Properties of Novel Fluorinated Compounds and Their Mixtures as Alternative Refrigerants

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Abstract

The liquid density, boiling point, critical temperature and critical density are investigated for chemicals; R-161 (CH_3CH_2F, bp = -37.3°C), CF_3SCF_3 (bp = -20.1°C), and E-218 ($CF_3OCF_2CF_3$, bp = -23.7°C); which are potential R-12 and R-22 alternatives. Since the range of applicability of chemicals may be improved by considering their mixtures, the vapor pressure of the mixtures; CH_3CH_2F/CF_3SF_5, CF_3CH_2F/CF_3SF_5, and $CF_3(CF_2)_2CH_2F$/isopentane; were also investigated. The critical mixing temperatures of the CH_3CH_2F/CF_3SF_5 mixtures are above -30°C which is greater than the normal boiling point (about -40°C). Therefore these mixtures would have limited application as R-12 and R-22 alternatives. The normal boiling point of the R-134a (CF_3CH_2F)/CF_3SF_5 mixtures at the azeotropic R-134a mole fraction 0.60 is -30.0°C. The critical mixing temperature of the latter mixtures is low enough not to be a factor in refrigerant applications. The $CF_3(CF_2)_2CH_2F$/isopentane mixtures have a normal boiling point of 15.1°C and a critical mixing temperature of about -5°C which may be acceptable for some applications as a R-11 alternative.

I. Introduction

In recent years a number of investigations have been identified chemicals which are alternatives to the chlorofluorocarbon (CFC) and hydrochlorofluorocarbon (HCFC) refrigerants for the purpose of protecting the stratospheric ozone layer. The authors' previous work [1,2] has centered around investigation of over 30 fluorinated propanes and butanes as alternatives for R-11, R-12 and R-114. A number of problems still remain, particularly with regard to identifying satisfactory R-22 and R-502 alternatives. This has motivated the investigation of mixtures, particularly azeotropic or near azeotropic mixtures [3,4].

With mixtures one hopes to retain the desirable properties of the pure components and minimize their shortcomings in refrigerant applications. The hydrocarbons or hydrocarbon derivatives with a low fluorine content are desirable for refrigerant applications because these have good environmental properties and higher critical temperatures than their highly fluorinated counterparts. Recently an azeotropic mixture of tetrafluoroethane (R-134a) and isobutane was patented for refrigerant applications [5].

In this work the ether compound, $CF_3OCF_2CF_3$, and the sulfur compounds, CF_3SCF_3 and CF_3SF_5, have been prepared for investigation of their potential as R-12 or R-22 alternatives either as pure chemicals or components of mixtures. The sulfur compounds are inert and nontoxic and are expected to have a higher solvating power for hydrocarbons than highly fluorinated hydrocarbons.

Fluoroethane or R-161 has a boiling point (-37.3^{o}C) and critical temperature 102.1^{o}C that are desirable for an R-22 alternative. However the flammability of fluoroethane lowers its potential as a refrigerant. R-134a (CF_3CH_2F) has a high critical temperature but its boiling point, -26.5^{o}C, is to high for it to serve as a replacement for R-22 in many applications. Mixtures of these compounds with CF_3SF_5 are examined to find if the desirable properties of these compounds are retained in the mixture and if the shortcomings are removed. Similarly the vapor pressures of the mixtures of isopentane

with nonflammable R-338mccq ($CF_3CF_2CF_2CH_2F$) are investigated to determine if the desirable properties of isopentane as an R-11 alternative are retained in the mixture.

II. Results for the Pure Chemicals

For a description of experimental methods the reader is referred to the authors' previous publications[1,2]. The pure chemicals studied in this work were synthesized to 99.5% purity, excepting R-161 (CH_3CH_2F) which was purchased from PCR Inc. In Table 1 we summarize the boiling point, heat of vaporization and critical properties for the pure chemicals. Tabulations of the measured liquid densities, ρ_ℓ, diameters, $\bar{\rho}[= (\rho_\ell + \rho_g)/2$ ρ_g being the vapor density], and vapor pressures are given in Tables 2 to 4. Parametric representations of the vapor pressures based on least squares calculations of the measured data are given in Tables 5 and 6. For temperatures below the boiling point, the vapor pressures are parametrically represented by the following equation,

$$\ln P = a + b \ln T + \frac{c}{T} \quad , \tag{1}$$

and for temperatures above the boiling the vapor pressures are represented by the Riedel or Lee-Kesler Equation [6],

$$\ln P = \alpha + \frac{\beta}{T_r} + \gamma \ln T_r + \delta T_r^6 \quad , \tag{2}$$

where T is the absolute temperature, P is the pressure, and T_r (= T/T_c , T_c being the critical temperature).

The two novel sulfur containing compounds, CF_3SF_5 and CF_3SCF_3 have boiling points and critical temperatures that would make them potential R-12 alternatives. The thioether compound, CF_3SCF_3, is expected to have a short atmospheric lifetime, however the acceptability of the atmospheric lifetime of these sulfur containing compounds remains to be investigated. Because these compounds are highly fluorinated they will likely form minimum boiling azeotropes with hydrocarbons and hydrocarbon derivatives with a low fluorine content.

The compound R-161 has a boiling point of -37.3°C and a critical temperature of 102.1°C. The critical pressure of 5046 kPa was obtained by extrapolating our measured data (Table 3) from 77°C to the critical temperature of 102.1°C using the parametric representation of the vapor pressure (Equation (2)) given in Table 6. The measured boiling point, critical temperature and critical pressure, (-37.3°C, 102.1°C and 5046 kPa) compare extraordinarily well with the literature values (-37.1°C, 102.16°C, 5028 kPa) of Wackher [7] and Booth et al. [8]. The boiling point and critical temperature are ideal for replacing R-22 which has a boiling point of -40.75°C and a critical temperature of 96°C. The high critical temperature of 102.1°C is particularly desirable and this contrasts most other R-22 replacements which generally have much lower critical temperatures than R-22.

The E-218 ether has a boiling point, -23.7°C that is suitable as an R-12 replacement however its critical temperature, 83.7°C, is low. It is nevertheless useful to examine mixtures containing E-218 because the exposed oxygen in highly fluorinated ethers give them greater solvent power than highly fluorinated hydrocarbon derivatives would have for HFCs with a low fluorine content.

III. Results for R-338mccq/isopentane, R-161/CF_3SF_5, and R-134a/CF_3SF_5 Mixtures.

The vapor pressure of the R-338mccq/isopentane mixtures were studied over the temperature range, 25°C to 120°C. Measurements were made at 5° temperature intervals and the data were fitted to Equation (2). The reduced temperature of Equation (2) for these mixtures was defined as,

$$T_r = T / T_c \quad , \tag{3}$$

$$T_c = XT_{c1} + (1 - X)T_{c2} \quad , \tag{4}$$

where X is the liquid phase mole fraction of R-338mccq, T_{c1} is the critical temperature of R-338mccq (160.5°C) and T_{c2} is the critical temperature of isopentane (187.8°C). The parametric representation of Equation (2) for the R-338mccq/isopentane mixtures is given

in Table 6. The mixture vapor pressures were also fitted to the following van Laar Equations by least squares methods described by Beyerlein et al. [2].

$$\gamma_1 = \exp\left[A\left(1 + \frac{AX}{B(1-X)}\right)^{-2}\right] , \tag{5}$$

$$\gamma_2 = \exp\left[A\left(1 + \frac{B(1-X)}{AX}\right)^{-2}\right] , \tag{6}$$

$$P = X\gamma_1 P_1^o + (1-X)\gamma_2 P_2^o \quad , \tag{7}$$

The least squares method utilizes vapor pressure data, the lever rule, pure component liquid densities [1,9] and vapor densities estimated using the modified corresponding states method [1,2]. The vapor phase mole fraction, y, is calculated from the above equations by,

$$y = \frac{XP_1^o\gamma_1}{P} \quad . \tag{8}$$

The results were ultimately used to correct the vapor pressure results in Table 6 to correspond to the liquid phase mole fraction. The corrections in all cases were less than 0.5%.

The least square results for the van Laar parameters are given in Table 7. The experimental vapor pressures and the results estimated from the van Laar analysis are plotted versus mole fraction for a representatiive temperature, 40°C, in Figure 1. The results indicate an azeotropic composition of 0.511 mole fraction at 20°C and the azeotropic concentration systematically shifts to 0.590 molefraction as the temperature is increased to 120°C. The boiling point of the mixtures of 15.1^{o}C is nearly 10°C below that of the pure components which is indicative of the very large deviations from ideality that are observed for mixtures of hydrocarbons with a highly fluorinated component.

The R-338mccq/isopentane mixtures exhibited a visible liquid-liquid phase separation at temperatures below -40°C which disappeared as the mixtures warmed above -10°C. The thermodynamic stability condition for the onset of a phase separation is given by,

$$\frac{\partial \ln \gamma_1}{\partial \ln X} \geq -1 \quad . \tag{9}$$

This stability condition can be rewritten in terms of the van Laar parameters with the following inequality,

$$2X(1-X)A^2B^2 \le [B+(A-B)X]^3 \tag{10}$$

The least squares fit of the van Laar parameters A and B to the reciprocal of the absolute temperature yields the following temperature dependence for the van Laar parameters,

$$A = -1.0758 + \frac{790.2}{T} \quad , \tag{11}$$

$$B = -1.4347 + \frac{959.6}{T} \quad , \tag{12}$$

By combining Equations (11) and (12) with inequality (10), one estimates the phase separation for the azeotropic composition 0.511 to begin at -5.º C for the R-338mccq/isopentane mixtures. The high phase separation temperature may reduce the desirability of these mixtures for refrigeration applications, but would not affect significantly their usefulness as industrial foam blowing agents.

The vapor pressures for the R-161/CF_3SF_5 and R-134a /CF_3SF_5 mixtures were measured as a function of temperature up to their normal boiling point and the parametric representation of the vapor pressures in terms of Eq. (1) is given in Table 2. Calculation of the van Laar parameters for R-161/CF_3SF_5 from these data indicates the critical mixing temperature is above -30°C for these mixtures. Since this is well above the normal boiling point (about -42°C), these mixtures may not be useful as an R-22 alternative.

The van Laar parameters were calculated using the vapor pressure data for the R-134a/CF_3SF_5 mixtures and these are given in Table 7. These mixtures have an azeotropic composition which estimated to be 0.600 mole fraction at -60°C and it shifts to 0.661 mole fraction as the temperature is increased to -20°C. From the temperature dependence of the van Laar parameters and the inequality (10) one estimates the onset of phase separation occurs at about -80°C. Since this is well below the boiling point of the these mixtures, -30.0^{o}C, phase separation should not be a factor in determining the potential of these mixtures as refrigerants. The boiling point of -30.0^{o}C indicates that these mixtures would be an excellent alternative to R-12 which has a boiling point of -29.8^{o}C.

Vapor pressures were also measured for R-143b (CF_3CH_3)/CF_3SF_5 mixtures which are 0.9 mole fraction in R-143b (CF_3CH_3). The R-143b has a fluorine content intermediate between R-161 and R-134a. Since these mixtures do not form an azeotrope, further investigations were not pursued at this time. The lack of an azeotropic composition may be explicable in terms of the large separation between the boiling points of the pure components (26.6°C) rather than in terms deviations from ideality. Additional investigations on this system are planned to obtain a complete picture of the mixing behavior of CF_3SF_5 with the various fluorinated ethane derivatives.

IV. Summary and Conclusions

The sulfur compounds, CF_3SF_5 and CF_3SCF_3, offer new possibilities for mixtures which are potential R-12 and R-22 alternatives. Mixtures of CF_3SF_5 with fluorinated ethanes of lower fluorine content, R-161 and R-134a, exhibited large deviations from ideality which is expected. Similarly large positive deviations from ideal mixing are obtained for highly fluorinated hydrocarbon derivatives with hydrocarbon derivatives of low fluorine content as is seen from the vapor pressure data on R-338mccq/isobutane mixtures. In such mixtures azeotropes are formed with a boiling point that is much lower than the pure component boiling points which can be a benefit in the search for R-22 replacements. These mixtures may also have a high critical mixing temperature and these must also be investigated to evaluate these mixtures for refrigeration applications.

Preliminary investigations of CF_3SCF_3 with R-161 indicate a minimum boiling azeotrope may occur at about 0.85 mole fraction with a normal boiling point of about -39°C. Unlike the R-161/CF_3SF_5 mixtures, the R-161/CF_3SCF_3 mixtures have a critical mixing temperature well below the boiling point. The R-161/CF_3SCF_3 mixtures and mixtures of fluorinated hydrocarbons with the ether compound, $CF_3OCF_2CF_3$, are the subject of future investigations.

List of Symbols

Roman

P = Vapor Pressure

T = Kelvin Temperature

T_c = Critical Temperature

$T_r = T/T_c$ = Reduced Temperature

X = liquid phase mole fraction

y = vapor phase mole fraction

(a,b,c) are parameters of the vapor pressure Eq. (1)

(A, B) are parameters of the van Laar Eqs. (7) and (8).

Greek

γ_i = activity coefficient of component I.

ρ_ℓ = liquid density

ρ_g = vapor density

$\bar{\rho} = (\rho_\ell + \rho_g)/2$ = diameter

(α,β,γ,δ) are parameters of the vapor pressure equation

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Table 1

Boiling Point, Heat of Vaporization and Critical Properties of Pure Compounds

ASHRAE Code	Chemical Formula	T_b(°C)	ΔH_{vap} (kJ/mol)	T_c(°C)	P_c (KPa)	ρ_c (Kg/m^3)
Pure Compounds						
------	CF_3SF_5	-20.3	20.18	108.1	3371	690
------	CF_3SCF_3	-21.9	21.33	103.6	--------	--------
R-161	CH_3CH_2F	-37.3	21.18	102.1	5046	302
E-218	$CF_3OCF_2CF_3$	-23.7	22.27	83.7	--------	640

Table 2

Vapor Pressure of Pure Chemicals Below the Boiling Point

R-161 CH_3CH_2F		E-218 $CF_3OCF_2CF_3$		E-143 CF_3OCH_3		CF_3SF_5		CF_3SCF_3	
Temp. (°C)	Press. (kPa)	Temp. (°C)	Press. (kPa)	Temp. (°C)	Press. (kPa)	Temp. (°C)	Press. (kPa)	Temp. (°C)	Press. (kPa)
-72.6	14.3	-61.0	14.9	-75.8	8.4	-49.8	23.6	-47.9	31.5
-68.8	18.5	-58.4	17.6	-69.9	12.3	-47.4	27.6	-45.0	36.0
-65.4	23.1	-56.1	20.1	-65.4	16.0	-45.3	31.3	-42.6	40.4
-62.7	27.2	-54.0	22.7	-61.5	19.7	-43.5	34.8	-40.0	45.7
-60.4	31.1	-52.3	24.9	-58.3	23.5	-41.7	38.3	-37.8	50.7
-58.3	35.1	-50.7	27.2	-55.0	27.6	-40.1	41.7	-35.4	56.3
-56.4	39.2	-49.3	29.6	-52.4	31.7	-38.9	44.7	-33.5	61.3
-54.6	43.2	-46.0	35.7	-49.7	36.3	-37.5	47.6	-31.8	66.5
-53.0	47.1	-45.0	37.2	-47.3	40.6	-36.2	50.5	-29.9	72.4
-51.3	51.5	-43.9	39.3	-45.2	44.8	-35.2	53.1	-28.2	78.0
-49.7	56.1	-42.9	41.5	-42.8	50.0	-34.2	55.7	-26.2	84.9
-48.3	60.4	-42.0	43.3	-40.8	55.1	-33.2	58.4	-24.9	89.9
-47.0	64.4	-41.1	45.3	-38.6	60.9	-32.1	61.3	-23.7	94.3
-45.8	68.5	-40.2	47.5	-36.8	65.5	-30.7	65.6	-22.6	98.5
-44.5	72.7	-39.3	49.6	-34.9	70.0	-29.9	68.0		
-43.4	76.7	-38.4	51.7	-32.8	77.1	-29.4	69.7		
-42.3	80.8	-37.6	53.9	-31.5	82.1	-28.9	71.3		
-41.1	85.6	-36.8	55.9	-29.9	87.7	-27.9	74.1		
-40.1	89.9	-36.0	58.4	-27.9	93.7	-27.1	76.7		
-38.9	94.8	-34.4	62.7	-26.6	98.9	-26.4	79.5		
-38.3	97.5	-33.5	65.3			-25.5	82.1		
		-32.7	68.3			-24.8	84.8		
		-31.6	71.5			-24.0	87.2		
		-30.4	75.1			-23.2	90.8		
		-29.5	78.5			-22.2	94.4		
		-28.5	85.5			-21.5	97.5		
		-25.7	92.5						
		-24.4	98.0						

Table 3

Vapor Pressure of R-161 and CF_3SF_5 between the Boiling Point and the Critical Temperature

R-161 CH_3CH_2F		**CF_3SF_5**	
Temp. (°C)	Pressure (kPa)	Temp. (°C)	Pressure (kPa)
15.65	738.1	15.24	380.1
20.00	833.3	20.25	443.3
25.16	959.3	25.16	511.8
30.08	1093.1	30.19	590.6
35.08	1242.4	35.11	675.6
40.17	1410.2	40.18	773.1
45.22	1593.1	45.24	880.1
50.02	1782.6	50.08	992.1
55.27	2006.0	55.15	1118.6
60.17	2228.9	60.16	1255.9
65.18	2444.8	64.93	1401.1
70.06	2739.8	70.33	1576.2
75.55	3053.9	75.26	1750.5
77.71	3196.5	80.20	1954.2
		85.25	2162.2
		90.28	2411.3
		95.30	2661.8
		100.26	2925.3
		105.32	3170.4

Table 4

Liquid Densities and Diameters for R-161,CF_3SF_5 and E-218.

R-161 (CH_3CH_2F)			**(CF_3SF_5)**			**E-218 ($CF_3OCF_2CF_3$)**		
Temp. (°C)	Diam. (kg/m^3)	Liquid Density (kg/m^3)	Temp. (°C)	Diam. (kg/m^3)	Liquid Density (kg/m^3)	Temp. (°C)	Diam. (kg/m^3)	Liquid Density (kg/m^3)
22.8	369.9	750.1	21.1	817.8	1563.5	29.3	717.3	1382.1
30.8	363.1	732.3	30.2	804.5	1525.9	35.1	707.4	1351.0
40.4	354.9	699.9	40.0	790.2	1482.5	39.4	700.1	1323.7
50.4	346.3	678.1	49.6	776.2	1435.2	45.2	690.2	1287.8
60.7	337.5	639.2	60.6	760.1	1375.9	50.0	682.0	1254.2
73.1	326.9	595.8	70.1	746.2	1313.4	54.0	675.2	1224.3
			79.3	732.8	1246.4	58.5	667.5	1191.9

Table 5

Parametric Representation of the Vapor Pressure
of Pure Chemicals and Mixtures Below the Boiling Point

Pure Chemicals					
Compound or Mole Fraction of (1)	a	b	c	Boiling Point (^oC)	% Deviation
CF_3SF_5	158.745	-22.1236	-8021.2	-20.3	0.5
CF_3SCF_3	14.8290	0.0	-2565.7	-21.9	0.4
CH_3CH_2F	51.4282	-5.6558	-3738.4	-38.7	0.3
$CF_3OCF_2CF_3$	47.3253	-4.9462	-3844.7	-23.6	0.4
CF_3OCH_3	32.8393	-2.8665	-3071.1	-26.0	0.6
R-161(1)/CF_3SF_5(2)Mixtures					
1.0	65.3707	-7.7700	-4315.8	-37.3	0.2
0.84	145.4160	-20.3979	-6870.9	-40.7	0.4
0.75	104.075	-13.8632	-5546.3	-42.1	0.4
0.60	52.1279	-5.6940	-3813.1	-42.4	0.4
0.20	34.4388	-3.2318	-2950.0	-28.2	0.5
0.0	158.7455	-22.1236	-8021.2	-20.3	0.5
R-134a(1)/CF_3SF_5(2) Mixtures					
1.0	51.1894	-5.3992	-4162.7	-25.6	0.6
0.85	98.1303	-12.7688	-5695.2	-29.0	0.4
0.50	48.7050	-5.0970	-3905.3	-30.5	0.4
0.2	52.2384	-5.7124	-3983.3	-26.7	0.3
R-143(1)/CF_3SF_5(2)					
1.0	63.1074	-7.4482	-4096.0	-46.9	0.2
0.9	51.7835	-5.6743	-3717.2	-46.3	0.4

Table 6

Parametric Representation of the Vapor Pressure of R-338mccq/isopentane mixtures for Temperatures Ranging from 25ºC to 120ºC.

Chemical or Mole Fraction (1)	α	β	γ	δ	% RMS Dev.
Pure Compounds					
R-161	13.1958	−4.6471	2.0678	−0.02231	0.5
CF_cSF_5	16.3152	−8.3712	−2.3649	0.1790	0.5
R-338mccq(1)/Isopentane(2) Mixtures					
1.0	15.1978	−7.0069	1.1637	−0.3147	0.4
0.905	12.6382	−4.5621	2.9881	0.0	1.0
0.801	18.5263	−10.7521	−5.7169	0.2727	0.8
0.703	14.5491	−6.2233	0.9849	−0.1995	0.2
0.516	17.8642	−10.1317	−5.4281	0.7424	0.5
0.398	17.0001	−9.1961	−4.2905	0.69719	1.0
0.294	14.1456	−5.6399	1.4998	−0.2939	0.3
0.150	11.9476	−3.4456	4.2300	−0.2054	0.3
0.050	12.0474	−3.3644	4.8385	−0.6802	0.4
0.0	11.4267	−2.4862	6.8235	−1.1221	0.6

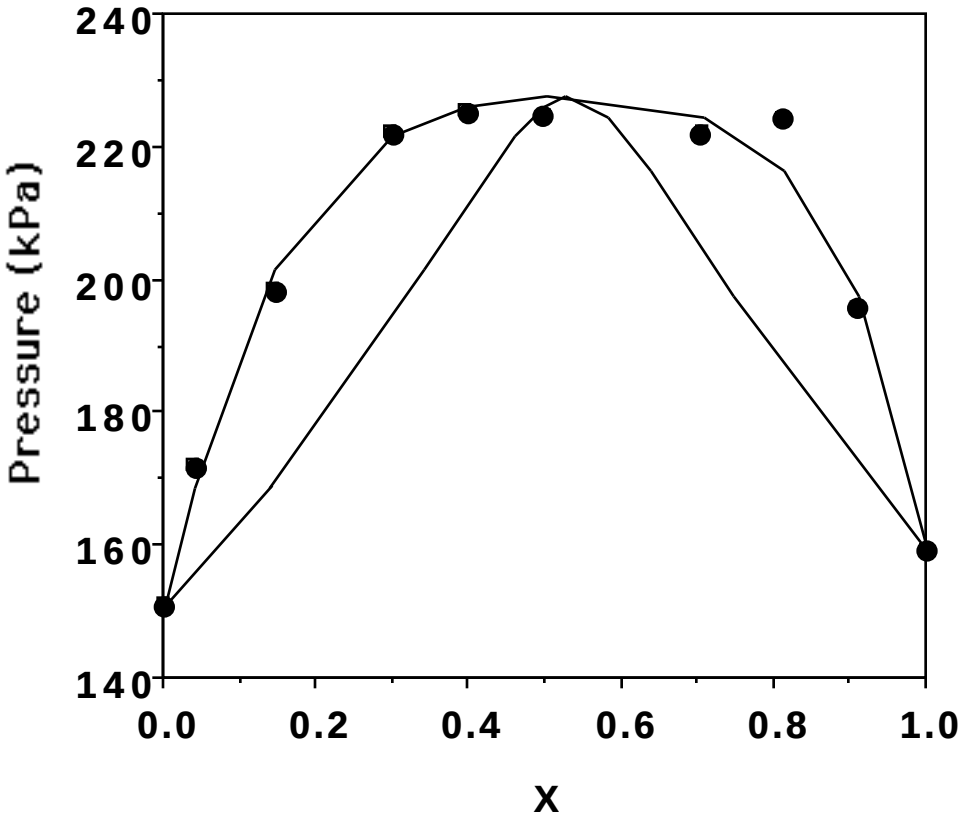
Table 7

Results of the van Laar Analysis of the Mixture Vapor Pressure Data

Temp. (ºC)	van Laar Parameters		Azeotropic Mole Fract.	Azeotropic Pressure	% RMS Deviation
	A	B			
R-338mccq(1)/Isopentane(2) Mixtures					
20.0	1.653	1.871	0.511	120.3	2.7
40.0	1.457	1.617	0.525	228.1	1.7
60.0	1.262	1.423	0.546	401.9	1.9
80.0	1.107	1.256	0.565	665.0	1.4
100.0	1.015	1.122	0.580	1042.2	0.9
120.0	1.007	1.050	0.590	1562.0	2.1
Normal Boiling Point at the Azeotropic Mole Fraction (0.511) = 15.1ºC					
R-134a(1)/CF_3SF_5(2) Mixtures					
-60.0	1.433	1.870	0.600	20.5	2.8
-50.0	1.170	1.770	0.611	37.3	2.0
-40.0	0.980	1.681	0.629	63.3	2.1
-30.0	0.879	1.539	0.649	101.4	2.6
-20.0	0.849	1.358	0.661	154.3	3.6
Normal Boiling Point at the Azeotropic Composition (0.63) = -30ºC					

Figure Captions

Figure 1. Vapor Pressure of R-338mccq/isopentane mixtures at 40°C. The points and upper solid line represents vapor pressure vs. liquid mole fraction and the lower solid line represents vapor pressure vs. vapor mole fraction.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160
140
Pressure (kPa)
0.0
0.2
0.4
0.6
0.8
1.0
x