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Works Technical Department Report

TEST PILE REACTIVITY LOSS
DUE TO TRICHTHLORETHYLENE

K. E. Plumlee, L. C. Heck &
R. A. Webb, Jr.

August 24, 1953

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Test Pile Reactivity Loss
Due to Trichlorethylene

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Test Pile Reactivity Loss
Due To Trichlorethylene

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ABSTRACT

The presence of trichlorethylene in the test pile caused a continual decrease in pile reactivity. A system which removed, purified, and returned 12,000 cfh helium to the pile has held contamination to a negligible level and has permitted normal pile operation.

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SUMMARY

Data collected after calibration indicated that the test pile reactivity was continually decreasing. This decrease was found to be due to trichlorethylene vapor which contaminated the sealed helium gas system enclosing the pile. An air purge through the gas casing reduced the contamination and increased the pile reactivity. The rate of trichlorethylene removal indicated that purging would be necessary for several months. The pile was subcritical during air purging.

A system which removed, purified, and returned 12,000 cfh helium to the pile has held contamination to a negligible level and has permitted normal pile operation.

Test pile reactivity had returned to the calibration level by late February 1953 and has continued to increase through June. The excess reactivity has apparently stabilized at 110% of the calibration value.

More than 7 kilograms of trichlorethylene were removed from the pile.

RECOMMENDATIONS

Recirculation of purified helium will be necessary for an indefinite period. Failure to control the trichlorethylene vapor content of the pile atmosphere will result in loss of reactivity.

Daily determinations of excess reactivity of the pile will provide an indication of the effectiveness of the purifying system.

Periodic analyses of cold trap condensates should be made. These analyses will indicate the rate of removal of moisture and trichlorethylene vapor from the pile.

DISCUSSION

Determining the Cause

Shortly after the calibration data had been collected a plot of reactivity vs time (see figure 1) indicated that the pile reactivity was continually decreasing. The probable causes were considered to be:

1. air leaking into the pile atmosphere
2. a temperature rise in the pile moderator
3. contamination in the pile atmosphere

Tests for the air content in the pile atmosphere (using an Orsat) proved that air leakage was an insignificant factor. Resistance thermometers were installed on December 24, 1952 in test hole 24 (under TA 3-19). Within a few days it was apparent that changes in pile temperature were negligible. (See table I, appendix).

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Since the pile walls and gas system were cleaned with trichlorethylene, the presence of trichlorethylene in the pile atmosphere was considered to be the most probable cause of the reactivity loss. A copper chip flame test of the exit gas showed the presence of halogens. A similar test on the helium supply yielded negative results. Samples collected through liquid nitrogen traps at the gas exit were analyzed and found to contain trichlorethylene and water. Liquid nitrogen traps on the helium supply did not collect trichlorethylene. As shown under the heading "Trichlorethylene Poisoning", Appendix, the observed loss of reactivity correlated with the loss calculated on the basis of measured trichlorethylene contamination.

Removing Trichlorethylene

Reverse Gas Flow, TA 3-22. Since there were low points where trichlorethylene may have been trapped in the feed lines, the gas flow was reversed. Helium gas entered the pile through the normal carbon dioxide vent and left the pile through the helium vent, the helium feed line, and the carbon dioxide feed line. The feed was increased to 10 cfh helium (see table II, appendix).

Liquid nitrogen traps were placed on the supply and at each exit. As shown in table III, appendix, the quantity of trichlorethylene trapped from the new discharge points was not greater than that trapped from the normal helium vent. Thus, it was concluded that no liquid trichlorethylene was trapped in the U-shaped feed lines.

East-West Air Purge, TA 3-23. ^{Heated} ~~Heat~~ and filtered air was taken into the pile through a manifold and the annuli around the liners of test holes 25, 26, and 27. Air was drawn out of the pile thru the helium exit by the vacuum cleaner, which is normally used for removing shot. A schematic diagram of the equipment arrangement is shown in figure 2. The seal pot, which controls pile pressure, was disconnected, but the pressure differential between the room and the pile was limited to 1.5 inches of water. The air was heated to 55°C on entering the pile, and the relative humidity was held as low as the building air conditioning system would maintain. The main air stream was near the bottom of the pile since trichlorethylene was suspected to be in the grout, packed under the base plates. During this period the pile reactivity became constant (see figure 2). Since the temperature was increasing, and a graphite-natural uranium pile has a negative temperature coefficient, the pile reactivity should decrease. This indicated that the reactivity had improved, if corrected for temperature changes.

South-North and East-West Air Purge, TA 3-27. To more thoroughly purge the graphite, air was blown into the pile through the annuli around the liners of test holes 1, 4, 5, 6, 7, 8, 9, 10 and 11 on the south side and removed from the same holes on the north side. This equipment arrangement is indicated in figure 3. Although most of the air circulated up and over the graphite, better ventilation of the metal channels was obtained since these extend north-south through the graphite. The east-west purge was continued, but heating was stopped. Pile reactivity increased steadily and at the point where the galvanometer reading became constant, calculations based on sub-critical flux measurements showed that most or all of the lost reactivity had been regained.

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Data on reactivity changes are plotted in figure 4 and tabulated in table IV, appendix.

Helium Recirculating System, TA 3-32. The gas casing was sealed and the air was displaced with helium beginning February 7. This permitted normal operation of the pile, but a significant increase occurred in trichlorethylene vapor content before the purification system installation was completed (see table V, appendix).

Beginning February 14, 1953, 12,000 cfh of helium was removed from the helium vent, purified, and returned to the pile through the normal carbon dioxide vent and a temporary fitting placed over the south end of test hole 1.

The purifying system consisted of a 24-inch by 24-inch by 9-inch bed of silica gel for moisture removal and a 27-inch by 27-inch by 36-inch bed of activated charcoal for trichlorethylene removal. About 90% cleanup was indicated at 12,000 cfh until the contaminant in the helium returned to the pile fell below 5 ppm, which was the lower limit of the sampling technique. Blower, filters, and other mechanical details appear on SRP dwg. S5-3-103 (see figure 6).

A slow increase in reactivity occurred during recirculation. (See figure 5 & table VII). The excess reactivity had apparently stabilized at about 110% of the calibration value during June 1953. Interruptions of recirculation have invariably been accompanied by loss of reactivity and increased trichlorethylene contamination.

The contamination level in the pile fell to less than 50 ppm of trichlorethylene by weight after a few days of recirculation. By April 30, 1953 the gas removed from the pile contained only 15 ppm of trichlorethylene. There has been no indication of substantial additional improvement.

Laboratory scale testing which preceded the installation of the equipment is reported in DPSP-53-202.¹ This indicates that complete removal of the trichlorethylene which may be absorbed in the graphite can eventually be accomplished by ventilating the pile with pure helium.

No test was performed to determine the products resulting from the irradiation of trichlorethylene. It has been assumed that the amount of these materials is small and that the activated charcoal has more affinity than the graphite for the volatile material. If these products are not absorbed in the purifying system they will eventually be removed by the normal helium feed.

1 "Sorption of Trichloroethylene on Pile Grade Graphite and Activated Charcoal" by F. T. Osika and P. G. McCarthy.

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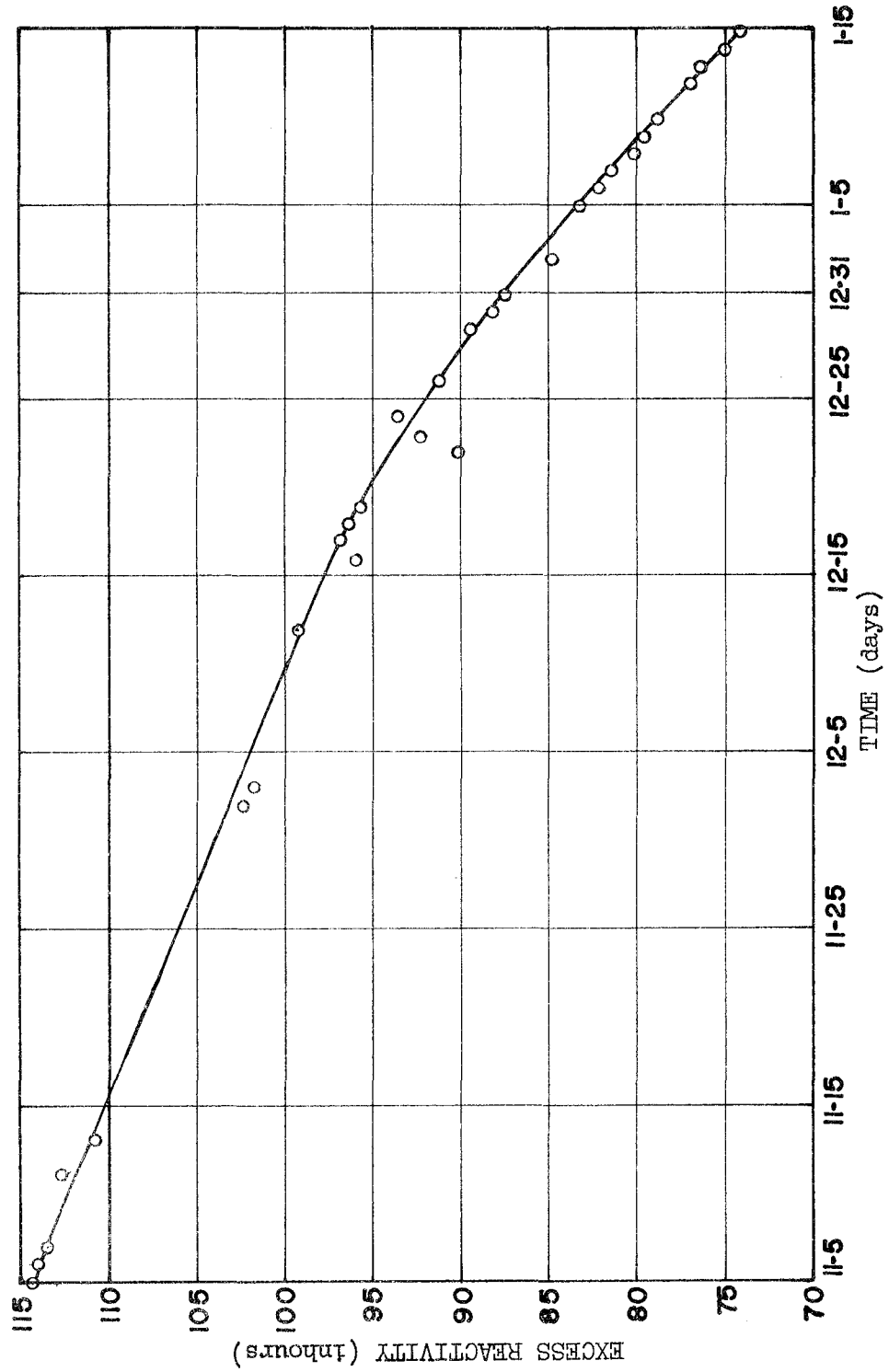


Figure 1. Loss in Reactivity Due to Trichlorethylene Poisoning

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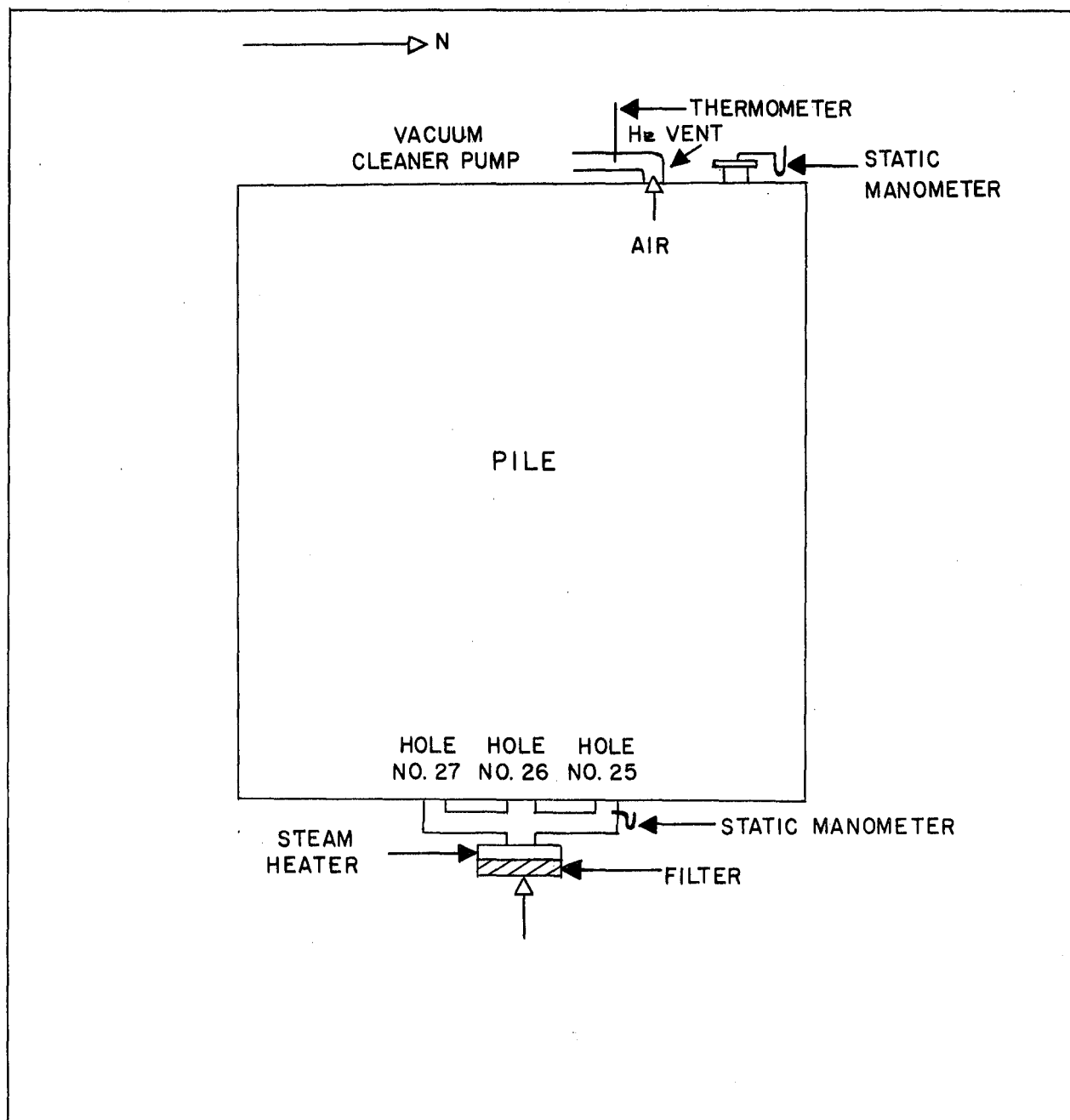


Figure 2. Air Purge Diagram

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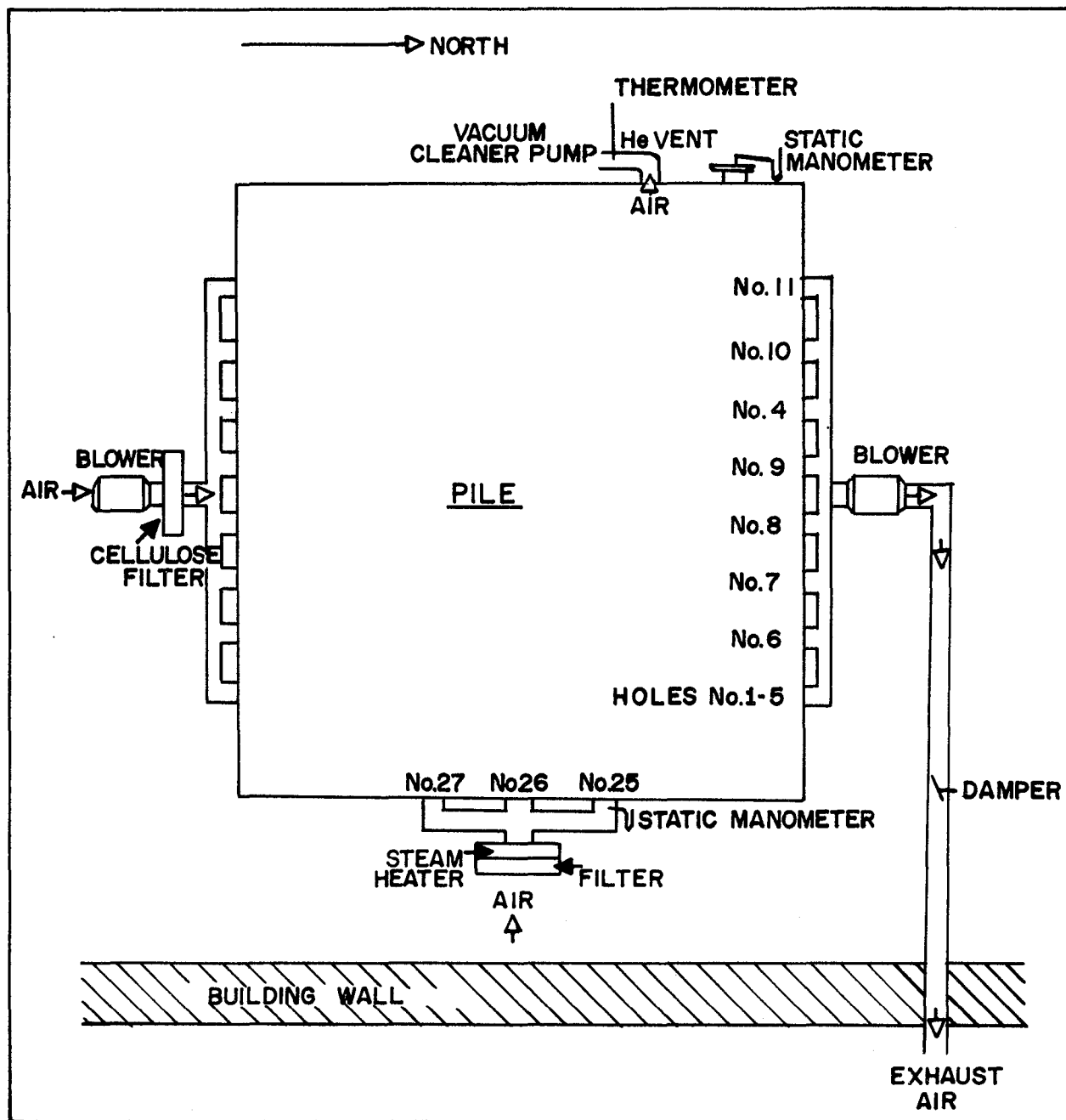


Figure 3. Air Purge Diagram Showing Test Holes

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TEMPERATURE OF PILE GRAPHITE °C

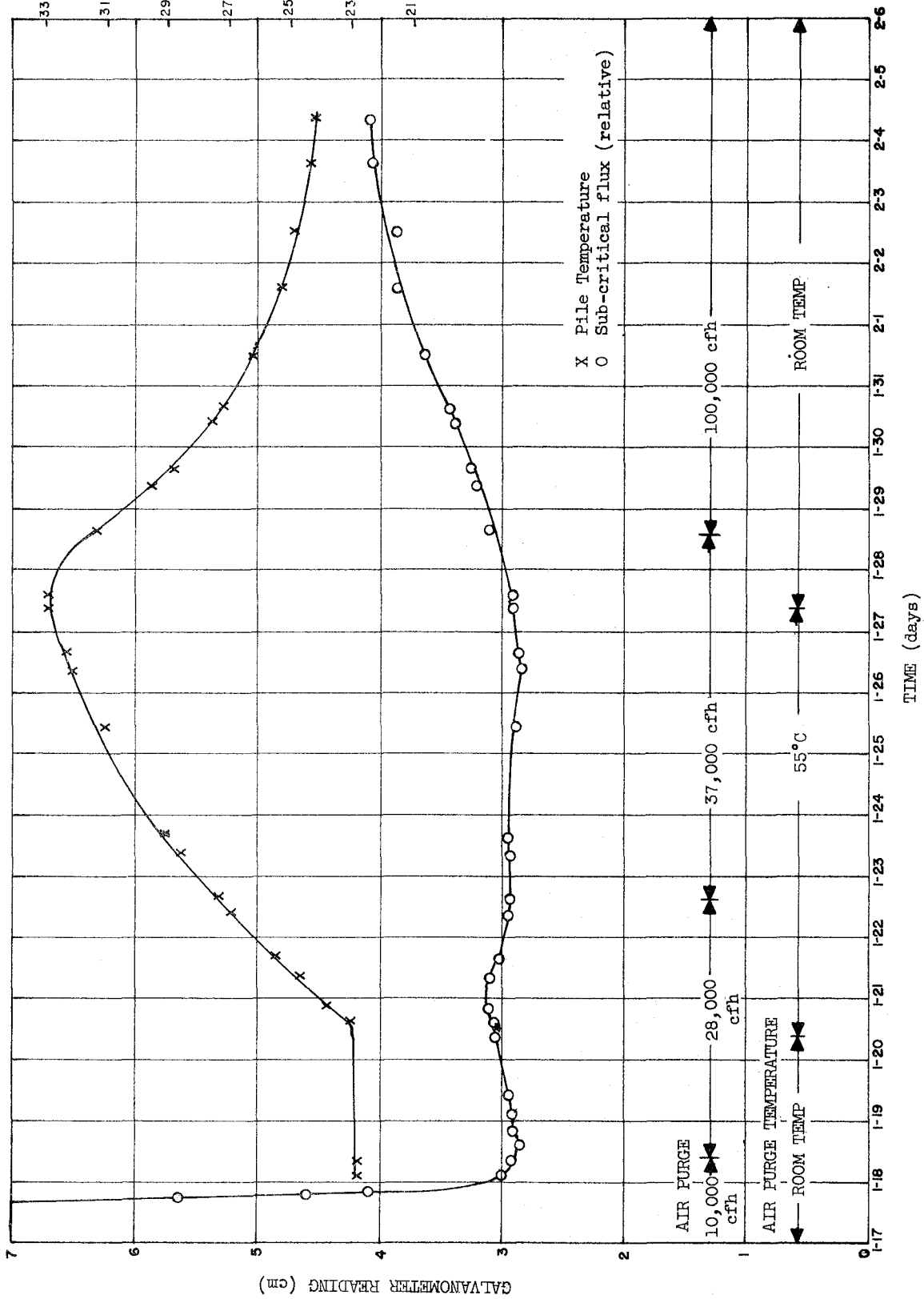


Figure 4. Gain of Reactivity Due to Air Purge

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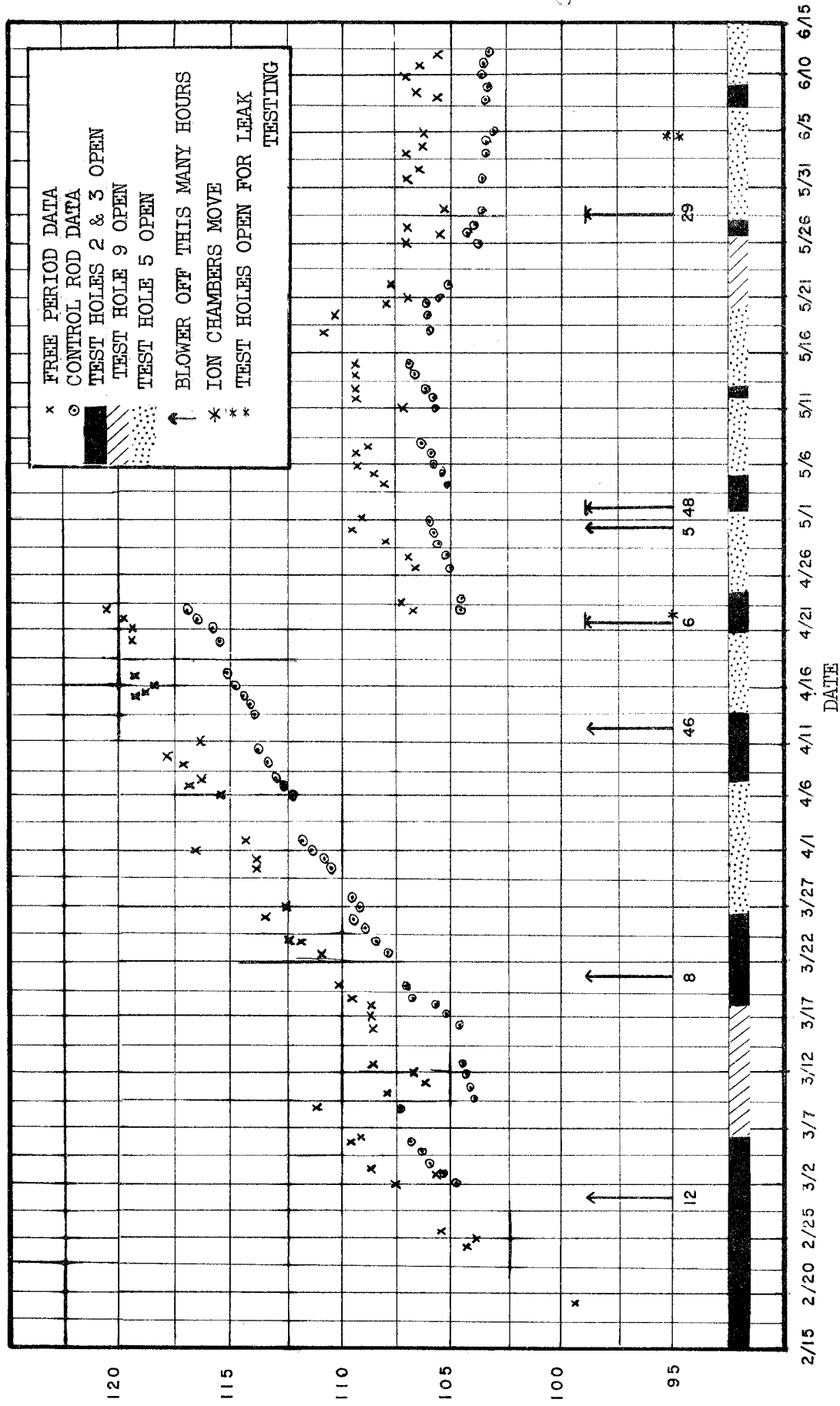


Figure 5. Helium Recirculating System - Reactivity Gain Due to Purge of Trichlorethylene

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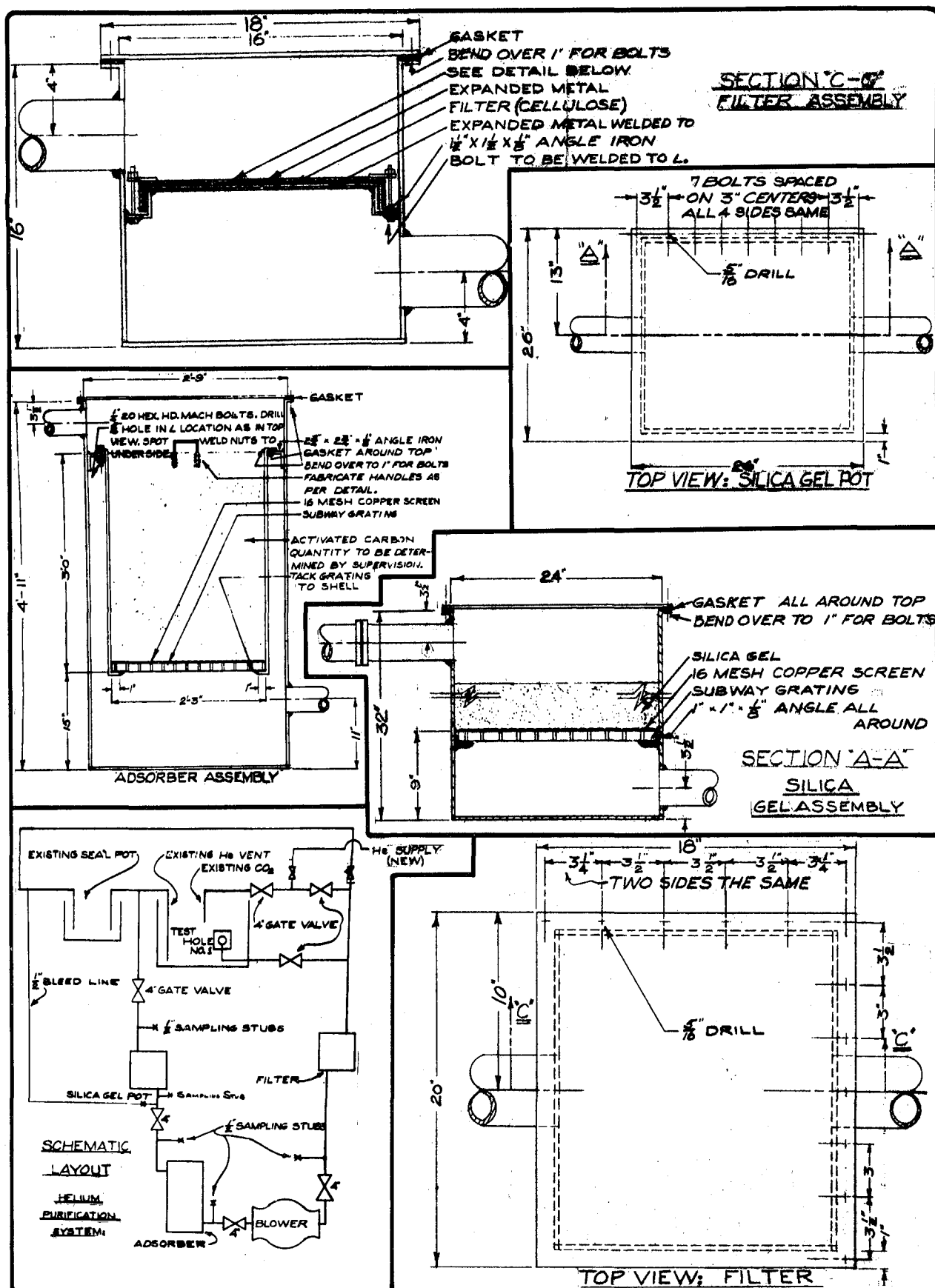


Figure 6. Mechanical Details of Helium Recirculation System. (These drawings are not to scale.)

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APPENDIX

TRICHLORETHYLENE POISONING

Calculation based on calibration measurements with standard absorbers showed that for uniform distribution over the active lattice 0.36 cubic centimeters trichlorethylene (liquid) would account for approximately 1 inhour loss in reactivity. For the 38 inhours lost, this would be equivalent to 106 cubic centimeters of trichlorethylene uniformly distributed throughout the pile.

At the time of the 38 inhour loss, the concentration of trichlorethylene in the exhaust gas was 0.11 cubic centimeters of liquid per cubic foot of gas, with a relatively low helium feed into the system. Inside the graphite block there are 1100 cubic feet of free space. Assuming that the 0.11 cubic centimeters/cubic foot holds throughout the pile; $(1100) \times (0.11) = 121$ cubic centimeters of trichlorethylene.

The calculated loss due to the observed concentration of trichlorethylene vapor agreed very closely with the observed decrease in reactivity.

About 3,000 cubic centimeters of water would be required to cause an equivalent loss.

The pile will reflect a change of one gram (or about 180 ppm by weight) of trichlorethylene in the pile atmosphere as a reactivity change of about 0.25 inhours.

This section contains several tables which are referred to in the main body of this report.

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Table I. Loss of Reactivity Due to Trichlorethylene Poisoning

Date	Excess Reactivity, inhours	Cumulative Loss/day, inhours	Helium Purity,*1 %	Barometric Press,*2 mm of hg	Moderator Temp, °C
11- 5-52	114.65		Nominal	754.5	
11- 6-52	114.0	0.65	100	755.2	
11- 7-52	113.67	.49		761.8	
11-11-52	112.76	.32		756.5	
11-13-52	110.93	.46			
12- 2-52	102.30	.46		758.5	
12- 3-52	101.80	.46		760.8	
12-12-52	99.36	.41		756.5	
12-16-52	95.98	.44		762.4	
12-17-52	96.85	.42	99.7	758.8	
12-18-52	96.49	.42	Helium Feed	760.7	
12-19-52	95.81	.42	erratic	760.8	
12-22-52	90.13	.52	82	758.2	
12-23-52	92.28	.46	99	757.2	
12-24-52	93.58	.43		759.6	
12-24-52	93.58*3	.43		760.7	
12-26-52	91.1	.46	92.5	760.4	23.32
12-29-52	89.4	.47	90.5	761.4	23.24
12-30-52	88.0	.48	81.5	758.5	23.19
12-31-52	87.4	.49	79.4	748.3	23.18
1- 2-53	84.8	.51	80.3	761.5	23.13
1- 5-53	83.1	.52	95.0	756.0	23.05
1- 6-53	82.0	.53		761.6	23.02
1- 7-53	81.5	.53	92.0	763.0	
1- 8-53	79.1	.56	95.4	758.0	22.99
1- 9-53	79.4	.54	96.2	743.1	22.97
1-10-53	78.9	.54	96.2	749.0	
1-12-53	76.8	.56		761.1	
1-13-53	76.2	.56		765.1	23.00
1-14-53	75.0	.57		764.8	23.00
1-15-53	74.0	0.57		763.5	

- *1 Helium purity was estimated on the basis of orsat analyses for oxygen. No correction for impurities other than air and moisture were considered.
- *2 Barometric pressure measured in Control room in millimeters of mercury at 23°C.
- *3 Resistance thermometers installed in test hole 24 resulted in a loss of 8.6 inhours. All readings after 12/24/52 contain 8.6 inhour adjustment to compensate for this effect.

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Table II. Helium Feed into Gas System

Date		Average Purge Rate, cfh
Start	End	
10/30/52	11/7/52	0.65
11/8/52	11/17/52	0.9
11/18/52	12/23/52	1.1
System	12/23/52	944 (at unknown rate)
Failure	12/24/52	10.0
12/25/53	1/6/53	1.0
1/6/53	1/16/53	9.0 (see TA 3-22)
1/17/53	2/6/53	Air purge (see table IV)
2/7/53	2/14/53	10.0
2/15/53	6/30/53	Makeup to system ~2* ¹
		Recirculation of purified helium *2

*1 Several system failures resulted in high purge rates for short periods.

*2 Sec TA 3-32.

Table III. Trichlorethylene Removed from Pile Before Air Purge

Time	*1 Date	Location	Time Collect, Hours	Total Flow, cu ft	Top Layer cc	Bottom Layer,*2 cc	Liquid per cu ft gas, gm	
							H ₂ O	TCE
	1-9-53	CO ₂ FP	2.33	8.8	2.35	1.55	.267	.258
1545	1-9-53	He Vent	1.33	10.0	3.00	1.70	.300	.249
1742	1-9-53	He FP	1.82	7.6	0.8	0.00	.105	
0834	1-10-53	CO ₂ FP	1.66	7.6	1.5	1.1	.197	.211
0956	1-10-53	He Vent	1.00	9.0	2.5	1.15	.277	.186
0855	1-12-53	He Vent	1.25	11.0	5.25	1.25	.477	.166
1059	1-12-53	CO ₂ FP	1.97	9.1	1.70	0.90	.186	.144
1355	1-12-53	He FP	2.57	11.9	1.30	0.00	.109	
0850	1-13-53	CO ₂ FP	2.08	9.0	2.1	1.1	.233	.179
1205	1-13-53	He Vent	1.00	9.1	2.2	0.7	.241	.111
0932	1-14-53	CO ₂ FP	2.00	9.6	3.3	1.0	.343	.152
1300	1-14-53	He Vent	1.00	9.3	2.6	0.6	.279	.094
0941	1-15-53	He FP	2.00	10.1	-	-	-	-
	1-15-53	He Vent	-	6.5	5.4	1.4	.830	.315

*¹ No flow data available on samples collected prior to 1-9-53. These samples were collected by passing pile gas through liquid nitrogen trap. The liquid was turned over to the Building 320-M control laboratory for qualitative analysis.

*2 Samples collected after 1-16-53 showed no bottom layer. The air purge began on this date, and purge rate was much higher than above.

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Table IV. Gain of Reactivity During Air Purging

Date	Time	Galvanometer Reading, cm	Temp, * °C	Purge Rate, cfh	Remarks
1-17-53	1604	8.70		9,900	Air purge from east end of holes 25, 26, & 27 around mg liners. Out helium vent to vacuum pump. Circulated air at room temp. See TA 3-23.
		5.65		9,700	
	2000	4.60	23.06	9,700	Conc. TCE in exhaust air
	2200	4.10	23.03	10,000	yielded 99 ppm in water or
1-18-53	0330	3.00		10,000	0.000018 gm/cf air*
	0935	2.90	23.04	10,000	
	1535	2.85		27,200	
	2135	2.90		27,200	
1-19-53	0332	2.90		27,400	Air going into pile heated to 130°F
	1130	2.95		27,400	
	0945	3.05		27,800	Conc. TCE in exhaust air
1-20-53	1500	3.05	23.16	27,800	yielded 69 ppm in water or
	2200	3.10	23.89	28,000	0.000018 gm/cf air*
1-21-53	0800	3.10	24.87	28,300	
	1600	3.01	25.65	28,000	
1-22-53	0900	2.95	27.11	27,600	
	1600	2.93	27.46	27,600	
	0835	2.92	28.73	37,000	Conc. TCE in exhaust air
1-23-53					yielded 70 ppm in water or
	1600	2.95	29.27	37,000	0.000018 gm/cf air*
1-25-53	1100	2.88	31.26	37,000	
1-26-53	0900	2.83	32.20	37,000	
	1600	2.86	32.40	37,000	
	0900	2.90	33.00	37,000	Cut off heat to circ. air
1-27-53					Started S-N purge; kept E-W purge
	1400	2.90	33.10	37,000	
1-28-53	1615	3.10	31.42	110,000	
1-29-53	0915	3.20	29.69	112,000	
	1600	3.35	29.02	101,000	
1-30-53	1000	3.39	27.64	101,000	
	1600	3.42	27.30	101,000	
1-31-53	1200	3.63	26.36	101,000	
2- 1-53	1507	3.85	25.43	101,000	
2- 2-53	1300	3.85	24.90	101,000	
2- 3-53	1600	4.05	24.42	101,000	
2- 4-53	0845	4.08	24.30	101,000	
2- 6-53	0900				Air purge ceased

* Samples tested by E. C. Dunlop

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Table V. Reactivity With Helium Atmosphere Before Helium Recirculation

Date	Start	Reactivity, in hours	Helium*1 Purity, %	Dura- tion, hours	Gas Flow, cu ft	Top Layer, cc	Bottom*2 Layer, cc
2- 9-53	0910			0.83	10.3	2	Trace*3
	1430	48.96	82	1.67	20.7	4	Trace*3
2-10-53	1040	65.98	85	5.00	35.2	7.2	0.7*3
2-11-53	1016	76.95	87	3.60	32.0	6.2	0.4*3
2-12-53	1015	87.33	90	3.83	37.3	3.3	0*4

* 1 Impurity present was air.

* 2 Highest concentration of TCE during this period was 0.029 gm/cf.

* 3 TCE Drops on side of tube could not be collected.

* 4 From He inlet.

Table VI. Summation of Minimum Quantities of Trichlorethylene Removed

Date	Rate, cfh	TCE Conc, gm/cf	C.F.	TCE, gm	Total
to 1-16-53					900
1-16-53 to 1-18-53	10,000	0.00013	42×10^4	54.6	955
1-18-53 to 1-19-53	27,000	0.00013	81×10^4	105.3	1,060
1-19-53 to 1-22-53	27,000	0.000018	186×10^4	33.5	1,093
1-22-53 to 2- 6-53	100,000	0.000018	36×10^6	648.0	1,741
2- 9-53 to 2-25-53	12,000	0.00023*	46×10^5	1,058.0	2,799
2-25-53 to 7-31-53	12,000	0.00010*	44×10^6	4,400.0	7,199

* Based on approximate cleanup in activated charcoal bed.

Table VII. Reactivity During Helium Recirculation

Date	Reactivity, inhours	Location	TCE, ppm	Approx RH, %	Sample, cu ft	TCE, mg	Approx Liq Vol, ml	Approx Mod Temp, °C
2-19-53	98.26	Exhaust	7.2	4.7	203	3.0	5	22.89
2-21-53		Exhaust	5.9		176.4	1.6		22.75
2-22-53		Exhaust	4.3		151.9	0.18		
2-24-53	104.1	Exhaust	4.2	2.9	200	0.18	3	22.53
2-25-53	103.3	Inlet	37.9	4.9	196	31.12	5	
2-26-53	105.37	Inlet	30.5	4.7	122	15.12	3	22.30
3-3-53	105.5	Exhaust	4.4	3.8	151	0.29	3	22.11
3-4-53	108.52	Inlet	4.7	3.4	169.4	0.52	3	
3-6-53	109.46	Inlet	44.8		210.9	40.3		22.00
3-10-53	107.6	Inlet	4.9	4.3	249.5	1.03	5.5	22.58
3-11-53	106.25	Exhaust	4.6	3.6	284.7	0.75	5.3	
3-12-53	106.69	Inlet	7.7		173	3.0		
3-13-53	108.11	Inlet	90.8		208	84.5		
3-16-53	108.11	Inlet	45.0	5.6	171	32.8	5	
3-18-53	108.11	Exhaust	5.0		155	0.78		
3-19-53	109.46	Inlet*1	170.8		150	117.09		
3-20-53	109.94	Inlet	52.7		64	14.6		
3-24-53	111.93	Exhaust	4.6		150	0.4		
3-25-53	112.45	Inlet	20.5		154	11.92		
3-26-53	113.67	Exhaust	4.1		162	0.11		
3-27-53	112.96	Inlet	5.8		164	1.35		
4-1-53	116.76	Inlet	17.1		190	11.7		22.32
4-2-53	114.55	Exhaust	4.1		145	0.6		
4-7-53	116.76	Inlet	10.7		113	3.52		
4-8-53	116.2	Exhaust	4.1		163	0.12		
4-10-53	118.5	Inlet	13.7	8.2	143	6.50	6.1	22.4
4-14-53	119.27	Inlet	29.1		132.8	15.6		22.36
4-15-53	118.91	Inlet	23.8		146	13.5		22.34
4-16-53	118.6	Exhaust	4.3		167	0.23		21.85
4-20-53	119.1	Inlet	15.9	8.5	146.4	8.13	6.5	
4-21-53	119.1	Exhaust	4.2	8.4	160	0.16	7.0	
5-6-53*2	109.0	Inlet	16.4	12.3	124	7.2	8.0	23.15
5-7-53	109.0	Exhaust	4.0	10.5	127	0.03	7.0	
5-12-53	108.84	Inlet	13.9	10.8	141	6.50	8.0	23.21
5-13-53*3	109.0	Exhaust	4	2.4	160	0.04	2.0	22.84
5-14-53	109.0	Inlet	19.9	4.7	142	10.6	3.5	
5-19-53	109.0	Exhaust	4.2	3.3	233	0.2	4.0	23.65
5-20-53	107.6	Inlet	9.8	3.5	190	5.2	3.5	23.31
6-2-53	106.11	Inlet	14.1	5.5	138	6.5	4.0	25.03
6-3-53	106.54	Exhaust	4.3	6.2	138.5	0.2	4.5	25.06

*1 Blower off

*2 Ion chambers moved 4-22-53 (13.1 inhour reduction in reactivity resulted)

*3 Silica gel changed 5-13-53