

Recent Developments in the Design of New Refrigerant Mixtures for Existing and Future Systems

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Introduction

The primary approach industry has taken to fill the void resulting from the elimination of the most important CFC's (i.e., CFC-11 and CFC-12) has been to develop other single component refrigerants (i.e., HCFC-123 and HFC-134a) which have very low or zero ozone depletion potential. These alternatives were attractive mostly because they had similar pressure-temperature property profiles relative to that which they were replacing and, thus, required minimal machinery design changes for adoption.

No such single component alternative has been discovered for HCFC-22 or CFC-502. As a result, the industry, with its needs to meet the various phase out dates internationally agreed upon, has begun an intensive effort to tailor various zeotropes (a.k.a. nonazeotropes) and, whenever possible, azeotropes to act as "drop-in" alternatives in existing equipment as well as the long term working fluids for new equipment. (Actually no chlorine-free refrigerant can be a true drop-in since a new lubricant is required.) Concern for the zeotrope's major disadvantage, that of unknown and unwanted composition shifting during leaking and charging situations, has caused the industry to focus only on those zeotropes with minimal temperature and composition shifts or glides during phase change. These minimal glide zeotropes have become known as "near-azeotropes" and behave in an "azeotrope-like" manner. Although no formal definition of "near-azeotrope" has been quantified, sufficient research has been conducted to know that a temperature glide greater than 1 °C will have a negative effect on the performance of a flooded shell and tube chiller. On the other hand, the true zeotropic benefits (i.e., Lorenz cycle glide matching through counter or cross-counter flow heat exchange) are usually not significant enough to redesign the system unless the glide is of the order of 5 °C or more. Thus, the boundary between near-azeotropes and zeotropes is probably between these two temperatures, depending on the machinery system and application. The most important mixtures currently being evaluated by the refrigeration machinery manufacturers are listed in Tables A1 and A2 of the appendix.

Mixtures offer the possibility to formulate a nonflammable mixture while using a flammable component. To achieve this, it is only necessary to maintain the mixture's composition such that the total amount of flammable component is less than that quantity that would be flammable in air. For some mixtures even this amount may be exceeded since there appears to be some flame quenching effect with some of the chlorine-free non-flammable components that are being considered. Thus, the use of limited amounts of the flammable fluids HFC-32 and HFC-143a, each of which have rather desirable thermodynamic properties, has been made possible. Such

developments and related terminology have led to both considerable opportunities and misunderstandings, so it is important to review the fundamentals of mixtures.

Fundamentals of Mixtures

An azeotrope is a mixture of fluids which has the same composition in the vapor phase and liquid phase, when in equilibrium. A binary azeotropic mixture is relatively unusual and a ternary or higher order azeotrope is rare indeed. In the past, at least seven binary azeotropes formed by halogenated hydrocarbons have been developed to the stage of commercialization in refrigeration machinery and at least three (i.e., CFC-500, CFC-502, CFC-503) are still in use. Several others are known but are of no particular advantage. Figure 1 is a graphical representation of the CFC-502 azeotrope which exemplifies the characteristics required of an azeotrope. The azeotropic condition exists at only one composition point in the two phase region for a given temperature and pressure. The mixture's vapor pressure at the azeotropic point must be either lower or higher than that of both of the components; in fact, at the maximum or minimum temperature, where the slope of this phase diagram is zero. In this case of minimum temperature there would exist a corresponding maximum pressure diagram. As can be seen in Figure 1, the azeotropic point exists only over a narrow range of composition for a given temperature and pressure condition. In the other regions the vapor and liquid would be of different compositions when in equilibrium. However, as shown in Figure 1 when this azeotrope is used within a refrigeration system, whose two phase operation is along the indicated saturation line the potential for glide (i.e., composition shift) is minimal (i.e., $< 0.5^\circ\text{C}$ glide) making it a rather desirable working fluid.

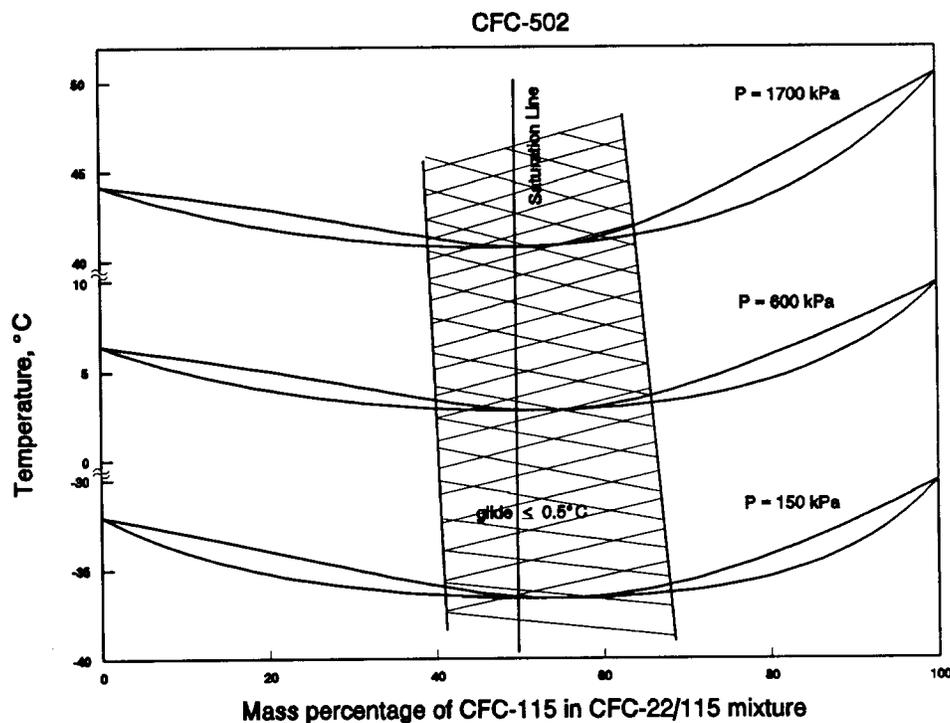
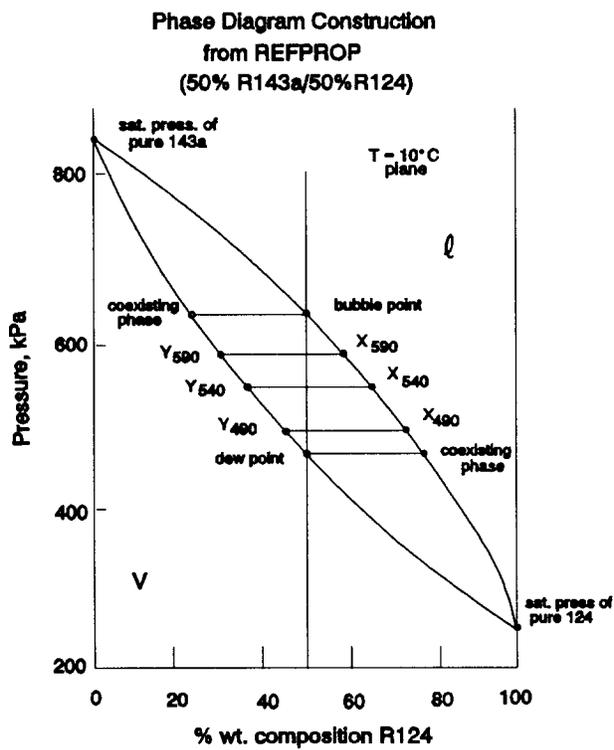


Figure 1

Zeotropes have no such equal phase composition point within the phase diagram and, thus, resemble the one shown in Figure 2. Because this diagram has pressure-composition coordinates the liquid region is above the two phase region and the vapor is below. The two-phase region is bounded by a bubble line and a dew line which are loci of the initial phase change points, i.e., bubble (X) and dew (Y) points, for each composition. Such data can be obtained from the refrigerant database REFPROP [1]. Figure 3 is the same binary mixture shown in the temperature-composition diagram. This diagram explicitly shows the temperature glide that occurs during the constant pressure transformation from the liquid to vapor phase which is due to the varying composition of the liquid (vapor) during the evaporation (condensing) process. It is rigorously defined as the change in fluid temperature during the heat transfer process. It is this temperature glide characteristic of zeotropes that make possible the Lorenz Cycle efficiency benefits in machinery designed to take advantage of matching it with the external heat transfer fluid's (e.g., air or water) temperature glide by way of counterflow arrangement of the heat exchangers. However, it also represents a potential pinchpoint disadvantage in counter-cross flow heat exchangers which currently occurs in most of the existing systems of today.



The composition shift that occurs in both the liquid and vapor during a zeotropic phase change is quantified by the values along the bubble and dew lines between the respective bubble point and dew point and the coexisting phase points. This variability in composition has significant influence on heat transfer as well as thermodynamic behavior of the fluid. Figure 4 illustrates the variation of the evaporative heat transfer coefficient for the R-502 mixture under different pressure conditions. For any given data set, the two pure component values (i.e., end points) and the azeotropic point (i.e., the peak of the hump) would form a straight line, much the way an ideal mixture would have its thermophysical properties aligned. By comparing this figure with Figure 1 (noting the ordinate's reverse scale) it can be seen that the greater the deviation from azeotropy the greater the degradation of the heat transfer coefficient from the ideal value.

This is due to:

- nonlinear property effects (i.e., viscosity and conductivity) for mixtures
- radial concentration gradients
- circumferential concentration gradients.

The composition difference between the liquid and vapor is the potential force for creating these concentration gradients and the molecular size of the more volatile component determines the resistance to the concentration gradient potential [2].

Phase Diagram Construction from REFPROP (50% R143a/50% R124)

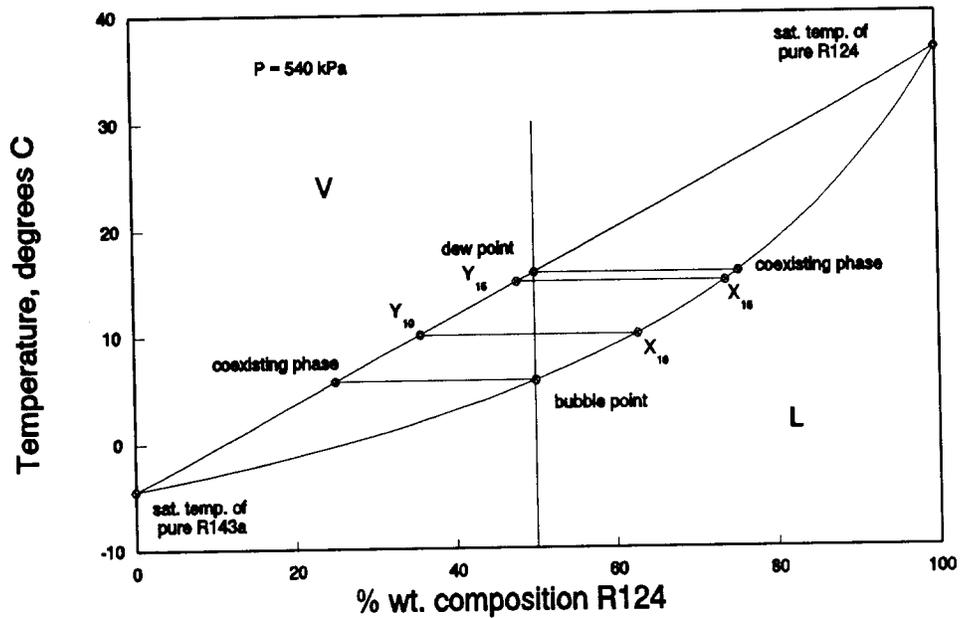
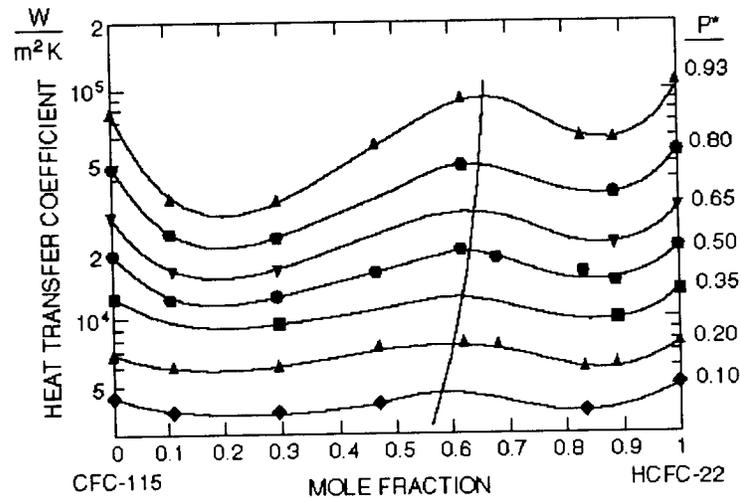


Figure 3



from Gorenflo and Bieling, Proc. Int. Cent. Heat Mass Transfer, pp 243-257, 1987

Figure 4

Zeotropic Mixtures of Near-Azeotropic Behavior

Since no single component alternative has been discovered for the higher pressure refrigerants HCFC-22 or CFC-502, the industry has begun to develop various mixtures of HCFCs for the short term (see Table A1) and HFCs for the long term (see Table A2), in an attempt to provide a working fluid of a thermodynamic performance equal to or greater than the CFC it is replacing. In addition, "drop-in" replacements for the CFCs have been developed for selected applications.

For retrofitting existing equipment, a capacity similar to that provided by the CFC is likely to be of prime importance. Capacity is primarily a function of suction density and latent heat, so the components of the mixture must be adjusted to offer a similar saturation pressure at the application's evaporator temperature, as previously existed with the CFC it is replacing. The resulting mixture composition may have a lower efficiency, particularly when the flammable components (e.g., HFCs 152a, 32, 143a, 142b, and HCs 290, 600a) must be limited so as not to make the mixture flammable even under the most severe "fractionation" conditions. Other factors such as compatibility with mineral oil or alkylbenzene lubricants also place restrictions on the design of mixtures. For this reason, primarily, the need for HCFCs has continued for use in alternatives for the transition period (see Table A1). Their chlorine content, albeit minor, is sufficient to allow the use of the lubricants with a long history of reliability and avoids concerns about thorough flushing of old oils or mixing effects with synthetic lubricants. Chlorine-free mixtures are, in some cases, also being proposed as potential retrofit alternatives but for the most part they are intended for newly designed systems. They have benefitted from lubricant research (i.e., polyalkylene glycol and polyol esters) that was conducted to meet the needs of HFC-134a. Being totally free of chlorine they meet all of the international restrictions resulting from the ozone problem and are the basis for the new equipment design for applications currently using CFC-12, CFC-502, and HCFC-22.

Since HCFC-22 constitutes the largest refrigerant market, by far, the focus of development has been on its alternative. In new commercial equipment, HFC-134a is a strong candidate for replacing HCFC-22. However, this refrigerant's vapor pressure is considerably lower than HCFC-22 and, thus, requiring larger component sizes for the same capacity machine. To approximate HCFC-22 vapor pressure the industry has formulated a zeotropic mixture of the higher pressure HFC-32 and the lower pressure HFC-134a. All zeotropes (including near-azeotropes) have a vapor pressure between that of its components. As seen in Figure 5, the mixture does not have its liquid and vapor saturation lines projected behind one another in the P-T plane as do azeotropes and single component refrigerants. Rather both the bubble and dew lines appear separately as dictated by the temperature glide during the phase change. For the HFC-32/134a (30/70% wt.) shown in Figure 5, the phase lines straddle that of HCFC-22 which suggests a near optimum simulation. Changing the composition or adding other components (e.g., HFC-125) can bring the phase lines closer together (i.e., reduce glide) or move them up or down the pressure scale to change thermodynamic performance. Such is the intent of the ternaries listed in Table A2 as alternatives to HCFC-22. Similarly, the CFC-502 alternative possibilities include a binary azeotrope and several ternary near-azeotropes and zeotropes.

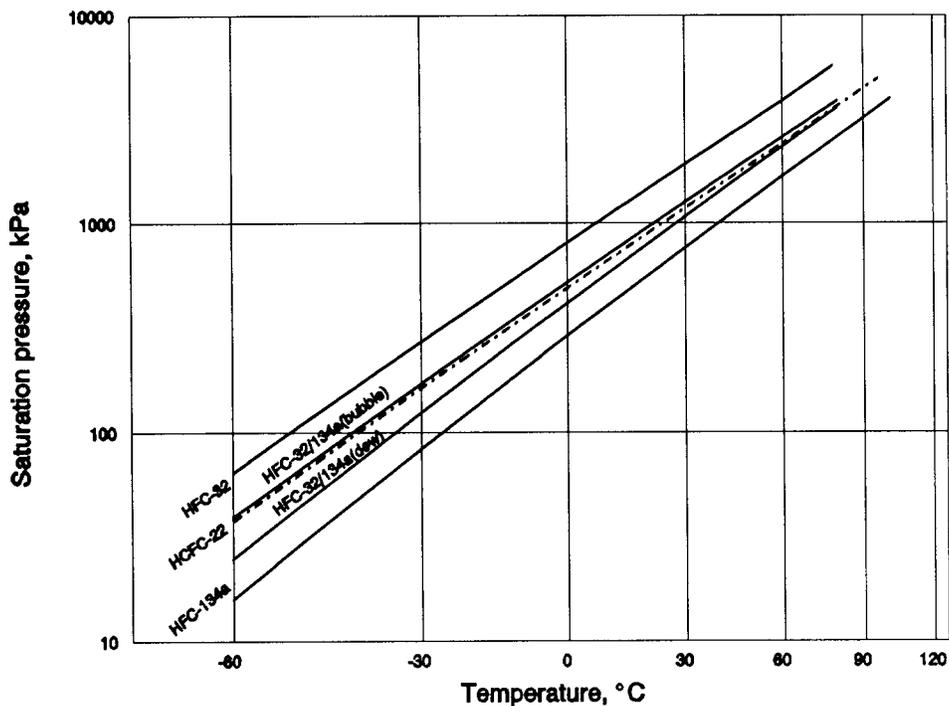


Figure 5

As noted earlier, HFC-32 is flammable and it is desirable, if not mandatory, that the mixture be nonflammable under all possible field conditions. The nature of zeotropes is such that upon a slow leak condition the composition of the vapor will be increased in the more volatile component, which for this case is the HFC-32, as the temperature decreases. Figure 6 illustrates that if a system or container with this HFC-32/134a (30/70% wt.) zeotrope were to leak vapor at approximately 50°C, the vapor would contain about 45% HFC-32, still nonflammable. However, a leak at about -25°C would yield a composition containing about 57% HFC-32, which is borderline flammable. For this reason U.S.A. companies have taken to developing several other zeotropes, with additional nonflammable components or smaller temperature glides, as HCFC-22 alternatives (see Table A2). Figure 7 illustrates that although the HFC-32/134a as formulated composition (30/70) is nonflammable by the ASHRAE standard with its 25°C test condition and is flammable according to the Underwriter's Laboratory (UL) with its 100°C test; it would clearly be flammable at the (57/43) leak composition by either test. The addition of 10% and 25% quantities of HFC-125 has the tendency to decrease the flammability as indicated in Figure 7; where the "coexisting phase" is the vapor leak composition from a system which was mistakenly vapor charged from a canister. In other words, the canister's vapor composition, was liquified in the refrigeration system and the new equilibrium vapor composition which favors the most volatile component even more, is what would leak out in case of a system failure. This latter case is not yet part of an official test procedure, but it is a criteria that some refrigerant manufacturers use. Assuming that a zeotrope that passes any flammability test is available, such as HFC-32/125/134a (23/25/52), an additional problem exists. Since system leaks and recharging often stem from containers where the mixture is in a two-phase equilibrium

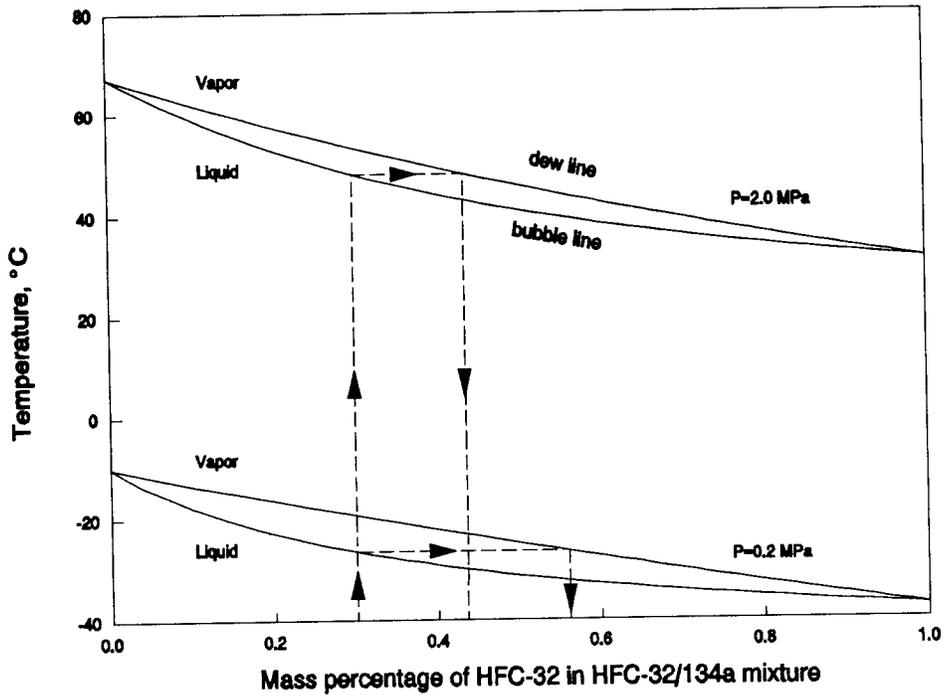


Figure 6

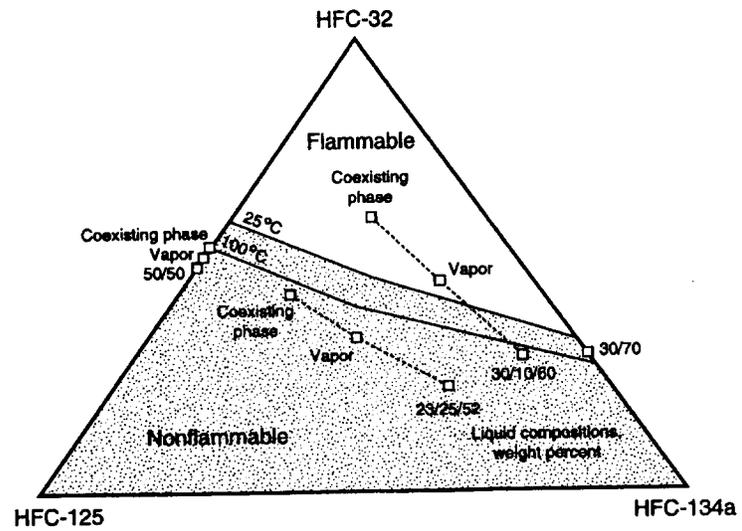


Figure 7

state (e.g., evaporators, condensers, storage cylinders) the composition will fractionate during phase change making it difficult to know what the new composition is exactly, particularly under field conditions. One solution is to simply recharge with liquid of the original composition and live with the consequences. Such procedures are being now evaluated by the chemical manufacturers. The effect on capacity of a series of leak/recharges is illustrated in Figure 8 for the zeotrope R-32/125/134a (23/25/52% wt.). For either case considered, it would appear that the capacity degradation would tend to level off somewhere in the 90%-of-original range. Alternatively, of course, the balance of any residual charge could be recovered for recycling and a completely new charge of original composition could be installed with no change in capacity. Perhaps for smaller systems (e.g., residential air conditioners) the latter procedure would be preferred. Again, the temperature glide shown in the Tables is an index as to how severe this phenomena is for a given mixture.

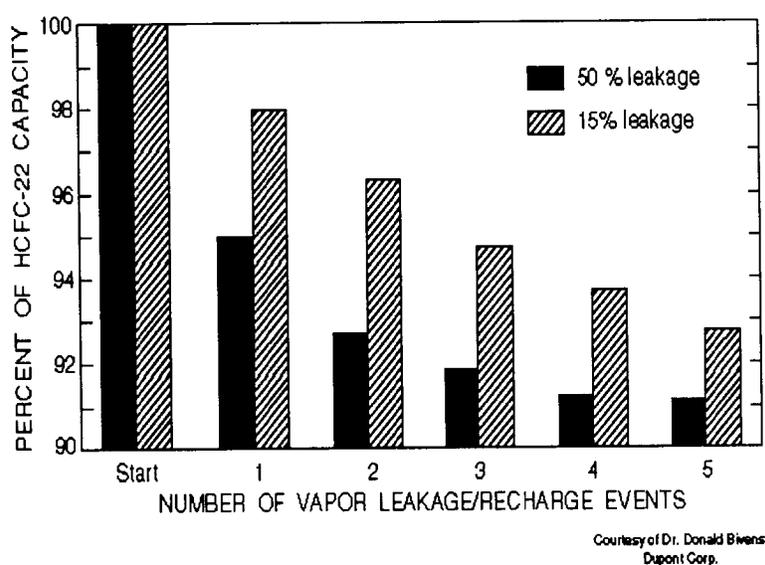


Figure 8.

In both commercial and residential unitary equipment two new mixture candidates have emerged in the U.S.A.: HFC-32/125 (50/50 and 45/55) and HFC-32/125/134a (23/25/52). The HFC-32/125, with its azeotrope-like behavior, is attractive for its traditional field handling practices; but also for its potential for smaller sized equipment and the possibility of improved COP. However, its vapor pressure lies close to HFC-32 which is considerably higher than HCFC-22, as can be seen in Figure 9. This means that the machinery manufacturers must determine if the economic trade-offs of compressor redesign and higher pressure containment, with its increased wall thicknesses and stronger sealant requirements, are offset by the small volumetric capacity requirements (i.e., reduced capital costs). It is also thought to be a possibility that the efficiency of such a system could be greater than its HCFC-22 counterpart because improved compressor efficiency and reduced pressure losses of the more dense fluid might compensate for the theoretical system efficiency decrease this fluid has relative to HCFC-22.

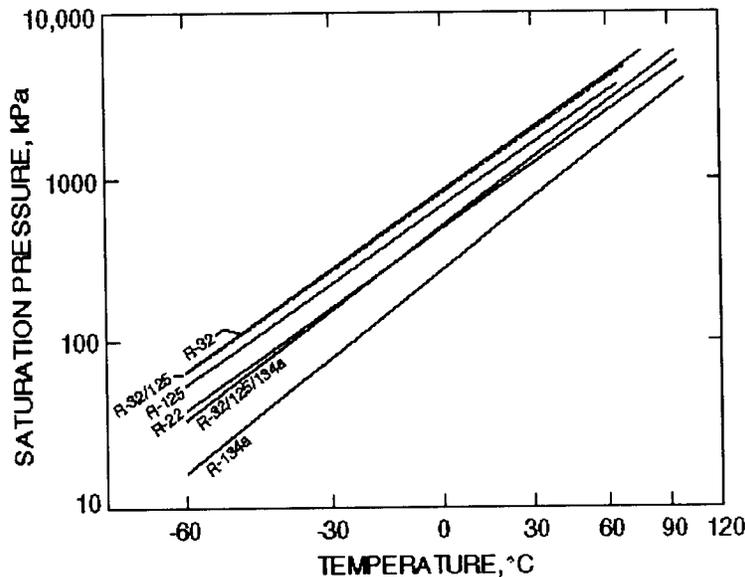


Figure 9

On the other hand, HFC-32/125/134a has been designed to have a similar vapor pressure as HCFC-22, so current equipment sizes are immediately available with no need for new product designs (albeit minor modifications may be necessary). It too lends itself to some minor efficiency gains through equipment redesign. The use of counterflow or cross-counterflow heat exchangers to take advantage of temperature glide matching is a possibility [3]. The larger specific heat fluids have a lower COP in the vapor compression cycle which can be improved (theoretically) up to but not above the COP of small specific heat fluids through the use of a liquid-line/suction-line heat exchanger [4]. Thus, at the present time it is not clear if there will be one or more refrigerant alternatives for HCFC-22. And given the two aforementioned alternatives, it would follow that the new machines could be quite different in size and/or configuration. For this somewhat staid and standardized industry that is used to producing a reliable, efficient, cost-effective product, these design choices offer radical changes indeed. Refrigerant mixtures in the form of azeotropes (ASHRAE 500 series) have been utilized by the industry for many years. These mixtures will have their phases at constant temperature throughout the evaporation and condensation processes and are in every way dealt with as though they were single component fluids. Unfortunately, they are quite rare and of all the mixtures listed in Tables A1 and A2 only one has shown azeotropic behavior, and that has been rigorously so only at very low temperatures.

Figure 10 illustrates the predicted equilibrium phase diagram for the only true azeotrope in these tables, HFC 125/143a (50/50). Although the azeotrope rigorously exists at only one composition for each pressure, the temperature glide region is so narrow for any composition that the phase diagrams appear to be a single line on this coordinate's scale. The azeotrope drifts to the left on the composition scale as pressure increases until it no longer exists (this is typical azeotropic behavior). This mixture is not actually an azeotrope in the temperature range of practice. This academic fact is of little practical consequence, however, since the components have normal boiling points that are only 1°C apart. Thus the temperature glide remains negligible throughout

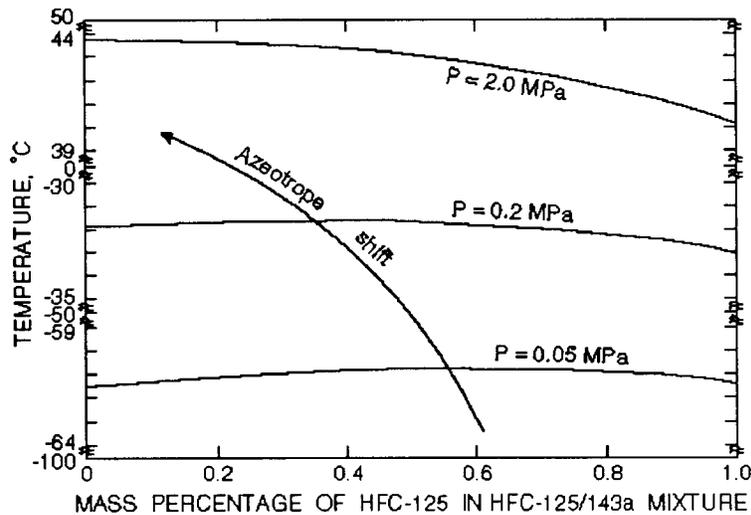


Figure 10

the temperature range of interest. As with all azeotropes, the vapor pressure curve lies outside the region of the individual components' curves. Figure 11 illustrates that it is a negative azeotrope; that is, it has a lower vapor pressure than either of its components, which is a rare phenomena for halogen azeotropes. This, however, is fortunate in that it brings R-125/143a closer to the R-502 vapor pressure which it is intending to replace.

In spite of the negligible temperature glide and very small composition glide R-125/143a has, the close proximity to the flammability region (see Figure 7) poses a problem. Given a liquid charge of (50/50) the equilibrium vapor composition will be borderline flammable under some test conditions (i.e., 100°C). For this reason, other chemical companies have chosen to add HFC-134a to this binary which will trap the flammable component HFC-143a between two nonflammable fluids. Of course this resultant ternary HFC-125/143a/134a (44/52/4) will have a small but measurable temperature glide (0.8°C). Figure 11 also illustrates that the vapor pressure simulation of the ternary is indistinguishable from that of the binary. One possible problem with either of these alternations, however, is the relatively high global warming potential of HFC-143a.

Other mixture alternatives for HCFC-22 and R-502 are usually a result of the trade-offs among performance cloning, flammability rating, and of course patent rights. A rather comprehensive summary of the details in the HCFC-22 and CFC-502 alternatives development is presented by Dr. Hickman in the ASHRAE Journal [5].

A New Mixtures Model

The need for predicting zeotropic mixtures composition under leak and recharge conditions has motivated an effort at NIST to develop a model entitled LEAK, based on the refrigerant properties program REFPROP [6]. The model predicts a histogram of equilibrium vapor and

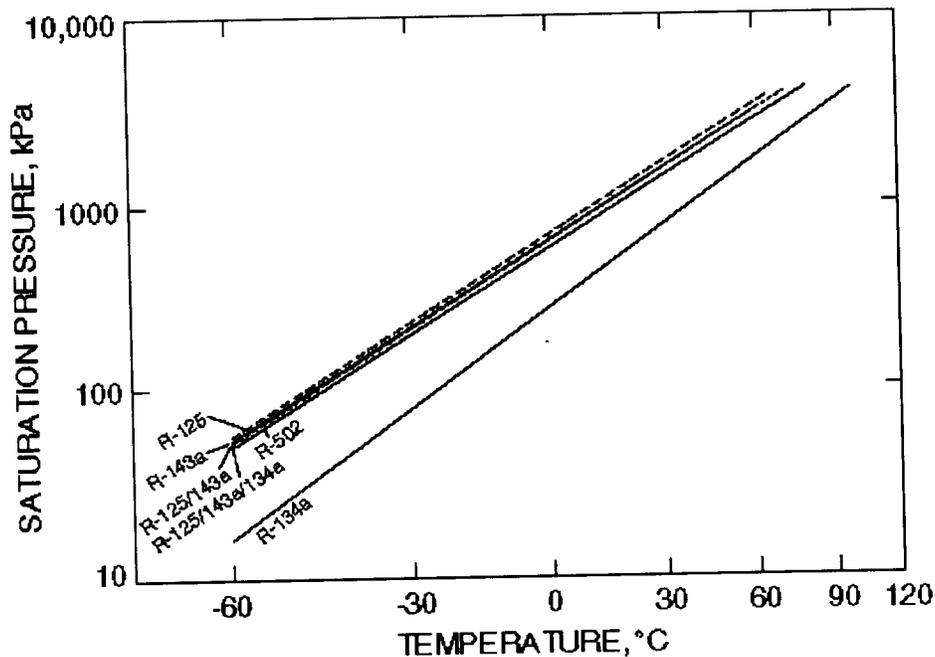


Figure 11

liquid compositions of a mixture, under isothermal or adiabatic conditions, for either a liquid or vapor leak from a container of an assigned temperature, initial composition and initial volumetric quality. It is also possible to simulate up to six cycles of an assigned percentage recharge of the original composition [7]. (See Figures below). The isothermal leak simulation is analogous to an ultra slow leak over a long period of time such that equilibrium conditions exist at all times. The model results agree very well with laboratory tests. The adiabatic leak simulation is analogous to a sudden fast leak. However, actual laboratory or field tests of such a fast leak will not necessarily correlate well with simulations because the leaking fluid will extract a significant amount of heat from the solid walled container and, thus, not be truly adiabatic. The adiabatic leak simulation represents an extreme (ideal) condition that may be of some value as a limiting case for a standard test. As with REFPROP 4.0, LEAK is currently capable of simulating any mixture up to five components from a list of 36 refrigerants. The precision of LEAK results is, of course, totally dependent on that of REFPROP. For the example discussed below the compositions predicted agreed with measurements from two different laboratories within a few percent. This was largely due to the fact that REFPROP has measured values for all the relevant mixing coefficients. When estimated mixing coefficient values are the only ones available less precision is likely. However, as REFPROP is routinely improved (5.0 is due out in the summer of '95) so will LEAK.

The example chosen is illustrated in the following figures. Figures 12 and 13 represent the schematic and process for a slow vapor leak under isothermal conditions. The overall

composition process line starts with a cylinder full of liquid and expands until it is 100% saturated vapor only. It can be seen that the net loss of the mass of fluid mixture causes both

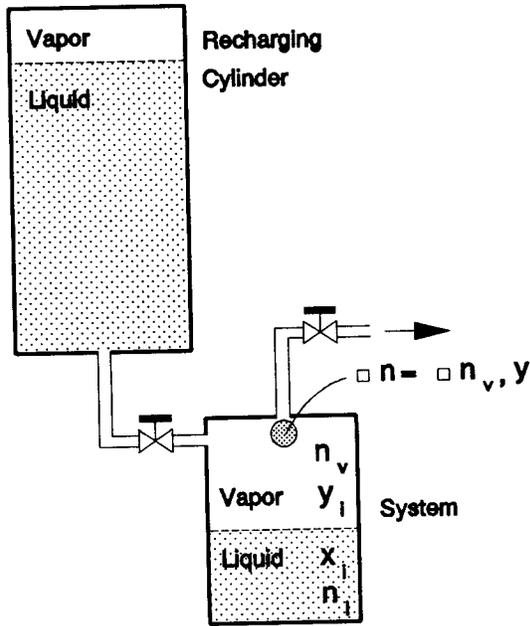


Figure 12

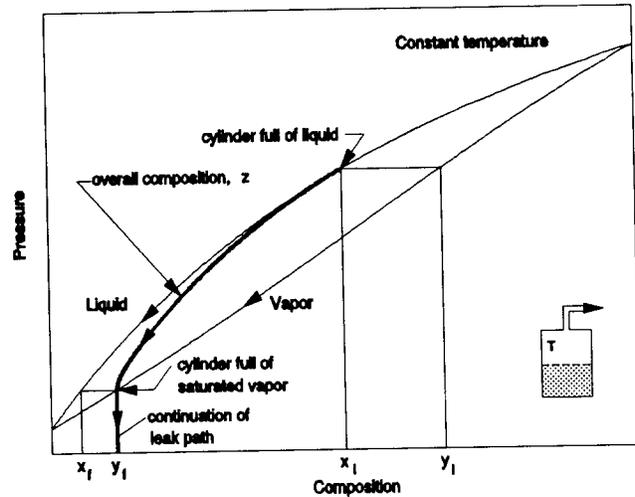
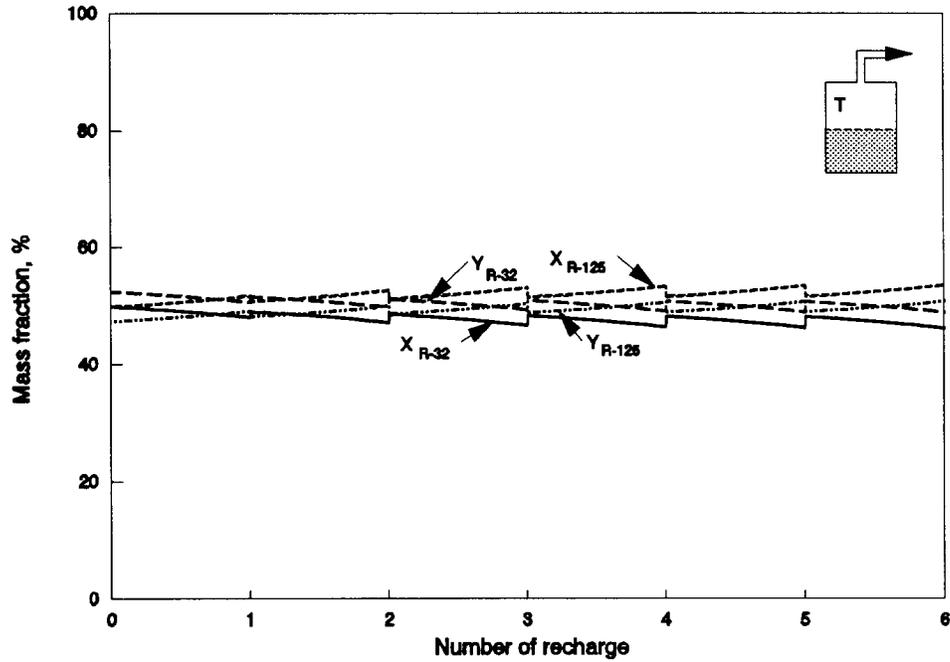


Figure 13

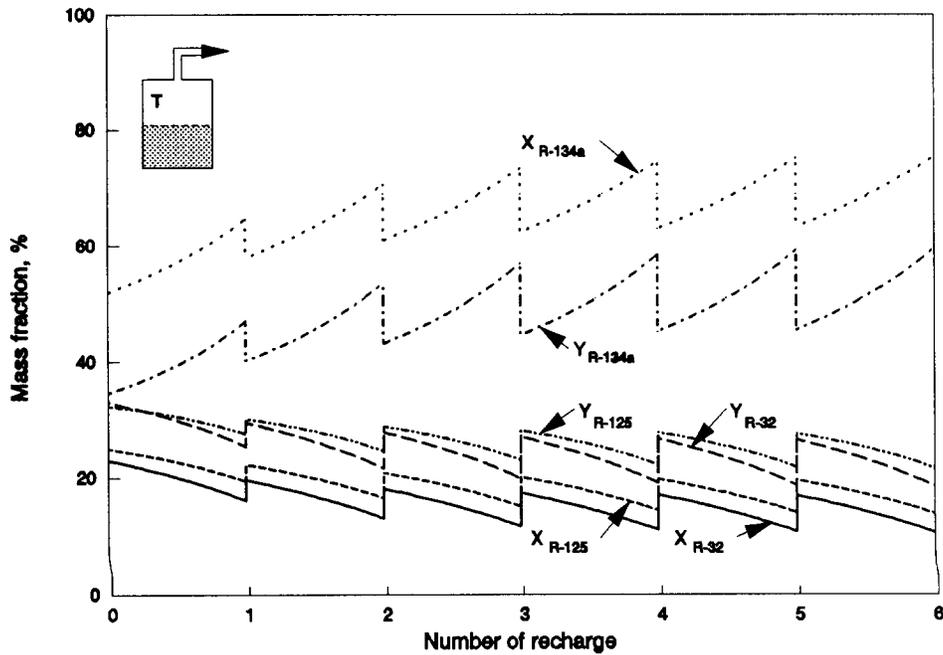
of the phases compositions to migrate towards a dominance of the lower volatile component. Figures 14 and 15, respectively, represent the binary and ternary alternatives for R-22, discussed above. The more realistic initial condition of 85% volumetric quality is considered through six cycles of 50% leak and recharge with the original composition. The binary with its extreme near-azeotropic behavior shows very little change in composition which is one of this mixture's most attractive attributes. The ternary, however, has an increase in the lowest volatility component in the liquid and vapor phases which causes a net decrease in the mixture's vapor pressure, and thus as the measured data of Figure 8 indicate, a net decrease in a refrigeration system's capacity.

It is hoped that such modeling efforts can be useful in minimizing the lengthy testing that slow leak conditions require. Given measured values for mixing coefficients, thermodynamic equilibrium states are generally a phenomenon that can be modeled with excellent precision.



Mass fraction change during the isothermal vapor leak/recharge process of the R-32/125 mixture at 20°C as a function of number of recharge. (During each leak process, mass fraction change is plotted with respect to the leaked mass percentage. System is recharged when 50% of refrigerant charge is leaked.)

Figure 14



Mass fraction change during the isothermal vapor leak/recharge process of the R-32/125/134a mixture as a function of number of recharge. (During each leak process, mass fraction change is plotted with respect to the leaked mass percentage. System is recharged when 30% of refrigerant charge is leaked).

Figure 15

Conclusions

It would appear that the zeotropic mixture, with all of its foibles, is to be with us in the immediate future if not indefinitely. Its flexibility offers near-cloning capabilities for at least the more important characteristics of those CFCs and HCFCs that are being phased out. The degree to which the zeotropes will be used will largely depend upon two factors: (1) whether the vapor pressure of the HCFC-22 alternative will be the same (i.e., the ternary HFC-32/125/134a) or will be considerably higher (i.e., binary HFC-32/125), (2) whether machine design changes come about that will capitalize on the Lorenz cycle and the variable capacity qualities inherent to zeotropes.

Currently most machine manufacturers are studying the manufacturing and materials costs associated with a higher pressure, smaller volume, residential air conditioner/heat pump that the azeotropic-like binary would make possible. This transition is not unlike the transition from CFC-12 to HCFC-22 of several decades ago. The goal of yet another smaller, cheaper-to-manufacture machine is very attractive. It is this goal that the author believes the manufacturers will find a way to achieve and, thus, relagate the zeotropic ternary to one of transition period working fluid only. For the R-502 alternative, both the binary and ternary mixtures discussed above are very azeotropic-like in their behavior (i.e., temperature glides $< 1^{\circ}\text{C}$). It is, therefore, unlikely that any of the zeotropic mixtures will prevail indefinitely, except for selected applications where a particular composition may have improved properties to meet a particular need (e.g., greater capacity or efficiency).

Nevertheless, the multitude of transition period fluids (i.e., those of Table A1) are providing the industry with considerable "zeotropic handling" experience. Undoubtedly, some of the difficulties will be solved or at least mitigated. If temperature glide matching counter flow heat exchangers and/or variable capacity distillation columns are developed in a cost effective manner, the energy savings may well prove to be significant enough to merit production of truly zeotropic machines. This is particularly true for those applications where conventional machines are reaching their efficiency limit, (e.g., water chillers) or if the global warming threat limits the use of HFCs, and flammable fluids, (e.g., hydrocarbons) are reconsidered for central cooling of residences [8]. Based on these possibilities the author believes that research on wide-glide zeotropes should continue.

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Table A1: Alternative Mixtures containing HCFCs

A S H R A E No. (Rating)	Composition (% weight)	temp. glide (°C) @ atm. pressure	replacement for	brand name
401 A (A1/A1)	R-22/152a/124 (53/13/34)	4.9	CFC-12	MP 39
401 B (A1/A1)	(61/11/28)	4.6	CFC-12	MP 66
401 C (A1/A1)	(33/15/52)	4.7	CFC-12	MP 52
402 A (A1/A1)	R-125/290/22 (60/2/38)	0.9	CFC-502	HP 80
402 B (A1/A1)	(38/2/60)	1.0	CFC-502	HP 81
403 A (A1/A1)	R-290/22/218 (5/75/20)	2.5	CFC-502	ISCEON 69S
403 B (A1/A1)	(5/56/39)	0.9	CFC-502	ISCEON 69L
*405 A (A1/A1)	R-22/152a/142b/C318 (45/7/5.5/42.5)	5.6	CFC-12	G2015
*406 A (A1/A2)	R-22/600a/142b (55/4/41)	9.9	CFC-12	GHG
	R-22/124/142b (60/25/15)	7.9	CFC-12	FX 56
	R-125/143a/22 (7/46/47)	0.5	CFC-502	FX 10

Table A2. Chlorine-Free Mixture Alternatives

ASHRAE No. (Rating)	Composition (% weight)	temp. glide (°C)† @ atm. pressure	replacement for	brand name††
404 A (A1/A1)	R-125/143a/134a (44/52/4)	0.8	CFC-502	HP 62, FX 70
407 A (A1/A1)	R-32/125/134a (20/40/40)	6.4	CFC-502	KLEA 60
407 B (A1/A1)	(10/70/20)	4.1	CFC-12, CFC-502	KLEA 61
*407 C (A1/A1)	(23/25/52)	7.1	HCFC-22	AC9000, KLEA 66
	(30/10/60)	7.4	HCFC-22	
	R-32/125/143a (10/45/45)	0.5	CFC-502	FX 40
	R-32/134a (30/70)	7.4	HCFC-22	
507 (A1)	**R-125/143a (50/50)	0.0	CFC-502	AZ 50
*410 A (A1/A1)	R-32/125 (50/50)	<0.1	HCFC-22	AZ 20
	(45/55)	<0.1	HCFC-22	AC 9100
*411 A (A1/A2)	R-1270/22/152a (1.5/87.5/11)	?	HCFC-22	G2018A
*411 B (A1/A2)	(3/94/3)	?	CFC-502	G2018B
	R-290/600a (50/50) varies	8.6	CFC-12	OZ 12
<p>* Approved by ASHRAE committee SPC 34 for public review, comments invited. ** Azeotrope exists for low evaporator temperatures.</p>				
<p>†Temperature glide = $T_{dew} - T_{bub}$ as predicted by REFPROP 4.0 (Actual temperature glide about 15% less for evaporators)</p>				
<p>††Manufacturers: DuPont - MP, HP, AC ATOCHEM - FX G - Greencool Allied - AZ Rhone-Poulenc - ISCEON GHG - Monroe Air Tech ICI - KLEA Foron - OZ</p>				