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**THE SOLUBILITY OF URANIUM HEXAFLUORIDE
IN PERFLUOROETHERS**

E. J. Barber

K-25 INFORMATION
RESOURCE CENTER
K-1002 MS 221

JULY 1984

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FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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July 15, 1984

K/PS--798

DE87 002525

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Prepared for the U.S. Department of Energy
under Contract No. DE-AC05-84OR21400

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INTRODUCTION

The polyperfluoroethers are compatible with uranium hexafluoride (UF_6) and are suitable for use in diffusion pumps and in mechanical vacuum pumps which rely on an oil as both the lubricant and the seal. The UF_6 is soluble in all fluids with which it is compatible. Because a number of vacuum pumps in the BOP facilities of the GCEP plant employ these perfluoroether oils as the working fluid and have oil chambers which are large,* questions have been raised as to the relationships governing the solubility of UF_6 in these materials and the maximum quantities of UF_6 which could be dissolved in these oils under credible accident conditions. This report summarizes these solubility relations and the interaction of the UF_6 solubility and the pumping capability of this type of vacuum pump. It will be shown that, whereas the solubility of UF_6 in Fomblin Y25 fluoroether fluid under a UF_6 pressure of 760 torr and at the pump operating temperature of 160°F is about 500 g of UF_6 per liter of oil, the system controls are such as to isolate the system from the pumps before the quantity of UF_6 dissolved in the perfluoroether exceeds about 10 g of UF_6 per liter of oil.

BASIC SOLUBILITY RELATIONSHIPS

The solubility of UF_6 in fluorocarbons is reasonably close to ideal¹ and an approximation of the solubility can be made using Raoult's law which states that

$$\bar{P}_1 = x_1 P_1^\circ \quad (1)$$

where $\bar{P}_1$ is the partial pressure of component 1,

x_1 is the mol fraction of component 1 and

P_1° is the vapor pressure of pure liquid component 1.

In actual practice Raoult's law is never precisely obeyed and a proportionality factor called the activity coefficient is employed to make the law exact; namely,

*While the pump oil chambers of some of the pumps have a capacity of 40 L of fluid, the chambers do not have to be filled for the pumps to operate satisfactorily. For this reason the quantity of oil in each pump can be limited, without adverse effects, to 27 L by administrative control.

$$\bar{P}_1 = \gamma_1 x_1 P_1^\circ \quad (2)$$

where γ_1 is the activity coefficient for component 1.

Thus, it is apparent that the solubility of a volatile or gaseous component (or solute) in a system in which the second component (or solvent) is essentially not volatile is proportional to the relative pressure of the volatile component; i.e.,

$$x_1 = \bar{P}_1 / \gamma_1 P_1^\circ \quad (3)$$

where $\bar{P}_1 / P_1^\circ$ is defined as the relative pressure X_1 of component 1.

Equation 3 is readily evaluated if the activity coefficient can either be assumed to be independent of x_1 which is normally not the case or an analytic expression may be developed to evaluate it. Hildebrand and Scott² developed an expression which has proven very reliable in estimating activity coefficients in systems of this kind from the earlier efforts of Hildebrand, Scatchard, Flory, and Huggins^{3,4,5,6}. This expression is

$$\ln \gamma_1 = \ln(\theta_1/x_1) + V_1 \theta_2^2 (\delta_1 - \delta_2)^2 / RT - \theta_2 (V_1/V_2 - 1) \quad (4)$$

where δ_1 is the activity coefficient of component 1,

x_1 is the mol fraction of component 1,

θ_1 is the volume fraction of component 1 defined as

$$\theta_1 = x_1 V_1 / (x_1 V_1 + x_2 V_2),$$

V_1 and V_2 are the molar volumes of components 1 and 2,

θ_2 is the volume fraction of component 2,

R is the universal gas constant in appropriate units,

T is the temperature in Kelvin, and

δ_1 and δ_2 are the solubility constant parameters or the "cohesive energy densities" of the components.

The solubility parameters are defined by the relations

$$\delta_1^2 = (\Delta E_1^{\text{vap}}/V_1) \text{ and } \delta_2^2 = (\Delta E_2^{\text{vap}}/V_2)$$

where ΔE_1^{vap} and ΔE_2^{vap}

are the internal energies of vaporization and are frequently estimated by the relation

$\Delta E^{\text{vap}} = \Delta H^{\text{vap}} - RT$ in which ΔH^{vap} is the heat of vaporization.

To solve Equation 2 for x_1 , it is noted that

$$\gamma_1 x_1 = \bar{P}_1/P_1^0 \quad (5)$$

and that the value of the right hand side of Equation 5 is fixed for a given temperature and partial pressure of the solute. Now using an initial value for x_1 from Equation 1 and Equation 4 to evaluate γ_1 , an improved value of x_1 is obtained. By repeating the cycle with each new value of x_1 , the difference between

$$\gamma_1 x_1 \text{ and } \bar{P}_1/P_1^0$$

can be made arbitrarily small. The last value of x_1 so calculated gives the mol fraction of UF_6 in the solvent for the given relative pressure and temperature.

The solubility in terms of g UF_6 dissolved per liter of solvent is obtained from the mol fraction x_1 for UF_6 by the relation

$$Z = [x_1/(1 - x_1)] * [M_{\text{UF}_6}/V_s] * [1000] \quad (6)$$

or

$$Z = [x_1/(1 - x_1)] * [M_{\text{UF}_6}/M_s] * [1000 \rho_s] \quad (7)$$

where Z is the solubility in terms of g UF_6 per liter of oil,

x_1 is mol fraction of component 1 (or UF_6),

M_{UF_6} and M_s are the molecular weights of UF_6 and the solvent,

V_s is the molar volume of the solvent equal to M_s/ρ_s , and

ρ_s is the density of the solvent.

In the systems under consideration enough data are available to estimate all the values needed to solve the equations and an experimental value for the solubility has been determined which will permit estimation of the reliability of the calculations. The vapor

pressures, heats of vaporization, molecular weights, densities, and solubility parameters needed are given or are estimated from existing data in Appendix A.

ESTIMATED SOLUBILITY OF UF_6 IN TWO POLYPERFLUOROETHERS

The solubility of UF_6 in the polyperfluoroethers, Krytox 143 AC Fluid (referred to hereafter as Krytox AC) manufactured by E. I. DuPont de Nemours and Co. and Fomblin Y25 Fluid manufactured by Montedison, has been estimated using the method described in the preceding section. The results are tabulated in Appendix B and are shown graphically in Figures 1, 2, and 3. Figure 1 shows the solubility in both liquids as a function of the relative pressure of UF_6 . The nature of the spread in the solubility data indicates that, in addition to the dependence on the relative pressure, there is a second order perturbation due to the temperature at a given relative pressure. Figures 2 and 3 illustrate the solubility of UF_6 in the Krytox AC and Fomblin Y25 fluids as functions of the temperature and partial pressure of the UF_6 . Also shown on these figures is the measured solubility of UF_6 in Fomblin Y25 fluid. As will be demonstrated from experimental measurements on perfluorocarbon and polychlorotrifluoroethylene oils, the estimated solubilities of these perfluoroether liquids are in excellent agreement with the measured value and have the proper dependence on the partial pressure of UF_6 and the temperature.

To a first approximation the solubility of UF_6 in these liquids at a given temperature is directly dependent on the partial pressure of the UF_6 over the oil. The solubility varies from less than a gram of UF_6 per liter at 2 torr to approximately 700 g of UF_6 per liter at atmospheric pressure and 150°F in Fomblin Y25 fluid. The solubility in Krytox AC fluid is a little lower being less than 500 g of UF_6 per liter at a UF_6 pressure of 760 torr at 150°F. The experimental solubility of UF_6 in Fomblin Y25 fluid at 150°F and 100 torr partial pressure of UF_6 is 36.2 g of UF_6 per liter⁷.

COMPARISON TO UF_6 SOLUBILITY IN OTHER FLUOROCARBONS

The solubility of UF_6 in a perfluorocarbon oil designated as C-2144 having the empirical formula $\text{CF}_3(\text{CF}_2)_{19}\text{CF}_3$ (mol wt = 1088) was experimentally determined at SAM labs at 140, 180, and 212°F.⁸ These data are listed in Appendix C and compared to the solubility data on Fomblin Y25 fluid in Figures 4 and 5. Also shown are similar data for the solubility of UF_6 in an-MFL oil (chlorotrifluoroethylene polymer) of molecular weight 790 ± 30 at 100, 140, 180, 212, and 220°F.⁹ These data are also tabulated in Appendix C. Errors in measuring the solubility of UF_6 in these oils are essentially as large as the difference between values estimated for the solubility of UF_6 in the two perfluoroethers. Figure 5 which shows the dependence of the UF_6 solubility on the

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Figure 1
SOLUBILITY OF UF_6 KRYTOX 143AC AND FOMBLIN Y25
POLYPERFLUOROETHER FLUIDS AS A FUNCTION OF
RELATIVE PRESSURE AT FOUR TEMPERATURES

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Figure 2
SOLUBILITY OF UF_6 IN KRYTOX 143 AC FLUID

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(U)

Figure 3
SOLUBILITY OF UF_6 IN FOMBLIN Y12 FLUID

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(U)

Figure 4
SOLUBILITY OF UF_6 IN C-2144 FLUOROCARBON OIL AND IN MFL OIL (POLYCHLOROTRIFLUOROETHYLENE) COMPARED WITH SOLUBILITY OF UF_6 IN FOMBLIN Y25 FLUID (NOTE TEMPERATURE RANGE FOR C-2144 OIL IS 140 TO 212°F AND FOR MFL OIL IS 100 TO 220°F)

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Figure 5

SOLUBILITY OF UF₆ IN PERFLUOROCARBON TYPE FLUIDS

relative pressure of the UF_6 systematizes the data and clearly indicates that UF_6 is more soluble in the MFL oil than in the C-2144 oil or the two perfluoroethers.

EFFECT OF DISSOLVED UF_6 ON THE OPERATING PRESSURE OF A ROTARY SEAL VACUUM PUMP

In a rotary seal vacuum pump the working fluid provides both the lubrication and seal against atmospheric pressure or other pressure against which the pump is working. One of the properties which the working fluid must have is a lower vapor pressure than the required ultimate pressure in the system being pumped upon, since the pressure in the system cannot be reduced below the pressure exerted by the oil regardless of the pumping efficiency of the pump. In the uses projected for the pumps using the Fomblin Y25 fluids, the pressures at which operation will normally be carried out are in the order of 137 μm . For the pressure of the Fomblin Y25 oil to be this low, the concentration of the dissolved UF_6 must be less than 0.1 g of UF_6 per liter of oil. In practice, in an emergency the system will be permitted to operate until the pressure in the system being pumped upon reaches 9 torr total pressure. At this point all parts of the system are isolated from each other so that no more UF_6 can reach the pumps. If one assumes that essentially all the 9 torr is due to UF_6 , the maximum concentration of UF_6 in the Fomblin Y25 fluid would be between 4 and 8 g of UF_6 per liter depending upon whether the pump was operating at its normal temperature of 160°F or minimum operating temperature of 120°F.

Since the gas being drawn into the pumps may be fairly pure UF_6 , a stream of dry air is provided beyond the pumps in the discharge stream to keep the UF_6 partial pressure in the discharge stream from rising so high as to cause sufficient UF_6 to dissolve in the oil to raise the oil pressure to unacceptable levels; i.e., 9 torr. In view of these controls it does not seem reasonable that the quantity of UF_6 dissolved in the Fomblin Y25 fluid in these pumps could exceed approximately 10 g/L in a system isolation condition. Figures 6 and 7 show how this limiting pressure of 9 torr determines the quantity of UF_6 which can be dissolved in a liter of oil.

DISCUSSION OF THE RESULTS

The comparison of the estimated solubility of UF_6 in the perfluoroethers, Krytox AC and Fomblin Y25 fluid, at 100 torr UF_6 pressure and 150°F with the known solubility of UF_6 in other fluorocarbon based oils^{8,9}, indicates that the estimations are reliable and accurate to within the spread of approximately 30% of the value normally obtained for this method of estimation with well-behaved materials of this kind. The degree of variation of the solubility with changes in temperature and partial and relative pressures of UF_6 is correctly predicted. See Figure 5.

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Figure 6

MINIMUM PUMP ENTRANCE PRESSURE AS A FUNCTION OF DISSOLVED UF_6
AND PUMP OPERATING TEMPERATURE

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Figure 7
MINIMUM PUMP ENTRANCE PRESSURE AS A FUNCTION OF DISSOLVED UF_6
AND PUMP OPERATING TEMPERATURE

At relative pressures of UF_6 approaching 1.0, the solubility of UF_6 is quite large, reflecting the close to ideal mutual solubility of the components in each other. Only at and above the triple point temperature and pressure for UF_6 can a relative pressure of 1.0 be reached since the relative pressure is determined by the lower of the ratios of the actual partial pressure or the pressure of the solid UF_6 to the vapor pressure (or extrapolated vapor pressure) of liquid UF_6 at the given temperature. Of course, pure solid UF_6 will be deposited if the partial pressure of the UF_6 in the vapor exceeds the vapor pressure of solid UF_6 at the temperature. However, in an earlier section it was noted that system isolation from the pumps occurs if the pressure in the working part of the system exceeds 9 torr. This condition effectively defines the maximum quantity of UF_6 which can be dissolved in the pump fluid at the time of isolation from the UF_6 source to be less than 10 g UF_6 per liter. Figure 7 permits determination of the maximum quantity of UF_6 dissolved per liter of Fomblin Y25 fluid for this or any other permissible operating pressure at temperatures of 160, 150, 120, and 77°F.

The system isolation control instrumentation thus constitutes the safety instrumentation for the system, preventing both (1) potential accidental damage to system components due to high pressure and (2) any possibility of depositing enough UF_6 either by solubility in the pump fluid or condensation of solid to cause a nuclear criticality in case of rupture of the pump cooling system into the pump. This instrumentation will cause system isolation if either a breach occurs in the system being pumped upon or the flow of dry air to the pump discharge line is interrupted or fails to remove UF_6 as fast as it enters the pumps.

CONCLUSIONS

The solubility of UF_6 in polyperfluoroethers at a given temperature can be predicted from the physical and thermodynamic properties of the UF_6 and the particular perfluoroether fluid involved to within about $\pm 20\%$ of the value. The dependence of the solubility on temperature and UF_6 partial pressure are also correctly indicated.

The maximum solubility of UF_6 in the Fomblin Y25 fluid employed in the rotary vane vacuum pumps in the BOP facilities for the GCEP at Portsmouth, Ohio, is effectively controlled by the system isolation instrumentation to be less than 10 g of UF_6 per liter.

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APPENDIX A

PHYSICAL AND THERMODYNAMIC DATA REQUIRED FOR ESTIMATION
OF THE SOLUBILITY OF URANIUM HEXAFLUORIDE
IN KRYTOX 143AC AND FOMBLIN Y25 FLUIDS

The data needed for the estimation of the solubility of UF_6 in Krytox AC and Fomblin Y25 fluids include the following:

1. the vapor pressures of liquid and solid UF_6 as a function of temperature,
2. the heat of vaporization of UF_6 ,
3. the vapor pressures of the two polyperfluoroethers as functions of the temperature of estimations of the heats of vaporization,
4. the liquid densities of UF_6 , Krytox AC fluid and Fomblin Y25 fluid, and
5. the number average molecular weights of the two fluoroethers.

Note: The molecular weight most often given for polymers is based on the viscosity which emphasizes the contribution of the high molecular weight molecules. In this case, the molecular weight needed is the one which influences the volatility. It can be estimated from the experimental heat of vaporization obtained from the slope of $\ln P$ versus $1/T$ plot and the incremental increase in the heat of vaporization associated with each incremental increase in the molecular weight. The use of the molecular weight determined in this way may result in a slight overstatement of the solubility. Use of the viscosity based molecular weights often leads to a gross understatement of the solubility.

The remaining parameters, such as the molar volumes and the solubility parameters, can be derived from the above.

Equations for these properties will be listed below for each material, respectively. The symbol t will be used for degrees centigrade and the symbol T for Kelvin.

Uranium Hexafluoride

Vapor pressure of solid¹⁰

$$\log_{10} P(\text{torr}) = 6.38363 + 0.0075377t - 942.76/(t + 183.416)$$

Vapor pressure of liquid¹⁰

$$\log_{10} P(\text{torr}) = 6.99464 - 1126.288/(t + 221.963)$$

Density of liquid¹¹

$$\rho(\text{g/ml}) = 1.670 + 0.15203(t_c - t)^{1/2}$$

t_c is the critical temperature in degrees centigrade

Heat of vaporization (from slope of $\ln P$ vs $1/T$)

$$\Delta H = 7300 \text{ calories per g/mol}$$

Molecular weight

$$M_{\text{UF}_6} = 352$$

KRYTOX 143AC FLUID¹²

Vapor Pressure and Heat of Vaporization

The vapor pressure of the Krytox Brand Fluids are shown in Figure A-1 in which the $\log_{10} P$ is shown as a function of $1/T$. The heat of vaporization determined from the slope is 23,600 calories per g/mol.

Density of Liquid

$$\rho_{\text{K-AC}}(\text{g/mol}) = 1.942 - 0.00172t$$

Molecular Weight

If the heat of vaporization of polyperfluorocarbons and the heat of vaporization of perfluoroethers are plotted as a function of the molecular weight, the values tend to fall upon the same curve which becomes essentially linear as the molecular weight increases. By dividing the value of the heat of vaporization for a particular material by the limiting slope of this curve, one obtains an estimate of the molecular weight. The value for perfluorocarbons is 800 calories per 50 molecular weight units. The effective molecular weight of the Krytox AC fluid is approximately 1500 on this basis. The molecular weight determined from the viscosity is 5700.

FOMBLIN Y25 FLUID¹³

Vapor Pressure and Heat of Vaporization

The vapor pressures of the Fomblin Brand Fluids are given in Figure A-2. The heat of vaporization of Fomblin Y25 fluid is 20,020 calories per g/mol.

Density of Liquid

$$\rho_{\text{F-Y25}}(\text{g/mol}) = 1.925 - 0.00179t$$

Figure A-1

VAPOR PRESSURE OF KRYTOX FLUIDS

Figure A-2
VAPOR PRESSURE OF FOMBLIN FLUIDS

Note: The reference gives the density of the Fomblin Y25 fluid at only one temperature. The equation showing the temperature dependence is based on the behavior of a Krytox fluid of similar molecular weight.

Molecular Weight

Using the same scheme as for the Krytox 143 AC, the molecular weight needed for these calculations is 1100. The molecular weight based on the viscosity is 3400.

Data used in solubility estimation at 160, 150, 120, and 77°F

For UF₆:

Temperature, °F	Density, g/ml	Molar volume, ml	$\delta(\text{UF}_6),$ (cal/mol) ^{1/2}	Vapor pressure, torr
160	3.588	98.10	8.213	1417.4
150	3.620	97.22	8.256	1195.2
120	[3.717]	[94.70]	8.386	686.3
77	[3.848]	[91.48]	8.563	271.7

[] indicates extrapolated values for liquid

For Krytox 143 AC fluid:

Temperature, °F	Density, g/ml	Molar volume, ml	$\delta(\text{K-AC}),$ (cal/ml) ^{1/2}
160	1.820	824	5.27
150	1.829	820	5.29
120	1.858	807	5.33
77	1.899	790	5.40

For Fomblin Y25 fluid:

Temperature, °F	Density, g/ml	Molar volume, ml	$\delta(\text{F-Y25}),$ (cal/ml) ^{1/2}
160	1.798	612	5.62
150	1.808	609	5.64
120	1.837	599	5.69
77	1.899	585	5.76

APPENDIX B

SOLUBILITY OF UF_6 IN KRYTOX 143 AC AND FOMBLIN Y25 FLUIDS AT
160, 150, 120, AND 77°F

The equations presented in the second section of the report have been used to estimate the solubility of UF_6 in Krytox AC and Fomblin Y25 fluids. The results of the calculation are summarized in this Appendix.

Table B-1. The solubility of UF_6 at 160°F

UF_6 pressure, torr	Mol fraction UF_6 in		Solubility, g/liter in	
	Krytox AC	Fomblin Y25	Krytox AC	Fomblin Y25
2	0.00142	0.00145	0.61	0.84
10	0.00708	0.00724	3.05	4.19
100	0.0686	0.0706	31.5	42.4
250	0.164	0.171	83.8	118.6
500	0.307	0.321	189.2	272
760	0.437	0.456	332	482

Table B-2. The solubility of UF_6 at 150°F

UF_6 Pressure, torr	Mol fraction UF_6 in		Solubility, g/liter in	
	Krytox AC	Fomblin Y25	Krytox AC	Fomblin Y25
2	0.00163	0.00167	0.70	0.97
10	0.00814	0.00837	3.52	4.88
100	0.0787	0.0813	36.7	51.1
250	0.187	0.195	98.7	140
500	0.346	0.366	227	337
760	0.494	0.531	419	654

Table B-3. The solubility of UF₆ at 120°F

UF ₆ pressure, torr	Mol fraction UF ₆ in		Solubility, g/liter in	
	Krytox AC	Fomblin Y25	Krytox AC	Fomblin Y25
2	0.00257	0.00270	1.12	1.59
10	0.0128	0.0135	5.66	8.04
100	0.122	0.129	60.6	87.0
250	0.283	0.304	172	257
494.5*	0.512	0.567	458	770

*Maximum vapor pressure of UF₆ at this temperature.

Table B-4. The solubility of UF₆ at 77°F

UF ₆ pressure, torr	Mol fraction UF ₆ in		Solubility, g/liter in	
	Krytox AC	Fomblin Y25	Krytox AC	Fomblin Y25
2	0.00513	0.00559	2.30	3.38
10	0.0255	0.0278	11.7	17.2
100	0.235	0.261	137	213
111.8*	0.261	0.290	157	246

*Maximum vapor pressure of UF₆ at this temperature.

Table B-5. The relative pressure of UF_6 for several temperatures and partial pressures of UF_6

Temperature °F	UF_6 pressure torr	UF_6 liquid pressure torr	Relative pressure
160	2	1417.9	0.00141
	10		0.00705
	100		0.0705
	250		0.176
	500		0.353
	760		0.536
150	2	1195.0	0.00167
	10		0.00837
	100		0.0837
	250		0.209
	500		0.418
	760		0.636
120	2	686.0	0.00292
	10		0.0146
	100		0.146
	250		0.364
	494.5*		0.720
77	2	271.7	0.00736
	10		0.0368
	100		0.368
	111.8*		0.412

*Maximum vapor pressure of UF_6 at this temperature.

APPENDIX C

THE SOLUBILITY OF UF_6 IN FLUOROCARBON OIL C-2144 AND IN MFL OIL
(POLYCHLOROTRIFLUOROETHYLENE)

The solubility of UF_6 in a perfluorocarbon with the empirical formula $\text{CF}_3(\text{CF}_2)_{19}\text{CF}_3$ has been measured at three temperatures. The results are tabulated in Table C-1. Experimental measurements of the solubility of UF_6 in a polychlorotrifluoroethylene with a molecular weight of 790 ± 30 have been made at five temperatures and are given here in Table C-2.

Table C-1. The solubility of UF_6 in C-2144 fluorocarbon oil

Temperature °F	UF_6 pressure torr	Relative pressure	Solubility g/liter oil
140	52.8	0.0528	35.9
	63.1	0.0631	39.5
	95.8	0.0957	49.1
	105.6	0.106	57.5
180	111.8	0.0571	46.7
212	83.6	0.0267	16.8
	131.5	0.0419	33.6
	147.5	0.0470	31.2
	150.6	0.0480	32.4

Table C-2. The solubility of UF_6 in MFL oil

Temperature °F	UF_6 pressure torr	Relative pressure	Solubility g/liter oil
100	38.30	0.0841	57.5
	38.30		69.5
	38.30		77.9
	74.02	0.163	155.8
	74.53	0.164	141.4
	72.46	0.159	134.2
	74.53	0.164	147.4
	76.60	0.168	149.8
	79.19	0.174	154.6
	80.74	0.177	155.8
	80.74		154.6
	152.2	0.334	340.3
	152.2		378.7
140	82.30	0.0823	64.7
	84.37	0.0843	67.1
	106.6	0.107	112.6
	139.2	0.139	146.2
	150.1	0.150	151.0
180	83.85	0.0428	51.5
	83.85		41.9
	83.85		43.1
	83.85		44.3
	83.85		49.1
	74.02	0.0378	49.1
	76.08	0.0388	44.3
	99.90	0.0510	49.1
	112.3	0.0573	59.9
	114.9	0.0587	53.9
	124.2	0.0634	58.7
	129.9	0.0663	73.1
	152.7	0.0779	73.1
212	118.5	0.0378	24.0
	147.5	0.0470	49.1
	150.1	0.0479	52.7
	83.85	0.0240	21.6
	83.85	0.0240	21.6
	124.7	0.0356	40.7
	152.7	0.0436	46.7

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