2	2.0	UF_6 inlet line plugged. Wall temp. increased to 1000°F. and was still rising.
15	132	260	380	300	660		0.50	None	Grey Green	23.6	75.7	75.3	2.2	Caked powder on walls, 6 inches from top of reactor; 25 lbs. of material removed from tower.
16	76	260	670	660	690		0.25	None	Green	23.8	75.6	74.9	1.5	UF_6 inlet line plugged.
17	75	260	710	760	840		1.00	None	Green	24.1	75.6	74.7	1.9	UF_6 inlet line plugged. Wall temp. increased to 1040°F.
18	286	20	780	840	920		0.50	None	Green	23.9	75.8	74.5	3.5	Reactor plugged near bottom. Wall temp. increased to 1150°F.
19	106	190	260	250	240		0.22	Heavy	Green	No Solid Product				No reaction; no temp. rise.
20	94	180	600	600	580		1.00	None	Light Green	23.7	75.7		2.1	Tower not examined. Wall temp. increased to 910°F.
21	92	30	510	550	680		1.00	None	Green	24.0	75.7	74.9	3.3	23 lbs. caked material on walls. Wall temp. increased to 875°F.
22	94**	130	630	640	560		1.00	None	Light Green	24.0	75.7	73.8	2.4	Reactor clean. Wall temp. increased to 800°F.
23	92	190	530	560	750		1.00	None	Light Green	24.0	75.6	73.8	2.4	30 lbs. caked material in tower. Wall temp. increased to 800°F.
24	147	120	550	550	420		0.67	None	Green	24.0	75.4	74.1	2.9	Reactor clean.
25	144	100	700	650	770		0.67	None	Dark Green	23.9	75.8	73.9	3.0	60 lbs. caked material in tower. Wall temp. increased to 1000°F.
26	147	100	700	650	240		0.67	None	Dark Green	24.1	75.9	74.8	2.9	9 lbs. caked material in reactor.
27	150	140	520	500	290	240	0.08	Heavy		No Solid Product				No reaction.
28	150	140	600	610	410	410	0.17	Heavy		No Solid Product				No reaction.
29	150	140	740	770	720	720	0.67	None	Green	24.5	75.5	73.2	3.3	19 lbs. caked material in 6-inch section; 12-inch section clean. Reaction in both sections.
30	161	100	660	670	460	540	0.58	None	Green	24.1	75.5	73.8	3.0	30 lbs. caked material in 6-inch section; 12-inch section clean. Reaction in both sections.
31	132	100	540	540	200	640	0.67	Trace	Green	23.1	75.2	72.9	2.9	All reaction in 12-inch section. Wall temp. increased to 750°F. Reactor clean.
32	166	120	630	620	240	700	0.67	Trace	Dark Green	24.1	76.1	73.5	2.9	All reaction in 12-inch section. Wall temp. increased to 800°F. Reactor clean.
33	147	60	660	750	250	850	0.52	None	Light Green	23.7	75.7	74.6	2.7	All reaction in 12-inch section. Wall temp. increased to 950°F. Reactor clean.
34	151	45	630	650	400	840	1.00	None	Green	23.9	75.9	74.3	2.5	All reaction in 12-inch section. Reactor clean.
35	158	10	620	640	200	930	1.00	Trace	Light Green	24.3	75.8	75.1	2.8	All reaction in 12-inch section. Wall temp. increased to 1075°F. Reactor clean.
36	94	10	640	650	220	920	2.00	Trace	Dark Green	23.9	75.6	72.8	2.3	All reaction in 12-inch section. Wall temp. increased to 1050°F. Reactor clean.
37	83	80	560	550	170	860	2.00	None	Green	24.2	75.8	73.4	2.6	All reaction in 12-inch section. Reactor clean.
38	130	180	560	550	270	860	0.67	None	Light Green	24.0	75.5	74.7	2.7	All reaction in 12-inch section. Wall temp. increased to 1035°F. Reactor clean.
39	189	100	570	580	340	860	0.58	None	Light Green	24.0	75.4	74.5	2.6	All reaction in 12-inch section. Reactor clean.
40	141	150	840	850	340	910	0.75	None	Light Green	23.9	75.7	74.6	3.3	All reaction in 12-inch section. Reactor clean.
41	142	80	750	750	450	790	1.00	None	Green	24.1	75.6	75.5	3.2	All reaction in 12-inch section. Reactor clean.

CONFIDENTIAL

TABLE IV (Cont'd.)

URANIUM HEXAFLUORIDE REDUCTION WITH HYDROGEN

Run No.	Feed Rate, Pounds UF_6/hr.	H_2 Excess, %	UF_6 Temp., °F.	H_2 Temp., °F.	Reactor Temp.*, °F. Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Length Of Run, Hours	UF_6 In Outlet Gas	Product Color	Product Analyses % F	% U	% U^{+4}	Packed Density, g./cc.	Remarks
42	76	330	150	150	320	390	600	730		0.25	Heavy	Black		74.7	67.4	2.0	Only 4 lbs. product.
43	43	670	740	740	400	560	810	840		0.40	Trace	Green					UF_6 inlet plugged.
44	152	64	700	665	320	605	710	970		0.75	None	Light Green	24.2	75.9	74.8	3.5	29 lbs. caked material in reactor.
45	132	71	640	650	310	570	725	900		0.85	None	Some Black	23.9	75.3	74.4	3.3	50 lbs. caked material in reactor.
46	111	106	555	550	185	235	690	995		0.65	None	Green	24.2	75.5	74.5	3.3	Temps. out of control. Reactor clean.
47	83	90	595	595	205	340	485	975		1.25	None	Green	24.1	75.6	74.2	2.8	Reaction moved to precipitator section. 7 lbs. caked material in reactor.
48	79	141	690	705	305	365	665	910		1.50	None	Green	24.1	75.7	76.0	2.6	9 lbs. caked material in reactor.
49	78	148	605	605	220	310	650	1070		1.33	None	Green	24.0	75.6	74.8	3.5	Lower reactor section cherry red. Reactor clean.
50	84	84	615	615	360	405	720	905		1.42	None	Black	24.1	75.7	74.4	2.7	23 lbs. caked material in reactor.
51	80	230	475	480	250	460	905	1000		0.70	None	Green	24.1	75.6	74.5	3.7	Run shut down when lower section reached 1100°F. Reactor clean.
52	74	145	510	505	325	530	875	925		2.00	None	Some Black	23.9	75.3	73.1	3.2	Slight inlet pressure build-up. Reactor clean.
53	44	207	600	600	325	535	600	900		1.10	None	Green	23.7	75.2	73.1	2.8	Poor temp. control in zones 1 and 4. Reactor clean.
54	120	85	760	760	260	285	275	755	845	0.85	Trace	Green	23.9	75.0	71.1	3.5	Reactor clean.
55	127	82	435	515	445	520	525	735	745	0.88	Trace	Black	23.4	74.4	59.4	3.8	Reactor clean.
56	79	256	285	355	910	890	865	950	920	0.78	Trace	Dark Green	23.7	75.2	72.8	2.6	Unable to control temp. in zone 1. Reactor clean.
57	89	70	325	350	735	900	945	1060	930	2.00	Trace	Green	23.9	75.5	74.3	3.3	Reactor clean.
58	84	468	760	780	340	425	450	695	900	0.55	None	Green	23.9	75.7	74.6	2.6	9 lbs. caked material in reactor.
59	83	104	865	795	290	415	450	1025	760	1.50	Trace	Green	23.8	75.6	74.2	2.9	7 lbs. caked material in reactor.
60	76	236	875	870	380	530	680	1030	880	0.75	None	Green	23.7	75.3	72.0	3.2	Lost reaction to precipitator section. Reactor clean.
61	83	138	485	515	480	540	565	935	740	1.00	None	Light Green	23.9	75.6	73.0	2.4	Reactor clean.
62	80	200	675	630	445	440	435	825	540	0.22	Heavy		No Solid Product				No reaction.
63**	73	0	720	725	720	820	815	885	775	0.55	Heavy	Black	No Sample Taken				Little reaction.
64**	80	256	660	660	570	560	815	1000	865	0.75	Heavy	Black	23.8	75.0	72.4	2.9	Reaction in precipitator.
65	33	250	710	640	895	945	960	1000	215	1.00	None	Black	23.6	74.6	69.7	3.1	Reactor clean.
66	113	70	620	580	500	580	660	740	735	0.28	Heavy	Black	No Sample Taken				Little reaction.
67	95	100	630	620	700	800	795	945	820	1.00	None	Light Green	24.1	75.7	75.8	2.8	12 lbs. caked material in reactor.
68	92	269	625	620	730	870	810	910	885	1.75	None	Light Green	24.2	75.8	75.5	2.9	Reaction dropped to zone 5. Reactor clean.
69	91	113	635	625	780	855	830	1110	700	3.50	Trace	Dark Green	24.1	75.7	74.0	3.7	Inlet pressure build-up.
70	91	118	670	645	740	885	850	940	685	3.72	None	Dark Green	24.1	75.7	73.3	3.2	150 lbs. caked material in reactor.
71	96	99	640	630	760	895	735	1045	740	5.00	None	Dark Green	24.0	76.0	74.5	3.2	68 lbs. caked material in reactor.
72	100	43	730	675	830	935	800	950	705	5.00	None	Light Green	24.1	75.8	74.7	3.2	Reactor clean.
73	96	92	615	605	865	875	560	1000	780	1.75	Heavy	Light Green	24.0	75.8	74.3	2.9	UF_6 appeared in outlet after 1.5 hrs.

* Average temperature at start of each run.

** Sufficient nitrogen, to lower the UF_6 concentration to 50%, was added before the reactor.

CONFIDENTIAL

CONFIDENTIAL

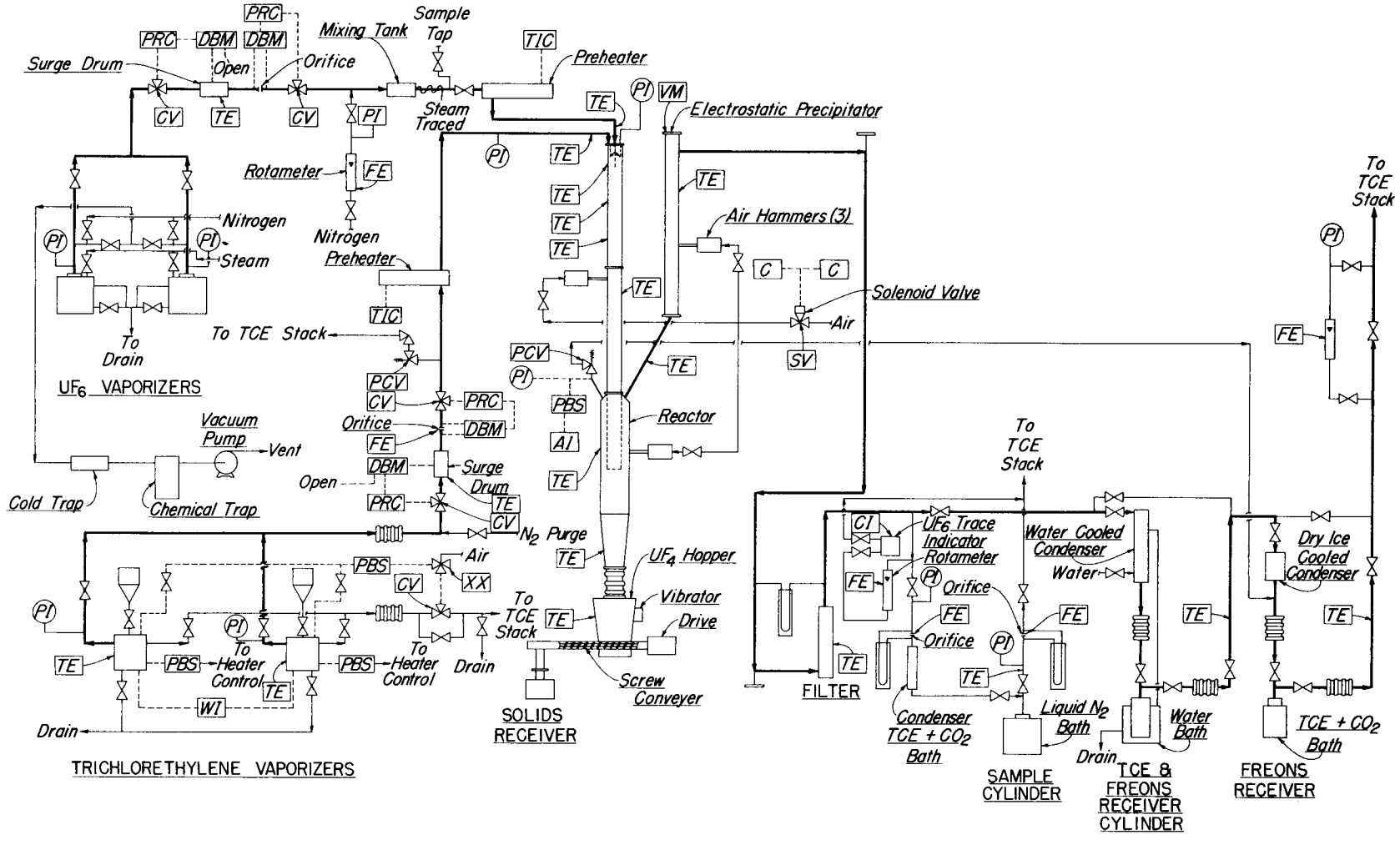

Figure 1

UF_6 REDUCTION PILOT PLANT
FLOW AND INSTRUMENTATION

CONFIDENTIAL

CONFIDENTIAL

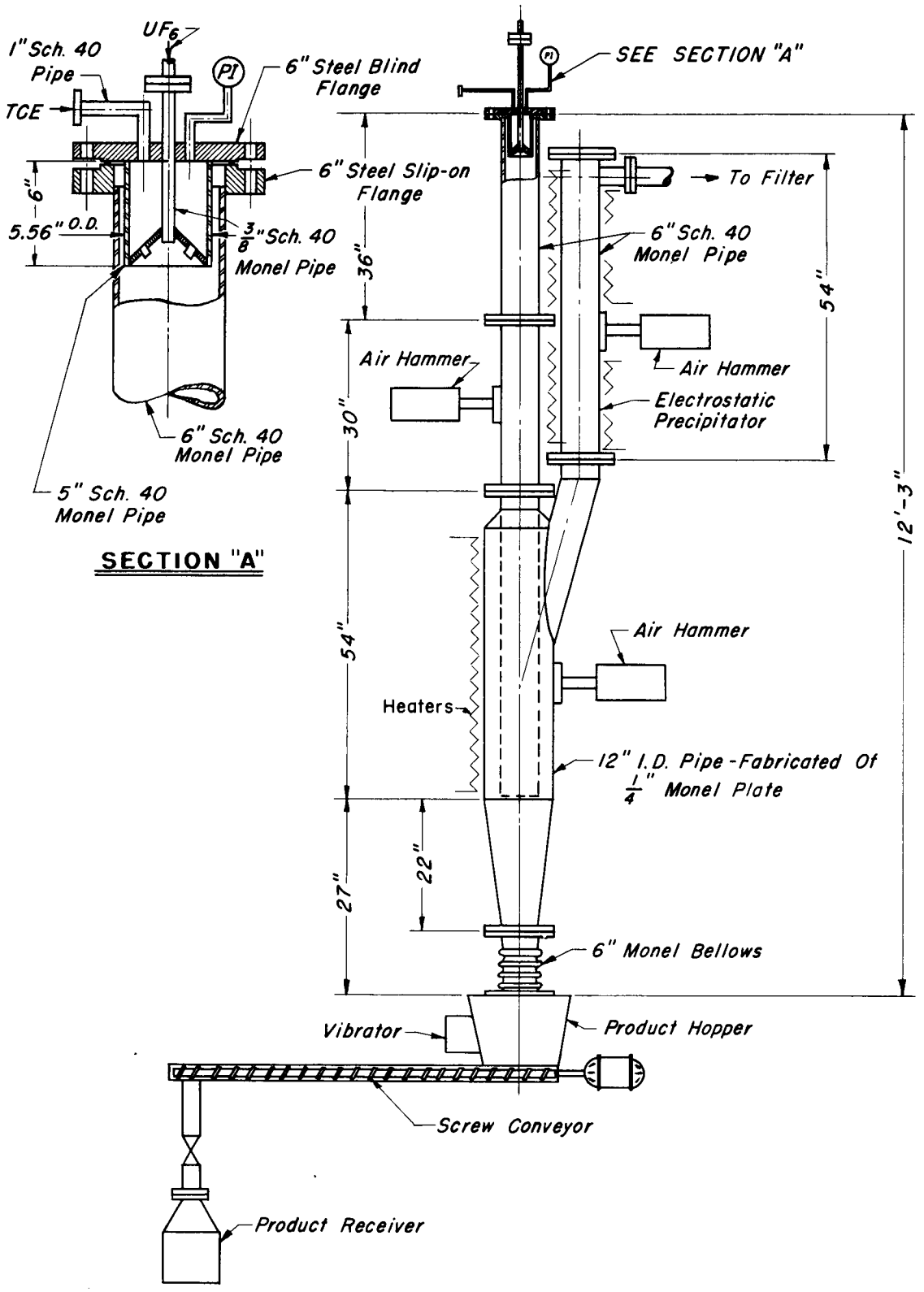

Figure 2
UF_6 REDUCTION REACTOR
ORIGINAL UNIT

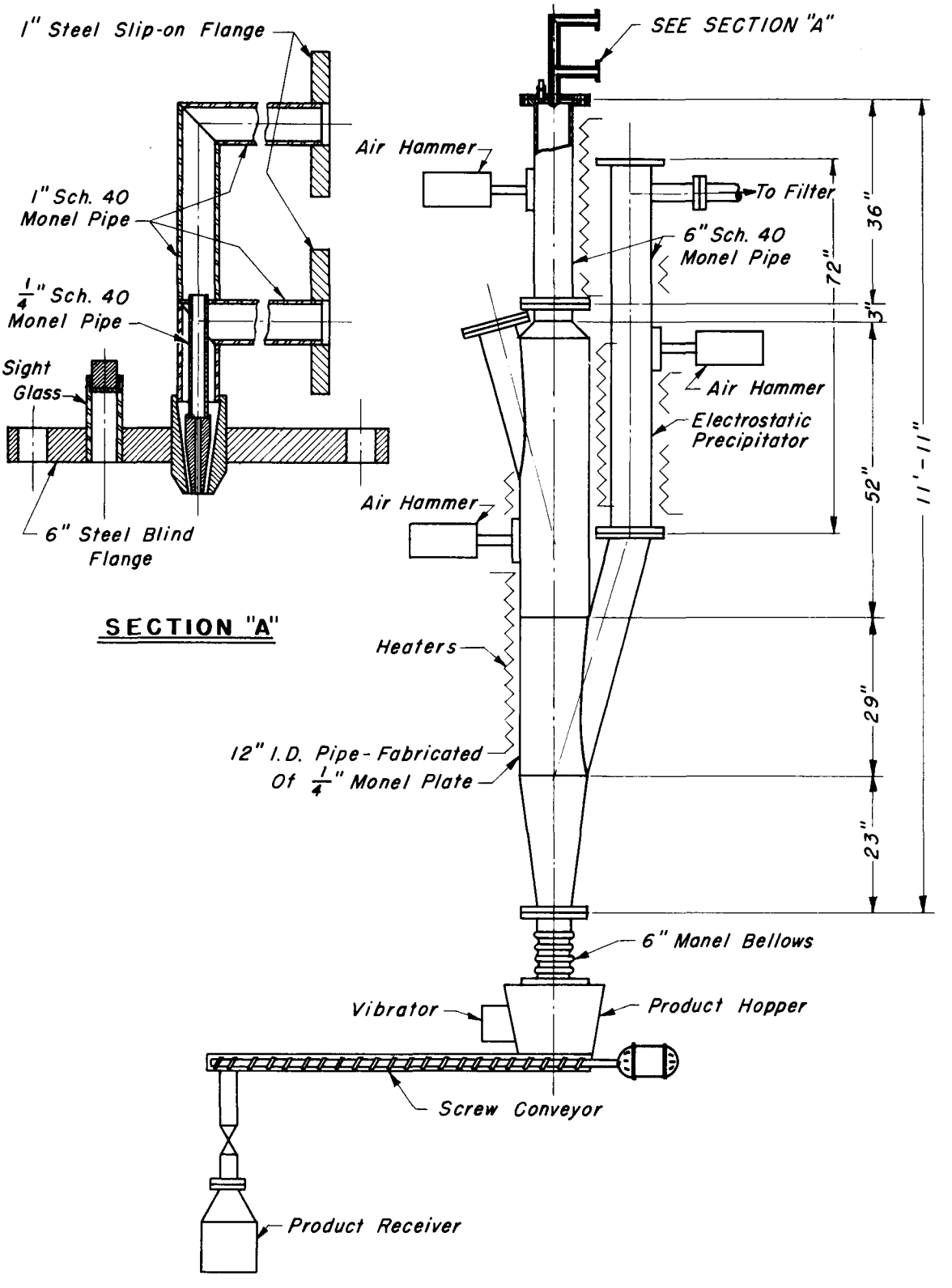

Figure 3

UF_6 REDUCTION REACTOR
FIRST REVISION

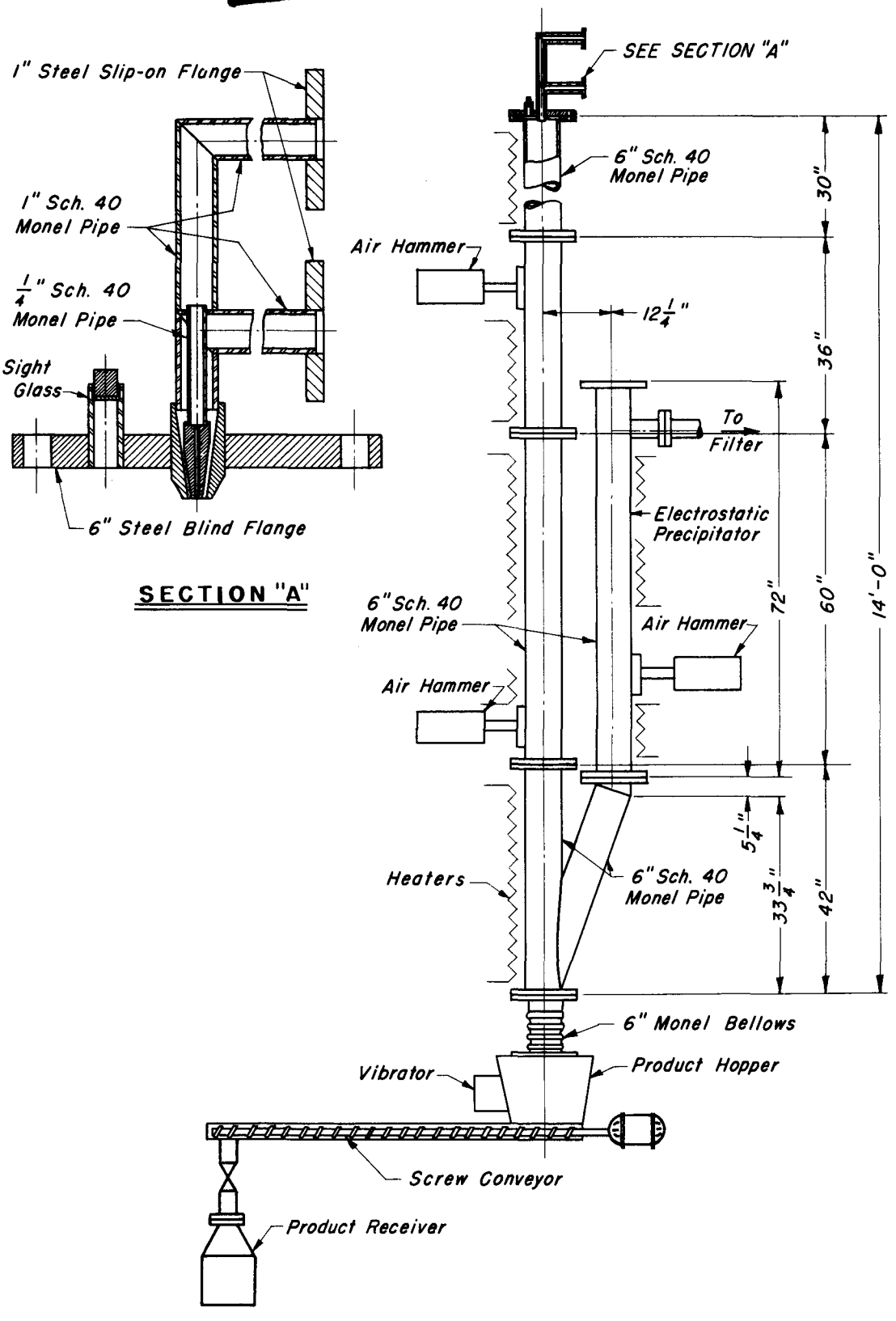

Figure 4

UF_6 REDUCTION REACTOR
SECOND REVISION