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Double Azeotropy in Binary Mixtures 1,1,1,2,3,4,4,5,5,5-Decafluoropentane High-Boiling Diastereomer + Tetrahydrofuran

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Pure component vapor pressures at 293 K to 328 K and binary vapor−liquid equilibrium (VLE) at 68.93 kPa have been measured for the two diastereomers of 1,1,1,2,3,4,4,5,5,5-decafluoropentane (HFC-43-10mee) using an ebulliometer. VLEs have been measured as a function of overall composition for binary mixtures of the high-boiling diastereomer of HFC-43-10mee + tetrahydrofuran (THF) at 26.69 kPa and 45.51 kPa using an ebulliometer. The VLE data were correlated using the NRTL equation. The coexistence curves for HFC-43-10HB + THF at 45.51 kPa exhibit a maximum and a minimum in temperature, establishing the existence of double azeotropes at this pressure.

Introduction

Binary double azeotropy is an unusual vapor−liquid equilibrium (VLE) phenomenon where two azeotropes exist at a given temperature or pressure. Double azeotropes have been found for five organic binary mixtures: benzene and hexafluorobenzene,[1,2] diethylamine and methanol,[3,4,5] acetic acid and isobutyl acetate,[6,7] ammonia and pentafluoroethane or HFC-125,[8] and 1,1,1,2,3,4,4,5,5,5-decafluoropentane (HFC-43-10mee) and THF.[9,10] Leu and Robinson[11] reported that 1,2-butylene oxide and methyl acetate exhibited two azeotropes at 298.15 K. However, Monton et al.[12] measured the VLE for the same system at 35 and 101.3 kPa and at 298.15 K and found no azeotropes. Monton et al.[12] claimed the data of Leu and Robinson[11] were in error because of impurities in the reagents and experimental errors.

HFC-43-10mee (CASN, 138495-42-8) exists as two diastereomers, the erythro diastereomer (a mixture of RS and SR enantiomers; CASN, 142347-08-8) and the threo diastereomer (a mixture of RR and SS enantiomers; CASN, 142347-07-7).[13] The sawhorse projections of the four stereoisomers are shown in Figure 1. Commercially available HFC-43-10mee consists of a mixture of about 90 % threo and 10 % erythro diastereomer. The vapor pressures of the two diastereomers were found to be significantly different from each other at one of DuPont plants after our previous work had been published. Since previous work from this laboratory established the existence of double azeotropy between tetrahydrofuran (THF) and the HFC-43-10mee diastereomeric mixture, we were interested in exploring the VLE of the individual diastereomers with THF. Vapor pressures for both diastereomers were measured over a limited temperature range using an ebulliometer. Binary VLE data for two diastereomers of HFC-43-10mee were measured at 68.93 kPa; binary VLE data for the threo or the high-boiling diastereomer (HFC-43-10HB) and THF were measured at 26.69 kPa and 45.51 kPa. The data were measured using two ebulliometers and correlated using the NRTL equation. The coexistence curves for HFC-43-10HB and THF at 45.51 kPa exhibited a maximum and a minimum in temperature, establishing the existence of double azeotropes at this pressure.

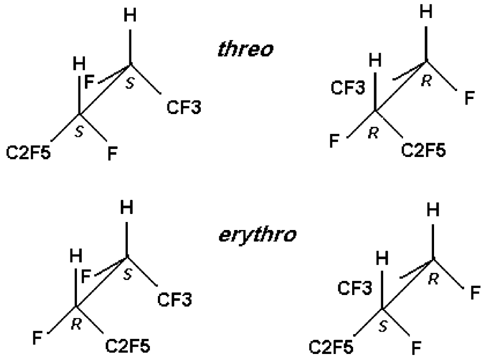

Figure 1. Sawhorse projections of the stereoisomers of HFC-43-10mee.

Experimental Section

Chemicals. Tetrahydrofuran (THF) with a purity > 99.9% was purchased from Aldrich. It was used without further purification. HFC-43-10mee or Vertrel XF was obtained from DuPont. The two diastereomers of HFC-43-10mee were separated and purified by distillation. The threo or high-boiling diastereomer (HFC-43-10HB) and the erythro or low-boiling diastereomer (HFC-43-10LB) had purities of about 99.89% and 99.06%, respectively. The purification and analysis of the diastereomers are given in the appendix.

Apparatus and Procedures. An ebulliometer measures equilibrium temperature for a pure compound or a mixture at a controlled pressure. Two ebulliometers were used for this work; one was the 221-cm^3 boiler described in our previous paper;[9] the other one (Figure 2) was a 129-cm^3 boiler with 0.2-cm^3 liquid holdup. Less materials were needed to use the 129-cm^3 boiler, also better mixing between the liquid and condensate was obtained because its capillary return line was extended into the liquid phase. The pressure of the boiler was kept constant by a Mensor PCS 400 pressure controller with 0.01% or 0.01 kPa

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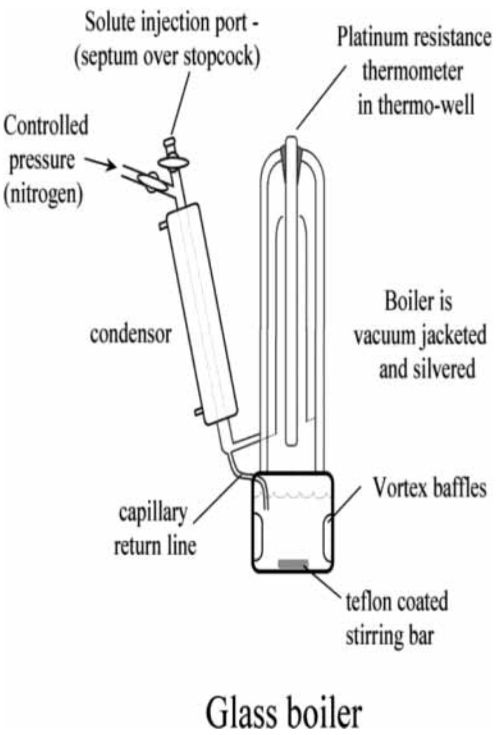

Figure 2. Schematic diagram for the 129-cm^3 boiler.

accuracy and was measured using a Paroscientific 740-15A pressure transducer with 0.01% or 0.01 kPa accuracy. The temperature was measured using a Hart Scientific Black Stack Base Unit with 0.007-K accuracy and a secondary PRT with 0.01-K accuracy (ITS-90 scale).

To measure binary VLE, one of the two components of known mass was added to the boiler and the equilibrium temperature was measured at a controlled pressure; successive isobaric equilibrium temperatures were measured over roughly one-half of the composition range by injecting known masses of the second component into the boiler. Equilibrium temperatures over the other half of the composition range were obtained by repeating the procedure starting with the other pure compound. The mass was measured to 0.0001 g for an addition less than 10 cm^3 and to 0.001 g for a larger addition. The estimated composition uncertainty was ±0.0001.

Data Reduction

The overall mixture composition, calculated on the basis of known loadings of the two components, was converted to equilibrium vapor- and liquid-phase compositions by accounting for the small amount of each component in the vapor phase and in liquid holdup as described by Kao et al.[9] and summarized below. The material balance equations for reduction of data measured by an ebulliometer are

$$n^{L} + n^{V} + n^{cond} = n_1 + n_2 \tag{1}$$

$$n_1 = x_1 n^{L} + y_1 n^{V} + y_1 n^{cond} \tag{2}$$

$$V_T = n^{L} v^{L} + n^{V} v^{V} + V^{cond} \tag{3}$$

where n_i is the number of moles of component i loaded to the boiler, n^{α} is the total number of moles in phase α, v^{α} is the molar volume of phase α, x_1 and y_1 are the liquid and vapor mole fractions of component 1, V^{cond} is the volume of liquid holdup, and V_T is the total volume of the boiler. An expression for the number of moles in the liquid phase is derived from eqs 1 and 3

$$n^{L} = [(n_1 + n_2 - n^{cond})v^{V} + V^{cond} - V_T]/(v^{V} - v^{L}) \tag{4}$$

v^{V}, v^{L}, and n^{cond} (number of moles in liquid holdup) are calculated as

$$v^{V} = RT/P \tag{5}$$

$$v^{L} = x_1 v_1{}^{L} + x_2 v_2{}^{L} \tag{6}$$

$$n^{cond} = V^{cond}/(y_1 v_1{}^{L} + y_2 v_2{}^{L}) \tag{7}$$

where R is the gas constant and $v_i{}^{L}$ is the liquid molar volume of pure component i.

The expression for the mole fraction of component 1 in the liquid phase is derived from eqs 1 and 2

$$x_1 = [n_1 - (n_1 + n_2 - n^{L})y_1]/n^{L} \tag{8}$$

A solution for x_1 is found at the pressure of interest by a conventional VLE calculation in which the equilibrium temperature and y_1 are first determined from the pressure and an initial guess for x_1. In this calculation, the ideal gas law is used for vapor-phase fugacity calculations and the NRTL (nonrandom two-liquid) equation[14] is used for liquid-phase fugacity calculations. The NRTL parameters are obtained by minimizing $[1/N \sum (T_{calc} - T)^2]^{1/2}$.

Results and Discussion

Vapor Pressures for Pure Components. Several vapor pressure data for pure THF, HFC-43-10HB, and HFC-43-10LB were measured at the same time as the binaries using the same ebulliometer. The measured vapor pressure for THF agreed well with the values calculated using the Antoine equation,[15] which is also given below as eq 9

$$\ln P/\text{kPa} = 14.0919 - 2768.38/(T/\text{K} - 46.9) \tag{9}$$

The measured vapor pressures for HFC-43-10HB and HFC-43-10LB were well correlated using

$$\ln P/\text{kPa} = 14.0522 - 2474.73/(T/\text{K} - 66) \tag{10}$$

and

$$\ln P/\text{kPa} = 14.1190 - 2519.337/(T/\text{K} - 55) \tag{11}$$

respectively. Table 1 gives comparisons of the measured and calculated vapor pressures for THF and two diastereomers of HFC-43-10mee. No vapor pressure data for diastereomers have been published before. The vapor pressure for HFC-43-10LB or the erythro diastereomer is 46.7% and 37.8% higher than the vapor pressure for HFC-43-10HB or the threo diastereomer at 293.15 K and 313.15 K, respectively. Such a huge vapor pressure difference is rarely found between stereoisomers. This unusually huge vapor pressure difference might be attributed to different dipole moments resulting from different geometries. The low-boiling diastereomer should only have a very small dipole moment, while the high-boiling diastereomer should have quite a large dipole moment. To the best of our knowledge, 2H,3H-octafluorobutane (HFC-338mee) is the only other reported component in the literature, which exists as diastereomers of very different volatilities. The normal boiling points for dl and meso diastereomers of HFC-338mee reported by ref 16 are 41 °C and 25 °C, respectively. In contrast to HFC-43-10mee and HFC-338mee, the dl and meso diastereomers of 1,2-dichloro-1,2-

Table 1. Vapor Pressure for Pure Components

T/K	P/kPa	P_{calc}/kPa	$(P_{calc} - P)/P$
		THF	
303.05	26.68	26.69	0.0003
305.11	29.09	29.09	0.0000
309.28	34.50	34.50	−0.0001
313.11	40.15	40.14	−0.0001
318.10	48.62	48.62	−0.0001
303.04	26.67	26.68	0.0002
316.36	45.51	45.52	0.0002
		4310HB	
295.82	26.69	26.69	0.0001
303.17	37.25	37.25	0.0001
305.11	40.56	40.55	−0.0002
313.05	56.54	56.55	0.0001
318.04	68.94	68.95	0.0002
323.24	84.10	84.09	−0.0001
327.95	99.96	99.97	0.0001
		4310LB	
293.17	34.49	34.51	0.0005
302.95	52.41	52.37	−0.0007
309.84	68.93	68.93	−0.0001
313.03	77.90	77.89	−0.0001
319.81	99.95	100.01	0.0005

Table 2. VLE Temperatures and Phase Compositions for HFC-43-10HB (1) + HFC-43-10LB (2) at 68.93 kPa

x_2	$y_{2,calc}$	T/K	T_{calc}/K	$(T_{calc} - T)$/K
0.0000	0.0000	318.04	318.03	−0.009
0.0941	0.1339	316.85	316.84	−0.012
0.1522	0.2076	316.19	316.20	0.011
0.1104	0.1551	316.71	316.66	−0.054
0.1511	0.2062	316.26	316.21	−0.047
0.2392	0.3093	315.33	315.34	0.008
0.3241	0.4009	314.52	314.58	0.062
0.4094	0.4877	313.76	313.88	0.121
0.5501	0.6220	312.91	312.82	−0.088
0.5933	0.6616	312.57	312.51	−0.057
0.6596	0.7208	312.08	312.05	−0.027
0.7484	0.7976	311.45	311.45	0.005
0.8195	0.8571	310.97	310.99	0.019
0.8957	0.9188	310.48	310.50	0.020
0.9586	0.9683	310.09	310.10	0.012
1.0000	1.0000	309.84	309.84	0.004

difluoroethane (CFC-132) have very similar volatilities; their normal boiling points reported in ref 17 are 59.4 °C and 59.9 °C, respectively.

Equations 9−11 were used to reduce the binary isobaric data. It is not recommended to use eqs 10 and 11 for HFC-43-10HB and HFC-43-10LB outside the limited temperature range measured for this work.

HFC-43-10HB/HFC-43-10LB at 68.93 kPa. The reduced NRTL parameters for HFC-43-10HB/HFC-43-10LB at 68.93 kPa are $\alpha_{12} = 0.44$, $\tau_{12}T = 205.22$ K, $\tau_{21}T = -136.72$ K. The measured and calculated VLE temperatures and phase compositions are given in Table 2 and are shown in Figure 3. The two diastereomers of HFC-43-10mee form a fairly ideal mixture, as expected. Knowing the vapor pressures and NRTL parameters for HFC-43-10HB/HFC-43-10LB, the HFC-43-10mee used by Kao et al.[9] to discover the double azeotropy in HFC-43-10mee/THF was found to contain 89% HFC-43−10HB and 11% HFC-43-10LB. Table 3 compares the vapor pressures for HFC-43-10mee measured by Kao et al.[9] to the pressures calculated for a mixture of 89% HFC-43-10HB and 11% HFC-43-10LB.

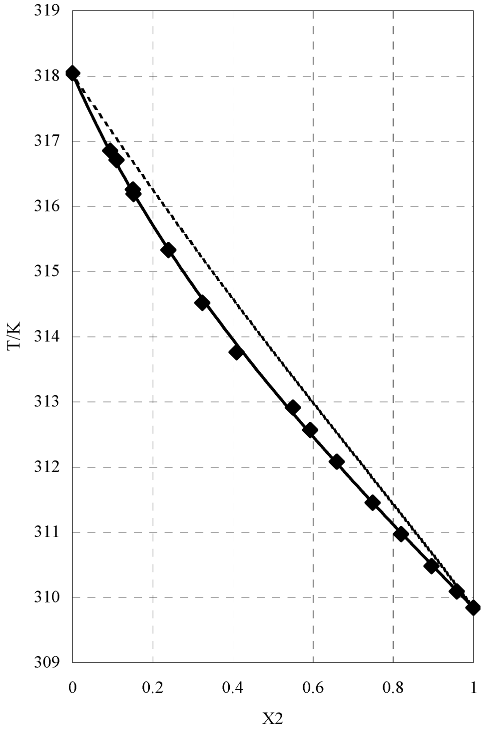

Figure 3. Vapor−liquid equilibrium temperatures and phase compositions for HFC-43-10HB (1) + HFC-43-10LB (2) at 68.93 kPa. The symbols denote measured temperatures; the solid and broken lines are the calculated liquid and vapor coexistence curves, respectively.

Table 3. Comparison between Measured[9] and Calculated Vapor Pressure for HFC-43-10mee

	T/K	P/kPa	P_{calc}/kPa	$(P_{calc} - P)/P$
4310mee	294.44	26.68	26.66	−0.0006
89%HB	303.60	40.34	40.29	−0.0011
11%LB	312.52	58.58	58.50	−0.0014
	325.44	95.86	95.96	0.0011

Table 4. VLE Temperatures and Phase Compositions for HFC-43-10HB (1) + THF (2) at 26.69 kPa

x_2	$y_{2,calc}$	T/K	T_{calc}/K	$(T_{calc} - T)$/K
0.0000	0.0000	295.82	295.82	−0.001
0.0523	0.0199	296.57	296.58	0.004
0.1010	0.0419	297.31	297.32	0.004
0.1844	0.0895	298.62	298.66	0.039
0.2646	0.1493	299.91	299.99	0.075
0.3640	0.2462	301.57	301.60	0.030
0.4411	0.3396	302.73	302.71	−0.025
0.5031	0.4248	303.49	303.43	−0.057
0.5653	0.5161	304.01	303.96	−0.052
0.6153	0.5910	304.22	304.21	−0.013
0.6440	0.6333	304.26	304.28	0.016
0.6999	0.7124	304.20	304.27	0.070
0.9629	0.9666	303.08	303.05	−0.033
0.9826	0.9827	303.05	303.03	−0.023
1.0000	1.0000	303.04	303.05	0.006

HFC-43-10HB/THF at 26.69 kPa and 45.51 kPa. THF was added successively to pure HFC-43-10HB at 26.69 kPa (the pressure at which the double azeotropy was found in HFC-43-10mee and THF) until the temperature passed the maximum. Then HFC-43-10HB was added to pure THF in two small increments at the same pressure, and the temperature increased slightly. The data were reduced using the NRTL equation and the ideal gas law. The measured and calculated VLE temperatures and phase compositions are given in Table 4 and are shown in Figure 4. The reduced NRTL parameters are $\alpha_{12} = 0.48$, $\tau_{12}T = -438.8$ K, $\tau_{21}T = 814.49$ K. The calculated coexistence curves exhibit a tiny minimum in temperature at approximately 98 mol % of THF in addition to a maximum in temperature at approximately 67 mol % of THF. The calculated temperature minimum was not observed experimentally; however, the difference between measured and calculated temperature at THF end was too small to

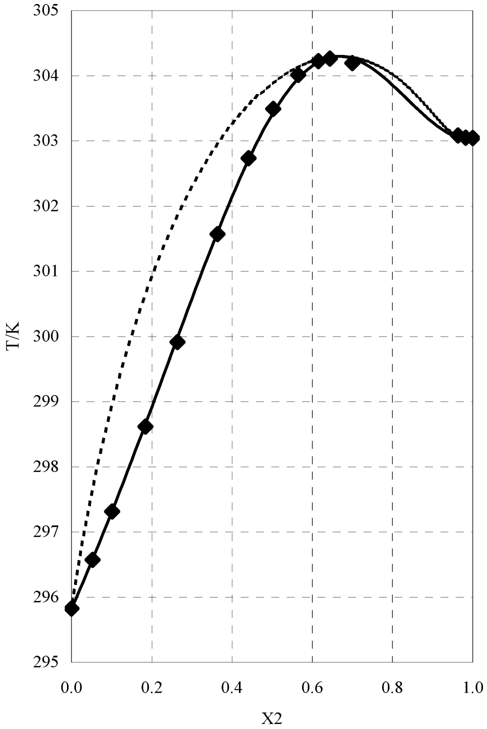

Figure 4. VLE temperatures and phase compositions for HFC-43-10HB (1) + THF (2) at 26.69 kPa. The symbols denote measured temperatures; the solid and broken lines are the calculated liquid and vapor coexistence curves, respectively.

conclude whether double azeotropes really existed at this pressure. Measurements at a higher pressure were needed to establish the existence of double azeotropes for HFC-43-10HB/THF.

The VLE experiment was next conducted at 45.51 kPa, which was chosen because prominent double azeotropes were predicted at this pressure by using the NRTL equation with parameters reduced from data at 26.69 kPa. THF was added successively to HFC-43-10HB until the temperature passed the maximum. The other half of the isobar was measured twice by adding HFC-43-10HB to pure THF. A temperature minimum at approximately 93 mol % of THF was observed for both runs. One of the runs covered from pure THF to 69.8 mol % of THF and overlapped with part of the data from the first half of the isobar. The data at 45.51 kPa clearly show that there is a maximum and a minimum in temperature and establish the existence of double azeotropes at this pressure. The data were reduced using the NRTL equation and the ideal gas law. The measured and calculated VLE temperatures and phase compositions are given in Table 5 and are shown in Figure 5. The reduced NRTL parameters are $\alpha_{12} = 0.48$, $\tau_{12}T = -422.616$ K, $\tau_{21}T = 761.62$ K. The calculated azeotropes are $x_2 = 0.7218$ at 316.58 K and $x_2 = 0.9445$ at 316.22 K. Both azeotropes are concentrated in THF the same as the double azeotropes found for HFC-43-10mee and THF at 26.68 kPa. Like the double azeotropes for HFC-43-10mee and THF, the double azeotropes for HFC-43-10HB and THF exist at a pressure for which the equilibrium temperatures of the two components are notably different and they only cover a narrow temperature and composition range at this pressure.

The double azeotropes for HFC-43-10HB + THF disappear at lower pressures due to the disappearance of the minimum temperature azeotrope as shown by the data at 26.69 kPa. The double azeotropes expect to disappear at higher pressures due to the collapse of two azeotropes to a tangent azeotrope and then no azeotrope at all as shown by the data for HFC-43-10mee + THF at 58.58 kPa by Kao et al.[9] The NRTL model predicts that there is no minimum-temperature azeotrope at about 13.8 kPa and that the tangent azeotrope exists at about 62 kPa.

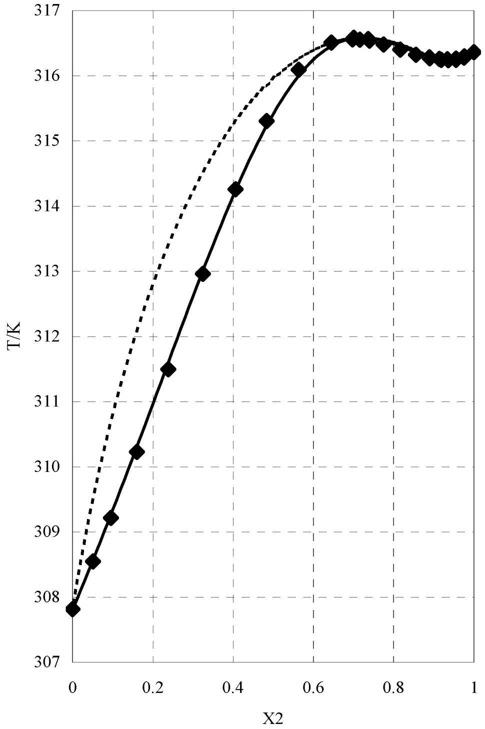

Figure 5. VLE temperatures and phase compositions for HFC-43-10HB (1) + THF (2) at 45.51 kPa. The symbols denote measured temperatures; the solid and broken lines are the calculated liquid and vapor coexistence curves, respectively.

Table 5. VLE Temperatures and Phase Compositions for HFC-43-10HB (1) + THF (2) at 45.51 kPa

x_2	$y_{2,\mathrm{calc}}$	T/K	T_{calc}/K	$(T_{\mathrm{calc}} - T)$/K
0.0000	0.0000	307.82	307.81	−0.006
0.0510	0.0213	308.55	308.57	0.019
0.0957	0.0431	309.21	309.26	0.050
0.1602	0.0804	310.23	310.30	0.076
0.2385	0.1365	311.50	311.60	0.103
0.3245	0.2134	312.96	313.00	0.043
0.4066	0.3026	314.26	314.24	−0.016
0.4840	0.3994	315.31	315.24	−0.070
0.5634	0.5076	316.09	316.00	−0.092
0.6446	0.6203	316.51	316.45	−0.056
0.7007	0.6949	316.58	316.58	−0.009
0.7366	0.7400	316.56	316.58	0.017
0.6979	0.6914	316.56	316.57	0.015
0.7158	0.7142	316.56	316.58	0.028
0.7400	0.7441	316.54	316.58	0.042
0.7753	0.7852	316.48	316.54	0.055
0.8166	0.8295	316.40	316.45	0.051
0.8553	0.8671	316.32	316.36	0.032
0.8899	0.8980	316.27	316.28	0.010
0.9188	0.9227	316.24	316.23	−0.009
0.9362	0.9374	316.24	316.22	−0.018
0.9552	0.9539	316.24	316.22	−0.025
0.9755	0.9729	316.28	316.25	−0.025
1.0000	1.0000	316.36	316.36	−0.004
0.8890	0.8972	316.28	316.28	−0.003
0.9136	0.9182	316.26	316.24	−0.021
0.9336	0.9351	316.25	316.22	−0.034
0.9553	0.9540	316.26	316.22	−0.043
0.9763	0.9738	316.30	316.25	−0.045
1.0000	1.0000	316.38	316.36	−0.025

Depending on the availability of HFC-43-10LB it is planned to have the VLE between HFC-43-10LB and THF measured in the future. Since the temperature difference between HFC-43-10LB and THF is larger than the difference between HFC-43-10HB and THF at the same pressure, it is less likely to find double azeotropes in HFC-43-10LB and THF.

Acknowledgment

The careful experimental work of Mr. David Guntrum, who assisted with the measurements, is greatly appreciated.

Appendix

A 5-L distillation flask containing a thermowell was charged with 3 498 g of commercial HFC-43-10mee. The flask was heated by an electric heating mantle controlled by a variac. The distillation flask was connected to a 152-cm vacuum-jacketed column (inner diameter about 12 mm) containing stainless steel Helipak B packing. Vapor reaching the top of the column was condensed by a coil in the distillation head containing a recirculating ethylene glycol/water mixture chilled to 5 °C. Head vapor temperature was measured by means of a thermocouple in a small thermowell. Distillate was collected by lifting a valve in the liquid return line via a magnet surrounded by a coil. The coil was energized using an adjustable Flexopulse timer, which was set the reflux ratio. The distillation was conducted at atmospheric pressure.

Distillation fractions were analyzed by gas chromatography using a 105-m Rtx-1 capillary column (Crossbonded dimethylpolysiloxane, 0.25 mm i.d. × 1.0 mm d.f., Cat. No. 10159, Restek Corp., Bellefonte, PA) with flame-ionization detection. The GC temperature program was $T_i = 40$ °C/10 min, ramp at 10 °C/min, $T_f = 200$ °C/50 min.

The threo diastereomer of HFC-43-10mee (1,142 g) was isolated at a head temperature of 55.6−55.8 °C; the purity was 99.89%. Early fractions rich in the erythro diastereomer were combined and redistilled three times to give the erythro diastereomer (118 g) at a head temperature of 47.6−47.7 °C; the purity was 99.06%.

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