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**Sulfur Hexafluoride Purification from
Mixtures With Air: A Process
Feasibility Study**

Joseph J. Perona
J. S. Watson

**OAK RIDGE NATIONAL LABORATORY
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WITH AIR: A PROCESS FEASIBILITY STUDY

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ABSTRACT

Studies were made of the purification of SF₆ vapor contaminated with air for application at the Holifield Heavy-Ion Research Facility. Liquefaction appears to be a good method for recovering about 90% of the SF₆ if it is badly contaminated (15% air), and an even greater fraction can be recovered from mixtures containing less air.

In cases where liquefaction is insufficient by itself, adsorption of SF₆ on activated carbon at -50°F is promising. Two carbon beds, each containing about 500 lb of carbon, should be sufficient. The refrigeration system for liquefaction and adsorption would have a capacity of about 2 tons.

As an alternative, the use of molecular sieves to trap out the air was investigated, but such a bed would require at least 15,000 lb of molecular sieves and very long cycle times. A large-scale desublimer was also investigated and appears workable, but it would require some development effort before the design could proceed with confidence.

1. INTRODUCTION

The Holifield Heavy-Ion Research Facility,¹ now under construction at Oak Ridge National Laboratory, will use SF₆ as an insulating gas. The accelerator is contained in a pressure vessel filled with approximately 240,000 lb of SF₆ gas at a pressure of about 100 psia. When entry into the pressure vessel is necessary, the SF₆ must be transferred to storage. The SF₆ will be stored as a liquid at ambient temperature in three 2000-ft³ tanks at about 350 psia.

The accelerator vessel will be evacuated before it is filled with SF₆ from liquid storage, but it cannot be evacuated completely. Therefore, the concentration of air in the SF₆ will gradually rise as the SF₆ is transferred back and forth, and the SF₆ will eventually require purification. Errors in operating procedures could also bring about contamination of the SF₆ with air. It is virtually certain that a purification process for the SF₆ will be needed during the many years of operation projected for the accelerator. This need arises not from a deterioration of insulating properties, but rather from the necessity of liquefaction at ambient temperature for storage.

Purification of SF₆ has been carried out on a small scale at other accelerator laboratories. At Chalk River, the SF₆ has been separated from air by freezing it out with liquid nitrogen.² The use of an activated charcoal adsorption column for purification from air and helium was described by Brassard³ in which the contaminated gas was introduced to the column in short bursts and the column was operated as a chromatograph. With the column at -20°C (-4°F), a 650-g burst of gas was admitted and the effluent for the following 1 min was vented as primarily contaminant gases. The SF₆ was then collected for the next 15- to 20-min period. The column was 8 in. in diam by 33 in. long and had a production rate of 100 lb/day. Other studies of air adsorption into Linde 5A molecular sieves have also been reported.⁴ Use of these methods was investigated for application to the present system.

2. SELECTION OF BASE CASES

The following incident is considered as a worst case: SF₆ is pumped from liquid storage into the accelerator without prior evacuation of the accelerator. The accelerator filling is stopped at the normal operating pressure of 85 psig, but it now contains 1-atm partial pressure of air. The composition of the accelerator tank is 207 lb-moles of air and 1200 lb-moles of SF₆, giving 0.15 m.f. of air.

This incident badly contaminates most of the SF₆ inventory and therefore serves as a "worst" case. The purification process must be capable of restoring the SF₆ inventory to a purity of about 99% in a period of 2 to 3 weeks. It should also be capable of routinely purifying a small recycle stream containing approximately 1% air. These two cases are denoted as Case 1, the worst case, and Case 2, the routine purification of slightly contaminated SF₆.

3. PROPERTIES OF SULFUR HEXAFLUORIDE

Many important thermodynamic and transport properties of SF₆ were presented by Milner⁵ and by a vendor booklet from Allied Chemical Co. Only those properties required for understanding the process descriptions that follow are reported here.

The vapor pressure is of paramount importance in considering vaporization and condensation processes (Table 1). Notice that at the melting point, -59.4°F (-50.7°C), the vapor pressure is above atmospheric. The sublimation temperature is -83°F (-63.8°C).

Table 1. Vapor pressure and density of SF₆

<u>Temperature</u> °F	<u>Temperature</u> °C	Vapor pressure (psig)	Liquid density (lb/ft ³)	Vapor density (lb/ft ³)
-83	-63.8	0		
-59.4 (mp)	-50.7	17.8		
-58.00	-50.0	19.1867	115.384	1.251110
-48.00	-44.4	27.5522	113.665	1.541793
-38.00	-38.8	38.4835	111.890	1.885678
-28.00	-33.3	49.1701	110.052	2.290409
-18.00	-27.7	62.8074	108.145	2.764523
-8.00	-22.2	78.5951	106.160	3.318272
0.0	-17.8	92.9093	104.510	3.825729
10.0	-12.2	113.0773	102.359	4.552402
20.0	-6.7	135.9532	100.097	5.395882
30.0	-1.1	161.7704	97.703	6.376521
40.0	4.4	190.7045	95.153	7.519135
50.0	10.0	222.9780	92.411	8.856692
60.0	15.5	258.8184	89.428	10.439324
70.0	21.1	298.4854	86.126	12.336124
80.0	26.7	342.2993	82.376	14.668706
90.0	32.2	390.7000	77.938	17.656662
100.0	37.8	444.3533	72.237	21.823471
110.0	43.3	504.4993	62.905	29.147339
114.15	45.6	531.8936	45.260	36.398193

Although the solubilities of oxygen and nitrogen in liquid SF₆ are important, no published data on solubilities were found; therefore, solubilities were estimated using the regular solution theory described in ref. 6. The relationship takes the form

$$-\ln X_i = \ln \frac{\lambda_1}{y_i P} + \lambda_2, \quad (1)$$

where

X_i = equilibrium solubility of gas i, m.f.;

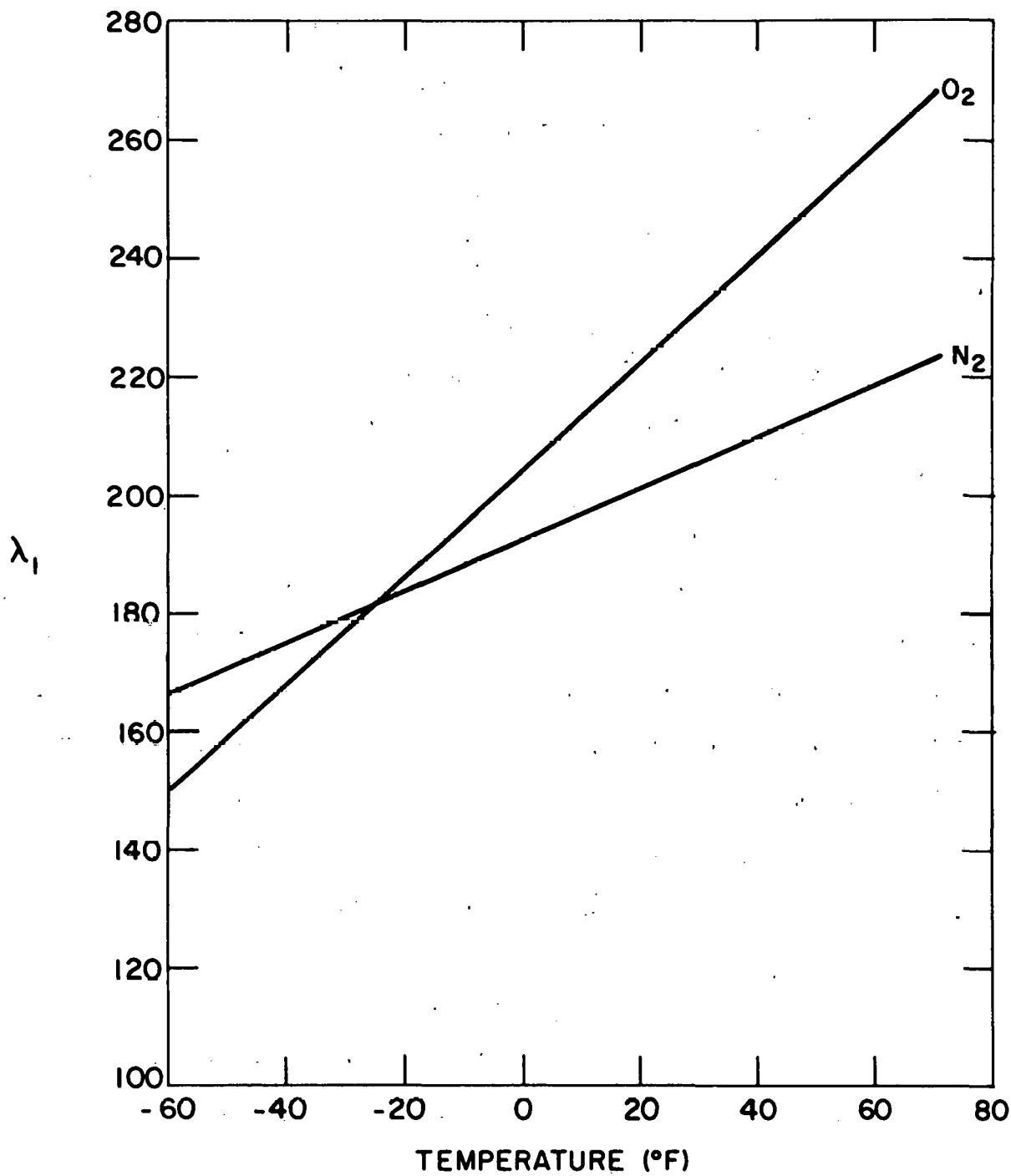
y_iP = partial pressure of gas i, atm; and

λ₁, λ₂ = constants that depend on gas and liquid properties and temperature.

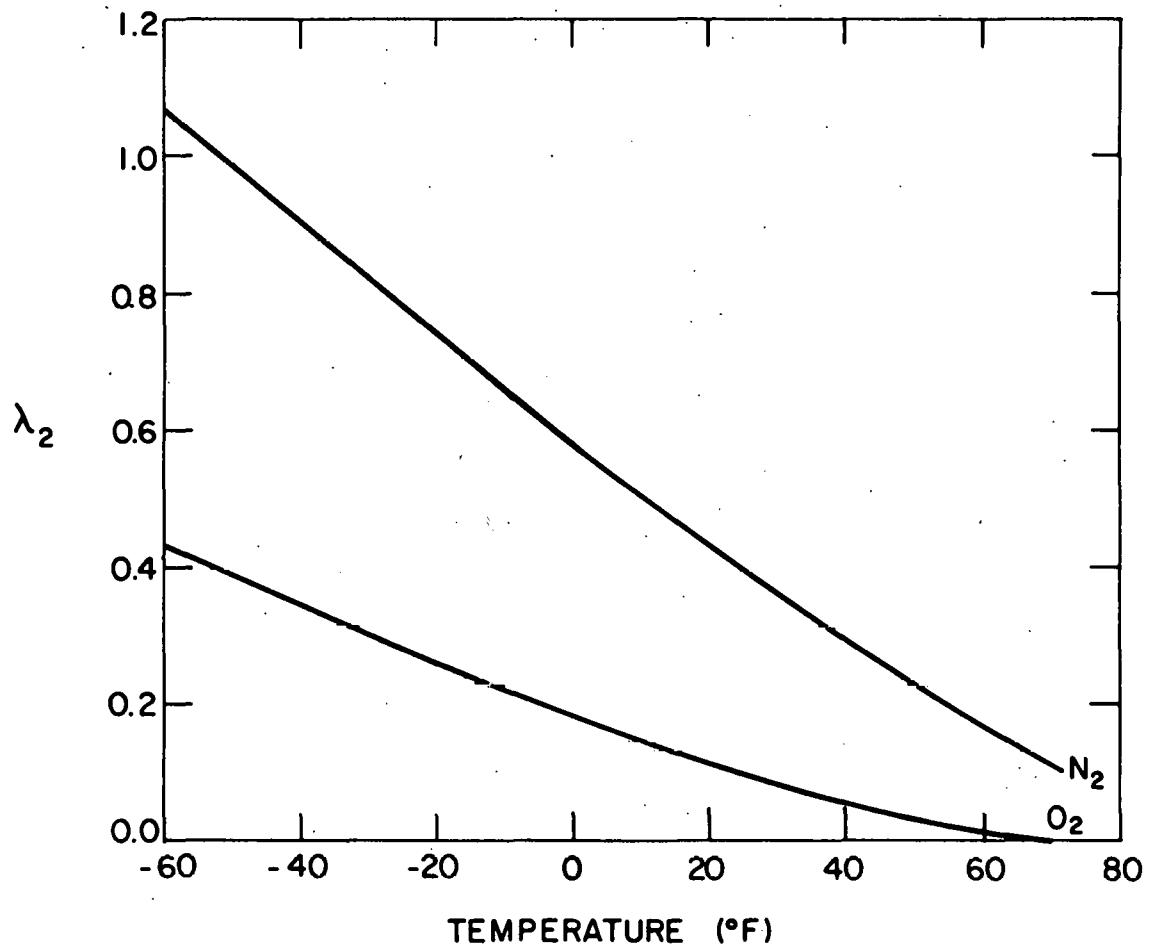
Calculated values for λ₁ and λ₂ for oxygen and nitrogen in liquid SF₆ are plotted in Figs. 1 and 2. If temperature and total pressure are chosen, the gas-phase composition is fixed by the vapor pressure of SF₆ and the ratio of oxygen and nitrogen in air. By choosing a range of pressures for several temperatures of interest, the solubilities shown in Fig. 3 were calculated. In these calculations, the oxygen and nitrogen in the gas phase were assumed to be present in the normal proportions found in air.

As the total pressure decreases to approach the vapor pressure of SF₆, the partial pressure of air and the solubility must approach zero. Hence the solubility curve for -50°F approaches an asymptote of 40 psia as the solubility becomes very small.

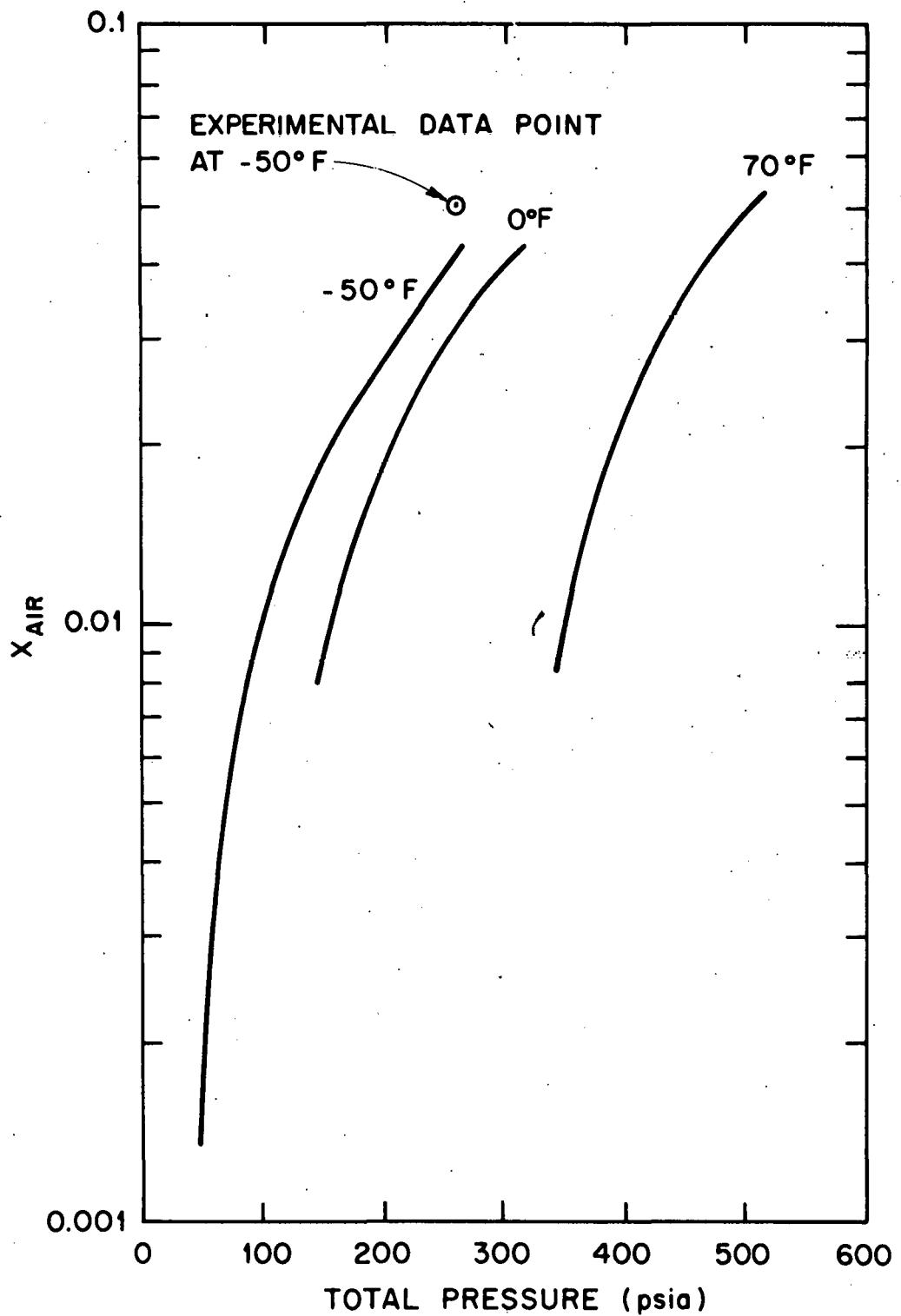
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Fig. 1. Values of λ_1 for O_2 and N_2 .

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Fig. 2. Values of λ_2 for O_2 and N_2 .

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Fig. 3. Mole fraction solubility of air in SF_6 .

4. AIR SOLUBILITY MEASUREMENT

The solubility of air in liquid SF₆ was measured to check the calculated solubilities. The apparatus used is shown in Fig. 4. The system between valves B and E and the sample cylinder were evacuated using a cryogenic molecular sieve pump (Varian VacSorb) to a pressure of less than 50 μ . The volume between valves A and B, containing air at ambient pressure (746 mm Hg), was connected to a cylinder of SF₆ at ambient temperature and cooled in a dry ice--chloroform bath. In this way, the A-B volume was pressurized to the vapor pressure of SF₆ (257 psig) and filled with a mixture of liquid SF₆ and air. Valve B was then opened slowly and the SF₆ liquid was allowed to flow into the volume tubing between valves B and C, thus yielding a liquid at -50°F which had been equilibrated with air at a partial pressure of 0.974 atm.

The B-C tube was allowed to warm to ambient temperature to vaporize the liquid, and the gas was then transferred to the sample cylinder.

Analysis by mass spectrometer yielded the following results:

	<u>mass %</u>
H ₂ O	0.05
N ₂ + CO	0.86
O ₂	0.17
Ar	0.01
SF ₆	<u>98.92</u>
	100.01

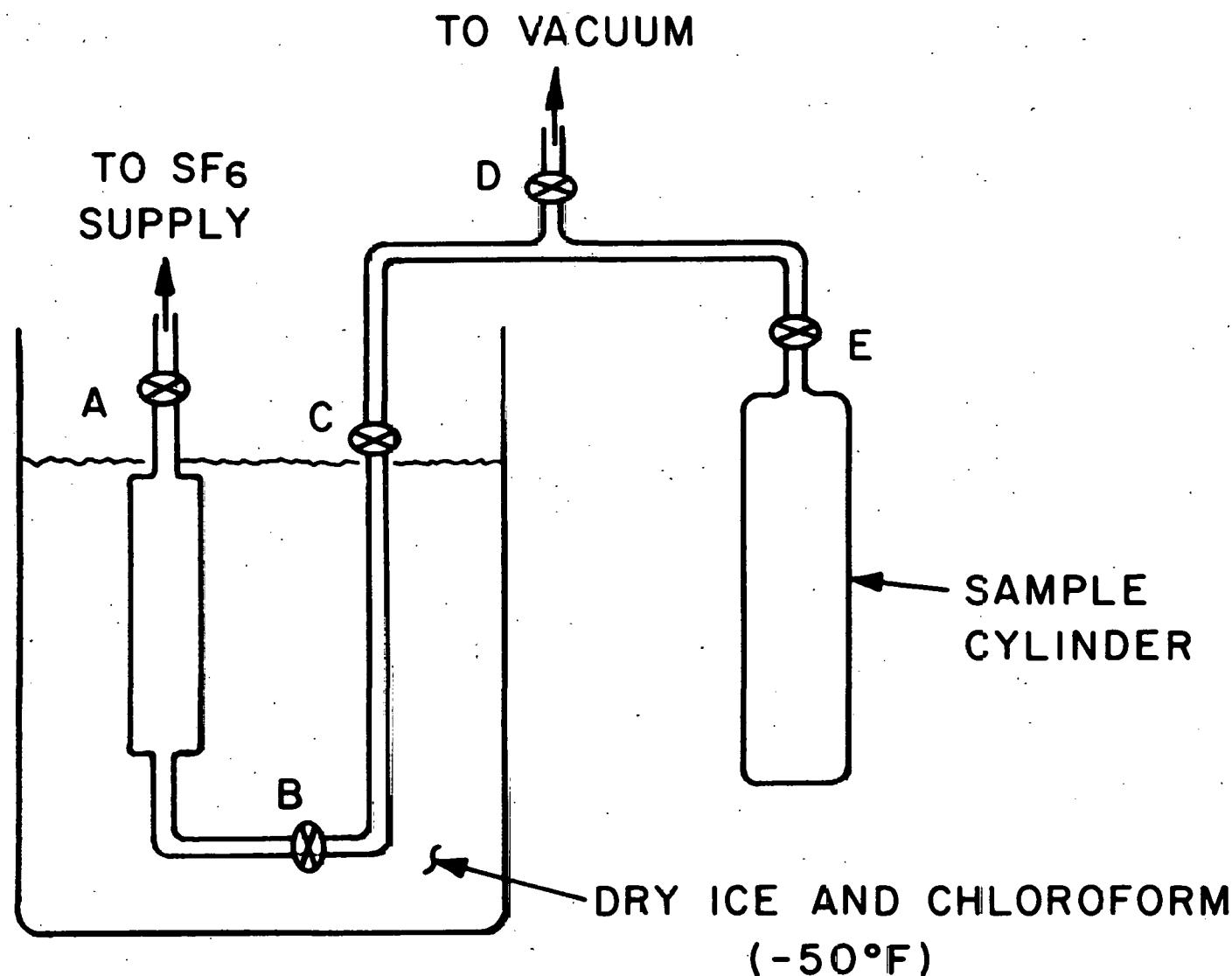


Fig. 4. Air-SF₆ solubility apparatus.

The solubilities are 0.043 m.f. for N_2 (assuming no carbon monoxide is present) and 0.007 m.f. for O_2 , giving a total solubility of 0.050 for air. From regular solution theory, the calculated values are 0.023 for N_2 and 0.018 for O_2 , giving a total air solubility of 0.041. These values are shown in Fig. 3.

5. PURIFICATION METHODS

5.1 Liquefaction

One of the simplest operations that can be performed with the contaminated gas is to cool it and produce a liquid phase. Two questions naturally arise: (1) how much of the SF_6 can be recovered by liquefaction, and (2) how pure will the liquid be? The distribution of gases and liquids obtained from an initial gas mixture can be estimated by the application of Dalton's and Raoult's laws:

$$y_{SF_6} P_T = X_{SF_6} P_v, \quad (2)$$

where

y_{SF_6} = mole fraction of SF_6 in vapor,

P_T = total pressure,

X_{SF_6} = mole fraction of SF_6 in liquid, and

P_v = vapor pressure of pure SF_6 .

The conditions that result in air solubilities greater than 1 or 2% are not of interest; therefore, we can take $X_{SF_6} \approx 1$ with the understanding that results are not accurate where this assumption does not hold. To a

good approximation, the composition of the vapor phase is thus fixed when the total pressure and temperature are specified.

Essentially all of the 207 moles of air remain in the vapor phase; the amount of SF_6 liquefied at a given total pressure and temperature can be calculated by obtaining the composition of the vapor phase from Eq. (2). The amount of SF_6 that is condensed is the difference between the original 1200 moles and the amount in the vapor. Figure 5 is based on this procedure and shows that over 95% of the SF_6 in Case 1 can be liquefied at moderate pressures if the mixture is cooled to 0°F or below. The solubility estimates in Fig. 3, however, indicate that the amount which can be liquefied is less than 90% at temperatures to -50°F if the liquid phase composition is to be limited to 1% air. A purification flowsheet for Case 1 utilizing liquefaction is given in Fig. 6, which is based on a 20-day cleanup period for this worst-case incident. After liquefaction, a vapor phase remains which is 40% SF_6 and amounts to 1050 lb/day of SF_6 . This stream requires further purification by some operation labeled "separator" in Fig. 6.

For Case 2, starting with a vapor containing 1% air, liquefaction is more efficient for the purification of SF_6 . At -50°F and a total pressure of 50 psia, over 96% of the SF_6 is liquefied and, as shown in Fig. 3, the composition of the liquid phase is only 0.0018-m.f. air.

5.2 Molecular Sieves

Molecular sieves with very uniform pore sizes are available commercially with pores too small for the SF_6 molecule to enter. A separation process can be devised in which the mixture of air and SF_6 passes through

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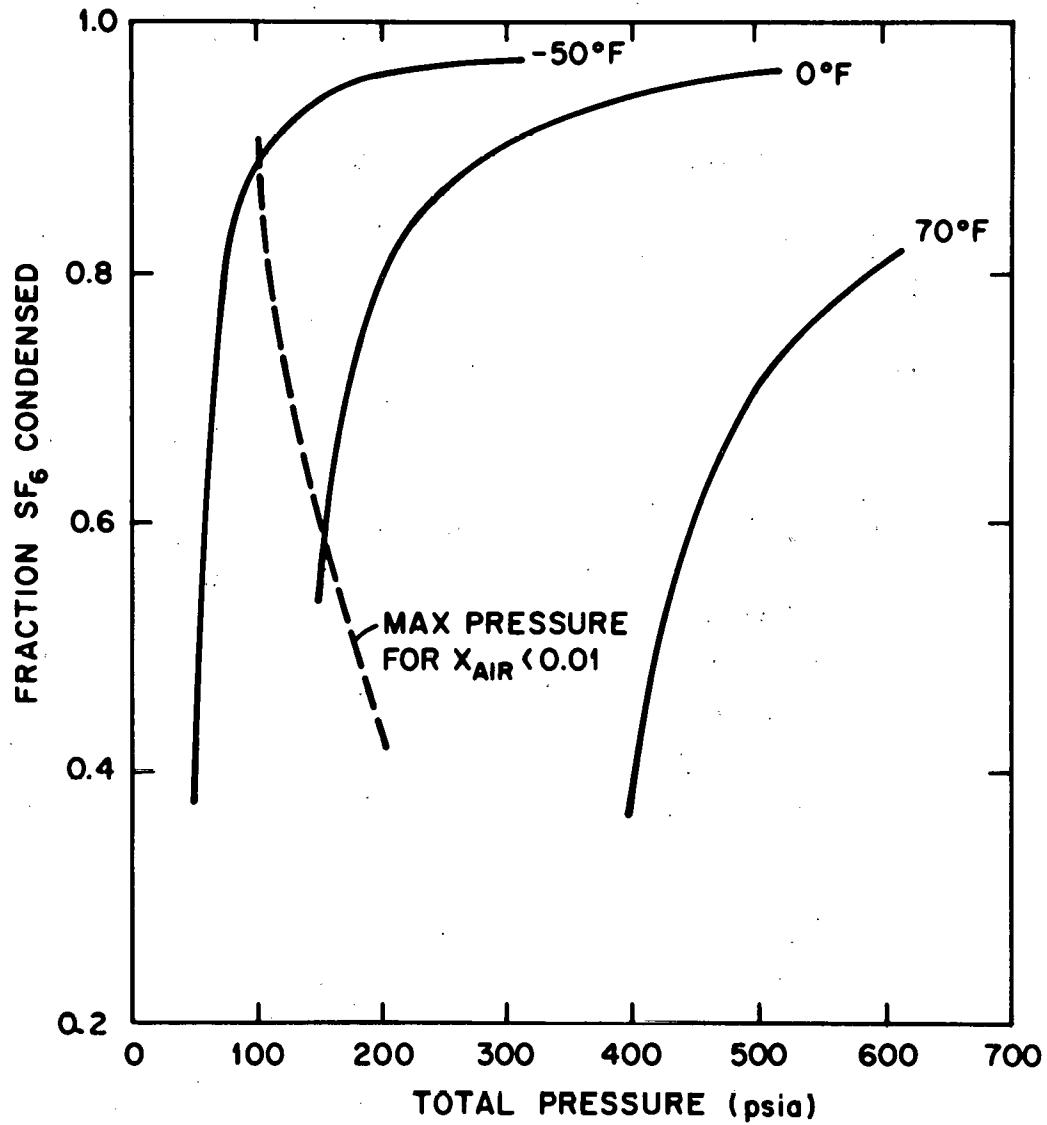
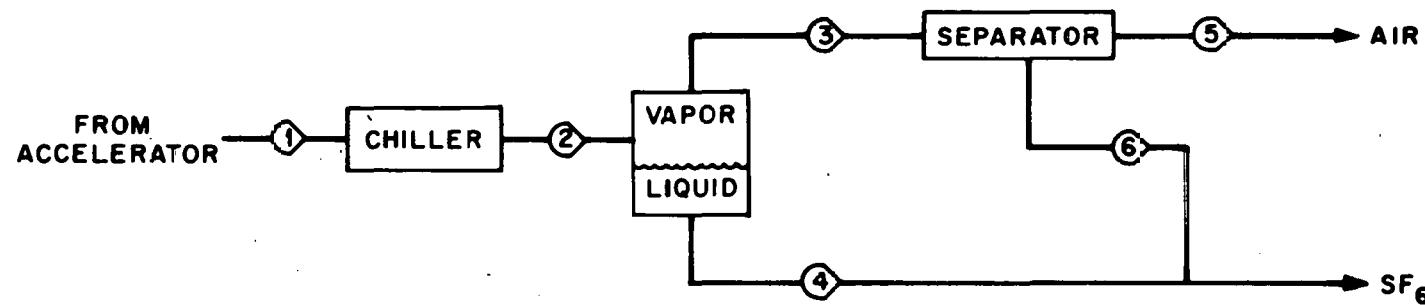


Fig. 5. Fraction of SF₆ condensed starting with Y_{air} = 0.15.



	1	2	3	4	5	6
TEMPERATURE (°F)	70	-50	-50	-50	70	
PRESSURE (psig)	85	85	85	85	85	0
FRACTION LIQUID	0	0.88	0	1.0	0	0
AIR IN VAPOR (m.f.)	0.15	0.60	0.60		0.96	0
AIR IN LIQUID (m.f.)		0.01		0.01		
FLOW RATE* (lb/day)						
SF ₆	8750	8750	1050	7700	50	1000
AIR	300	300	285	15	285	0

* 20-DAY CLEANUP

Fig. 6. Flowsheet for Case 1.

a bed of molecular sieve, and the air is trapped out while the SF₆ passes through. If the "separator" in Fig. 6 is assumed to be a bed of molecular sieve, it can be seen that as the gas stream passes through the bed and loses air, it becomes saturated and liquefies. The liquid might cause problems by blinding the pore openings, preventing the gaseous air from entering. After the bed is saturated with air, it would be taken off stream, depressurized, and warmed to release the air in preparation for another cycle.

The required size of a bed of molecular sieves for this application was discussed with engineers at Linde (Division of Union Carbide, Cleveland, Ohio). The bed cannot be operated below approximately -50°F because the SF₆ would freeze and plug the bed. Equilibrium loadings for specially dehydrated (activated) molecular sieves at -50°F are 2.5 lb of oxygen and 6 lb of nitrogen per 100 lb of molecular sieves. Practical design values recommended by Linde are 1 lb of oxygen and 4 lb of nitrogen per 100 lb of molecular sieves. For the condition presented in Fig. 6, a minimum bed size of 15,000 lb is required. Molecular sieves, which are ceramic oxides, are very poor conductors of heat, and temperature cycling of such a large bed would require prohibitively long periods of time. Therefore, the use of molecular sieves does not appear to be a good solution to the problem.

5.3 Desublimation

Freezing out SF₆ from a mixture with air has been practiced on small batches of gas such as at Chalk River. The feasibility of desublimation on a large, continuous scale was investigated for the present application. Desubliming is not a widely practiced operation, and the engineering of

desublimers is not well established in comparison with most other unit operations. However, a great deal of the existing expertise at ORNL is the result of routine desublimation of UF_6 . A design procedure that includes a computer program⁷ was applied to the desublimation of krypton.⁸ Hence the design procedure that is available has been tested on two different chemical systems and has been verified experimentally on both of them.

The desublimer would be the separator in Fig. 6. To recover 90% of the SF_6 entering the desublimer (99% of the SF_6 leaving the accelerator), the partial pressure must be reduced to 4 psia, which requires a temperature of -115°F. Allowing for heat transfer resistance, a cooling fluid temperature of -130°F or below is needed.

The desublimer can have many geometrical configurations, but one of the simplest and most effective is the finned tube (Fig. 7). As the superheated gas flows through the tube, the gas near the cold surface is cooled to the point where the partial pressure of SF_6 exceeds the vapor pressure and solids are deposited on the cold surfaces. The loss of SF_6 from the gas near the surfaces gives rise to a transverse mass transfer driving force, and the rate of mass transfer becomes one of the most important rate phenomena occurring in the desublimer. At the same time, the bulk gas is cooled as it flows along the tube and may generate SF_6 "snow" when it becomes subcooled. Both phenomena must be considered in the design. The greatest unknowns in the calculations lie in the values of density and thermal conductivity of the deposited solids.

The design program⁷ assumes that the snow is deposited at the same axial position at which it is formed. High gas velocities cause the

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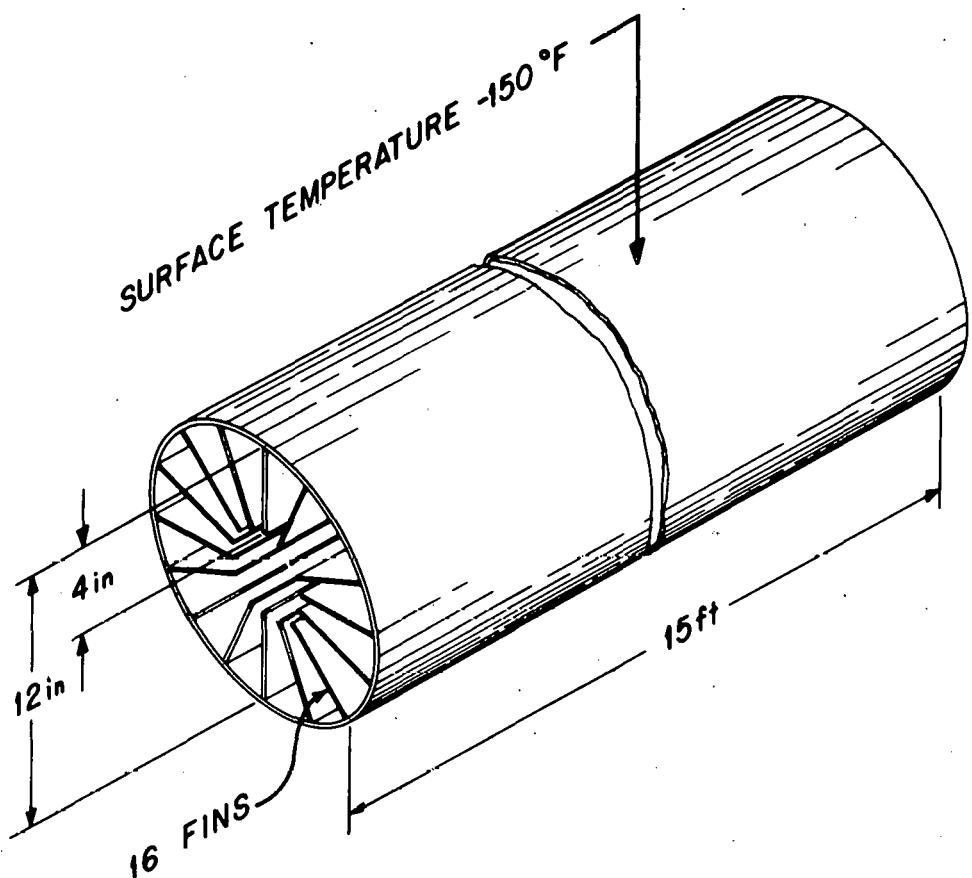


Fig. 7. Finned-tube desublimer.

snow to be carried further along the tube. Desublimers of UF_6 are fitted with filters at the gas outlets to prevent loss of snow. A rule of thumb derived from UF_6 experience is that the accumulation of snow on the filter is small if the superficial gas velocity is kept below 0.5 ft/sec. In one run made with a gas velocity that was about twice this value, there was no significant snow accumulation at the gas outlet.⁸

Guidelines for estimating the density of the deposited solids are very rough. A frost-to-liquid density ratio of 0.69 has been reported.⁷ This does not agree with the generally accepted meteorological ratio of 10 in. of snow per 1 in. of rain. A ratio of 0.5 was assumed for krypton, but it was not directly confirmed by experiments.⁸ If we assume a ratio of 0.5 for SF_6 , we obtain a solids density of 57 lb/ft³.

The volume of a 12-in.-diam by 15-ft-long desublimer is about 12 ft³. If the solids density is assumed to be 57 lb/ft³, such a desublimer would hold at maximum 670 lb of SF_6 solid. Since a desublimer cannot be filled completely, let us assume that a desublimer of this size could be loaded with 525 lb of SF_6 . If this is compared with stream 3 in Fig. 6, we find that two cycles of loading per day would handle the flow.

The operation of a 12-in.-diam desublimer that is 15 ft long and has 16 radial fins was investigated for a coolant fluid temperature of -150°F . The heat transfer coefficient (h) between the gas and the wall was calculated with the following equation:⁹

$$\frac{h}{LG} = 0.67 \left(\frac{LG}{\mu} \right)^{-1/2} \text{Pr}^{-2/3}, \quad (3)$$

where L is the axial length of the fin. High coefficients can be obtained with small values of L , which keep the boundary layer from becoming well developed. Even though our exchanger was 15 ft long, L could be made small by specifying that the fins were cut and rotated every 6 in. or foot of length.

The results of the computer study show that the desublimer would reach a loading of 525 lb in about 2.5 hr. The gas velocity would remain well below the 0.5-ft/sec limit. The effluent gas composition (m.f. of SF_6) during the loading period ranged from 0.007 after 15 min to 0.048 after 2.5 hr and resulted in an SF_6 recovery for the desublimer in excess of 90%. Solids deposition profiles during the 2.5-hr period are plotted in Fig. 8. Our calculations indicate that this is a workable design.

Because the thermal conductivity and density of the deposited solids are only estimates, the sensitivity of the design to these variables was investigated. A thermal conductivity of 0.1 Btu/hr.ft. $^{\circ}$ F was used in the previous calculations, which is the same value that seems to work for UF_6 desublimers. Computer runs were made in which the thermal conductivity was decreased by a factor of 10 to 0.01 Btu/hr.ft. $^{\circ}$ F and in which the density of the deposited solids was decreased by a factor of 2 to 30 lb/ ft^3 . The effects of these changes were similar. The design as it stands would not work because the effluent gas concentration of SF_6 would become excessive after about 1.5 hr. The hardware could be made to work by using three cycles per day, collecting 350 lb/cycle in about 1.5 hr. If the low values of both density and thermal conductivity are applied at the same time, a larger desublimer would be required.

Based on the design calculations, the use of a desublimer appears feasible for the purification of SF_6 . However, the uncertainties of

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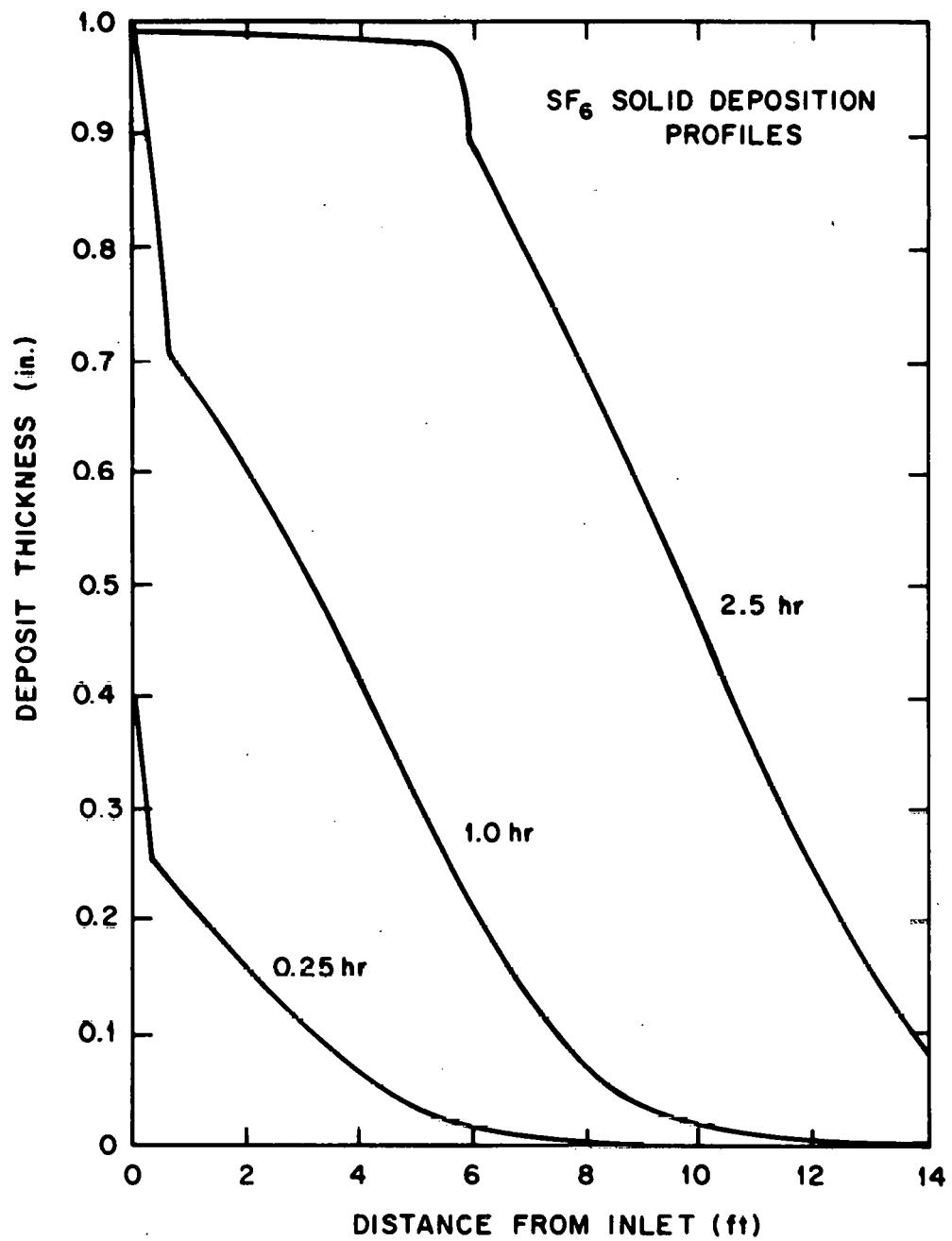


Fig. 8. Solid deposition profiles of SF₆.

solids properties would require that a small-scale desublimer be built and tested against design calculations before design of a full-scale desublimer could proceed.

5.4 Adsorption onto Activated Carbon

The affinity of SF₆ for activated carbon has already been noted. A conventional process arrangement would be to pass the SF₆-air mixture through the bed and allow the SF₆ to be adsorbed and the air to flow through. When the bed is saturated and SF₆ begins to break through, the bed would be taken off stream and warmed to release the SF₆. No data on equilibrium loadings of SF₆ onto activated carbon were available in a literature search.

A simple apparatus was assembled to measure the adsorption capacity of activated carbon for SF₆ (Fig. 9). Dry air and SF₆ gases were metered into a cooling coil and passed into a column packed with activated charcoal. The 2-in.-diam by 2-ft-long column contained 430 g of activated charcoal. The gases and column were cooled and maintained at -50 to -100°F before the start of a run. The effluent gases from the column passed through a gas density analyzer (manufactured by GOW-MAC), which provided a continuous analysis of the gas composition.

Ten runs were made with gas phase compositions that ranged from 0.068-to 0.58-m.f. SF₆ (Table 2). Breakthrough curves were very sharp for all cases; essentially no SF₆ was in the effluent gas for the first 30 min, after which the effluent composition suddenly rose from 0 to 100% SF₆ within 1 to 2 min. A sample curve is shown in Fig. 10. This behavior is very desirable in an adsorption system and would permit high utilization of the bed. Bed loadings are also encouragingly high,

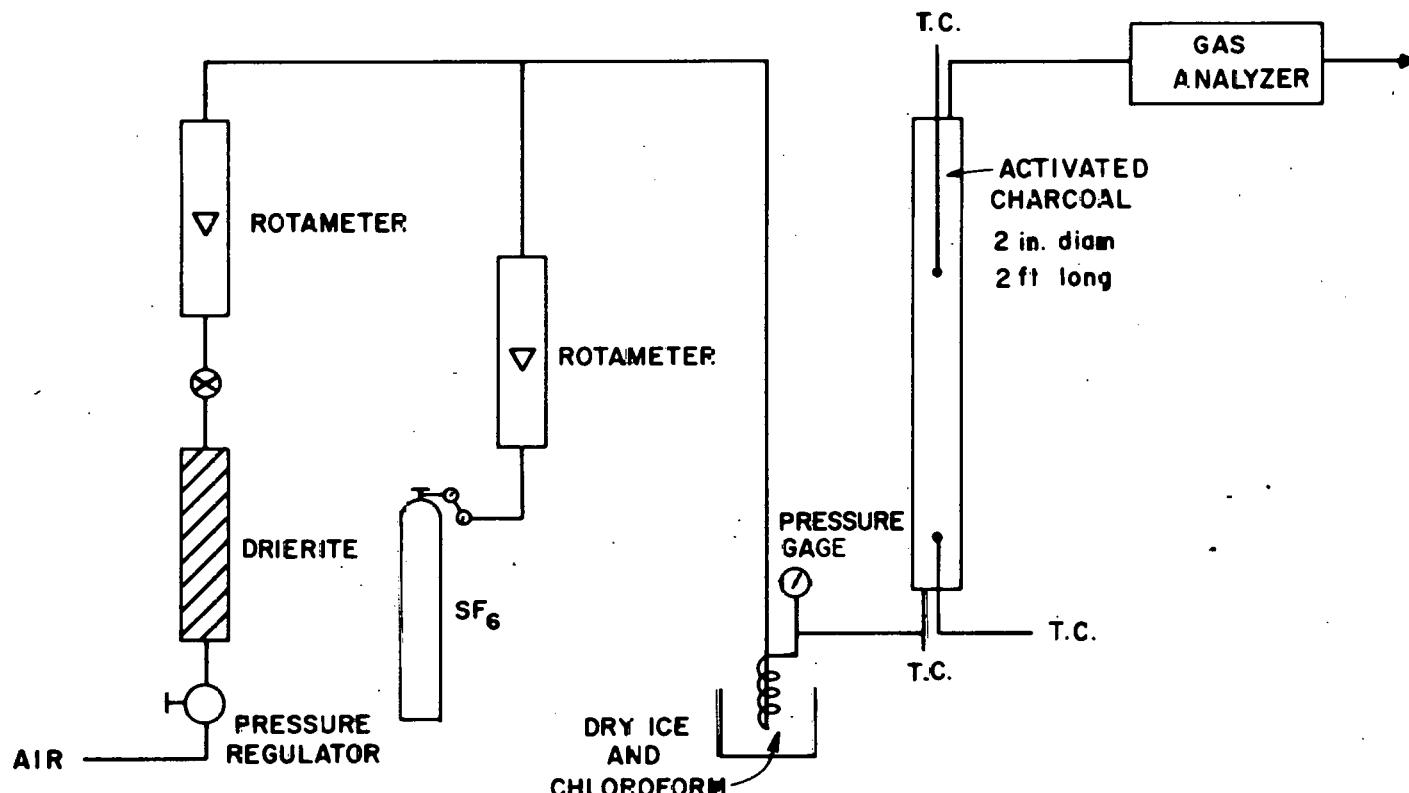
Fig. 9. Apparatus for activated carbon adsorption of SF_6 .

Table 2. Adsorption of SF₆ onto activated carbon

Run	Feed composition (m.f., SF ₆)	Inlet pressure (psia)	Inlet temperature (°F)	Time to breakthrough (min)	Partial pressure SF ₆	Loading (g SF ₆ /g C)
2	0.251	28.4	-63	31.5	7.14	1.48
3	0.336	21.7	-63	34.8	7.29	1.43
4	0.423	20.7	-62	30.0	8.77	1.20
5	0.780	17.7	-62	36.5	10.27	1.32
6	0.370	28.7	-51	25.8	10.80	1.03
7	0.205	24.2	-76	29.4	4.97	1.09
8	0.165	20.7	-65	50.3	3.41	1.32
9	0.118	22.7	-73	57.1	2.69	1.07
10	0.068	27.2	-55	113.3	1.86	1.12

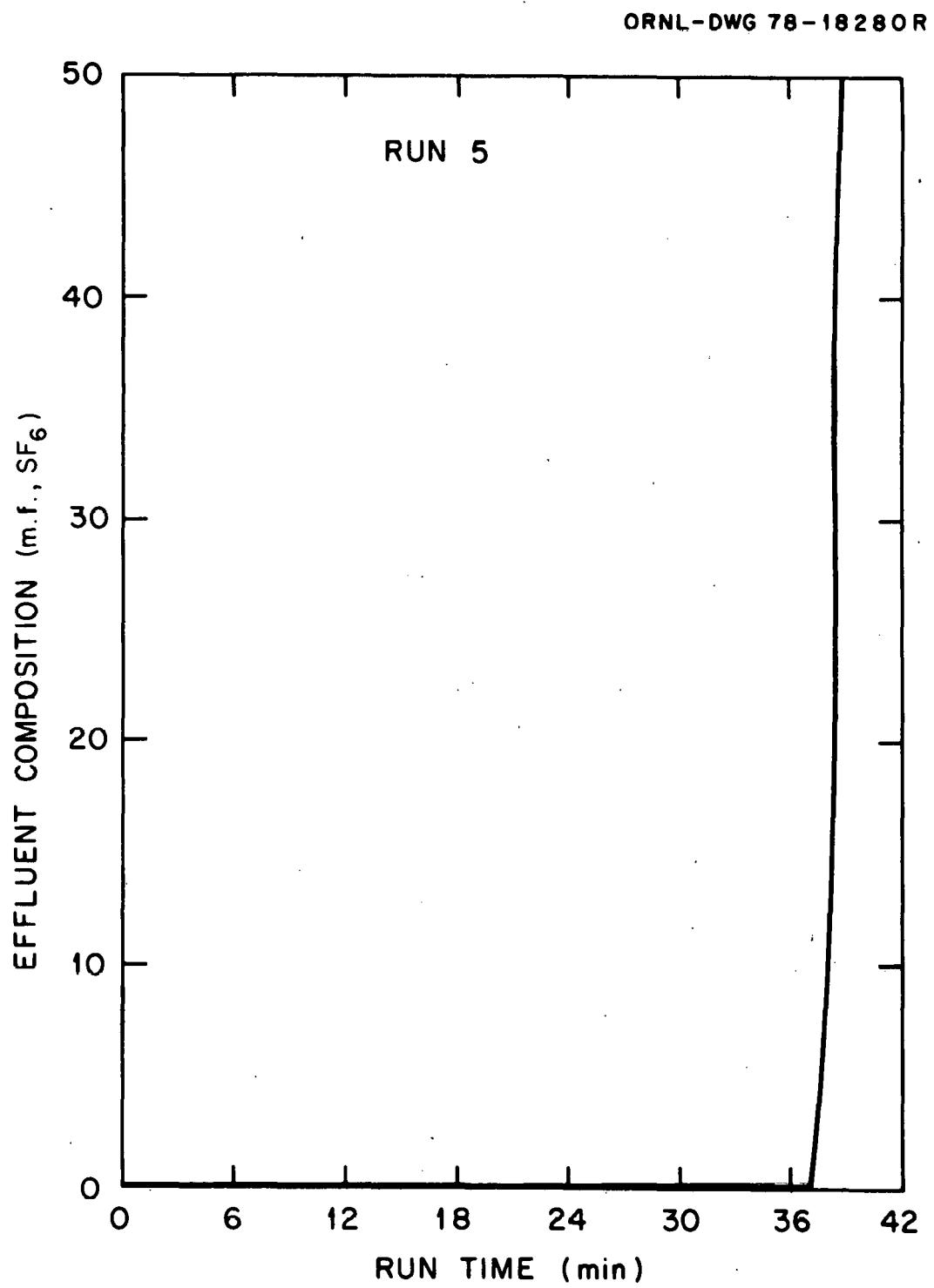


Fig. 10. Breakthrough curve for Run 5.

ranging from 1.0 to 1.48 g of SF₆ absorbed per g of carbon for all runs.

A large heat-of-adsorption effect was noted. As shown in Fig. 11, the temperature near the center of the bed suddenly rose above 0°F as the mass transfer zone reached that part of the bed, then slowly fell to almost that of the inlet temperature at breakthrough. Thus the loadings cannot be taken as isothermal data.

The loading data in Table 2 do not correlate with partial pressures of SF₆, as might be expected. The loadings are so high that most of the void volume of the charcoal bed is filled with SF₆ in these runs, and thus the expected correlation was not obtained.

Favorable results have been obtained in preliminary feasibility calculations for a purification system that uses charcoal adsorption for the separation (see Fig. 6). The separator could consist of two charcoal beds, one of which is on stream and loading for 12 hr, while SF₆ is recovered from the other by a temperature cycle. Each bed requires a capacity of 525 lb of SF₆, which means the bed must contain 525 lb of activated charcoal for a loading ratio of 1.0. Using a charcoal density of 35 lb/ft³, each bed would have a volume of only 15 ft³. These volumes are very reasonable.

A refrigeration system to cool 8750 lb of SF₆ per day and 300 lb of air per day to -50°F is also reasonable. The enthalpy of SF₆ gas at 70°F is 53 Btu/lb; SF₆ gas at -50°F is 43 Btu/lb, and SF₆ liquid at -50°F is -2 Btu/lb (based on saturated liquid at -40°F). The total refrigeration requirements are less than 20,000 Btu/hr for cooling and liquefying gases. Cooling requirements for temperature cycling

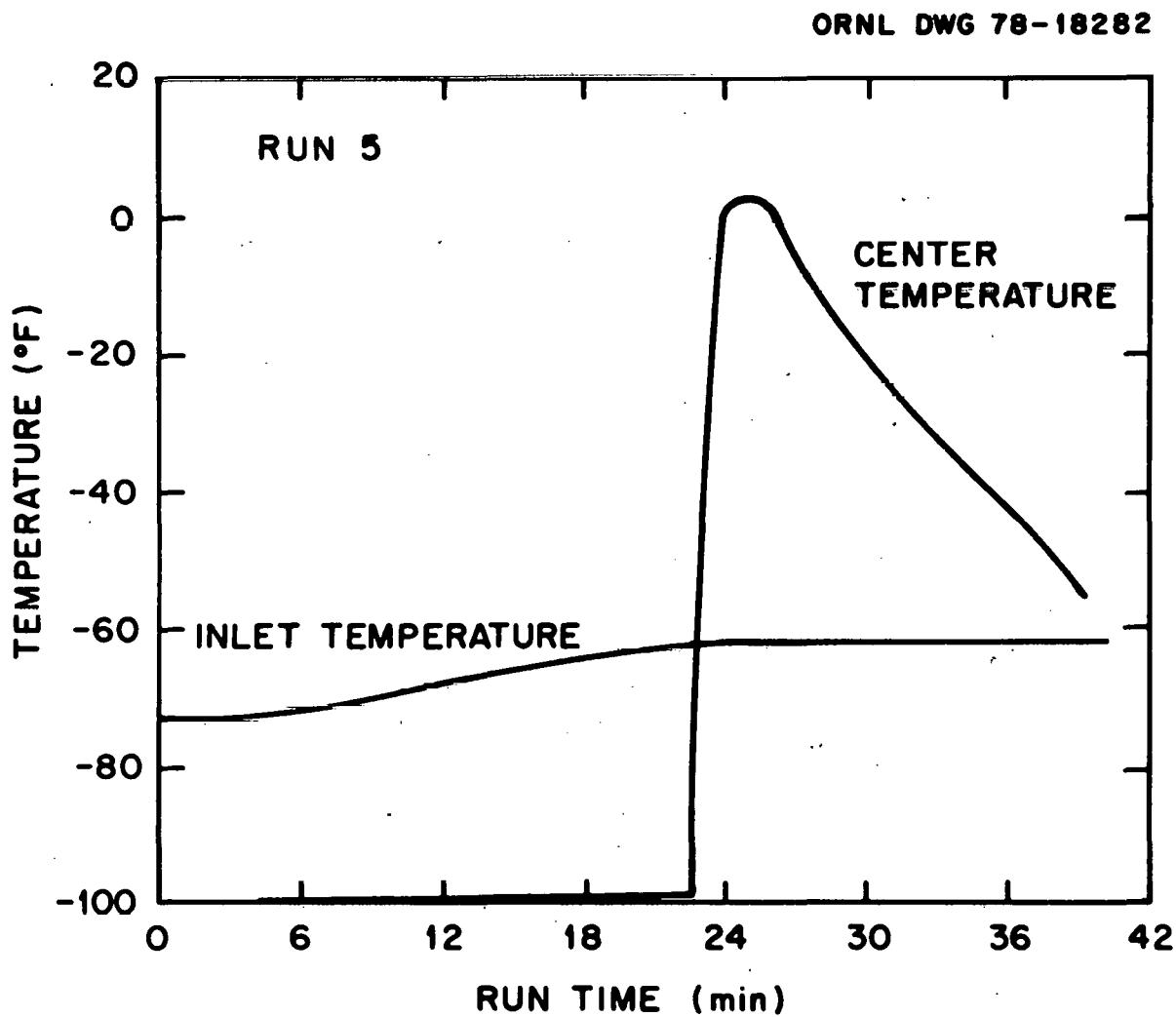


Fig. 11. Charcoal-bed temperatures for Run 5; breakthrough at 37 min.

the carbon bed would be only about 20% of this. Thus, 2 tons of refrigeration should be adequate. A rough estimate of the cost of such a system indicates a cost of about \$15,000.

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