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Sponsored by C. E. Newlon

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ABSTRACT

The knowledge of the hydrogen concentration of hydrogen fluoride (HF)/uranium hexafluoride (UF_6) gas mixtures when cooled below condensation or desublimation temperatures is necessary in determining fissile moderation for criticality safety considerations in UF_6 desublimation processes. The HF/UF_6 gas mixtures used in this study were initially at 126°F (52°C) and a maximum of 607 torr before cooling to 60°F, 46°F, 32°F, 15°F and 0°F. Starting HF/UF_6 gas mixture volume ratios were 10/90, 50/50, and 90/10. Visual evidence of liquid HF occurred only in the cooling of 90/10 mixtures at or below 32°F. Maximum relative enhancement (factor of 3) of HF in the cooled mixture occurred at 15°F from a 10/90 initial HF/UF_6 gas mixture.

CONTENTS

SUMMARY

An empirical study of HF-UF_6 gas mixtures cooled to various temperatures was performed to better understand nonequilibrium desublimation processes relative to criticality considerations. The gas mixtures were prepared using a gas pipette maintained at a constant temperature. The gas mixture was admitted to a cooled Fluorothene tube where the mixture was cooled until the pressure of the mixture in the gas pipette stabilized. The material in the Fluorothene tube was then isolated and further cooled to -310°F using liquified air. The tube was then removed from the system, and the content of the tube was analyzed for weight percent UF_6. The remainder of the net weight is assumed to be HF. The resulting data are summarized by HF-UF_6 phase distributions and hydrogen:uranium ratios. Visual evidence of liquid HF in the Fluorothene tube occurred at high initial HF concentrations and at cooling temperatures at 32°F or below. Maximum HF enhancement in the cooled Fluorothene tube occurred at 15°F from low initial HF concentrations.

INTRODUCTION

Data relevant to the estimation of hydrogen:uranium ratios for fissile moderation control of cooled gas mixtures in the range of 0° to 60°F is necessary for better understanding potential criticality problems associated with the operation of a gaseous diffusion enrichment facility. Nonequilibrium data on this mixture was not available in the current literature, although several studies of the phase equilibria for HF-UF_6 systems at various temperatures and pressures have been performed, principally by researchers at the Oak Ridge Gaseous Diffusion Plant in the early phase of the plant's history. This study complements existing phase equilibria data, and was performed to simulate potential nonequilibrium situations in the plant; eg, gas mixtures condensed from piping into cylinders or into deadend systems.

The data presented herein provide additional information from which those dedicated to criticality control may estimate the fissile moderation caused by the hydrogen in HF in conjunction with the uranium in UF_6. However, it is noted that the data obtained in this study may be specific to the equipment and procedures employed. Existing equipment and analytical methods were used to gather the data for this study. Estimates of precision of existing data were obtained by repetition of some data sets.

EXPERIMENTAL

EQUIPMENT

Gas Pipette

A schematic diagram of the automatic gas pipette is shown in Figure 1. The pipette is a 0.95-liter volume Monel container enclosed in a thermally insulated chamber. Temperature was maintained at 126 ± 4°F and was measured using an inert gas thermometer. The pressure within the pipette

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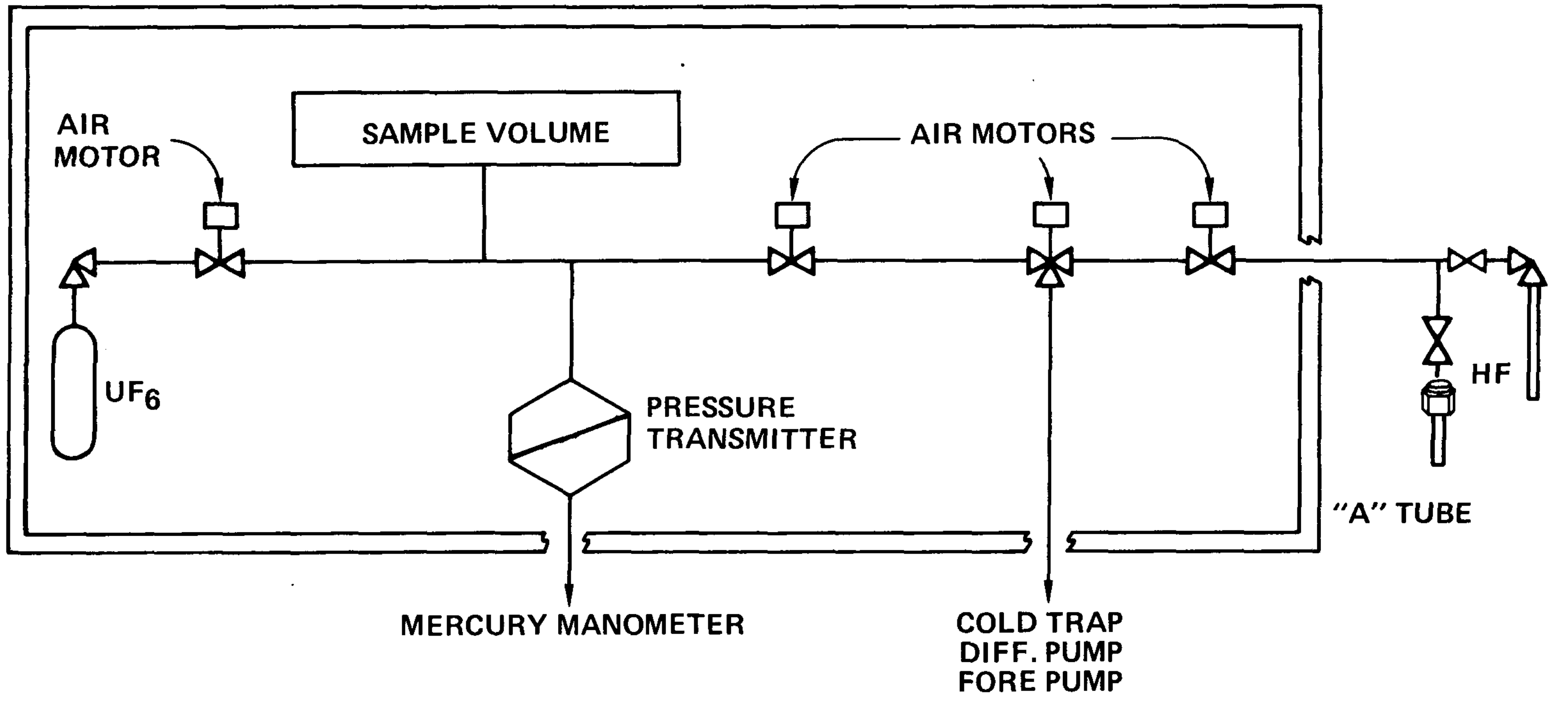

Figure 1
GAS PIPETTE

was measured using a 0 to 760-mm-Hg pressure transmitter in conjunction with an automatic digital readout mercury manometer. All valves within the insulated container are air-operated globe valves with Fluorothene spindle seats and a phosphor-bronze bellows. The vacuum system in conjunction with the gas pipette is comprised of a liquid air-cooled trap followed by a water-cooled oil diffusion pump and a fore pump.

Hydrogen Fluoride Container

The container used for dispensing and weighing the quantities of HF is shown in Figure 2. The 3/8-in. nickel tube is closed on one end by silver brazing and the other end by a small needle valve with a phosphor-bronze bellows. The weight of the tube and contents were below 200 g, permitting weighing to 0.1 mg on an analytical balance.

Collection Tube

A sketch of the collection tube is shown in Figure 3. The portion of the tube exposed to the collected sample and the closure gasket is fabricated of Fluorothene. The closure fittings are Monel.

MATERIALS

Hydrogen Fluoride

The HF was obtained commercially and was analyzed for impurities using infrared spectrophotometry. No active infrared compounds were observed in the 1,500 cm^{-1} to 600 cm^{-1} range. Other impurity levels were less than 0.1 wt % as analyzed by mass spectrometry. The HF was frozen into the HF container using liquid air, then warmed to the dry ice slush temperature, at which time residual air components were evacuated.

Uranium Hexafluoride

The UF_6 was distilled from a parent container and desublimed into a 150-cc, valved Monel container maintained at 32°F. The UF_6 in the 150-cc cylinder was 99.998 wt % UF_6 as determined by freezing point depression analyses.

PRECEDURE

Stabilizing Pipette and Sampling Lines

The gas pipette and sampling lines were heated to 126°F and evacuated to 20μ. The UF_6 was admitted to the pipette and the pressure noted. A change of pressure indicates reaction of UF_6 with system components or residual compounds in the system. This procedure was repeated four times with no pressure change noted after the second UF_6 admission.

Gas Mixture Preparation

The HF container was weighed to ±0.1 mg on an analytical balance. The weighed container was placed on the outlet side of the pipette. A collection tube was also placed on the outlet side, but isolated from

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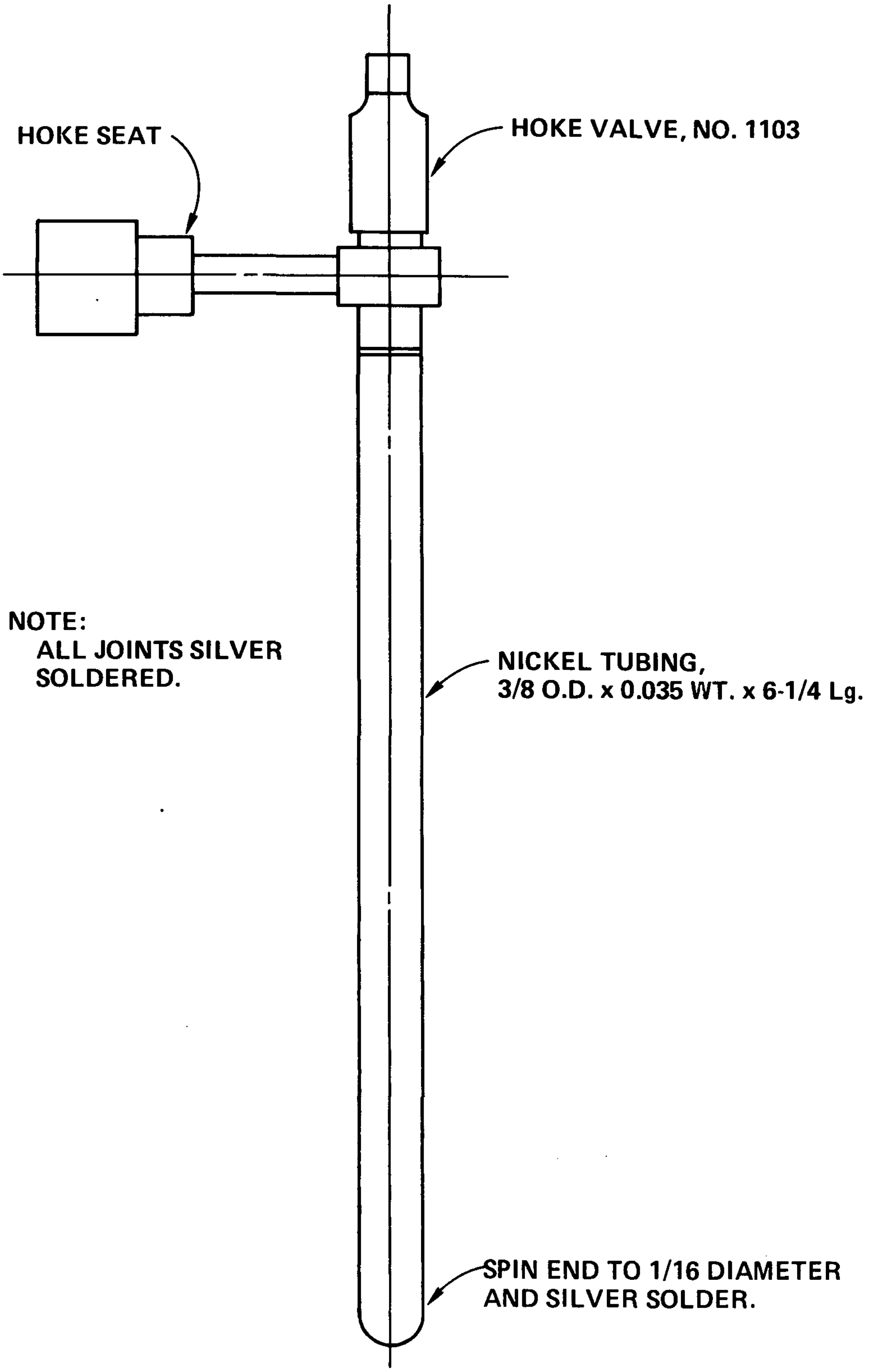

Figure 2

HYDROGEN FLUORIDE CONTAINER

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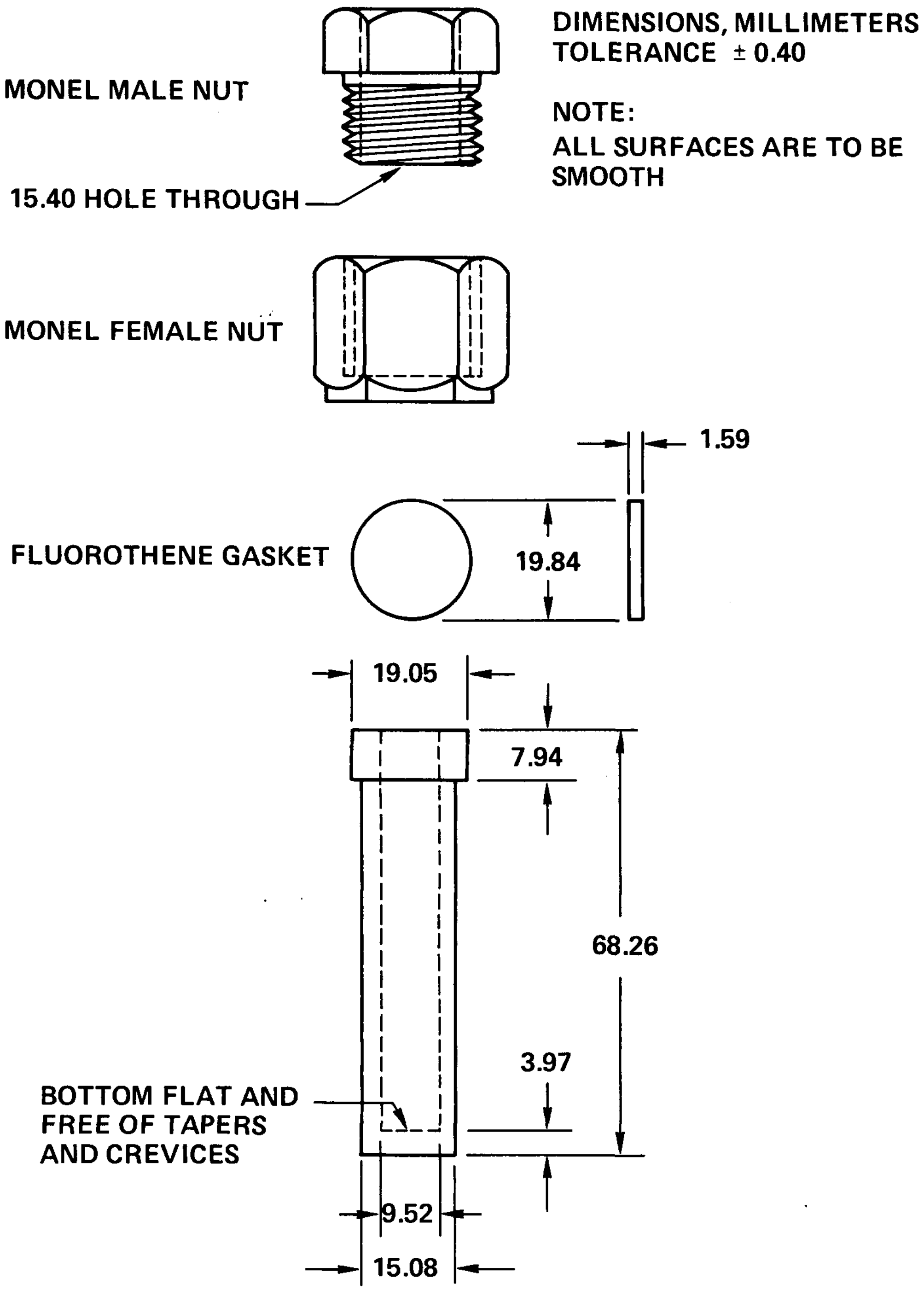

Figure 3

FLUOROTHENE SAMPLE TUBE

the HF container (Figure 1). The UF_6 container was placed on the inlet side of the pipette, where it remained throughout the study. The inlet and outlet lines, collection tube, and pipette were evacuated to 20µ with the enclosed portion of the system heated to 126°F. With the UF_6 container isolated from the pipette, the collection tube isolated from the outlet line, and the outlet line isolated from the evacuation system, the HF container was opened expanding HF into the pipette until the desired pressure, as measured on the manometer, was attained. The pipette was then isolated from the outlet line, and the HF container was cooled with liquid air to condense the residual HF into the HF container. The HF container was closed, removed from the outlet line, and weighed to ±0.1 mg on an analytical balance.

The UF_6 container was opened to the inlet line. The pipette was then opened to the UF_6, and UF_6 was admitted until the desired pressure was obtained. The pipette was then isolated to the UF_6 inlet line. The resulting HF-UF_6 mixture was allowed to remain in the pipette for at least one hour before opening the pipette to the collection tube.

The collection tube was cooled to the desired temperature with ice-cooled water (32°F) or with dry ice (CO_2) cooled dichloretetrafluoroethane (<32°F). The cooling temperatures were controlled to ±2°F (except for 32°F which was determined by ice water temperature at better than ±0.1°F). The mixture in the sample volume was opened to the cooled collection tube and permitted to cool in the collection tube until the decrease in pressure ceased as indicated on the manometer (usually attained in <30 min). The sample volume was then isolated from the outlet line, and the collection tube was cooled in liquid air for at least 6 min. The collection tube was then removed from the system and sealed closed with the Fluorothene gasket and the Monel fittings.

Analyses

The collection tube with the sample was warmed to ambient temperature and analyzed for UF_6 using the precise gravimetric method.* That which was not analyzed as UF_6 is HF. Results of the analyses was then converted to weights, mole percentage, and hydrogen:uranium ratios for tabulation and correlation with observed pressures, temperatures, and weights.

RESULTS

The observed results with forced residuals based on the concept of a binary system are presented in Table 1. The quantities with a single asterisk are observed valves (i.e., weight of HF in mix; analyses of UF_6 in cooled fraction; pressure HF, UF_6 and remaining mix; and temperature). The values for total grams cooled in the collection tube includes not only the desublimed UF_6 and condensed HF but also includes the HF-UF_6 vapors and atmospheric air admitted when sealing the collection tube.

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*W. D. Hedge (Compiler) *Laboratory Procedures for the Oak Ridge Gaseous Diffusion Plant,* K-2-6203 Procedure Number 2.767.04.01. (1968)

Table 1

OBSERVED HF/UF_6 DATA

Observed Properties	Mix			Cooled Fraction			Pipette Pressure, Torr* Mix		
	HF, g*	UF_6, g	Total, g	HF, g	UF_6, g*	Total, g**	HF/UF_6/Total	Remaining	t, °F*
White Solid	0.0684	8.9515	9.0199	0.0511	6.7663	6.8174	65/542/607	173	0
White Solid	0.0626	8.4115	8.4741	0.0528	5.2916	5.3444	61/510/571	167	0
White Solid	0.2529	4.1728	4.4257	0.1559	2.8316	2.9875	250/253/503	182	0
White Solid/	0.5394	1.4699	2.0093	0.3723	1.0759	1.4482	484/89/573	176	0
Clear Liquid	0.3730	1.1891	1.5621	0.1708	0.6984	0.8692	329/72/401	199	0
White Solid	0.0466	6.9106	6.9572	0.0224	1.5040	1.5264	46/419/475	265	15
White Solid	0.2242	3.5460	3.7702	0.0742	1.4167	1.4909	215/215/430	270	15
White Solid/ Clear Liquid	0.3596	0.6267	0.9863	0.1015	0.2459	0.3474	342/38/380	260	15
White Solid	0.0692	8.6707	8.7399	0.0203	0.7393	0.7596	64/525/569	366	32
White Solid	0.0456	6.7787	6.8243	0.0061	0.3282	0.3343	45/411/456	353	32
White Solid	0.2541	4.0916	4.3457	0.0489	0.9411	0.9900	248/248/498	378	32
White Solid/	0.4854	0.0579	1.4433	0.1304	0.2932	0.4236	450/58/508	370	32
Clear Liquid	0.4351	0.8579	1.2930	0.0759	0.2372	0.3131	420/52/472	371	32
White Solid	0.0694	7.1272	7.1966	0.0041	0.2625	0.2666	65/432/497	417	46
White Solid	0.1888	3.0677	3.2565	0.0050	0.1328	0.1378	186/186/372	354	46
White Solid	0.4512	0.8082	1.2594	0.0077	0.0584	0.0661	444/49/493	470	46
White Solid	0.0519	7.4219	7.4738	0.0016	0.2828	0.2844	50/450/500	430	60
White Solid	0.4706	0.8524	1.3230	0.0110	0.0523	0.0633	450/52/502	487	60

*Measured quantities

**Includes the vapor portion above the solid or liquid in collection tube

Table 2 presents calculated values obtained from the observed data in Table 1. Included in the table are the mole percentage of HF and UF_6 of the gas mixture with corresponding hydrogen:uranium ratios, the percentage of HF, UF_6 and total of mixture cooled in the collection tube with the corresponding hydrogen:uranium ratios, and the cooling temperature and remaining pressure for reference to observed data in Table 1.

The quantity of HF and UF_6 cooled to the collection tube as a percentage of each in the gas mixture is graphically displayed in Figure 4. Figure 5 is a comparable graphical display of the total quantity cooled to the collection as a percentage of the total in the gas mixture. Figure 6 is a graphical display of the distribution of HF relative to UF_6 in the collection tube as a percentage of each from the gas mixture. Figure 7 indicates the position of isotherms of hydrogen:uranium ratios of gas mixture versus sample in collection tube. The hydrogen:uranium ratio versus temperature by original gas mixture concentration is presented in Figure 8.

The grams of UF_6 in the original mix as indicated in Table 1 is calculated using the ideal gas law with the molecular weight of UF_6 taken as 352. The pressure used for each entry was the pressure indicated in Table 1, which is obtained as a difference between the observed pressure of HF in the pipette, maintained at 126°F, and the total pressure in the pipette after intoduction of the UF_6.

The mole percentage values of HF and UF_6 listed in Table 2 are obtained from the respective weights listed in Table 1 using 20 for the molecular weight of HF and 352 for UF_6. The hydrogen:uranium ratios listed are mole ratios; ie., moles of hydrogen per mole of uranium, and are obtained using the mole percentage of HF and UF_6. The data graphically displayed in Figures 4, 5, and 6 are based on percentage by weight of the quantity of HF, UF_6, or the total transferred from the pipette to the cooled collection tube. Since the data are based on mass as determined by weight, it is reasonable that the percentage of the mix as total transferred as indicated in Figure 5 should be essentially the same as that transferred as UF_6 in Figure 4. This is due to the more than 17 factor (352/20) difference in molecular weights of UF_6 versus HF. Also from Figure 6, it is apparent that only the 10/90 HF/UF_6 mixtures were enhanced with HF upon cooling, the maximum enhancement occurring at 15°F.

The pressure remaining in the pipette after cooling the sample in the collection tube is indicative of the number of moles of HF and UF_6 in the remaining fraction. The quantity of HF and UF_6 in the remaining fraction can be calculated from the difference between that in the initial mixture and that in the cooled fraction analyzed in the collection tube. It should be noted that HF has a tendency to polymerize at relatively high pressure (>100 torr) and low temperature. Polymerization is apparent in the study by using the ideal gas law and the actual weights of HF used (Table 3) in conjunction with observed pressure. This procedure produces an average molecular weight of 22.4 ± 0.4 (95% C.L.) for HF relative to the theoretical value of 20. This value is used in Table 3 for conversion of remaining grams of HF to moles of HF. In so doing, the agreement of observed pressure and the number of moles to calculated pressure and number of moles agree within experimental error; ie., differences of -11 ± 86 and 0.00063 ± 0.00414, respectively.

Table 2

CALCULATED DATA

M% HF/UF_6		% Cooled Fraction			H/U Ratio			Pressure, Torr	
Mix	Cooled Fraction	HF	UF_6	Total	Mix	Cooled Fraction	t, °F	Original Mix	Remaining
12/88	12/88	75	76	76	0.14	0.14	0	607	173
12/88	15/85	84	63	63	0.14	0.18	0	571	167
52/48	49/51	62	68	68	1.1	0.96	0	503	182
67/13	86/14	69	73	72	6.7	6.1	0	573	176
35/15	81/19	46	59	56	5.7	4.3	0	401	199
11/89	21/79	48	22	22	0.12	0.27	15	475	256
53/47	48/52	33	40	40	1.1	0.92	15	430	270
91/9	88/12	28	39	35	10	7.3	15	380	260
12/88	33/67	29	8.5	8.7	0.14	0.49	32	569	366
11/89	25/75	13	4.8	4.9	0.12	0.33	32	456	353
52/48	48/52	19	23	23	1.1	0.92	32	498	378
90/10	89/11	27	31	29	9.0	8.1	32	508	370
90/10	85/15	17	28	24	9.0	5.7	32	472	371
15/85	22/78	5.9	3.7	3.7	0.18	0.28	46	497	417
52/48	40/60	2.6	4.3	4.2	1.1	0.67	46	372	354
91/9	70/30	1.7	7.2	5.2	10	2.3	46	493	470
11/89	10/90	3.1	3.8	3.8	0.12	0.10	60	500	430
91/9	79/21	2.3	6.1	4.8	10	3.8	60	502	487

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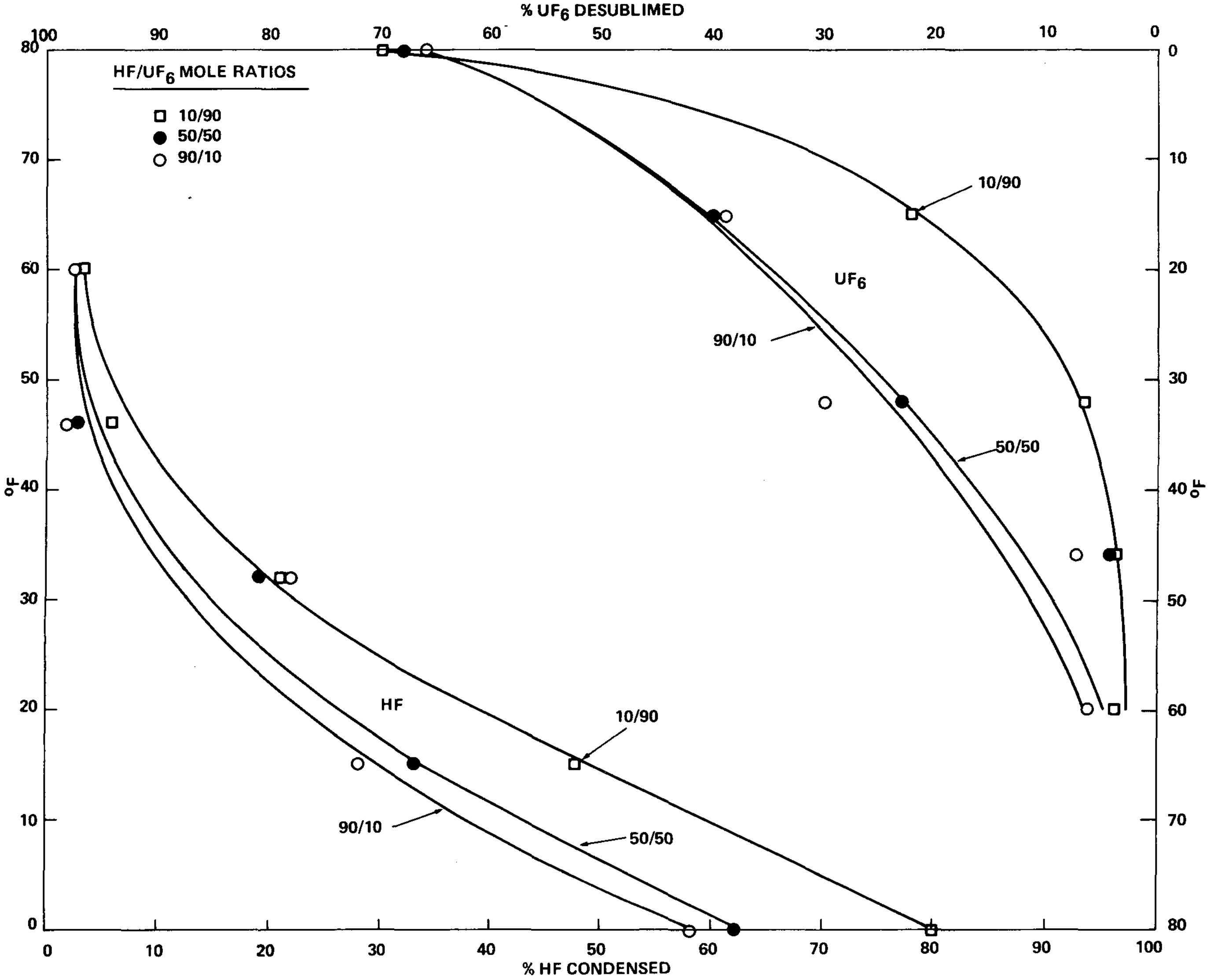

Figure 4

PERCENTAGE OF HF AND UF_6 IN GAS MIXTURE COOLED TO COLLECTION TUBE

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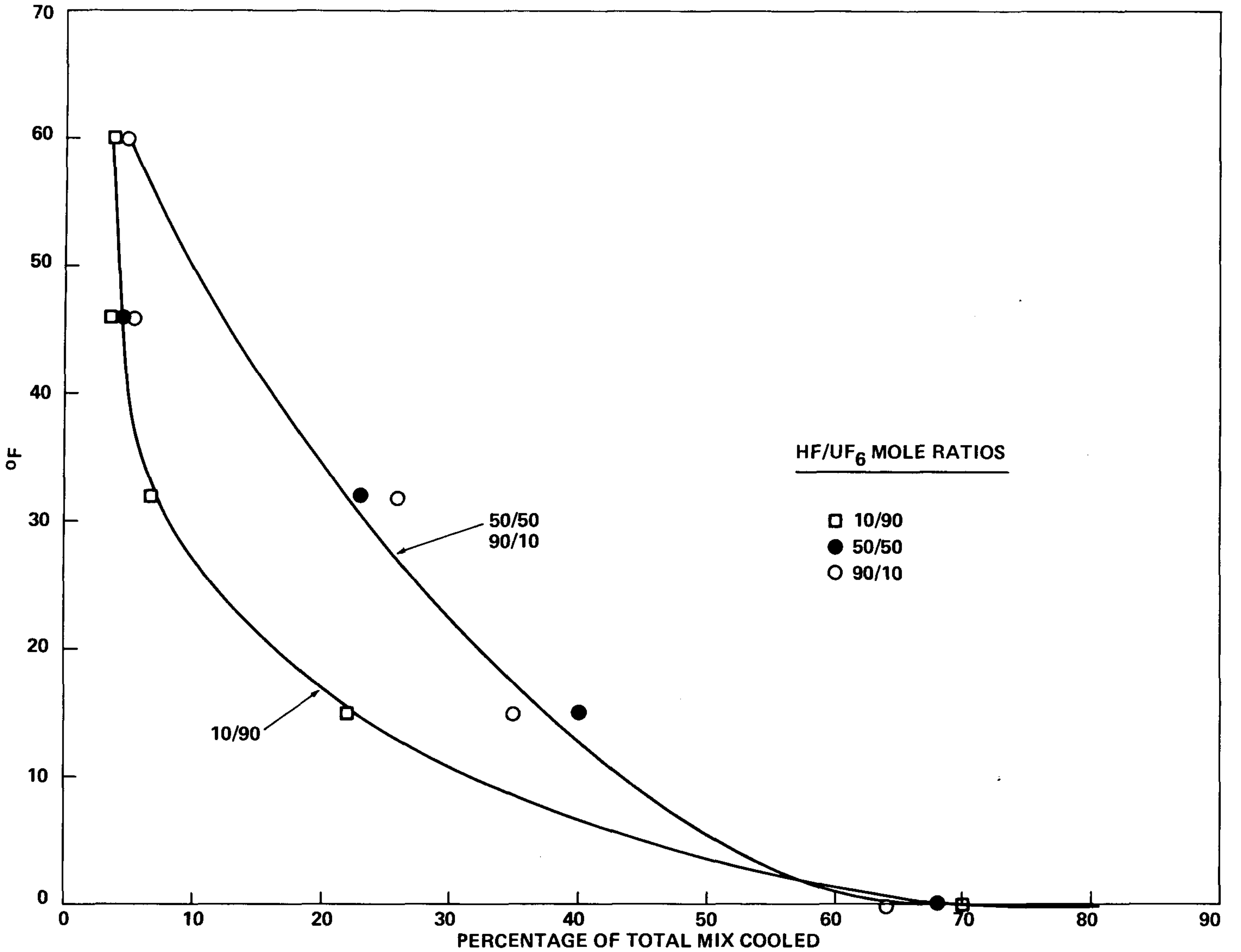

Figure 5

PERCENTAGE OF GAS MIXTURE COOLED TO COLLECTION TUBE

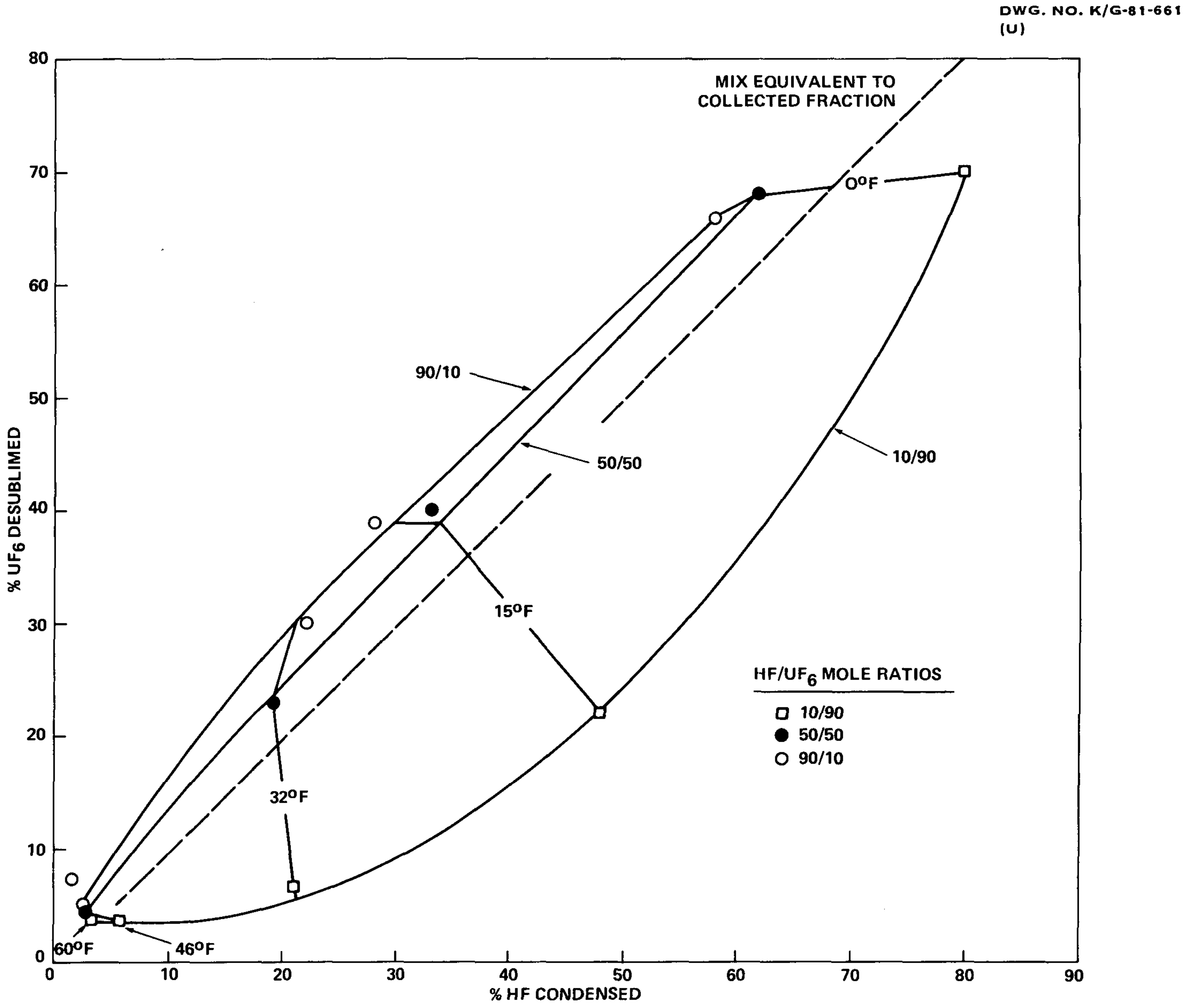

Figure 6

HF-UF$_6$ DISTRIBUTION IN COLLECTION TUBE AS RESPECTIVE PERCENTAGE OF GAS MIXTURE

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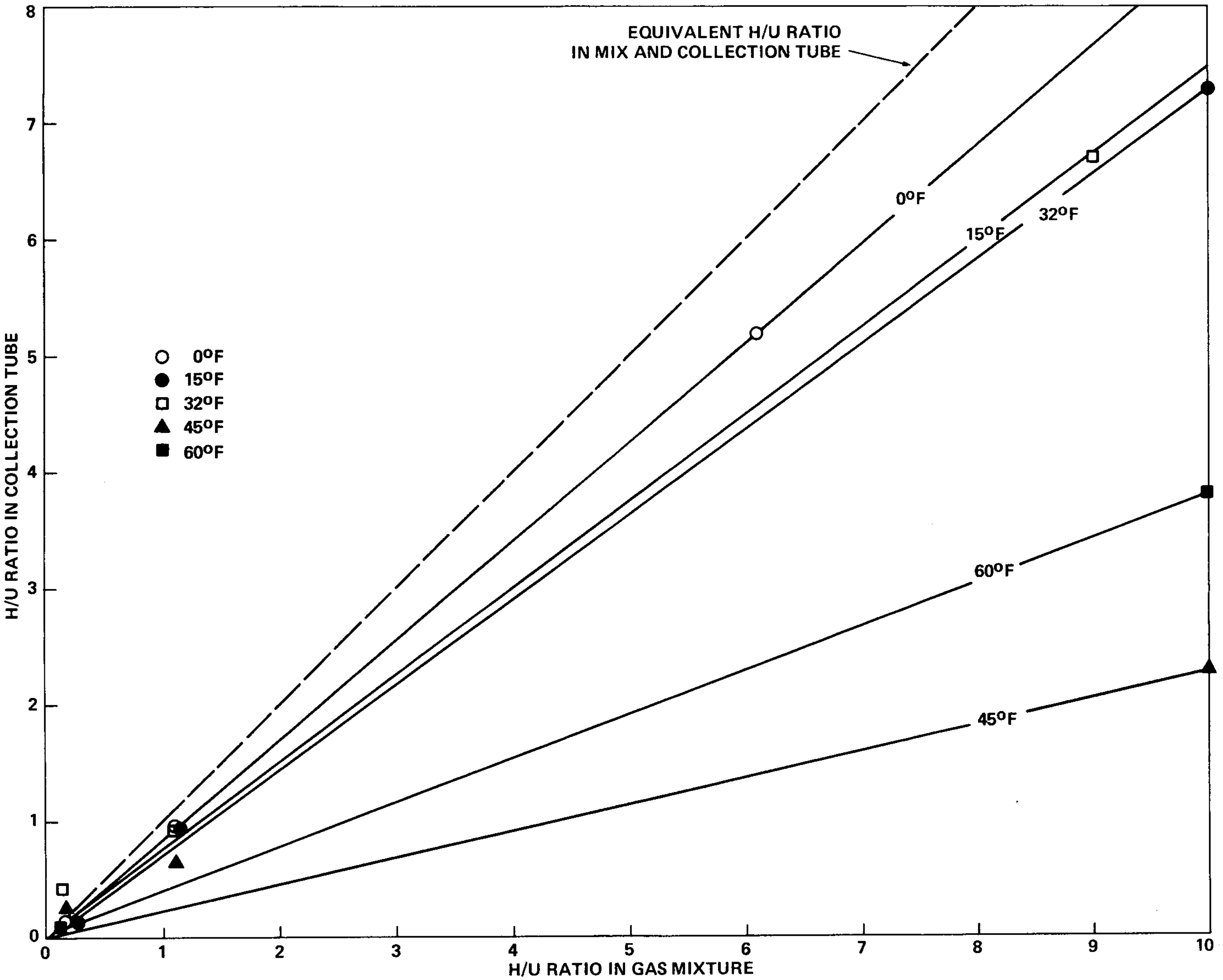

Figure 7

ISOTHERMS OF H/U RATIOS OF GAS MIXTURE VERSUS COLLECTED SAMPLE

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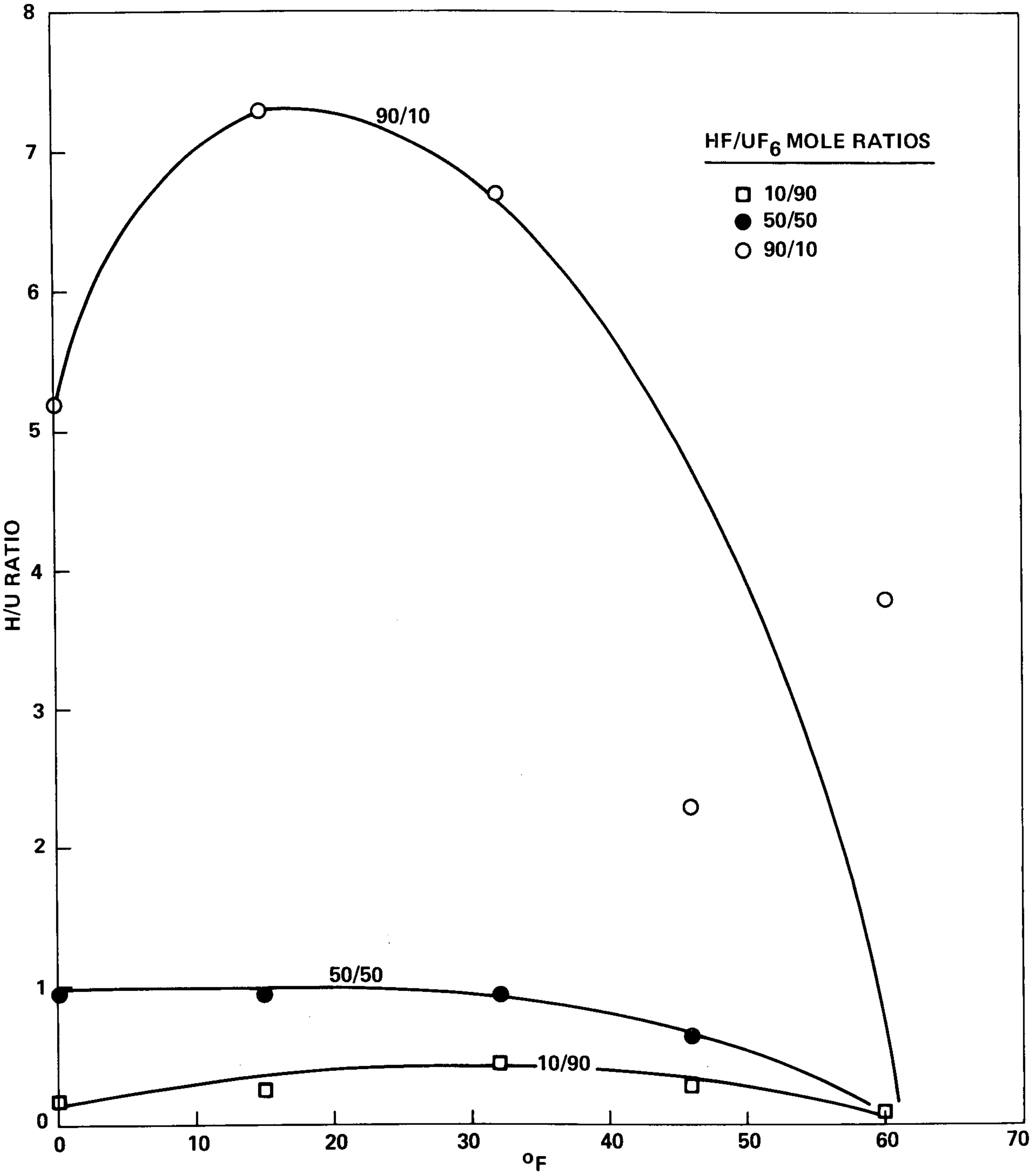

Figure 8

H/U RATIO VERSUS COOLING TEMPERATURE FOR SAMPLE IN COLLECTION TUBE

Table 3

REMAINING FRACTION DATA

MW HF	°F	Remaining Fraction HF, g	Remaining Fraction UF_6, g	Remaining Pressure, Torr Observed	Remaining Pressure, Torr Calculated	Moles Remaining Observed HF**	Moles Remaining Calculated UF_6	Total Moles Observed	Total Moles Calculated	M% HF/UF_6	Total Original Pressure
22.4	0	0.0173	2.1852	173	142	0.000772	0.00620*	0.00698	0.00853	12/88	607
21.9	0	0.0098	3.1199	167	189	0.000438	0.008863	0.00930	0.00824	5/59	571
21.6	0	0.0970	1.3412	182	165	0.004330	0.003810	0.00814	0.00898	55/44	503
23.8	0	0.1671	0.3940	176	174	0.00746	0.001119	0.00858	0.00868	88/12	573
24.2	0	0.2022	0.4907	199	211	0.009026	0.001394	0.01042	0.00982	88/12	401
		Saturation Vapor Pressure: 176*									
21.6	15	0.0242	5.4066	256	333	0.001080	0.015360	0.01644	0.01263	7/93	465
23.3	15	0.1500	2.1293	270	259	0.006696	0.006049	0.01275	0.01332	55/45	500
22.4	15	0.2581	0.3808	260	255	0.011522	0.001082	0.01260	0.012824	92/8	380
		Saturation Vapor Pressure: 255*									
23.1	32	0.0489	7.9314	366	501	0.002183	0.022532	0.02472	0.01805	10/90	589
21.6	32	0.0395	6.4505	353	407	0.001763	0.018325	0.02009	0.01741	10/90	456
21.9	32	0.2052	3.1505	378	367	0.009161	0.0089502	0.01811	0.01864	69/31	496
23.0	32	0.3550	0.6647	370	389	0.017736	0.001888	0.017736	0.01825	90/10	508
22.1	32	0.3592	0.6207	371	361	0.016036	0.001763	0.01780	0.01830	91/9	472
		Saturation Vapor Pressure: 382*									
22.8	46	0.0653	6.8647	417	456	0.002915	0.019502	0.02242	0.02057	14/86	497
21.7	46	0.1838	2.9349	354	335	0.008205	0.008338	0.01654	0.01746	52/48	372
21.7	46	0.4435	0.7498	470	445	0.019799	0.007130	0.02193	0.02318	91/9	493
21.2	60	0.0503	7.1391	430	457	0.002246	0.020282	0.02253	0.02121	11/89	500
22.3	60	0.4596	0.8001	487	462	0.02052	0.002273	0.02279	0.02155	91/9	502

* Assumes additive partial pressure of HF and UF_6 in the collection tube.

** Uses 22.4 for the molecular weight of HF and 1.0 liters for volume.

The vapor pressure versus temperature of the pure components are listed below.

t, °F	Vapor Pressure Torr HF[(a)]	Vapor Pressure Torr UF_6[(b)]
60	690	58
46	493	32
32	364	18
15	247	8
0	172	4

(a) $\log_{10} Pm = 8.38036 - \frac{1952.55}{335.52 - t}$, Jerry R. L. and Davis, Jr., W., *The Vapor Pressure Association, and Heat of Vaporization of Hydrogen Fluoride,* Journal of Physical Chemistry, Vol. 57, pp. 55-57 (1953).

(b) $\log_{10} Pm = 6.38353 + 0.0075377\ t = \frac{94276}{t - 183.416}$, Dewitt, R., *Uranium Hexafluoride: A Survey of the Physisco-Chemical Properties,* GAT-280 p. 81 (1960).

Figure 7 is a graphical display of the hydrogen:uranium ratios presented in Table 2. The lines are visually drawn between 0 and the largest hydrogen:uranium ratio for each of the five different temperatures studied. The position of the 45 and 60°F temperatures appear reversed. There is only one point for each of the 15, 45, and 60°F making the position of these lines highly subject to variation; ie., differences between hydrogen:uranium values in gas mixture and hydrogen:uranium values in collection tube can vary by ±3.3 (95% C. L.) as determined from the agreement of the high hydrogen:uranium ratio repetitive runs in Table 2). It is, however, noted that in no case above a hydrogen:uranium ratio of 1.0 that the ratio in the collection tube was higher than the H/U ratio in the gas mixture.

The reversal of the 45 and 60°F lines in Figure 7 is also noted in Figure 8. Assuming the smooth curve for the 90/10 HF/UF_6 ratios versus temperature is correct, then the 45°F point is low and the 60°F point is high. Also from Figure 8, the hydrogen:uranium ratio for mixtures containing 50 mole percentage of HF and below have ratios of 1.0 or less between 0 and 60°F, and also tend to converge at 60°F (and probably also at some temperature below 0°F).

In addition to the hydrogen:uranium ratio being of importance in criticality consideration, material proximity is also of concern. As noted in Table 1, liquid HF is noted in collection tubes obtained from gas mixtures of 90/10 HF/UF_6 at below 32°F. Figure 9 is a photograph of one such instance during the course of the study.

QUALITY ASSURANCE

The pipette volume was determined prior to this study using known volumes and determined to be 0.95 liters. Lines, valving, and residual volume of the collection tube are estimated to be an additional 0.05 liter. The weights of HF were measured to 0.1 mg using an analytical balance, thus the gross weight is known to better than ±0.2 mg. The HF and UF_6 were analyzed by mass spectrometry and infrared spectrophotometry for impurities, and both were at least 99.9 wt % pure. In addition, the UF_6 was measured by freezing point depression which indicated more than 99.99 wt % UF_6. The temperature of the coolant used with the collection tube was checked using two mercury thermometers to ±2°F, and the interior of the gas pipette was measured using a gas-filled and a mercury-filled thermometer and was controlled within ±4°F. The molecular weight of anticipated polymerized HF* was determined to be 22.4 ± 0.4 (95% C. L.) from measurements of the weight of HF versus the resulting pipette pressure. The agreement of observed pressure compared to ideal additive vapor pressure was 13 ± 26 torr (95% C. L.) for those points where saturation existed (0, 15, and 32°F). Agreement of observed versus calculated remaining pressure is -11 ± 10 torr (95% C. L.), indicating a small bias. This is also reflected in the agreement in the number of residual moles of the mixture remaining compared to the calculated number of moles based on observed remaining pressure, 0.00068 ± 0.00057 (95% C. L.).

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*Jerry R. L. and Davis, Jr., W., *The Vapor Pressure Association, and Heat of Vaporization of Hydrogen Fluoride,* Journal of Physical Chemistry, Vol. 57, pp. 55-57 (1953).

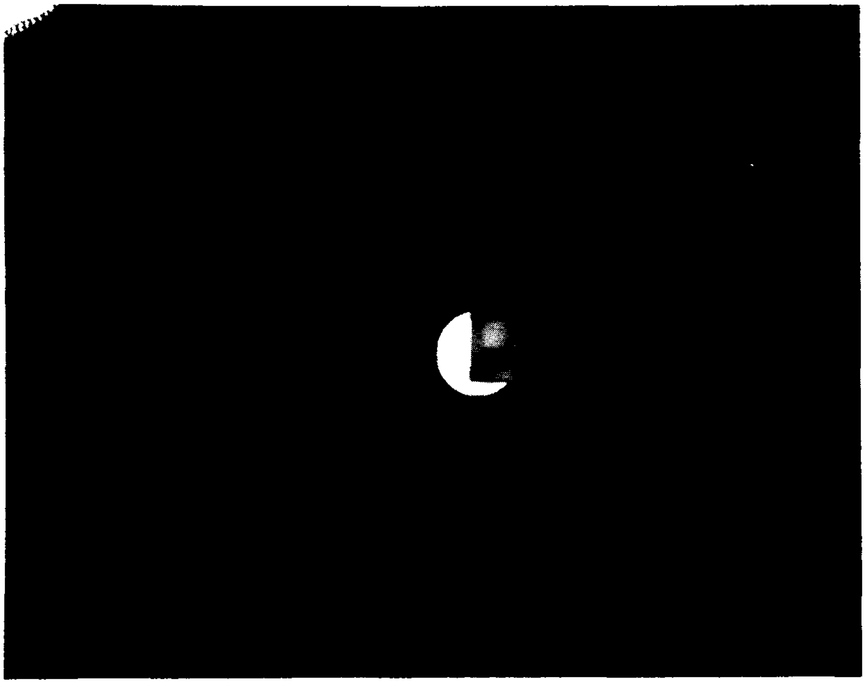

Figure 9

HF IN COLLECTION TUBES OBTAINED FROM GAS MIXTURES OF 90/10 HF/UF_6 BELOW 32°F

The measurement of uranium in the sample cooled in the collection tube was accomplished using a method normally associated with analyses of $\leq$99.9% UF_6 for 10 g samples, where the precision of measurement is ±0.1% of the value. However, the samples analyzed during this study were well below the 99.9% UF_6 content and many were below 2 g in total weight. An estimate of the analyses of UF_6 in the samples encountered in this study is of the order of no more than ±1%.

As a check for the concentration of the remaining fraction, some of the runs in this study were sampled into a 500-ml Monel container and submitted for mass spectrometer analyses. The HF and UF_6 mixtures are particularly difficult to sample using single-valued containers caused by the substantial difference in the vapor pressures of HF and UF_6, and the inherent capacity for HF to adhere to the walls of containers.

The following is a tabulation of the calculated remaining fraction for eight of the runs with corresponding mass spectrometer analyses.

HF		UF_6	
Calculated	Mass Spectrometry	Calculated	Mass Spectrometry
86	84	14	15
89	89	11	11
33*	2*	67*	98*
12	8	88	92
81	76	19	24
22	12	78	88
85	86	15	14
15	6	85	94

*Omitted from further calculation

95% C. L. of Single Difference: ±11
Mean Difference: 4 ± 4

CONCLUSIONS

The following conclusions may be inferred from this study:

1. Enhancement of HF in cooled fractions of HF-UF_6 gas mixtures occurs only from 10/90 HF/UF_6 gas mixtures, thus increasing the hydrogen:uranium ratio. The increase, however, produced hydrogen:uranium ratios <0.6.

2. The usual pressure of liquid HF in the collection tube occurs only at $\leq$32°F from initially high (90/10) mixtures of HF/UF_6.

3. HF does polymerize at 126°F and 500 torr. The polymerization is such that the molecular weight was measured to be 22.4 ± 0.4 rather than 20.0.

4. There is an insignificant difference in the separative behavior of HF/UF_6 gas mixtures between 50/50 and 90/10 when cooled from 126°F to between 0 and 60°F.

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