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James Jay Baustian

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Iowa State University, 1988
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Development of a sensor
for the continuous measurement of oil concentration
in a refrigeration system

by

James Jay Baustian

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of the
Requirements for the Degree of
DOCTOR OF PHILOSOPHY
Major: Mechanical Engineering

Approved:

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NOMENCLATURE

a  = absorptivity
A  = absorbance
b  = spectrophotometric path length
b_n = constants for regression equations
c  = concentration in spectrophotometer sample
c_a = acoustic velocity
C  = weight fraction of oil
C_p = constant pressure specific heat
C_P = molar constant pressure specific heat
I  = light intensity
I_0 = zero concentration light intensity
k  = thermal conductivity
K  = viscometer constant
M  = molecular weight
p  = pressure
PL = prediction limit
R  = universal gas constant
SG = specific gravity
t  = transmittance
T  = temperature
T_b = normal boiling point temperature
v  = specific volume
W  = weight fraction of refrigerant
y  = mole fraction
Greek Symbols

\( \beta \) = coefficient of thermal expansion

\( \theta \) = dimensionless temperature in regression equations

\( \theta \) = angle of incidence (Appendix 3)

\( \mu \) = dynamic viscosity

\( \rho \) = density

\( \rho_b \) = viscometer ball density

\( \sigma \) = surface tension

\( \tau \) = viscometer transit time

\( \phi \) = volume fraction

Subscripts

\( c \) = critical

\( m \) = mixture

\( o \) = oil

\( r \) = refrigerant

Superscripts

\( g \) = gas

\( \ell \) = liquid
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CHAPTER 1
INTRODUCTION

Because of the important effects of oil circulation on refrigeration system performance, there is considerable current interest in obtaining a method of continuously measuring the quantity of oil in a flowing oil-refrigerant mixture. The results of a research project seeking to develop such an oil concentration sensor are reported herein. Background information on the importance of oil concentration measurement and an organizational description of the research project are presented in this chapter.

The Effects of Oil on Refrigeration System Performance

The primary function of the oil present in a vapor-compression refrigeration system is to lubricate the moving parts of the compressor. In addition, the oil serves as the seal between the suction and discharge sides of the compressor and as a compressor coolant. Most of the oil remains in the compressor during normal system operation. However, a portion of the oil is carried through the entire refrigeration system by the flowing refrigerant. Knowledge of the amount of circulating oil is necessary for the accurate calculation of refrigeration system capacity, estimation of thermal and hydrodynamic performance of system components, prediction of fluid properties in the system, and prevention of operational problems caused by excessive oil circulation.

As the oil-refrigerant mixture circulates through the evaporator, most of the oil remains liquid and does not contribute to producing refrigeration. Thus, circulation of the oil reduces the maximum mass
Figure 1.1. Loss of capacity in R-12 system due to presence of oil [1]

flow rate of actual working fluid and, hence, the total system capacity. In addition, as the refrigerant boils in the evaporator, the boiling temperature of the remaining oil-rich mixture increases. This increase in the evaporation temperature raises the overall evaporator temperature, which further reduces the refrigeration system capacity. The effects of oil circulation on system capacity are illustrated in Figure 1.1 [1]. This plot shows that in a typical system using R-12 and an unspecified oil, the system capacity is steadily decreased as the circulating oil concentration is increased. Obviously, knowledge of the oil concentration in the circulating working fluid is necessary for the accurate prediction of actual system capacity.
The presence of oil in the refrigeration system also has a significant effect on the thermal and hydrodynamic performance of refrigeration system components, such as evaporators and condensers. For example, in a recent survey paper Schlager et al. [2] noted that heat transfer in evaporator tubes can be either enhanced or inhibited by the presence of oil, depending on the amount of oil present. The presence of oil also increases pressure drop through system components. Decreased heat transfer coefficients and increased pressure drops result in a reduction in system efficiency. The magnitude of the heat transfer degradation and pressure drop increase both depend on the amount of oil circulating in the refrigeration system.

Correlations for estimating the aforementioned heat transfer and pressure drop effects require numerical values for fluid properties such as viscosity, density, and specific heat. These fluid properties of the working fluid in a refrigeration system deviate significantly from pure refrigerant values due to dissolved oil. The changes in fluid properties caused by the oil also produce errors in refrigeration system mass flow rate measurements. Most flowmeters, including variable-area-orifice meters and turbine flowmeters, are affected by changes in fluid density, viscosity, or both. Thus, knowledge of the flowing oil concentration is necessary for determination of the actual properties of the oil-refrigerant mixture flowing in the system. These property values can subsequently be used for the correction of flowmeter readings and the estimation of heat transfer rates and pressure drops in the refrigeration system.
In addition to affecting performance, the circulation of oil may be responsible for certain operational problems in refrigeration systems. The most obvious problem is inadequate lubrication of the compressor due to excessive oil circulation. If an excessive amount of oil is circulating with the refrigerant, there could be a serious shortage of lubricating oil in the compressor crankcase. Other operational problems can be caused by the breakdown of circulating oil into sticky residues and by the corrosive products of chemical reactions between the circulating oil and the refrigerant. To aid in preventing these operational problems and estimating the actual system performance, a method of measuring the oil concentration in the circulating oil-refrigerant mixture is required.

**Standard Method for Determining Oil Concentration**

To support the need for oil concentration measurement, the American Society of Heating, Refrigerating, and Air Conditioning Engineers (ASHRAE) now maintains ASHRAE/ANSI Standard 41.4-1984 [3], which describes a method for the gravimetric determination of the oil concentration in an oil-refrigerant mixture flow stream. This standard method entails removing a sample of the oil-refrigerant mixture from the liquid line (condenser outlet stream), weighing the sample, boiling away the refrigerant component, and weighing the residue.

In addition to its obvious inconvenience, this method suffers from two significant inadequacies. First, a representative sample of the mixture can only be withdrawn when conditions in the liquid line are steady. As a result, this method is useless for the investigation of
transient oil circulation. Second, this method requires a relatively large sample, approximately one pound of the mixture. The removal of such a large sample significantly affects the operation of all but very large systems. An in-situ instrument for determining oil concentration in the liquid line would be a clear improvement over the sample-removal method, especially for measurements in smaller systems and for the study of transient oil circulation.

Description of the Research Project

The intent of this research project, which has been accomplished with ASHRAE sponsorship, was to respond to the need for an improved method of measuring the oil concentration in a refrigeration system. Brief descriptions of the research project objective, scope, and tasks are presented below.

Objective

As suggested by the project title, the objective of this research is to develop an instrument for the measurement of oil concentration in a flowing oil-refrigerant liquid mixture. The instrument is to be capable of operating under the conditions existing in the liquid-line (condenser outlet piping) of a refrigeration system. These conditions typically involve pressures up to 300 psia and temperatures from 70 to 120 F.

Scope

In order to ensure that the results of this project would be applicable to a variety of actual refrigeration systems, specific guidelines for this research were established with the assistance of
industry professionals [4]. Brief statements of these guidelines are presented below.

**Oil Types** Three types of refrigeration oils were included in this study. The first classification was those natural petroleum oils having a low percentage of aromatic hydrocarbons, often called paraffinic oils. Oils in the second major group are also refined from petroleum, but have relatively high percentages of aromatic ring (benzene-like) molecules in their structures. These oils are usually characterized as naphthenic refrigeration oils. Oils in the final group are synthetically produced and consist of aromatic structures with appended straight-chain, or alkyl, groups. Because of this structure, these oils are most often referred to as linear alkylbenzenes. The effects of lubrication promoters such as tricresyl-phosphate, which are commonly added to all types of refrigeration oils, were also to be considered.

**Refrigerants** Three widely used halocarbon refrigerants, R-12, R-22, and R-502, were included in this study. In addition, R-113 was included because of its very high normal boiling point. This high boiling point made R-113 a very easy fluid to use in bench-top experimental work.

The original project specifications also included the hydrocarbon refrigerant R-290, commonly known as propane. However, because of laboratory safety considerations, this highly flammable refrigerant was later dropped from the project with the approval of the sponsor.

**Range of oil concentrations** Oil-refrigerant mixtures consisting of up to 30% oil by mass were considered in this study. However, some
oil-refrigerant combinations do not form true single-phase solutions at all thermodynamic states when the oil concentration is high. Consequently, oil concentrations high enough to produce immiscible mixtures at 130 F liquid temperature were not included in this study.

**Accuracy** The obvious output unit of an oil concentration measurement system is the weight percent of oil in the mixture. When the output is expressed in this manner, the allowable error in the measurement was to be one weight-percent oil. The confidence level for this error specification was to be 95%. This specification means that at oil concentrations of around one weight percent, the error in the measurement would be ±100% of indication. At oil concentrations around 30 weight-percent, the error would be ±3% of indication.

**Effects of contamination** The effects of normal refrigeration system contaminants such as water, waxes from oil breakdown, and the products of oil-refrigerant reactions were to be considered in the development of the oil concentration sensors.

**Other factors** It is expected that any oil concentration sensor developed would be applicable not only in the laboratory but in refrigeration systems in the field as well. Consequently, it was desirable that the oil concentration sensors be simple, reliable, somewhat rugged, low-cost, easy to calibrate, simple to install, and convenient to use.

**Research tasks**

**Identification of oil concentration measurement methods** Direct determination of oil mass fraction can only be made through sample removal, as described previously. To make an in-situ determination, the
oil concentration must be inferred from the measurement of a physical property of the oil-refrigerant mixture in the liquid line. Consequently, the identification of potentially successful oil concentration measurement methods entailed the search for oil-refrigerant liquid mixture properties that possess two key attributes. First, the property must vary significantly with changes in oil concentration. Second, an accurate means of measuring the property in a flow stream must be feasible. It should be stressed that these two attributes are equally vital to a successful oil concentration measurement scheme. Properties which vary greatly with oil concentration but for which no means of accurate in-situ measurement is available are of no interest. Similarly, highly sensitive property measurement techniques are of no use if the measured property is not a function of oil concentration. Only when these two factors are mutually considered can an estimate of a proposed method's oil concentration measurement accuracy be obtained.

The identification of oil concentration measurement methods involved both a literature survey and preliminary laboratory measurements of oil-refrigerant mixture properties. The literature survey is presented in Chapter 2, and results of the laboratory investigations are contained in Chapter 3.

Selection of methods for continued development Practical considerations required that only the most promising methods for in-situ oil concentration measurement be developed into actual sensors. Obviously, the estimate of a method's measurement accuracy was the most important factor in this selection. However, many other factors were included in
the selection criterion, as discussed in the final section of Chapter 3.

**Development and testing of oil concentration sensors**

Four methods were selected for development into actual oil concentration sensors. In some cases, existing instruments were adapted for use under refrigeration system liquid-line conditions, while other methods required the construction of prototype instruments from basic components. Detailed descriptions of the development and testing of three of the sensors are presented in Chapters 5 through 7. Despite extensive development work, the fourth sensor was not successful and was never evaluated under refrigeration system conditions. The description of this sensor is presented in Appendix 3.

The performance of each instrument developed was to be tested under actual liquid-line conditions. These performance tests required the construction of an experimental flow loop capable of simulating a wide variety of liquid-line conditions. Chapter 4 contains details of the flow loop design and construction and a general description of the performance tests.

**Comparison and recommendation**

The final research task was to analyze and compare the performance test results. Chapter 8 contains a summary of the performance test comparisons and recommendations for using each of the instruments to measure oil concentration.
CHAPTER 2
A LITERATURE SURVEY OF OIL-REFRIGERANT MIXTURE PROPERTIES AND PROPERTY MEASUREMENT METHODS

Results of the literature survey were used to identify and select methods for oil concentration measurement. The literature survey encompassed the twelve properties of oil-refrigerant liquid mixtures listed below.

- density/specific gravity
- vapor pressure
- specific heat
- viscosity
- thermal conductivity
- surface tension
- acoustic velocity
- coefficient of thermal expansion
- electrical conductivity/resistivity
- dielectric constant
- light absorption
- refractive index

Early in the project, it became obvious that the amount of data in the literature concerning these properties was not sufficient to determine the variations of the properties with oil concentration. Consequently, the primary objective of the literature survey became the selection of mixture properties for further investigation in the laboratory. Although laboratory investigations of each of the twelve properties listed above would produce a great deal of important data,
such investigations were simply not possible within a reasonable amount of time. The literature survey was useful in eliminating some of these properties from consideration as oil concentration measurement methods without laboratory investigations.

An additional objective of the literature survey was to provide data for comparison with laboratory measurements. The literature contains few actual values of oil-refrigerant liquid-phase mixture properties for direct comparison. However, for most of the properties measured in the laboratory it was possible to find either a few data points for direct comparison or a general predictive relationship for liquid mixture properties which could confirm the trends in the measurements.

Fulfillment of the above objectives of the literature survey involved examining many types of literature. Technical papers concerning single- and two-phase heat transfer in oil-refrigerant mixtures proved to be good sources for thermophysical and transport property data. Additional property information for oils, refrigerants and oil-refrigerant mixtures was obtained from product bulletins of oil and refrigerant manufacturers. Technical papers concerning compressors and lubrication in refrigeration systems were also useful. Papers on the topic of hermetically sealed refrigeration compressors were particularly useful in obtaining electrical properties of the oil-refrigerant mixtures. In addition to data developed specifically for liquid mixtures of oils and refrigerants, general predictive relationships for the various properties of liquid mixtures were also sought. Technical papers and texts concerning the general theory of liquids and properties of the
liquid state were often useful in this regard.

A third objective of the literature survey was the review of instrumentation that showed potential for adaptation to oil concentration measurement. Specifications concerning the accuracy of these devices were vital to the selection of measurement methods for further development. Information regarding property measuring instruments was taken from instrument manufacturers' specifications and papers in the open literature describing potentially adaptable measurement techniques.

The final objective of the literature survey was the identification of potential problems with various measurement techniques. For example, some properties of oil-refrigerant mixtures are not entirely stable, usually because of decomposition of the oil or chemical reactions between the oil and refrigerant. Obviously, an oil-refrigerant mixture property that varies considerably over the normal life of a refrigeration system would not be a suitable basis for an oil concentration sensor. Technical papers and product bulletins from the oil and refrigerant manufacturers were useful in estimating the effects of property instability, chemical reactions, and contamination.

Density/Specific Gravity

Because of the importance of liquid density in many engineering calculations, values for the liquid density of refrigerants and oils were easily located in the literature. Even in the liquid state, density varies somewhat with temperature. Table 2.1 presents the approximate values of density for the refrigerants and a typical oil at a common temperature in specific gravity units.
Table 2.1 shows that the halocarbon refrigerants are 23% to 68% heavier than the oil, while propane is 49% lighter. These large differences in the densities of the pure components indicated that an oil concentration system based on density measurement could be feasible.

<table>
<thead>
<tr>
<th>Fluid type</th>
<th>Specific Gravity</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>1.23</td>
<td>5</td>
</tr>
<tr>
<td>R-22</td>
<td>1.11</td>
<td>5</td>
</tr>
<tr>
<td>R-502</td>
<td>1.11</td>
<td>5</td>
</tr>
<tr>
<td>R-290</td>
<td>0.46</td>
<td>5</td>
</tr>
<tr>
<td>R-113</td>
<td>1.51</td>
<td>5</td>
</tr>
<tr>
<td>Naphthenic Oil</td>
<td>0.9</td>
<td>6</td>
</tr>
</tbody>
</table>

ASHRAE [7] gives a predictive relationship for the densities of oil-refrigerant liquid mixtures. Making the assumption that both volume and mass are additive quantities for the mixture, this relationship is easily derived theoretically.

\[
\rho_m = \frac{\rho_r}{1 - (1 - W)(1 - \rho_r/\rho_o)}
\]  

ASHRAE [7] also points out, however, that the additive volume assumption is usually disobeyed by oil-refrigerant mixtures. Depending on the refrigerant, some mixtures are more dense than predicted by Equation 2.1, others less dense. A multiplicative factor, which is in itself a function of oil concentration, is often employed to correct Equation 2.1.

The literature review also revealed several devices that are designed for in-line determination of density in a flow stream. One of
the devices explored used the absorption of gamma rays by the flowing fluid as an indication of density [8]. This device, however, was designed for installation in very large process lines and was found to be prohibitively expensive.

Another device, examples of which were available from several manufacturers, was an in-line densimeter that used measurements on a vibrating U-tube through which the fluid flowed as an indication of density, mass flow rate, or both. An example of one of the devices, adapted from the manufacturer's product literature [9], is presented in Figure 2.1. This device, which is both a densimeter and a mass flow meter, consists of a U-tube which is excited into vibration as in Figure 2.1a. The reactions of the fluid forces to the vibrating motion of the tube cause the tube to twist, as in Figures 2.1b and 2.1c. The magnitudes of these reaction forces are directly proportional to the mass flow rate of fluid through the tube and, hence, the amount of twist is also proportional to the mass flow rate. The natural frequency of vibration of the tube is proportional to the fluid density. The twist angle and natural frequency are measured by magnetic proximity sensors. A simultaneous measurement of mass flow rate and density is thus accomplished. Although the exact principle of operation varies among the manufacturers, all of these devices have similar claimed density measurement accuracies of ± 0.001 specific gravity units.

The significant variation of density with oil concentration calculated from Equation 2.1 and the low densimeter measurement uncertainties indicated that a density-based oil concentration
Fig. 2.1. Diagram of vibrating U-tube densimeter
measurement was possible. Consequently, laboratory measurements were performed to determine the actual variation of density with oil concentration for mixtures of R-113 and a 150 SUS naphthenic refrigeration oil.

**Vapor Pressure**

Dissolving oil in a pure refrigerant lowers the vapor pressure of the refrigerant. Plots of vapor pressure versus the weight fraction of oil for constant temperatures were found in references 6, 7, and 10. Unfortunately, these curves showed only minor changes in refrigerant vapor pressures with the addition of as much as 40 weight-percent oil. Also, since the fluid state at the condenser outlet is all liquid during normal operation, no obvious method for vapor pressure measurement was evident. This lack of a measuring instrument, coupled with the small effect of oil concentration on vapor pressure, led to the elimination of vapor pressure as a possible basis for an oil concentration measurement system.

**Specific Heat**

Values for the specific heats of refrigerants and refrigeration oil are presented in Table 2.2. The specific heats of the halocarbon refrigerants are some 23% to 46% lower than a typical naphthenic oil, while propane's specific heat is 70% higher than oil. Jensen and Jackman [11] demonstrated an approximate relationship for the specific heat of an oil-refrigerant mixture based on the specific heats of the components:

\[
C_{pm} = C_{pr}W + C_{po}(1 - W)
\]  

(2.2)
Using this equation, these authors have shown that the specific heat of a 25 weight-percent solution of a typical naphthenic oil with R-113 will have a specific heat approximately 25% higher than the pure refrigerant. While this is a significant increase in the specific heat due to the addition of oil, it was not judged to be sufficient in light of the errors involved in specific heat measurements. In the case of a flowing fluid, simultaneous measurement of a heat flux, a small temperature difference, and a mass flow rate would be necessary to determine the specific heat. The product literature examined contained no references to such an in situ calorimeter. Consequently, specific heat of the oil-refrigerant mixtures was eliminated as a potential basis for oil concentration measurement.

**TABLE 2.2. Specific heats of liquids at 122 F**

<table>
<thead>
<tr>
<th>Fluid Type</th>
<th>Specific Heat, Btu/lbm-F</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>0.248</td>
<td>5</td>
</tr>
<tr>
<td>R-22</td>
<td>0.332</td>
<td>5</td>
</tr>
<tr>
<td>R-502</td>
<td>0.315</td>
<td>5</td>
</tr>
<tr>
<td>R-290</td>
<td>0.731</td>
<td>5</td>
</tr>
<tr>
<td>R-113</td>
<td>0.234</td>
<td>5</td>
</tr>
<tr>
<td>Naphthenic Oil</td>
<td>0.430</td>
<td>11</td>
</tr>
</tbody>
</table>

**Viscosity**

Values for the viscosities of pure oils and refrigerants are readily available in the literature. The viscosities of liquids are, however, very temperature dependent. For purposes of comparison, Table 2.3 presents the viscosities of liquid oils and refrigerants at a common
Table 2.3 shows that the viscosity of a refrigeration oil is over fifty times that of even the most viscous of the refrigerants. This very wide difference in the component viscosities indicated that a measurement of oil-refrigerant mixture viscosity could possibly be used to infer oil concentration.

<table>
<thead>
<tr>
<th>Fluid type</th>
<th>Viscosity, cp</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>0.197</td>
<td>5</td>
</tr>
<tr>
<td>R-22</td>
<td>0.186</td>
<td>5</td>
</tr>
<tr>
<td>R-502</td>
<td>0.157</td>
<td>5</td>
</tr>
<tr>
<td>R-290</td>
<td>0.092</td>
<td>5</td>
</tr>
<tr>
<td>R-113</td>
<td>0.577</td>
<td>5</td>
</tr>
<tr>
<td>150 SUS Naphthenic Oil</td>
<td>32.0</td>
<td>6</td>
</tr>
</tbody>
</table>

Published data for the viscosities of oil-refrigerant mixtures were considerably less plentiful than data for the pure components. The data located were generally for oil-rich mixtures, with oil concentrations in the range of 70 weight-percent and higher. Mixture viscosities in this range are of considerable interest in studying the lubrication of compressors. A limited amount of data was given by Jensen and Jackman [11], along with a discussion of predictive relationships for oil-refrigerant mixture viscosities. A number of such relationships were located in the literature; four of them are presented below.

\[
\frac{1}{\mu_m} = \frac{\phi_r}{\mu_r} + \frac{\phi_o}{\mu_o} \tag{2.3}
\]

\[
\ln \mu_m = y_r \ln \mu_r + y_o \ln \mu_o \tag{2.4}
\]
Equations 2.3 through 2.6 predict the mixture viscosity based on the viscosity and concentration of each component. Equations 2.3 and 2.4 were developed from theoretical considerations for "ideal" mixtures, in which the molecules are dissociated and the component viscosities are well within an order of magnitude. Equation 2.3 [12] is based on the principle that the fluidity, which is the reciprocal of the viscosity, is an additive property when weighted by the volume fractions of the components. Equation 2.4 uses the natural logarithm of the viscosity as the additive property, with the weighting function being the mole fractions. This equation was originally the work of Arrhenius [13], who used the mass fractions instead of the mole fractions. Kendall [14] later observed that the equation described a broader group of liquid mixtures if the mole fractions were substituted. Jensen and Jackman [11] developed Equation 2.5 by curve-fitting their measurements of oil-refrigerant viscosities. The data of Jensen and Jackman were for mixtures of naphthenic oils and R-113 in concentrations of ten weight-percent and less. The final equation is also an empirical relationship and was developed by Kendall and Monroe [15] after they examined a large set of data and noticed the forms of the earlier work in Equations 2.3 and 2.4. Kendall and Monroe also used mole fractions as the weighting factor and chose the third root of viscosity as the additive function.

\[ \mu_m = \mu_r \exp[(1 - \omega)(\mu_r/\mu_o)^0.3] \]  

Equation 2.5

\[ \mu_m^{1/3} = \gamma_r \mu_r^{1/3} + \gamma_o \mu_o^{1/3} \]  

Equation 2.6
Because of the large difference in the viscosities of the two components, even the most conservative of the above equations produces a fairly sharp viscosity change with oil concentration. However, the equations give very different results, indicating that not all of them can be correct for oil-refrigerant mixtures. A comparison of the four equations appears in the next chapter.

In addition to the property information obtained from the literature, a review of commercially available viscometers revealed two types designed for measurements in a process line. A schematic diagram of one type, adapted from the manufacturer's product literature [16], is presented in Figure 2.2. This device operates on the same principle as many popular laboratory viscometers, measuring the viscous drag exerted on a stationary cylinder by the surrounding fluid. The fluid is set in motion by a concentric cylinder driven by a motor. In this version of the instrument, the components are mounted in a bypass line and the fluid flows through the viscometer, as shown in Figure 2.2. Unfortunately, devices of this type are designed to measure viscosities much greater than those predicted for the oil-refrigerant mixtures. The manufacturer gives the minimum range for this viscometer as 10 to 30 cp, whereas oil-refrigerant mixtures were predicted to have viscosities approximately one hundred times smaller. Discussions with the manufacturer revealed little hope of successfully reducing this viscometer's working range.

The second type of process viscometer was considerably more promising. A schematic of this device, also adapted from the manufacturer's literature [17], is presented in Figure 2.3. The device
Fig. 2.2. Diagram of in-line rotating-cylinder viscometer
Fig. 2.3. Diagram of magnetic viscometer
(a) bypass installation
(b) sensor internals
mounts in the bypass configuration shown in Figure 2.3a. Magnetic coils are used to move the stainless steel bobbin to the top of its travel while the inlet valve is open, thereby drawing in a charge of the fluid from the process line. The inlet valve is then closed, and the bobbin is forced back through the stagnant fluid, again under magnetic forces from the coils. The position of the bobbin as a function of time can also be measured by the magnetic coils. If the magnetic force is held constant, the time required for the bobbin to travel a fixed distance through the fluid can be correlated as a linear function of the fluid viscosity. An internal temperature sensor is also included. Direct contact with the manufacturer of this viscometer revealed that an instrument with a range of 0.1 to 2 cp and an accuracy of +2% of indication could be obtained.

**Thermal Conductivity**

Table 2.4 presents values for the thermal conductivities of pure refrigerants and oils. These data show that the pure oil thermal conductivity exceeds that of the refrigerant by 46% to 100%. While this difference in pure component thermal conductivities is relatively large, a predictive relationship from Jensen and Jackman [11] indicates that the thermal conductivity of the mixture increases rather slowly with oil concentration:

\[ k_m = k_W + k_o (1 - W) - 0.72(k_o - k_R)(1 - W) W \]  

(2.7)

This equation shows that for a ten weight-percent solution of naphthenic oil and R-113, the thermal conductivity increases less than three percent.
TABLE 2.4. Thermal conductivities of liquids at 122 F

<table>
<thead>
<tr>
<th>Fluid type</th>
<th>Thermal Conductivity, Btu/hr-ft-F</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>0.035</td>
<td>5</td>
</tr>
<tr>
<td>R-22</td>
<td>0.044</td>
<td>5</td>
</tr>
<tr>
<td>R-502</td>
<td>0.031</td>
<td>5</td>
</tr>
<tr>
<td>R-290</td>
<td>0.047</td>
<td>5</td>
</tr>
<tr>
<td>R-113</td>
<td>0.040</td>
<td>5</td>
</tr>
<tr>
<td>Naphthenic Oil</td>
<td>0.072</td>
<td>11</td>
</tr>
</tbody>
</table>

In addition to the potentially small change in the thermal conductivity over the oil concentration range of interest, the measurement of this property in a refrigeration system seemed rather difficult. Standard methods for measuring the thermal conductivities of liquids use a stagnant column of the test fluid. A heat flux is imposed, and a small temperature difference is then measured over the fixed-length column of the test fluid. The inherently high errors in measurements of this type, combined with the potentially small variation of the thermal conductivity with oil concentration, indicated that the success of an oil concentration sensor based on thermal conductivity measurement was very doubtful. Consequently, no further investigation of thermal conductivity was attempted.

Surface Tension

Table 2.5 lists surface tension values for some of the refrigerants and oils of interest. Although the test temperatures are not all the same, these values reflect that the surface tensions of liquid oils are significantly greater than those of liquid refrigerants. Jensen and
Jackman [11] give the following equation for the surface tensions of mixtures of a typical naphthenic oil and R-113:

\[
\sigma_m = \sigma_r + (\sigma_o - \sigma_r) \sqrt{1 - W}
\]  

(2.8)

Using this equation, it can be seen that a ten weight-percent mixture of R-113 and the naphthenic oil has a surface tension some 18% greater than the pure refrigerant. Compared to most of the other properties in this study, this represents a very large change in the property due to the presence of oil in the mixture.

Despite the promisingly large change in the surface tension due to oil in the mixture, only bench-top devices for the measurement of surface tension were found in the literature [11]. These devices are somewhat delicate, and no method for reproducing their function in a flow stream environment was found. Consequently, the investigation of surface tension was not pursued further.

TABLE 2.5. Surface tensions of liquids

<table>
<thead>
<tr>
<th>Fluid Type</th>
<th>Surface Tension</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>8.9 (80 F)</td>
<td>18</td>
</tr>
<tr>
<td>R-22</td>
<td>8.0 (80 F)</td>
<td>18</td>
</tr>
<tr>
<td>R-290</td>
<td>6.6 (77 F)</td>
<td>19</td>
</tr>
<tr>
<td>R-113</td>
<td>15.3 (117.8 F)</td>
<td>11</td>
</tr>
<tr>
<td>Naphthenic Oil</td>
<td>25 (117.8 F)</td>
<td>11</td>
</tr>
</tbody>
</table>

Acoustic Velocity

The data available for the acoustic velocity in liquid refrigerants and oils were quite limited. The few values which were available are presented in Table 2.6. These data indicate that the acoustic velocity
in oil is some two to three times higher than in a liquid refrigerant. This large difference in the acoustic velocities of the pure components indicated a possible basis for oil concentration measurement.

TABLE 2.6. Acoustic velocities of liquids

<table>
<thead>
<tr>
<th>Fluid type</th>
<th>Acoustic Velocity, ft/s</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>1421</td>
<td>20</td>
</tr>
<tr>
<td>R-290</td>
<td>2106</td>
<td>5</td>
</tr>
<tr>
<td>Typical Oil</td>
<td>4000-4500</td>
<td>21</td>
</tr>
</tbody>
</table>

No in-line device for the measurement of liquid acoustic velocities was located in the literature. In addition, no standard laboratory device for the determination of liquid acoustic velocities was located. Consequently, the proposed prototype device was adapted from equipment originally designed for other purposes. The prototype device uses an ultrasonic transducer to transmit a burst of ultrasonic energy across the flow stream. The energy is then reflected to the transducer, which also functions as a receiver to detect the return echo. The acoustic velocity in the liquid can then be calculated from the known distance over which the ultrasonic burst traveled and the measured transit time of the burst. Since equipment for performing such measurements was available and the potential variation of acoustic velocity with oil concentration was relatively high, laboratory measurements of the acoustic velocities of oil-refrigerant mixtures were planned.

No predictive relationships for the acoustic velocities of liquid mixtures based on the component properties were found in the literature.
An equation for the acoustic velocity in seawater which included the effect of salinity was located [22]. An empirical equation for the velocity of sound (in m/s) in pure water at atmospheric pressure as a function of the Celsius temperature was also located [23]:

\[ c_a = 1402.7 + 488(T/100) - 482(T/100)^2 + 135(T/100)^3 \]  \hspace{1cm} (2.9)

This equation provided a useful calibration standard for subsequent experimental work.

Also located in the literature were two correlation methods for the acoustic velocity in pure liquids based on molecular structure [24]. Another method [25], modified somewhat using the original author's own work, was found to be useful for calculating acoustic velocities in pure refrigerants. The result is a semi-empirical relationship for the acoustic velocity in a liquid:

\[ c_a^2 = \frac{RT_b}{M} \left[ 101.6 - 82.4 \left( \frac{T}{T_b} \right) \left( \frac{\bar{\rho}_p}{\bar{\rho}_p - R[0.5 - 0.4(T/T_b)]} \right) \right] \]  \hspace{1cm} (2.10)

The use of absolute temperatures is required in Equation 2.10. This equation was checked using values for the acoustic velocity of R-11 [5]. These calculations showed that Equation 2.10 is within 3.2% of the data for saturated liquid R-11 over a band of temperatures from -63.7 to 74.9 F. Using this equation and data from the R-113 property tables [5], acoustic velocities for liquid R-113 at 77 F and 86 F were calculated to be 2333.7 and 2281.5 ft/s, respectively. These calculations were useful in calibrating the experimental setup, as presented in the next chapter.
Coefficient of Thermal Expansion

ASHRAE [5] reports the expansion coefficient of liquid R-12 and a typical oil to be 0.0056/F and 0.0013/F, respectively. Thus, the coefficient of expansion of a typical refrigerant is four times greater than that of the oil. However, the literature examined contained no standard devices or methods for measuring the coefficient of expansion of a liquid, much less an in-situ type of device.

The most promising way of accomplishing this measurement would be to apply the thermodynamic definition of the expansion coefficient,

$$\beta = \frac{1}{\nu} \frac{\partial \nu}{\partial T}$$

and measure a change in volume as a controlled temperature change is imposed on the sample. However, this method would necessitate the temporary capture of a sample from the flow stream, thereby eliminating any possibility of real-time measurement. In addition, the error in measuring the volume change of the sample is likely to be high and the imposed temperature change difficult to control. Because of this lack of adaptability to flow stream measurement, no further research concerning the thermal coefficient of expansion was planned.

Electrical Conductivity/Resistivity

Values for the volume resistivity—a property that is related inversely to electrical conductivity—of refrigerants and oils are shown in Table 2.7. The resistivity values for two of the pure refrigerants are seen to be within 7% of the resistivity of a typical refrigeration oil. This leaves little potential for significant variation in
electrical conductivity with oil concentration in a mixture of the two liquids. Beacham and Divers [26], however, report a measurable change in the resistivities due to the addition of one percent by volume of a naphthenic refrigeration oil. These values are also shown in Table 2.7. This result indicated some potential for the use of an electrical resistivity measurement as an indication of oil concentration.

TABLE 2.7. Volume resistivities of liquids at ambient temperatures [26]

<table>
<thead>
<tr>
<th>Fluid Type</th>
<th>Pure Fluid</th>
<th>With 1% by Volume naphthenic Oil</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Volume Resistivity x 10^{-3}, megohm-cm</td>
<td></td>
</tr>
<tr>
<td>R-12</td>
<td>5017</td>
<td>5029</td>
</tr>
<tr>
<td>R-22</td>
<td>8319</td>
<td>8331</td>
</tr>
<tr>
<td>R-290</td>
<td>7384</td>
<td>7371</td>
</tr>
<tr>
<td>R-113</td>
<td>4565</td>
<td>4572</td>
</tr>
<tr>
<td>Naphthenic Oil</td>
<td>7747</td>
<td></td>
</tr>
</tbody>
</table>

These promising results for the electrical resistivities of oil-refrigerant mixtures were cast in doubt when other problems with this type of measurement were considered. First, the extremely high resistivity values indicate that both oils and refrigerants are poor conductors of electricity. Consequently, conductivity cells of rather complex design [26,27] are required for these measurements. Very high excitation voltages, which could constitute a considerable danger in refrigeration systems in the field, are also required. A brief series of experiments revealed that electrical conductivity meters of the type routinely used for water purity testing were of inadequate range for measurements in oil-refrigerant mixtures.
Of added concern was the long-term stability of the electrical resistivity in oil-refrigerant mixtures. Eiseman [27] reports that oil-refrigerant mixture samples removed from a refrigeration system with 0.2 weight-percent oil in R-22 had resistivities two orders of magnitude lower than pure R-22. Eiseman suggests that the reason for the drop in resistivity is ionization in the oil-refrigerant mixture during system operation. This apparent lack of stability of the resistivity, combined with the aforementioned problems associated with its measurement, eliminated electrical resistivity or conductivity as potential bases for an oil concentration sensor.

**Dielectric Constant**

The literature concerning the dielectric constant of refrigerants and refrigeration oils is extremely limited, despite the importance of this property in hermetically sealed systems. In addition, the values in the literature indicated a very sharp temperature dependence of the dielectric constant. Table 2.8 presents the values of dielectric constant which were found in the literature. The test temperatures for the values in this table were approximately 86 F.

<table>
<thead>
<tr>
<th>Fluid type</th>
<th>Dielectric Constant</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>2.13</td>
<td>18</td>
</tr>
<tr>
<td>R-22</td>
<td>6.11</td>
<td>18</td>
</tr>
<tr>
<td>R-290</td>
<td>1.27</td>
<td>26</td>
</tr>
<tr>
<td>R-113</td>
<td>2.44</td>
<td>18</td>
</tr>
<tr>
<td>Naphthenic Oil</td>
<td>2.0-2.3</td>
<td>26,27</td>
</tr>
</tbody>
</table>
As seen in the table, the dielectric constants of R-12 and R-113 are similar to that of oil, while R-22 and propane have dielectric constants three times higher and 50% lower, respectively.

A factor which encouraged the use of a dielectric constant measurement was the simplicity of the usual measurement technique found in the literature. As described by Smyth [28], the dielectric constant can be determined by first measuring the capacitance of a test capacitor with the fluid in question filling the space between the cell plates. This measured value is then divided by the capacitance of the same cell filled with air at standard conditions. An impedance bridge circuit is the usual way of measuring the unknown capacitances, although other techniques are used for more precise measurements [28,29]. Because of this simple and potentially accurate measurement method, laboratory investigations with oil-refrigerant mixtures were planned.

**Light Absorption**

Literature concerning the light absorption properties of oil-refrigerant mixtures is essentially nonexistent. The general literature on spectrophotometric techniques, however, revealed that light absorption measurement is a standard method of determining the concentration of dissolved components in solutions. Therefore, some form of spectrophotometric measurement seemed promising for the determination of oil concentration.

The general law which is most useful in light absorption tests for concentration determination is usually called Beer's Law, although it is
often called the Beer-Lambert equation [30]. Other references [e.g., 31] insist that the work is most properly attributed to Beer and Bouguer. In any case, the most useful forms of the relationship are presented in Equation 2.12:

\[ A = \ln(1/t) = \ln \left( \frac{I_0}{I} \right) = abc \]  

(2.12)

This equation defines the absorbance, \( A \), as the logarithm of the inverse of the transmittance, \( t \). The transmittance, in turn, is defined as the ratio of the intensities of the light transmitted through the pure solvent to the light transmitted through the sample, \( \frac{I_0}{I} \). The absorbance is further shown to be directly proportional to the concentration of the dissolved substance, \( c \), through two parameters, \( a \) and \( b \). The parameter \( a \), absorptivity, is a physical property of a particular pure substance. This property is usually a function of the wavelength of incident light and temperature. The parameter \( b \), the length of the optical path through the solution, is usually a known constant. Thus, once the absorptivity is determined for a particular solution via standardization tests with solutions of known concentration, the concentration of an unknown solution can be calculated from the measured absorbance (or transmittance) and the remaining parameters.

This scheme seemed uniquely suited to the oil concentration determination task, even without the benefit of actual absorbance data for support.

In the case of the naphthenic oils, a visible range spectrophotometric method was suggested by the simple fact that the oil possesses a visible color and the refrigerants do not. The naphthenic oils are yellow in color, meaning that they absorb light in the blue region of the
visible spectrum. Because the refrigerants are colorless, solutions of varying color intensity result from mixing naphthenic oils and refrigerants.

The paraffinic and synthetic oils are essentially colorless, indicating that visible range spectrophotometry would produce no useful results. Thus, for light absorption measurements to be of benefit in concentration determination for mixtures of these oils, wavelength bands in the ultraviolet or infrared regions would need to be explored.

The literature did reveal that light absorption measurements on flowing fluid streams were possible. The instrument manufacturers' catalogs contain many types of flow-through test cells. These cells, typically called cuvettes, are not designed for fluids under pressure or at elevated temperatures. Even with this lack of a readily available method for flow stream measurements in a refrigeration system, a laboratory investigation of light absorption properties was determined to be in order.

Refractive Index

Values for the refractive indices of pure liquids obtained from the literature are given in Table 2.9. The values in Table 2.9 show a rather small variation of the refractive indices of the pure components, ranging from about 10% to 20%.

Figure 2.4, adapted from Takeo and Hattori [32], depicts a refractometer constructed from an optical fiber. As shown in Figure 2.4a, the sensor itself is merely a bend in the fiber with the surrounding cladding removed. Source light is transmitted down the fiber
Fig. 2.4. Diagram of optical fiber refractometer
(a) probe
(b) measurement system
as shown in Figure 2.4b. When the light rays reach the bend in the fiber with the cladding removed, some of the rays will be refracted back inside the cable while others will be refracted into the surrounding medium. An attenuation of the source light is thereby produced, and if the radius of the bend in the fiber is kept constant, the amount of attenuation is dependent only on the refractive index of the medium surrounding the bend. The amount of attenuation can be measured by a photodetector as in Figure 2.4b, the output of which can then be calibrated directly for refractive index measurement. Takeo and Hattori [32] have also shown that the sensitivity of the device is controllable by the selection of the fiber diameter and the bend radius. Also reported in this paper is the measurement of refractive indices correct to the third decimal place. Bergman et al. [33] successfully used a fiber-optic probe of this type to infer the local salinity in a stratified salt solution. The successes reported in these two references, together with the noted differences in the refractive indices of the pure components of the oil-refrigerant mixtures, indicated that a laboratory investigation of the mixture refractive index would be a promising endeavor.

**TABLE 2.9. Refractive indices of liquids at 77 F**

<table>
<thead>
<tr>
<th>Fluid type</th>
<th>Refractive Index</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-12</td>
<td>1.285</td>
<td>18</td>
</tr>
<tr>
<td>R-22</td>
<td>1.234</td>
<td>18</td>
</tr>
<tr>
<td>R-290</td>
<td>1.34</td>
<td>18</td>
</tr>
<tr>
<td>R-113</td>
<td>1.357</td>
<td>18</td>
</tr>
<tr>
<td>Naphthenic Oil</td>
<td>1.50</td>
<td>6</td>
</tr>
</tbody>
</table>
CHAPTER 3
PRELIMINARY PROPERTY MEASUREMENTS

Because the data available in the literature were so limited, the task of establishing property gradients with oil concentration relied heavily on laboratory investigations. As discussed in the previous chapter, the following oil-refrigerant mixture properties were selected for laboratory investigation:

- density/specific gravity
- viscosity
- acoustic velocity
- dielectric constant
- light absorption
- refractive index

An important consideration in designing the preliminary property measurement experiments was to maximize the number of properties that could be investigated within a reasonable time. To simplify these property measurements, R-113 was chosen as the refrigerant component of the mixtures. The high normal boiling point (117.8 °F) of R-113 eliminated the need for pressurized test chambers to maintain the mixtures in the liquid state. Thus, the experiments could make use of readily available bench-top property measurement instruments. Most of the equipment was available either within the Iowa State University Department of Mechanical Engineering or through the ISU Research Equipment Assistance Program.

The six property investigations presented in this chapter are
organized in a common format, which includes a description of the experimental equipment and procedure, results of the property measurements, and a discussion of the potential adaptability of the property measurement to oil concentration sensing. Comparisons with predictive relationships for the properties are presented where such relationships were available. The chapter concludes with a brief summary section which includes recommendations for prototype development of four oil concentration sensors.

Density/Specific Gravity

Experimental procedure

A very simple experimental procedure was followed for the density measurements. Direct gravimetric measurements of mixture densities were accomplished by weighing known volumes of the oil-refrigerant mixture samples at selected temperatures. This procedure was somewhat complicated by the evaporation of liquid R-113 at room temperatures and pressures, a process that was accelerated when the temperatures were elevated. To minimize this evaporation, glass bulbs with very small openings were used as the sample containers. The openings were further reduced by cementing a 1 mm I.D. stainless steel tube within each vial opening. The vials were of 15 ml nominal volume, with the exact volume of each vial being determined by calibration with distilled water. These calibrations were repeated at several temperatures and no measurable change in the vial volumes due to temperature was observed.

All vial calibration and density determination weighings were
performed with an Alnsworth 24N 160 g Electronic Balance. This balance was certified accurate to 0.00001 g. After calibration, seven of the vials were filled with mixtures of R-113 and a 150 SUS naphthenic oil in concentrations of 0, 1, 5, 10, 20, 30, and 100 weight-percent oil. The vials were filled at room temperature, which was in all cases below 77 F. The vials were then submerged almost totally in the well of a Haake B81 constant temperature bath and their temperatures raised to 77 F. As the mixtures expanded, the excess fluid was allowed to escape from the vials through the 1 mm opening. After attaining thermal equilibrium with the bath, each vial was removed and thoroughly dried on the exterior. The vials were then weighed and the densities calculated using the weights of the vials and mixtures, the known weights of the empty vials, and the known volumes of the vials. The tests were then repeated at temperatures of 86, 95, 104, 113, and 117.8 F. Random replications of the tests were performed to confirm the results.

Results

The experimentally determined mixture densities are presented in Figure 3.1 in specific gravity units. Because the combinations that were replicated produced very precise results, only mean values of the experimental determinations are presented on this plot. These data reflect an average decrease in the oil-refrigerant mixture densities of approximately one percent for each weight-percent increase in the mixture oil concentration. A temperature sensitivity of approximately -0.2%/F to -0.3%/F is also indicated. Although the temperature effect is only about one-tenth the magnitude of the oil concentration effect, a
Fig. 3.1. Specific gravities of 150 SUS naphthenic oil/R-113 liquid mixtures
temperature measurement in concert with the density measurement would be necessary for the inference of oil concentration.

Figure 3.2 compares the experimentally determined densities at 113°F with those predicted by Equation 2.1. Some departure from the ideal mixture density is evident, particularly at the higher oil concentrations. This indicates that the components were associated in solution, which violates the additive volume assumption used in the derivation of Equation 2.1. However, because of the small magnitude of departure from ideal mixture behavior, no correction factors for Equation 2.1 are recommended herein.

**Density-based oil concentration measurement**

Figure 3.3 presents a comparison of the ASHRAE-specified prediction limits with the projected prediction limits for the vibrating U-tube densimeter described in the previous chapter. Both sets of prediction limits are based on the best-fit curve through the density measurements at 113°F. This temperature was chosen because it best represents the slightly subcooled state in the liquid line of a refrigeration system. One set of prediction limits has been constructed about this curve using the ASHRAE statement of required measurement accuracy. The other set of prediction limits represents the estimated performance of the proposed oil concentration sensor and is based on the densimeter manufacturer's accuracy specification. The method of constructing these prediction limits is detailed in Appendix 1. The use of these prediction limits is very simple. As long as the prediction limits for the proposed instrument are inside the band formed by the ASHRAE prediction limits,
Fig. 3.2. Comparison of ideal and measured specific gravities for 150 SUS naphthenic oil/R-113 mixtures
Fig. 3.3. Densimeter prediction limit comparison
the combination of the sensor uncertainty and the sensitivity of the property to oil concentration is sufficient for oil concentration measurement. In this case, it is evident that the vibrating U-tube densimeter is sufficiently accurate at low oil concentrations, but not at the upper end of the range. The proposed sensor is, however, very close to meeting the specification over the entire range.

Other factors influencing the potential adaptability of a vibrating U-tube sensor include the effects of temperature, bubbles in the flow stream, and the expected pressure drop through the instrument. Densimeters typically include an integrated temperature sensor for reporting the temperature within the tube, thereby providing a method of accounting for the observed temperature effect on oil-refrigerant mixture density. Clearly, a measurement of this type is a poor choice for two-phase environments, since the vibrating U-tube densimeter bases its measurement on the actual mass in the tube. Any bubbles in the flow stream would, at least temporarily, decrease the bulk density of the mixture and give an erroneously high estimate of the oil concentration. In addition, the densimeter itself may cause the bubbles to occur, since the pressure drop through the densimeter could cause flashing in barely subcooled or saturated liquid flow streams. Therefore, precautions to ensure sufficient subcooling through the densimeter would be necessary in order to use this instrument as an oil concentration sensor.
Viscosity

Experimental procedure

Two methods were used to measure the viscosities of the liquid mixtures. The first involved the use of a common laboratory device, the Canon-Fenske Routine (C-F) viscometer. This method involves timing the efflux of a known quantity of fluid through a capillary tube as it flows under the influence of gravity alone. The measured efflux time is then multiplied by a calibration constant to obtain the viscosity. The devices are of glass construction and designed to be submerged in a liquid bath for control of the test temperature.

Two problems became apparent after a limited number of measurements were made with the C-F viscometer. First, these viscometers had certified calibrations at only two temperatures, 100 and 210 F. There is no provision, either in the manufacturer's instructions or the standard [34] which governs the use of these devices, for performing tests at other temperatures. Second, these viscometers are open to the atmosphere, presenting the possibility of refrigerant evaporation at elevated temperatures. Because of these two problems, viscosity data obtained with the C-F viscometer were not used in the analysis and are not reported herein.

Another type of viscometer proved to be very successful. A simplified falling-ball viscometer, manufactured by the Gilmont Instrument Company, was purchased for this study. The device consists of a precision-bored glass tube into which the fluid is introduced. After filling the tube, a steel or glass ball is inserted and the tube is
sealed with a screw-cap. Once sealed, the tube can be submerged in a water bath to obtain steady test temperatures. The clearance between the ball and tube is very small, resulting in Stokes flow over the ball as it falls slowly under the influence of gravity. Two sets of marks are provided on the tube, and the time needed for the ball to pass between the marks is recorded as the indication of viscosity. The viscosity of the fluid is then calculated from an equation provided by the manufacturer of the viscometer:

$$\mu_m = K \tau (\rho_b - \rho_m)$$

(2.13)

In this equation, the constant $K$ is determined by calibrating the device with fluids of known viscosity in the same range as the test fluid. The density of the ball is given by the manufacturer, but the density of the test fluid must be determined. In the oil-refrigerant experiments, the ideal mixture density from Equation 2.1 was used to reduce the viscosity data. A different calibration constant $K$ was determined for each test temperature, using viscosity values from ASHRAE [5] for pure R-113 as the calibration standard.

The remainder of the experimental setup included various clamps and stands to hold the viscometer in a water bath. The clamp/stand system was shock-mounted to eliminate the effects of vibration through the lab bench and was equipped with a leveling device to ensure verticality of the viscometer tube. The bath consisted of a Haake B81 constant temperature bath which recirculated water through a four-gallon Pyrex jar. The latter was necessary because the Haake bath did not have transparent sides for viewing the falling ball.
The tests were conducted using mixtures of a 150 SUS naphthenic oil and R-113 in concentrations of 0, 1, 5, 10, 20, and 30 weight-percent oil. Test temperatures were 68, 77, 86, 95, 104, 113, and 117.8 F. Viscosity measurements for each combination of oil concentration and temperature were performed ten times.

Results

The results of the viscosity testing are presented in Figure 3.4. Due to the extremely tight grouping of the replicated tests, only the mean values of the ten readings at each test combination are presented on this and subsequent plots. Figure 3.4 shows that the variation of viscosity with oil concentration is quite significant, as expected from the results of the literature search. The average increase in viscosity over the range of oil concentrations tested is 4% for each weight-percent increase in the oil concentration. This is the largest average gradient with oil concentration of any of the properties tested in this study. As expected, the oil-refrigerant mixtures show a significant decrease in viscosity with increasing temperature, which is typical of most liquids. The average change in viscosity is approximately -1%/F to -2%/F.

Figure 3.5 is a comparison of the data with the predictive liquid mixture viscosity relationships from Equations 2.3 through 2.6. As can be seen from the plot, Equations 2.3 [12] and 2.4 [13,14] substantially underpredict the mixture viscosities. Equation 2.5 fits the experimental data a little better, but is simply an empirical curve-fit by Jensen and Jackman [11] of their own data. Jensen and Jackman's data were collected at 117.8 F, which is the reason for the choice of temperature in this
Fig. 3.4. Viscosities of 150 SUS naphthenic oil/R-113 liquid mixtures
Fig. 3.5. Comparison of viscosity measurements at 118 F with four predictive relationships for mixture viscosities
comparison. Figure 3.5 shows that Equation 2.6 [15] describes the current data much better than the other relationships.

**Viscosity-based oil concentration measurement**

The estimated prediction limits for the magnetic viscometer are compared with the ASHRAE specifications in Figure 3.6. The prediction limits for the viscometer are shown to fall within the band formed by the ASHRAE prediction limits throughout the range of interest. This result indicated that viscosity measurements with this device could be used to infer oil concentration with the desired accuracy.

Several unique problems could affect the potential use of the viscometer for measurements in a refrigeration system. First, the small volume of fluid taken through the viscometer bypass loop moves quite slowly. As a result, a significant time lag would be introduced into the oil concentration measurement. However, an advantage of the slow flow rate through the bypass loop is that additional subcooling could be supplied to the oil-refrigerant mixture. Thus, with carefully designed supply piping, this sensor could be used in situations where the main flow stream is two-phase. Other potentially significant effects on the viscometer operation could be caused by contaminants and two-phase flow in the sensor itself. Because the clearance between the viscometer bobbin and spindle is very small, solid particles or sticky residues from oil breakdown could cause sensor malfunction. The presence of the vapor phase within the viscometer itself would also result in faulty operation. The viscometer also offers the advantages of an internal temperature sensor and rugged, field-suitable construction. The internal temperature
Fig. 3.6. Viscometer prediction limit comparison
sensor would provide information necessary for the inference of oil concentration from the viscosity measurement.

**Acoustic Velocity**

**Experimental procedure**

The variation of acoustic velocity due to oil concentration in the mixtures was measured using an ultrasonic transducer designed for detecting very small changes in the lengths of bolts subjected to varying high stresses. The device is known simply as an ultrasonic bolt gauge and was manufactured by Raymond Engineering. The transducer consists of a single piezoelectric crystal which acts as both the transmitter and receiver of an ultrasonic signal. The transducer is surrounded by a very strong permanent magnet for the purpose of attachment to a steel bolt. When the transducer is placed in acoustic contact with an object, the transmitted signal propagates through the object until it strikes a sharp change in acoustic impedance, such as the steel/air interface at the end of a bolt. Part of the signal is reflected from the interface, and the electronics unit of the bolt gauge is designed to time the interval between the initial transmission and the return of the first reflection. Knowing the travel time of the signal and the acoustic velocity of the medium, the length of the object can be calculated in a manner identical to classical sonar soundings.

In the oil-refrigerant experiments, a test cell of known length was constructed from copper tubing, as shown in Figure 3.7. A steel washer bonded in place near the top of the cell provided a mounting surface for
Fig. 3.7. Diagram of acoustic velocity test cell
the transducer. The test procedure involved filling the cell with the

test fluid to a level just above the transducer mounting surface,
attaching the ultrasonic transducer, and submerging the cell in the well
of a constant temperature bath. With this arrangement, the ultrasonic
signal propagated through the test fluid, struck the bottom of the cell,
and reflected to the transducer, thereby providing an indication of the
cell length. The instrument driver and display electronics package
included a compensation setting to account for differences in the
acoustic velocities of different bolt materials. This setting was
adjusted so that the acoustic path length measured by the instrument with
pure R-113 in the cell at 77 F corresponded to the known length of the
cell, approximately 1.9 in. The setting remained constant for all
subsequent tests. The bolt gauge displayed the apparent length of the
test cell with a resolution of 0.0001 in.

The tests were conducted at temperatures of 77 and 86 F, using
mixtures of R-113 and either a 150 SUS naphthenic, paraffinic, or
alkylbenzene refrigeration oil. Tests were conducted for a variety of
oil concentrations from 0 to 30 weight-percent, with the most complete
testing being performed using the naphthenic oil mixtures.

To convert the test cell lengths to values of acoustic velocity, the
acoustic velocity at a reference condition was necessary. In this case,
the calculated value for pure R-113 at 77 F calculated from Equation 2.10
was used as the reference. The calibration method involved simply
multiplying this reference acoustic velocity by the ratio of the known
test cell length to the indicated test cell length at any test
combination. The method was checked using distilled water at various temperatures and Equation 2.9 for the acoustic velocity in water. This check of the calibration method gave excellent results.

As a final check on the test method, an oscilloscope was connected in parallel with the bolt gauge electronics unit across the output of the transducer. In this manner, the time between the transmitted ultrasonic burst and the first return signal was measured on the oscilloscope screen. This procedure was followed for pure R-113 at 77 and 86 F, with the results being within 3.5% of the values calculated from Equation 2.10. This result verified the standard value of R-113 acoustic velocity which was used as the basis for the calculations.

Results

Figure 3.8 presents the results of the acoustic velocity tests for the naphthenic oil/R-113 mixtures. This plot shows that the acoustic velocity increases more than 1% for each weight-percent rise in the oil concentration. Using the two test temperatures available, it appears that the acoustic velocities of the mixtures change by about -1%/F.

Figure 3.9 shows the effect of oil type on the acoustic velocities. Obviously, enough difference in the oil compositions exists so that a separate calibration for each oil type would be necessary for an oil concentration sensor. If the range of interest were confined to concentrations below ten weight-percent oil, however, a single oil-type calibration may suffice.
Fig. 3.8. Acoustic velocities of 150 SUS naphthenic oil/R-113 liquid mixtures
Fig. 3.9. Effect of oil type on oil/R-113 liquid mixture acoustic velocities at 77 F
Acoustic-velocity-based oil concentration measurement

The estimates of the prediction limits for a prototype device are presented in Figure 3.10. This plot shows that acoustic velocity measurement could be an excellent basis for an oil concentration sensor. In the construction of this plot, the measurement uncertainty in the bolt gauge was taken to be ±0.001 in, which was an order of magnitude greater than the instrument's resolution. Even with this conservative estimate of the measurement accuracy, the prediction limits fall well within the ASHRAE specified band. It is interesting to note that if the measurement uncertainty estimate is relaxed by another order of magnitude to ±0.01 in, the prediction limits are still very close to the ASHRAE specified limits. Thus, from an accuracy standpoint, the adaptation of this device shows great promise as an oil concentration sensor.

A device of this type could be mounted to monitor acoustic velocity either directly in the main flow stream or in a small receiver. A temperature measurement at the same location would be required, since a significant temperature dependence of the acoustic velocity has been observed. The effects of contamination are likely to be minor with the possible exception of solid contaminants in the acoustic path. Either small particles or a bubble in the flow stream could disrupt the acoustic signal.
Fig. 3.10. Acoustic velocity sensor prediction limit comparison
Dielectric Constant

Experimental procedure

The dielectric constants of the oil-refrigerant mixtures were measured using a simple capacitance cell and an impedance bridge. As shown in Figure 3.11, the cell was based on a variable-area air-gap capacitor of the type used in radio tuning circuits. The particular capacitor chosen consisted of 35 parallel plates and possessed a maximum capacitance of 103 pF with air filling the capacitor plate spaces. The capacitor was permanently adjusted to the maximum setting and firmly positioned on insulating mounts inside a small glass jar. The screw-type jar lid was modified to include a Teflon gasket and a BNC-type electrical connection. The connector was attached to the capacitor using flexible connections, thereby permitting the removal of the lid for cleaning and sample introduction. The sealed construction allowed the cell to be submerged in a constant temperature bath so that the effects of temperature on the cell capacitance could be measured. The cell also included a port for the insertion of a thermistor probe to ensure thermal equilibrium with the bath water before taking a capacitance reading.

The cell assembly was connected to a Hewlett-Packard 4262A LCR Meter (impedance bridge) via a 65 pF coaxial cable. The LCR meter was operated in its capacitance bridge mode using an excitation frequency of 1 kHz. This instrument provided a digital display of the attached capacitance resolved to 1 pF. After measuring the capacitances of the coaxial cable and the empty cell, the cell was filled with various oil-refrigerant mixtures, including mixtures containing naphthenic, paraffinic, and
Fig. 3.11. Diagram of capacitance test cell
alkylbenzene refrigeration oils. For each test fluid, the cell capacitance was measured at a number of temperatures from 77 to 113 F.

The parallel-plate construction of the test capacitor enabled the measured capacitance values to be easily converted to fluid dielectric constants by simply dividing the measured capacitance at each test combination by the capacitance of the empty test cell. As noted in the experimental results presented in Figures 3.12 and 3.13, this produced an "apparent" dielectric constant, since some small plate-edge and plate-to-cell interaction effects were not considered in the calculations of the dielectric constants.

Results

Figure 3.12 presents the variation of the dielectric constant in mixtures of a 150 SUS naphthenic oil and R-113. Limited measurements taken in mixtures of the other oils and R-113 indicated no significant oil type effect. As seen in Figure 3.12, the dielectric constants of the oil-refrigerant mixtures decrease by an average of 2.5% over the range of oil concentrations tested. This plot also shows that the temperature effect on the dielectric constant is even greater than the oil concentration effect.

Capacitance-based oil concentration measurement

As seen in Figure 3.12, the measurement system failed to detect a difference in the measured dielectric constant between the pure refrigerant and a one weight-percent oil concentration mixture. It should be noted, however, that this setup used the simplest means of
Fig. 3.12. Dielectric constants for 150 SUS naphthenic oil/R-113 liquid mixtures
measuring an unknown capacitance, the impedance bridge circuit. This circuit was capable of resolving the measured capacitance to only 1 pF. Improved bridge circuits used with capacitance probes in fluidized bed measurements have produced measurement resolutions of 0.02 pF [29]. Other researchers have shown that electrical heterodyne circuits can resolve unknown capacitances to within 0.06 pF [28]. Using this more conservative estimate of the attainable measurement resolution, the prediction limit comparison plot of Figure 3.13 was prepared. This plot shows that measurement accuracy sufficient to allow the use of a capacitance probe to infer oil concentration may be possible.

Among the other factors influencing the potential adaptability of a capacitance probing technique is a temperature dependence greater than the oil concentration effect. The large temperature dependence makes accurate temperature measurements very important in preserving the overall accuracy of the measurement. Other factors include the fact that the dielectric properties may be severely affected by contaminants such as water [26] and ionization of the oil molecules [27]. Both of these contaminants could cause significant conductance to occur in the mixtures which, according to Smyth [28], can cause very large errors in the determination of the dielectric constant by the test cell or capacitance probe method.

**Light Absorption**

**Experimental procedure**

Standard spectrophotometric procedures were followed in all of the tests described in this section. A number of different spectrophotometer
Fig. 3.13. Capacitance probe prediction limit comparison
test setups were used because of the different wavelength ranges to be explored. The initial work involved a visible range spectrophotometer and solutions of a 150 SUS naphthenic oil and R-113 at room temperature. The transmittance of light through samples containing these components was measured at wavelengths from 340 nm to 480 nm in steps of 20 nm. Oil concentrations in the solutions were 0, 1, 5, 10, 20, and 30 weight-percent. The test instrument was a Bausch & Lomb Spectronic 20 equipped with the standard range photodetector cell. At each test wavelength, the instrument light intensity was adjusted to give 100% transmittance with pure R-113 in the sample tube. Four replications of each wavelength-concentration combination were performed.

Variations of the tests with this spectrophotometer included extending the range into the near infrared in an attempt to detect the presence of either the paraffinic oil or the synthetic oil in the mixtures. Using an accessory kit which included a special photodetector tube and filter, the range of the instrument was extended to 900 nm. However, tests at wavelengths between 500 nm and 900 nm failed to produce any indication of the presence of any oil type in the mixtures. Another series of experiments involved heating the naphthenic oil mixtures to 110 °F before testing and then observing the spectrophotometer output as the temperature of the sample cooled to ambient. No temperature effect was observed during these tests.

Because of the failure of the visible-range spectrophotometer to detect the presence of the clear paraffinic and synthetic oils, tests using an ultraviolet spectrophotometer were also performed. The test
procedure was much the same as with the visible range tests, except that the UV spectrophotometer was a dual-beam instrument, meaning that the reference sample of pure R-113 was present in the machine at all times. The test light beam, containing the sample of interest, was then compared to the reference beam containing the pure R-113 as the wavelength was continuously varied. Another difference in these tests was that quartz cuvettes were used as the sample containers. Quartz containers are necessary for ultraviolet spectrophotometry because optical glass, as used in the visible range tests, absorbs ultraviolet light. The cuvettes used were of rather unusual design in that they were equipped with ground glass stoppers. By using these special cuvettes, the possibility of evaporative loss of the refrigerant component of the mixtures during testing was eliminated. The ultraviolet tests involved wavelength scans from 400 nm downward to around 220 nm. Samples of varying concentrations containing R-113 and each of the oils were tested in the ultraviolet range, with the number and type of tests ultimately being governed by the results, as presented in the next section.

Results

Figure 3.14 presents the results of the visible range tests for the naphthenic oil mixtures. As is obvious from this plot, the visible range light absorption is very sensitive to oil concentration, with the extent of sensitivity dependent upon wavelength. The wavelengths around 400 nm appear to be the most promising for oil concentration determination over the entire range of interest. As mentioned previously, these results were found to be essentially independent of temperature.
Fig. 3.14. Visible-range light transmission measurements in 150 SUS naphthenic oil/R-113 liquid mixtures at room temperature.
To contrast the ultraviolet range absorption behavior of the various oils, Figure 3.15 presents the absorption spectra for 0.5 weight-percent concentration oil/R-113 mixtures of the three oils under investigation. As can be seen from the naphthenic oil spectrum, the wavelength band from 360 nm to 400 nm, which produced successful results with the visible range spectrophotometer, corresponds to a region of rather gently decreasing absorbance with increasing wavelength. This result is consistent with the earlier Spectronic 20 data. It was hoped that similar regions of absorbance could be located in the alkylbenzene and paraffinic oil mixture absorption spectra. While the alkylbenzene absorption spectrum exhibits similar behavior at wavelengths near 320 nm, the paraffinic oil mixture curve is seen to possess only very abrupt changes in absorbance with respect to wavelength. These sharp changes in absorbance with respect to wavelength make precise control of the incident light wavelength absolutely necessary—a situation both difficult and expensive in practice.

While searching for a suitable bandwidth in the paraffinic oil mixture absorption spectra, an interesting and disturbing phenomenon was observed. Some of the paraffinic oil mixture samples used in the course of the investigation had been prepared some weeks previous to the tests and were carefully stored in glass bottles with sealed screw-type caps. Figure 3.16 presents the absorption spectrum of a one weight-percent oil concentration example of these aged solutions, designated as Sample 1 in the figure. The absorption spectrum labeled Sample 2 in Figure 3.16 was a mixture of identical oil type and concentration, but prepared only
Fig. 3.15. Ultraviolet/visible absorption spectra for oil/R-113 liquid mixtures
Fig. 3.16. Ultraviolet/visible absorption spectra for two samples of 150 SUS paraffinic oil/R-113 liquid mixtures.
minutes before the spectrophotometric testing. It is clear from this comparison that some additional absorbing specie is present in the aged sample, possibly the product of a reaction between the refrigerant and the oil and/or the bottle and sealing cap materials. Another possibility is an interaction between one of the materials and the additive butylated-triphenyl-phosphate (BTPP), which was later found to be present in the oil. Subsequent gravimetric analyses of the aged samples revealed the oil mass fractions to be correct in all cases, despite the disruptions in the absorption spectra. However, the unexpected appearance of this extra absorbance band in the spectrophotometric results, even under carefully controlled test conditions, cast considerable doubt on the possible use of this method as the basis for an oil concentration sensor.

Figure 3.16 also shows that the unknown contaminant had little effect on the absorption spectrum of the paraffinic oil mixture below approximately 275 nm. Consequently, an attempt was made to exercise the more standard spectrophotometric practice of using the wavelength corresponding to the primary peak in the absorbance spectrum for the purpose of solute concentration determination. Figure 3.17 shows the effect of oil concentration on the absorption spectra of mixtures containing R-113 and the paraffinic oil. As is obvious from these plots, the ultraviolet absorption spectra are greatly influenced by the oil concentration. However, the characteristics of the peaks in these absorbance spectra present additional problems with this oil concentration measurement method. First, the peaks are very narrow, once
Fig. 3.17. Effect of oil concentration on the absorption spectra of 150 SUS paraffinic oil/R-113 liquid mixtures
again requiring control of the incident light wavelength to within a nanometer or two to ensure accurate results. Second, the very size of the peaks is also a problem. Equation 2.12 shows that an absorbance of 2.0 corresponds to a transmittance of only 1%. In other words, over 99% of the incident light at the peak frequency is being absorbed by a relatively weak solution of just 2 weight-percent oil concentration. This leaves very little additional variation in absorbance for the solutions of higher concentration. Additional tests revealed that a 5 weight-percent solution of this oil caused all absorbance readings from 230 nm to 270 nm to register beyond the range of the spectrophotometer. In order to extend the useful range of this measurement to higher oil concentration, special cuvettes with shorter path lengths would be necessary.

Light-absorption-based oil concentration measurement

The prediction limit comparison plot of Figure 3.18 was prepared from a portion of the visible range spectrophotometry results for the naphthenic oil mixtures. Manufacturer's specifications for the accuracy of the Spectronic 20 were used to establish the prediction limits for the oil concentration sensor. Despite the very sharp changes in light absorption due to low concentrations, Figure 3.18 shows that the accuracy requirements cannot quite be attained at the higher oil concentrations. The choice of incident light wavelength would not help this sensitivity problem at the higher concentrations, since the curves in Figure 3.14 are equally flat above 20 weight-percent oil. Still, the prediction limits are within range at the lower oil concentrations that are likely to be of
Fig. 3.18. Visible-range spectrophotometer prediction limit comparison
the greatest practical interest.

The results of the ultraviolet absorption tests cannot be evaluated in a manner similar to the preceding data for the visible range tests. The primary purpose of the ultraviolet light-absorption experiments was to determine if the early success with the visibly colored naphthenic oil-refrigerant mixtures could be extended to the colorless oils. The problems outlined by the discussion of Figures 3.15 through 3.17 seem to indicate that similar success cannot be easily obtained. In addition, it appears that the light absorption spectra of oil-refrigerant mixtures are probably not very stable. Borchardt [35] has shown that significant darkening of both synthetic and mineral oils occurs because of reactions with R-12. This darkening would affect nearly any type of light absorption technique, thereby causing unpredictable errors in the oil concentration estimation.

In addition to the problems with darkening due to reactions in the oil-refrigerant mixtures, contaminants such as water and solid particles would also adversely affect light absorption measurements in an oil-refrigerant flow stream. The effect of bubbles in the flow stream would also be detrimental to a measurement of this type, since the bubbles would cause unpredictable scattering of the light passing through the sample. While the effect is likely to be greater attenuation of the light signal, causing an erroneously high oil concentration determination, the exact effect of a bubble on light absorption measurements is unknown.

No device for in-line light absorption measurement in a
refrigeration system could be located. Modification of a laboratory spectrophotometer would be very expensive, especially if the ultraviolet test range were to be included so that all oil types could be measured. Even simple spectrophotometers are rather expensive pieces of equipment. In addition, because of the delicate nature of spectrophotometric equipment, such a device would be suitable only for application in the laboratory.

Refractive Index

Experimental procedure

The refractive indices of the oil-refrigerant mixtures were measured using a Bausch & Lomb Dual-Prism Abbe refractometer. This device was equipped with ports to allow the circulation of water over the prisms for precise control of the sample temperature. The experiments made use of these ports, with the water being supplied by a Haake B81 recirculating constant temperature bath. A thermometer port was also supplied in the upper prism holder, and a thermistor probe was used to monitor the prism temperature at this location. In addition to the temperature control, the refractometer required modifications to prevent the evaporation of the refrigerant from the small test sample volume between the prisms. To accomplish this, the prisms were sealed from the atmosphere using a brass gasket. With the gasket in place and the prisms in the closed test position, a very small passage remained for the injection of the sample via a hypodermic needle. This experimental setup was tested using distilled water and pure R-113 as standards and was found to produce
accurate results.

The tests were conducted using mixtures of R-113 and naphthenic, paraffinic, and alkylbenzene oils in oil concentrations of 0, 0.5, 2, 5, 7.5, 15, 20, 25, and 30 weight-percent oil. The 20 weight-percent samples were later eliminated for the naphthenic oil because of an error in mixture preparation. The tests were conducted at 77, 95, and 113 F, with each test combination being performed twice.

Results

The data from the refractive index measurements are presented in Figures 3.19 through 3.21. The symbols on these plots represent mean values of the two trials at each test combination because the scatter is indistinguishable on plots of this scale. These figures show an average increase of four percent in the mixture refractive index as the oil concentration is increased to 30 weight-percent. Although this is a very small change in the measured property, the sensitivity of the property to oil concentration must always be considered in light of the attainable measurement accuracy, as will be discussed later. An average temperature sensitivity of 0.05%/F is also indicated. Although this is a low temperature sensitivity in an absolute sense, it is on the same order as the oil concentration effect. Obviously, a refractive-index-based oil concentration sensor would require a temperature measurement as well as the refractive index measurement. Bergman et al. [33] have shown that a fiber-optic refractometer can be equipped with a thermocouple for temperature measurement at the same location as the refractive index measurement.
Fig. 3.19. Refractive indices of 150 SUS naphthenic oil/R-113 liquid mixtures
Fig. 3.20. Refractive indices of 150 SUS paraffinic oil/R-113 liquid mixtures
Fig. 3.21. Refractive indices of 150 SUS alkylbenzene oil/R-113 liquid mixtures
The data of Figures 3.19 through 3.21 are combined to show the effect of oil type at a temperature of 95°F in Figure 3.22. This plot shows that a separate calibration for each oil type would be necessary, although perhaps not if the oil concentration range of interest were 10 weight-percent and lower.

Refractive-index-based oil concentration measurement

Figure 3.23 presents the prediction limit projections for the prototype device. Using the ±0.001 uncertainty given by Takeo and Hattori [32], this plot shows that the prediction limits for the refractive index measurements fall comfortably within the requirements throughout the oil concentration range of interest. Based on this result, a fiber-optic refractometer appeared to be an excellent choice for an oil concentration sensor.

As presented before, a temperature measurement would also be necessary with the refractometer. Also, this device shows excellent potential for use in flow streams containing bubbles. Because the bubbles would have a vastly different refractive index than the liquid, the disruptions in the signal due to bubbles could be eliminated by filtering. In addition, contaminants such as water and waxes should have little effect on this localized measurement technique. The effect of an overall darkening of the oils, as mentioned in the discussion of light absorption measurements, could be significant in refractive index measurement as well. It should also be noted that the halocarbon refrigerants are incompatible with the plastic optical fibers which are often used for refractometers. Careful selection of fiber materials
Fig. 3.22. Effect of oil type on refractive indices of oil/R-113 liquid mixtures at 95 F
Fig. 3.23. Optical fiber refractometer prediction limit comparison
Selection of Methods for Development into Oil Concentration Sensors

Discussions concerning the potential adaptability of each of the properties for oil concentration measurement have been presented in the individual property sections. The following section briefly summarizes these discussions in a comparative format and also introduces some new considerations in the selection of methods for continued development. The chapter concludes with the recommendation of four methods for continued development.

Six instruments have been proposed for the measurement of oil concentration: a densimeter, a viscometer, an acoustic velocity sensor, a capacitance probe, some form of modified spectrophotometer, and a fiber-optic refractometer. In evaluating the potential success of these devices as oil concentration sensors, a prediction of the accuracy of each device was the most important consideration. The effects of mixture temperature, bubbles in the flow stream, and contaminants were also considered. Also important in the selection were the suitability of the proposed sensors for use in the field and, of course, cost.

Accuracy

The prediction limit comparison plots of Figures 3.3, 3.6, 3.10, 3.13, 3.18, and 3.22 indicate that all of the proposed methods could be sufficiently accurate over at least a portion of the range of interest. The acoustic velocity probe, refractometer, and viscometer were not only
acceptable, but also showed potential for higher accuracy than specified. Although meeting the specified accuracy requirements was the most important consideration in selecting measurement methods for development into oil concentration sensors, none of the methods were eliminated on the basis of accuracy considerations alone.

**Temperature sensitivity**

Determining the sensitivity of the properties to temperature was one of the main objectives of the preliminary property investigations. However, this factor was not useful in eliminating any of the proposed methods from further consideration. With the possible exception of the light absorption measurements, all of the methods were found to have significant temperature sensitivity. Thus, a concurrent temperature measurement would be necessary for essentially all of the proposed oil concentration sensors.

**Two-phase vapor-liquid flows**

Because the usual state in a refrigeration system liquid line is only slightly subcooled, the effects of an occasional bubble on the proposed sensors were considered. The densimeter would have the most trouble with two-phase flows, since the density determination is based on a measurement of the actual mass within the sensor tube. In addition, the densimeter may contribute to the two-phase problem because of the relatively high pressure loss through the vibrating U-tube. It should be noted that the remaining sensors also cannot measure oil concentration in a two-phase environment. However, it may be possible to eliminate the
problem by mounting these other sensors either in a small receiver or a bypass circuit from the main flow stream.

Contamination

The possible effects of normal refrigeration system contaminants was the most useful consideration in eliminating certain sensors from further development. The experiences encountered concerning the observed instability of the light absorption spectra and the addition of similar observations by other researchers [35] contributed to the elimination of the spectrophotometric methods of oil concentration measurement. The capacitance probe was eliminated from consideration solely by the probable effects of contamination. Beacham and Divers [26] and Eiseman [27] showed that water and ionic components of oil-refrigerant mixture reactions can significantly increase the conductance of the oil-refrigerant mixtures. Because any appreciable conductance causes large errors in the determination of dielectric constants by capacitance probing [28], the capacitance probe was dropped from consideration for further development. The effects of these normal ionic oil-refrigerant mixture contaminants are unlikely to effect properties on which the remaining four proposed sensors are based.

Field usage

The most suitable of the proposed sensors for application to refrigeration systems in the field would be the densimeter and viscometer because these devices are ready-to-use, field-tested instruments developed for other purposes. The acoustic velocity probe would also be
applicable in the field, since it would most likely be assembled from industrial-quality ultrasonic components designed for the non-destructive-evaluation (NDE) of engineering materials. The refractometer could be suited to field operation if the laser light source described in the prototype were replaced with another type of source, which is quite possible. Any type of modified spectrophotometer, due to its fragility and complexity, would no doubt be suitable only for use in the laboratory.

Cost

The specifics of instrument cost have also not been previously discussed. The densimeter/flowmeter is available in complete form, including specific gravity, temperature and mass flow rate displays, for approximately $4600. The viscometer can be purchased either complete with viscosity and temperature displays for $2550 or with board-level electronics for $1600. The latter option would require a digital multimeter for data display. All of the densimeter and viscometer packages presented above also include outputs suitable for computer data acquisition. A prototype system for acoustic velocity measurement, including an ultrasonic transducer and pulser/receiver electronics, could be purchased for approximately $3000. A 100 MHz oscilloscope would also be needed to complete this sensor system. Thus, when support instrumentation such as voltmeters and oscilloscopes is included, the cost of the above alternatives is roughly the same.

Because the fiber-optic refractometer is strictly a prototype device, estimation of the cost of a single unit is difficult. Sufficient
components to assemble a refractometer similar to that of Takeo and Hattori [32] would cost approximately $800, excluding the cost of a modest digital voltmeter for data display. Thus, the refractometer could perhaps be assembled for significantly less than the above instruments.

The visible and ultraviolet spectrophotometers used for the measurements cost approximately $1500 and $12000, respectively. The cost of the extensive modification of either of these instruments for use in a refrigerant flow stream is difficult to estimate. This high potential cost, coupled with the likely instability of the light absorption properties due to normal oil-refrigerant reactions, led to the elimination of the spectrophotometric methods.

Recommendation

As presented above, only two of the proposed oil concentration measurement schemes could be easily eliminated from further development. Consequently, the remaining four sensors were recommended for continued development and testing. The predicted accuracies of the densimeter and viscometer compared well with the requirements. These two devices are also existing, ready-to-use instruments which would require no further development. Some unique design work would be necessary to incorporate an ultrasonic transducer into a refrigerant flow stream, but the high potential accuracy of this measurement was the compelling factor in its selection for continued development. The fiber-optic refractometer would require very extensive construction and development, as well as unique adaptation to refrigeration system use, but offered the advantages of high accuracy and low cost.
A specialized flow loop test facility was required to test the performance of the four prototype oil concentration sensors. This chapter presents details of the design and construction of the flow loop. A general description of the procedures followed for the flow loop experiments is also presented. The three chapters following contain discussions of the adaptation of the densimeter, viscometer, and acoustic velocity sensor to the flow loop environment, as well as test results specific to each sensor. The fourth oil concentration sensor, the refractometer, was not fully tested in the flow loop because it exhibited poor repeatability in preliminary flow loop experiments. A complete discussion of the development of refractive index sensors appears in Appendix 3.

Flow Loop Description

The purpose of the flow loop was to provide conditions simulating a refrigeration system liquid line (i.e., condenser outlet) for the evaluation and calibration of oil concentration sensors. Although an actual refrigeration unit could have been used for these tests, the flow loop offered the considerable advantage of more precise control over temperature, pressure, and flow rate. Even more importantly, it would not have been possible to control the oil concentration of the flowing oil-refrigerant mixtures in an actual refrigeration system. The following paragraphs present the specifications used in designing the
flow loop, an overview of the flow loop operation, and descriptions of the components and construction.

**Flow loop design requirements**

Although the sensors developed in this project are to be widely applicable, it was not practical to design a flow loop that could simulate liquid-line conditions in all types of refrigeration systems. The project was also limited in the types of refrigerants and oils to be tested because of practical considerations. Consequently, most of the design parameters for the flow loop were established by consideration of the type of system to be simulated and the test fluids of interest.

**Type of system to be simulated** As a case of general interest, a residential central air conditioner of approximately 5 ton capacity was chosen as the system to be simulated. The liquid line in such a system is often a 3/8 in copper tube that carries oil-refrigerant mixtures at flow rates of approximately 10 lbm/min. The oil-refrigerant mixture is usually subcooled by 2 to 6 F, and a representative temperature range in this line was chosen for design purposes as 70 to 120 F. With the temperature range and subcooling level selected, pressure requirements for the various flow loop components could be determined for the refrigerants of interest.

It is important to note that even though a specific system was simulated, the temperature ranges described above apply to much of the refrigeration equipment currently being used with the refrigerants of interest. In addition, the pipe size and flow rate selected for this study does not limit the applicability of the results because the oil
concentration sensors selected for study are not affected by either pipe size or flow rate.

**Test fluids**  The refrigerants of interest in this project were R-12, R-22, and R-502. Refrigerant property tables [5] show the saturation pressures for R-12, R-22 and R-502 at 120 F to be approximately 172, 274, and 297 psia, respectively. Based on these values, 300 psia was used as the working pressure.

In selecting components for the flow loop, the chemical incompatibility of the refrigerants with many elastomers and plastics was often a limiting factor. In addition to the refrigerants mentioned above, the chemical compatibilities of R-113 and R-11 were also taken into account because these low-pressure refrigerants were used as solvents to clean the loop at the completion of a test series. Information concerning chemical compatibilities of the refrigerants was obtained from product bulletins of the refrigerant manufacturers [36,37,38].

**Flow loop layout and operation**

A schematic diagram of the flow loop is presented in Figure 4.1. The flow loop used a positive displacement pump to recirculate the oil-refrigerant mixtures and a simple bypass loop for controlling system flow rate. The advantage of a pumped system over a compressor-driven system was that the absence of phase changes in the flowing mixtures ensured uniform oil concentration throughout the flow loop, provided that the oil-refrigerant liquid mixtures were in the miscible range.

As shown in Figure 4.1, a pressure vessel with a thermostatically controlled water jacket was arranged with its single discharge port
Fig. 4.1. Diagram of flow loop
attached to the pump discharge line. By regulating the saturation
temperature of the stagnant oil-refrigerant mixture within this vessel,
the system pressure was controlled. The dashed lines in the diagram
depict piping that was not used during the experiments, as explained in a
later section. In addition to its pressure control function, the vessel
also served as a dampener to decrease the pulsations from the positive-
displacement pump.

A heater was mounted in the flowstream ahead of the test section to
regulate the fluid temperature. The test section design allowed a
variety of oil concentration measuring devices to be installed and
tested. A downstream chilled-water heat exchanger was included to ensure
that the fluid was sufficiently subcooled to prevent flashing at the pump
inlet. Provisions for charging included service ports in the pressure
vessel piping and pump suction lines. A drain port was also included at
the system low point. As indicated in the diagram, the loop was instru-
mented with temperature and pressure sensors at critical locations.

The flow loop employed a novel method of oil injection and mixture
sample removal. Using the service port near the test section exit, a
small double-acting hydraulic cylinder was attached to the flow loop.
This device acted as a syringe for the extraction of a sample or the
addition of oil. To take a sample, the double-acting cylinder was first
back-charged with bottled nitrogen to a pressure higher than the flow
loop pressure. After securing the Swagelok tube connections between the
cylinder and the flow loop, the series shutoff valves were opened. The
nitrogen pre-charge was then bled away and the cylinder slowly filled
with the liquid oil-refrigerant mixture. The valves were then closed, the tubing connection was disassembled, and the filled cylinder was removed, cleaned and weighed. The oil and refrigerant components were then separated by heating and the oil residue was weighed to determine the concentration. To increase the oil concentration in the system, oil was added by filling the cylinder with oil, reconnecting it, opening the valves, and forcing the oil into the flow loop using gas pressure.

Flow loop component selection and construction

Selecting components for the flow loop required special care, due mainly to the high pressures required and the incompatibility of halocarbon refrigerants with most elastomer and plastic materials. A description of the major components in the flow loop is presented below, along with specifics of the flow loop's interconnecting piping and structure.

Pump A special type of recirculating pump was required to withstand the high inlet pressures in the flow loop. A positive-displacement, diaphragm-type pump was chosen because of previous success in a similar Heat Transfer Laboratory application. The particular pump selected was a Wanner Engineering D10B, which used three hydraulically actuated pistons to power neoprene pumping diaphragms. The pump was driven by a 1750 rpm, 1/3 hp single-phase electric motor with a ten-to-one V-belt speed reduction. This combination provided an output of approximately 1 gpm at 300 psia discharge pressure. The flow rate in the system was controlled by a 3/8 in V-stem control valve in the pump bypass loop. The pump was isolated by 1/2 in ball valves and was connected to
the flow system piping through refrigerant-compatible flexible hoses.

**Pressure vessel** An upright liquid receiver of common design was selected to serve as the system pressure vessel. The receiver displaced approximately 275 in³ and was equipped with three ports near what is customarily its top end. In the present application, the receiver was inverted to ensure that liquid covered the side discharge port. The other side port, which attaches to an internal standpipe, was used to connect the top end of a liquid-level gauge, as depicted in Figure 4.1. This port was also useful for circulating the oil-refrigerant mixture through the vessel while additional oil was being added, thereby ensuring uniform oil concentration throughout the system. This mixing process, which was performed before the start of a test series, used the auxiliary piping shown as dashed lines in Figure 4.1. The remaining port in the receiver was used to connect a thermocouple probe and a 300 psia Acco pressure gauge. The pressure gage was calibrated with a dead-weight tester and found to meet its rated uncertainty of ±0.25% of indication. Also included in the pressure vessel piping was the flow loop relief valve, which was set at 320 psig.

A plastic water jacket, which surrounded the vessel completely except in the areas around the piping connections, was bonded directly to the steel pressure vessel. The temperature within the vessel was controlled by circulating water from a Haake B81 constant temperature bath through the jacket.

The vessel was attached to the flow loop piping using Swagelok tubing connectors to facilitate removal of the pressure vessel for
thorough cleaning at the close of an experimental series. The connecting piping also included 1/2 in ball valves for isolation and directing the flow during the mixing operation.

**Heater** A U-bend section of the tubing was heated uniformly by an electric heating tape. Immersion heaters were not used because of their typically high heat fluxes, which could result in accidentally raising the oil-refrigerant mixtures above the degradation temperature. A Thermolyne heating tape of 832 W capacity was selected and attached to the tubing with aluminum tape. The heater section was then heavily insulated with preformed fiberglass pipe insulation. Thermocouple probes were installed in the flow stream near the heater inlet and outlet. Power to the heater was controlled with a variable transformer, and actual heater input power was measured with a Scientific-Columbus Exceltronics XL5C5A2 watt transducer.

**Test section** The test section consisted of a 2 ft section of 3/8 in copper tubing with Swagelok tubing connections at each end. Detachable instrument pods with pressure gauges, thermocouple probes, and sight glasses were connected to each end of the copper pipe. The test section was modified as needed to accommodate one or more oil concentration sensors along its length. A port for sampling and oil injection was included in each test section.

**Oil injector/sampler** Two hydraulic cylinders, displacing approximately 5.4 and 18.8 in³, were employed for injection and sampling. Toggle-operated valves with 1/16 in orifices were used between the cylinder and the flow loop. During sampling or injection, the valves
were temporarily connected with a short length of 1/8 in copper tubing and Swagelok tubing connectors. This system provided convenient injection and minimized dead volume during sample removal.

**Heat exchanger** A 2.5 ft section of 3/4 in copper tubing was used to jacket the flow piping near the outlet of the test section. This component removed the heat introduced by the upstream heater and prevented flashing through the flowmeter and at the pump inlet. The temperature of the water supplied to the heat exchanger was approximately 55 F.

**Flowmeter/densimeter** This device served as both the system flowmeter and as one of the oil concentration sensors. The particular device used was a Micro Motion D25 vibrating U-tube flowmeter with the optional DT7 Densimeter attachment [9]. Location of this instrument outside the test section did not allow precise control of the fluid temperature within the meter, but the device was equipped with its own internal temperature sensor. Thus, while the temperature at the density measurement location was not precisely controlled, it was precisely measured.

**Instrumentation** All thermocouples used in the loop were 6 in copper-constantan (T-type) probes with 1/16 in stainless steel sheaths. The probes were fully inserted into the flowstream to minimize errors and were attached with special bored-through Swagelok tubing connectors. Readout was accomplished with a 10-channel Fluke 2176A thermocouple thermometer. The manufacturer’s specifications for this thermometer state a measurement uncertainty of ±0.4 F.
Except as noted for the pressure vessel location, the pressure gauges were simple 3 in diameter Marsh service gauges. A Hewlett-Packard 3457A digital multimeter was dedicated to the flow loop for reading oil concentration instrument outputs. Digital displays for the flow rate and flowing density were also included.

**Flow piping**
Except where necessary to accommodate equipment, the flow piping was 3/8 in "L" series hard copper tubing (1/2 in O.D.). Most of the piping system was soft-soldered together, but Swagelok tubing connectors were included as needed to make the flow loop modular. Any of the major components could be individually detached. Pipe thread connections to equipment in the flow loop were sealed with a popular compound for refrigeration systems. Each time the flow loop was charged with refrigerant, the system was checked for leaks using an electronic halocarbon leak detector. The leak-checks were performed with R-22 gas pressurized to 300 psia with bottled nitrogen. The entire piping system was insulated with urethane foam pipe insulation.

**Supporting structure**
The flow loop was constructed over a modular steel frame that occupied a floor space approximately 2 ft by 4 ft. The tallest point in the framing was the pressure vessel support at about 6 ft. This frame supported all of the system components except the pump, which rested on the floor. A photograph of the flow loop is presented in Figure 4.2.

**General Description of the Flow Loop Experiments**
In many respects, the procedures followed for all of the oil concentration performance tests were identical. Because of this similarity,
Fig. 4.2. Photograph of flow loop
these general procedures are summarized below and are not repeated in the later descriptions of particular performance tests.

After thoroughly cleaning the flow loop with R-113 or R-11, the system was evacuated for several hours to an indicated pressure of 0 psia on the pressure vessel gauge. The flow loop was then charged with 6.00 ± 0.02 lbm of liquid refrigerant, which was measured with a 60 lbm capacity electronic balance. The flow loop pump was started and the flow rate adjusted to 6.0 lbm/min. Readings from the oil concentration sensor being tested were then recorded as the temperature of the pure refrigerant in the test section was varied from 70 to 120 F. The test section temperature was controlled using the upstream heater and the downstream cold-water heat exchanger. During these tests, the pressure vessel temperature was adjusted to an average of 4 F above the desired test section temperature to ensure that the flowing fluid remained liquid. Although it would have been simpler to maintain the system at a single high pressure during the experimental runs, conducting the tests using barely subcooled liquids was seen as a more faithful simulation of the intended service conditions.

After completing the pure refrigerant tests, oil was added to the flow loop using the hydraulic cylinder injection system described previously. Sensor performance tests were conducted in solutions containing 1, 2, 5, 10, 20, and 30 weight-percent oil. After each oil injection, the fluid was circulated throughout the entire system to ensure complete mixing of the oil and refrigerant. The range of test section temperatures for all tests was 70 to 120 F. A typical test
series, from initial refrigerant charging to completion of the 30 weight-percent tests, required approximately 16 hours. Whenever possible, two oil concentration sensors were tested concurrently, thereby reducing the time and cost of the performance tests.

**Refrigerants and oils**

The refrigerants used in flow loop experiments were R-12, R-22, and R-502. These fluids were obtained in 50 lb net weight cylinders and were manufactured primarily by the DuPont Freon Products Division. Some of the R-22 used was manufactured by Racon Chemical Company. The specifications for these products were, however, identical for both suppliers.

In addition to the refrigerants listed above, R-113 was used in a single preliminary series of flow loop experiments. This low-pressure refrigerant was obtained in 60 lb drums under its solvent trade-name, Freon TF. This chemical was also manufactured by DuPont.

Because of its overwhelming popularity for refrigeration system use, the majority of the flow loop experiments were performed with a 150 SUS naphthenic mineral oil. The particular oil used, designated RO3, was provided by the manufacturer, Calumet Refining Company.

A 150 SUS linear alkylbenzene refrigeration oil was also used in the flow loop experiments because of its superior miscibility in R-502. This oil was manufactured by DuPont under the trade name Zephron, and was provided by Kramer-Trenton Company. It should be noted that this oil is no longer produced by DuPont or the present owner of the trademark, Shrieve Refining Company. However, a very similar product is currently
available from Shrieve Refining with the trade name Zerol.

Preliminary flow loop experiments

A brief series of experiments was conducted with no oil concentration sensor in the test section to evaluate the operation of the completed flow loop. These experiments used R-113 and naphthenic oil mixtures. These mixtures required only very slight pressurization to remain in the liquid phase at temperatures up to the anticipated 120 °F test section limit. These low-pressure tests showed that the flow loop was capable of producing the desired conditions with no unexpected difficulties. The oil-injection and sampling procedures were also tested during these experiments and found to be highly satisfactory. The success of these tests indicated that performance testing of the oil concentration sensors could begin with no modification to the flow loop or test procedures.

Flow loop tests of the vibrating U-tube densimeter, viscometer, and acoustic velocity sensor are reported in the next three chapters. As stated previously, the refractometer was tested briefly in the flow loop, but the tests were not completed because of poor refractometer output repeatability. Despite continued development, the repeatability of the refractometer could not be improved. A complete discussion of the refractive index sensor development is presented in Appendix 3.
CHAPTER 5
DENSIMETER PERFORMANCE TESTING

As presented in Chapter 2, instruments that continuously monitor the density of a flowing liquid are commercially available. The preliminary property measurements and prediction limit comparisons of Chapter 3 showed that a vibrating U-tube densimeter could be successful as an oil concentration sensor. Consequently, a series of performance tests for one of these devices was planned and executed. This chapter presents a description of the densimeter, its installation in the flow loop, results of the performance tests, and an analysis of oil concentration inference from densimeter measurements.

Densimeter Description and Installation
Vibrating U-tube densimeters with acceptable specifications were available from four manufacturers. Among the four alternatives, the density measurement principle and the claimed accuracy were very similar. Due to these similarities, the choice of an instrument was based on cost. The Department of Mechanical Engineering had previously purchased a Micro Motion D25 mass flow meter for use in another experimental refrigeration system. In order to measure density with this instrument, additional signal conditioning and display components are needed. These components are provided in an optional attachment, the Micro Motion DT7 Densimeter. By using the available mass flow meter and purchasing only the densimeter attachment, the cost for this instrument was approximately $1000. The cost of a complete densimeter package from any of the four manufacturers would have been at least $4500.
A complete mathematical formulation of the densimeter operating principal has recently been published by the manufacturer [39]. Referring to Fig 2.1a, the U-tube through which the fluid flows is forced to vibrate at its natural frequency by electromagnetic coils. The drive signal for the coils is a low frequency square wave, and the period of vibration of the U-tube is measured by magnetic proximity sensors between the square wave excitation pulses. The manufacturer's discussion shows that the unknown density of the material in the tube can be calculated from the measured period of oscillation, the measured temperature, two calibration reference values, and several instrument constants. This calculation is performed by the densimeter's microprocessor and the result is presented on a digital liquid-crystal display. The two reference values are set during the calibration procedure, as will be described in a later section. One of the instrument constants used in the density calculation, the tube material spring constant, is temperature dependent. This temperature dependency is compensated by the microprocessor using the known temperature variation of the spring constant and a measured value of the tube wall temperature.

This measurement scheme produces an accurate, temperature-compensated measurement of the flowing fluid density. The rated uncertainty of the DT7 densimeter is ±0.01 in specific gravity units. It should be noted that more recent versions of the densimeter from Micro Motion have a rated uncertainty five times smaller. The integral temperature measurement of the DT7 densimeter has an uncertainty of ±3 F when the factory calibration is used. Laboratory calibration improved
the temperature measurement uncertainty to $\pm 1$ F at a confidence level of 99%. The Micro Motion sensor also provided a measurement of the true mass flow rate with an uncertainty of $\pm 0.4\%$ of indication.

The densimeter was installed in the flow loop at the location indicated in Figure 4.1. Installation of the flowmeter/densimeter outside the test section was preferred for two reasons. First, this instrument was used during all the tests as the system flowmeter in addition to being one of the oil concentration sensors. Thus, providing for convenient removal of the device by locating it in the test section was not necessary. Second, the pressure drop through the densimeter was known from the manufacturer's specifications to be approximately 1 psia for the anticipated test conditions. Thus, locating the densimeter downstream from the chilled-water heat exchanger ensured that the flow through the densimeter remained liquid despite the pressure loss, even when the test section conditions were barely subcooled. Choosing this location for the densimeter did not allow the density measurement temperature to be set as conveniently as if the instrument were in the test section, but the integral temperature sensor relieved the need for precise temperature control of the fluid flowing through the densimeter.

As received from the manufacturer, the sensor was equipped with Cajon VCO piping connections. These O-ring-sealed, zero-clearance connectors permit removal of the sensor without disassembling the adjacent piping. The connection to the 3/8 in copper flow loop piping was completed with Cajon VCO to female pipe thread adapters and Swagelok male tubing connectors. After completing the installation in the piping
system, the sensor unit was thermally insulated with fiberglass batting as recommended by the manufacturer.

The microprocessor electronics package of the densimeter was installed on the rear of the display panel shown in Figure 4.2. Connection to the sensor was accomplished with a shielded cable provided by the manufacturer. The display panel also housed the DT7 densimeter electronics and display package, as well as a small digital indicator for the mass flow rate. The flowmeter/densimeter system also provided 0-5 VDC or 4-20 mA analog signals of flow, density, and temperature. These signals would be very useful if the device were used with a computer data acquisition system, but were not needed in this application.

**Densimeter Calibration**

As stated earlier, calibration adjustment of the densimeter with two known density inputs is required. These reference values are adjusted by means of two trimming potentiometers located under the front cover of the densimeter display. The manufacturer recommends using air as one of the reference values and a liquid of known density as the other. Since the densimeter resolves the measured density to only three places after the decimal in specific gravity units, setting one of the potentiometers to indicate zero with air in the tube introduces no error. In the experiments presented below, the densimeter zero was actually set after the flow loop had been evacuated in preparation for charging with refrigerant. The densimeter power was engaged at least thirty minutes prior to calibration or use, as recommended by the manufacturer.
After charging the flow loop with the refrigerant to be used in the experimental series, the pump was started and the temperature in the pressure vessel adjusted to approximately 75 F. The flow loop heater and heat exchanger were adjusted so that the temperature indicated by the densimeter display was approximately 70 F. When equilibrium was attained, the second densimeter calibration potentiometer was adjusted so that the densimeter display indicated the known value of the pure refrigerant specific gravity at the measured temperature. Reference values for the specific gravities of the pure refrigerants were taken from the ASHRAE refrigerant property tables [5].

For each test series, the densimeter calibration was checked in the manner described above. In most cases, the densimeter settings required no adjustment after the original calibration. After checking the calibration, a series of tests was performed with only the pure refrigerant in the flow loop. The temperatures in the test section for these tests were 70 to 120 F, as described in Chapter 4. The temperatures at the densimeter location were slightly less than the test section temperatures due to the intervening heat exchanger. To demonstrate the accuracy of the densimeter and the validity of the two-point calibration scheme, Figure 5.1 is presented. This graph shows the specific gravities of pure refrigerants plotted as a function of temperature. The symbols are the values measured by the densimeter, while the lines are best-fit curves drawn through the ASHRAE reference values. This comparison of the densimeter performance with reference values for the refrigerant verifies that the densimeter was functioning
Fig. 5.1. Comparison of densimeter measurements with reference values for pure liquid refrigerants
properly and was correctly calibrated.

**Experimental Results**

The general procedures for the flow loop experiments were described in Chapter 4. No additions to these procedures are needed in reporting the results of the densimeter performance tests. It should be noted that densimeter readings were recorded only when the temperature indication on the densimeter display had attained a steady value. Again, because of the location of the densimeter downstream from the heat exchanger, the temperatures in the densimeter were somewhat lower than those in the test section. Consequently, the oil-refrigerant mixture at the densimeter location was subcooled by an additional 3 to 5 F compared to the test section. As stated previously, this additional subcooling was necessary to compensate for the pressure drop through the densimeter.

Results from the experiments are presented in Figures 5.2 through 5.5. Figure 5.2 presents data for mixtures of R-113 and naphthenic oil, which was not an oil-refrigerant combination of practical interest. This graph is presented for reference only, and no statistical analyses of these data have been performed. The series of flow loop experiments represented by these data were actually the first conducted. The purpose of these low-pressure R-113 experiments was to test the operation of the flow loop as completely as possible before proceeding with the refrigerants requiring elevated pressures.

Figures 5.3 through 5.5 present the data used in analyzing the performance of the densimeter as an oil concentration sensor. Figure 5.3 presents densimeter measurements of mixtures for R-12 and naphthenic oil.
Fig. 5.2. Flow stream densimeter measurements in 150 SUS naphthenic oil/R-113 liquid mixtures
Fig. 5.3. Flow stream densimeter measurements in 150 SUS naphthenic oil/R-12 liquid mixtures
Fig. 5.4. Flow stream densimeter measurements in 150 SUS naphthenic oil/R-22 liquid mixtures.
Fig. 5.5. Flow stream densimeter measurements in 150 SUS alkylbenzene oil/R-502 liquid mixtures.
Measurements in mixtures of R-22 and naphthenic oil are shown in Figure 5.4. Finally, Figure 5.5 presents densimeter measurements using mixtures of R-502 and alkylbenzene oil. Figures 5.2 through 5.5 are plotted with temperature as the abscissa variable because the temperature level could not be precisely set at repeatable values. As presented before, this was a consequence of locating the densimeter outside the flow loop test section.

Multiple regression analysis was used to fit an equation to each of the data sets of Figures 5.3 through 5.5. The solid lines on these figures were generated from the regression equations. The equations were of the following form:

\[ SG = b_0 + b_1 C + b_2 \theta \]
\[ + b_3 C^2 + b_4 C \theta + b_5 \theta^2 \]
\[ + b_6 C^2 \theta + b_7 C \theta^2 + b_8 C^2 \theta^2 \]

where

\[ \theta = T/T_{ref} \]

Absolute temperatures must be used in Equation 5.1, and the reference temperature is 529.67 R. The coefficients are given for the three data sets in Table 5.1.

As can be seen from Figures 5.3 through 5.5, these regression equations fit the data quite well. This visual assessment was confirmed by an analysis of variance performed on each of the fitted equations. In each case, the analysis showed a very low residual error and a multiple correlation coefficient of over 0.999. In addition to the excellent fit, the equations are of an easily grasped algebraic form, being merely the
product of second-order polynomials in each independent variable (concentration and temperature). A discussion of the regression analysis is presented in Appendix 2.

TABLE 5.1 Coefficients for Equation 5.1

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>R-12/naphthenic</th>
<th>R-22/naphthenic</th>
<th>R-502/alkylbenzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>b0</td>
<td>0.624438</td>
<td>-0.954262</td>
<td>-2.604452</td>
</tr>
<tr>
<td>b1</td>
<td>8.662384</td>
<td>19.562827</td>
<td>38.726160</td>
</tr>
<tr>
<td>b2</td>
<td>2.407836</td>
<td>5.283569</td>
<td>8.797123</td>
</tr>
<tr>
<td>b3</td>
<td>-22.505005</td>
<td>-32.131390</td>
<td>-100.594657</td>
</tr>
<tr>
<td>b4</td>
<td>-21.507901</td>
<td>-42.100150</td>
<td>-80.549271</td>
</tr>
<tr>
<td>b5</td>
<td>-1.706437</td>
<td>-3.126579</td>
<td>4.958584</td>
</tr>
<tr>
<td>b6</td>
<td>48.802555</td>
<td>67.006517</td>
<td>200.413096</td>
</tr>
<tr>
<td>b7</td>
<td>12.346718</td>
<td>22.210729</td>
<td>41.520900</td>
</tr>
<tr>
<td>b8</td>
<td>-26.296189</td>
<td>-34.929758</td>
<td>-100.140162</td>
</tr>
</tbody>
</table>

There are essentially no data in the open literature for comparison with these measurements. As presented in Chapter 3, the densities of oil-refrigerant liquid mixtures cannot be accurately predicted with an ideal mixing relationship, such as Equation 2.1. The data presented in this chapter were compared with predictions from Equation 2.1, and deviations of up to 6% were found to occur. Factors which correct Equation 2.1 for oil-refrigerant mixtures containing R-12 and R-22 have been published [6], but were not comparable because the oil type used in these published experimental data was not specified.

**Inference of Oil Concentration from Densimeter Measurements**

The primary objective of the performance tests was to determine the accuracy with which each of the sensors could be used to measure oil concentration. The performance data and regression equations presented
in the last section were the first steps in making this determination. Prediction limits for the regression curves were then constructed and compared with the ASHRAE-specified prediction limits, much in the same manner as presented for the preliminary property measurements. In this case however, the prediction limits which were compared to the specification were not estimates but statistically derived measurements of the sensor's performance. A detailed description of the construction and comparison of the prediction limits is presented in Appendix 2.

The results of the statistical analyses and prediction limit comparisons are presented in Figures 5.6 through 5.8. The coordinates have been arranged to reflect the manner in which these graphs would be used to infer oil concentration from a densimeter measurement. Specific gravity is plotted as the abcissa independent variable with temperature as the independent cross variable. Oil concentration, which is the desired unknown, is plotted as the dependent variable. To use these plots, the temperature and specific gravity coordinates measured with the densimeter would first be located on the graph. The oil concentration would then be read from the ordinate. Alternatively, the measured values of specific gravity and temperature can be substituted into Equation 5.1 with the appropriate coefficients. The result of the substitution is a quadratic equation in C, the oil concentration. Solution of the quadratic for the unknown oil concentration in then a simple matter. In most cases, only one of the two solutions for the oil concentration would be a positive value. In those rare cases where two positive roots occur, the oil concentration cannot be inferred from the specific gravity
Fig. 5.6. Inference of oil concentration from densimeter measurements in 150 SUS naphthenic oil/R-12 mixtures
Fig. 5.7. Inference of oil concentration from densimeter measurements in 150 SUS naphthenic oil/R-22 mixtures.
Fig. 5.8. Inference of oil concentration from densimeter measurements in 150 SUS alkylbenzene oil/R-502 mixtures
The uncertainty in the oil concentration measurement can also be determined directly from these plots. Regions of uncertainty are indicated on each of these plots and identified by boldface Roman numerals. The regions are bounded by heavy dashed curves. The measured values of specific gravity and temperature taken from a densimeter reading would correspond to one of the regions of uncertainty. Region I represents the conditions under which an uncertainty of $\pm 1$ weight-percent can be attained. Oil concentrations inferred from points located in Region II would have uncertainties between $+1$ and $+2$ weight-percent. Region III, which appears only on the R-22/naphthenic oil and R-502/alkylbenzene oil plots, represents conditions under which the uncertainty is greater than $+2$ weight-percent. It should also be noted that some combinations of specific gravity and temperature in Region III of Figures 5.7 and 5.8 do not correspond to a unique value of oil concentration. Therefore, attempts to infer oil concentration from measured values in Region III should be avoided.

The performance tests have shown that a vibrating U-tube densimeter can successfully be used to measure oil concentration under refrigeration system liquid-line conditions. Obviously, certain restrictions apply to this conclusion. First, adequate precautions must be taken to ensure that sufficient subcooling exists in the liquid line to compensate for the pressure drop through the densimeter. Second, the desired accuracy of $+1$ weight-percent cannot be attained under all conditions. In particular, the oil concentration in mixtures containing the lighter R-22
and R-502 at temperatures above about 90 F can be measured with an uncertainty no less than +2 weight-percent. Still, there are many applications which employ either heavier refrigerants (such as R-12) or lower liquid-line temperatures. For these latter conditions, a densimeter measurement of the type described in this chapter would be an accurate, repeatable, and relatively trouble-free method of measuring oil concentration. It should also be noted even better results may be possible with the more sensitive versions of this densimeter which have recently been introduced.
CHAPTER 6
VISCOMETER PERFORMANCE TESTING

The data obtained from the literature review and the bench-top experiments showed that the viscosity of an oil-refrigerant mixture is a strong function of the oil concentration. In addition, the literature review revealed a new type of in-line viscometer suitable for use in the liquid line of a refrigeration system. The prediction limit comparison presented in Chapter 3 showed that a viscosity measurement with this new sensor could be used as an indication of the oil concentration in the flowing mixture. Based on these findings, a viscometer of this type was obtained on loan from the manufacturer and tested under a variety of liquid-line conditions. This chapter contains a description of the viscometer operation and installation, results of the performance tests, and an evaluation of the success of the viscometer as an oil concentration sensor.

Viscometer Description and Installation

The viscometer used in these experiments operates on a unique principle and is available from only one manufacturer, Cambridge Applied Systems, Inc. (CAS). The viscosity measurement package consisted of the sensor shown in Figure 2.3 and a remote electronics package to drive the sensor and perform output signal conditioning. The sensor is quite small, with a diameter of 2 in and a length of 3.9 in, excluding the threaded connections. As shown in Figure 2.3a, the sensor must be connected in a bypass to the main flow stream. Although a vertical orientation is shown in this diagram, the sensor can be inclined at any
angle that does not place the outlet below the inlet. Once connected, the sensor measures the viscosity of the flowing liquid intermittently, as described below.

Figure 2.3b depicts the sensor in a horizontal position with the bobbin in position for viscosity measurement and the inlet check valve closed. The bobbin is machined from stainless steel and is hollow with very thin walls. Consequently, the bobbin is very light, making it almost entirely insensitive to the small vibrations inherent in piping systems. The removable spindle which houses the bobbin and other internal parts is non-ferrous and surrounded by magnetic coils. From the position shown in Figure 2.3b, the bobbin is accelerated by the magnetic coils through the fluid until it impacts the plastic solenoid spacer. During its travel, the position of the bobbin is monitored by the magnetic coils, much in the same way that a linear variable differential transformer (LVDT) monitors the position of its conductive core.

Because the bobbin moves only under the influence of the magnetic force from the coils, the time required for the bobbin to travel between two reference points can be linearly correlated with the viscosity of the fluid in the viscometer. This is essentially the same operating principle as a falling-ball viscometer, except that the gravitational force on the ball is replaced by the magnetic force from the coils on the bobbin. For orientations other than horizontal, a small gravitational force is also exerted on the bobbin, but this force can be neglected if the sensor is calibrated and used in the same orientation. The viscometer cycle is completed by energizing coils A and C, which opens
the inlet valve and pulls the bobbin to the original position. This motion of the bobbin expels the previous fluid sample and draws a fresh charge of the liquid through the inlet. The time required to complete this cycle varies linearly from 1.5 seconds at the lower end of the calibrated viscosity range to 30 seconds at the top of the range.

The range of the viscometer is primarily a function of the bobbin dimensions. Because the expected viscosities of the oil-refrigerant mixtures were very low, the range for the instrument used in these experiments was 0.1 to 2 cp. This range was considerably less than any other application attempted with this viscometer, and the bobbin was custom-made by Cambridge Applied Systems for this application. Despite the unique range of the instrument, the manufacturer was confident that the rated accuracy of +2% of indication or +0.5% of full scale could be maintained. The sensor includes an internal temperature probe, which will be discussed in a later section. The cost of the sensor and electronics package is approximately $1600. The system used in these experiments, however, was loaned to the project at no charge by Cambridge Applied Systems. In addition to the viscometer sensor and electronics package, a digital voltmeter and a 12 VDC, 1.7 A power supply are required to complete the measurement system.

The sensor was installed in the flow loop test section as shown in Figure 6.1. The urethane foam piping insulation which covered the viscometer and connecting piping was removed for this photograph. The sensor was provided with 1/8 NPT threads at both ends. Swagelok elbow adapters were used to connect the sensor to 1/4 in copper tubing. These
Fig. 6.1. Viscometer installed in flow loop test section
connections were then enlarged and teed into the 3/8 in test section piping.

Thermocouple probes were also included in the bypass piping as shown in Figure 6.1. The sensing tips of these probes were located in the Swagelok elbows very near the viscometer inlet and outlet openings. These probes were included after preliminary experiments showed that the temperature measured by the internal viscometer RTD sensor was a poor indication of the fluid temperature. A comparison of the temperature measurements near the viscometer for a typical experimental series is presented in Figure 6.2. The inlet and outlet thermocouple readings and the internal sensor reading are plotted as functions of the temperature measured in the test section main flow stream.

The significant difference between the readings was caused by two shortcomings in the sensor design. First, the thermal resistance between the fluid in the viscometer and the RTD was too large for the RTD to measure the fluid temperature accurately. Most of this resistance was caused by the unbonded contact between the RTD and the outside of the spindle tube. The resistance was further increased by the tube wall and the low convection heat transfer coefficient within the viscometer. In addition, the RTD was influenced by heat generated in the magnetic coils during operation of the device. It was this effect which caused the RTD readings to be higher than the actual fluid temperature under most conditions. Because of these errors in the RTD readings, the mean of the viscometer inlet and outlet thermocouple readings was used as the temperature corresponding to the viscosity measurement. It should also
Fig. 6.2. Comparison of temperature measurements near viscometer during a typical test series
be noted from Figure 6.2 that all of the temperature sensors indicated significant cooling of the fluid bypassed through the viscometer.

The viscometer connects to the electronics package through a shielded cable provided by the manufacturer. A small enclosure was constructed for the circuit cards of the electronics package. This enclosure included connections for the sensor, the power supply, and the DC voltage outputs of the circuit card. A single pair of output terminals was used, and a manually-actuated switch was included to select either the viscosity or temperature channel. A 12 VDC, 3 A power supply was constructed to provide power to the viscometer system. The viscosity and temperature outputs were monitored with a Hewlett-Packard 3457A digital multimeter.

**Viscometer Calibration**

A single potentiometer was provided for adjustment of the viscometer voltage output. This trimmer was located on the electronic circuit cards and was actually an adjustment of the drive current of the magnetic coils. Thus, adjustment of the trimmer effectively changed the magnetic force exerted on the bobbin. The trimmer was adjusted at the beginning of each experimental series with pure refrigerant in the flow loop. Reference values for the viscosities of pure liquid refrigerants were taken from the ASHRAE refrigerant property tables [5]. The temperature during the calibration adjustments was approximately 70 F. The trimmer was adjusted until the voltmeter display settled steadily on the reference value of the refrigerant viscosity in centipoise. This adjustment was then verified by comparing the measured viscosities of the
pure refrigerants with reference values at elevated temperatures. These comparisons have been combined into the single plot of Figure 6.3. The symbols on this plot represent the measured viscosities of the pure refrigerants and the lines are best-fit curves through the reference values. This graph shows that after calibration the viscometer was capable of detecting the small variations in viscosity caused by modest changes in fluid temperature.

Experimental Results

A general procedure for the flow loop experiments was presented in Chapter 4. No deviations from this procedure were necessary during viscometer performance testing. However, it should again be emphasized that the temperature taken to correspond to the viscometer measurements was the mean of the thermocouple readings at the viscometer inlet and outlet. Referring to Figure 6.2, this mean temperature was several degrees lower than the test section temperatures due to the additional cooling obtained by extracting the test fluid from the main flow stream. Thus, the tests were conducted for main flow stream temperatures of 70 to 120 F, even though the temperature band indicated on the data plots is somewhat narrower.

The viscometer operated very reliably during the performance tests. The presence of vapor in the bypass loop was found to interrupt the viscometer operation, but normal operation was restored once the vapor condensed. Because of the slow progress of the fluid through the bypass loop, the viscometer responded very slowly to temperature changes in the main flow stream. Although not specifically tested, the viscometer would
Fig. 6.3. Comparison of viscometer measurements with reference values for pure liquid refrigerants
be equally slow in detecting a transient change in the main flow stream oil concentration. Thus, the overall transient response of the viscometer system as an oil concentration sensor must be considered quite poor. However, since sudden changes in oil concentration in the liquid line occur only under abnormal conditions, the slow transient response would not severely limit the usefulness of the viscometer as an oil concentration sensor.

The experimentally measured viscosities are presented in Figures 6.4 to 6.6. The oil-refrigerant mixture combinations corresponding to these data were R-12/naphthenic, R-22/naphthenic, and R-502/alkylbenzene, respectively. Because viscosity is the property being measured, it is important to note again that all of the oils used in these experiments were of the 150 SUS viscosity classification.

The data plots once again include solid lines which represent a regression equation for each data set. The equations were of the following form:

\[ \mu = b_0 + b_1 c + b_2 \theta + b_3 c^2 + b_4 \theta c + b_5 \theta^2 \]  

where  
\[ \theta = \frac{T}{T_{ref}} \]

Using the coefficients given in the following table, Equation 6.1 gives the mixture viscosity in centipoise. The equation requires the use of absolute temperatures and the reference temperature is 529.67 R.
Fig. 6.4. Flow stream viscometer measurements in 150 SUS naphthenic oil/R-12 liquid mixtures
Fig. 6.5. Flow stream viscometer measurements in 150 SUS naphthenic oil/R-22 liquid mixtures
Fig. 6.6. Flow stream viscometer measurements in 150 SUS alkylbenzene oil/R-502 liquid mixtures
TABLE 6.1 Coefficients for Equation 6.1

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>R-12/naphthenic</th>
<th>R-22/naphthenic</th>
<th>R-502/alkylbenzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>b₀</td>
<td>0.731510</td>
<td>0.653265</td>
<td>0.706933</td>
</tr>
<tr>
<td>b₁</td>
<td>3.441837</td>
<td>6.219701</td>
<td>3.233726</td>
</tr>
<tr>
<td>b₂</td>
<td>-0.504179</td>
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<td>b₃</td>
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<td>b₅</td>
<td>-6.407845</td>
<td>9.501119</td>
<td>-4.941047</td>
</tr>
</tbody>
</table>

These regression equations fit the experimental data very well, as shown in Figures 6.4 to 6.6. The analysis of variance for the regression equations revealed very low residual errors and multiple correlation coefficients of over 0.999. These equations are very similar in form to those used for the density correlations in the last chapter, with the exception that the temperature dependence has been reduced to a first-order polynomial. For details of the regression analysis, the reader is once again referred to Appendix 2.

No data were available from the literature for comparison with these measurements. As presented in Chapter 2, the published oil-refrigerant liquid mixture viscosity data concern the oil-rich mixtures which occur in compressor crankcases. The predictive relationships for mixture viscosities presented in Equations 2.6 to 2.9 were compared to the viscometer measurements. The comparisons were performed for all of the oil-refrigerant combinations tested and for several temperatures in the test range. The equations were not found to predict the mixture viscosities accurately under these conditions. Except for very low oil concentrations, the differences between the data and the equations were
greater than 5%.

Inference of Oil Concentration from Viscometer Measurements

Using the property data and regression equations, estimates of the accuracy with which oil concentration can be predicted from viscometer measurements were developed. The analysis was performed in the same manner as presented for the densimeter in the previous chapter. The results of the analysis are presented in Figures 6.7 to 6.9. These graphs show oil concentration plotted as a function of two independent variables, temperature and viscosity. Regions of uncertainty are once again bounded by heavy dashed lines. Figure 6.7, which is for R-12/naphthenic oil mixtures, shows no uncertainty region boundaries because the prediction uncertainty in the entire domain was found to be +1 weight-percent or less. The remaining two oil-refrigerant combinations required the use of two uncertainty regions on the prediction plots, with the maximum uncertainty being +2 weight-percent.

Factors other than accuracy must also be considered in evaluating the viscometer as an oil concentration sensor. Certain drawbacks to the viscometer's bypass arrangement were noted during the tests. These drawbacks consisted of the additional temperature probes required for accurate temperature measurements and the very slow response to changes in the main flow stream conditions. The viscometer manufacturer had previously noted the problems with the internal temperature sensor and new viscometers with improved built-in temperature sensors were in the prototype stage at the time of this writing. In addition, new sensors
Fig. 6.7. Inference of oil concentration from viscometer measurements in 150 SUS naphthenic oil/R-12 mixtures.
Fig. 6.8. Inference of oil concentration from viscometer measurements in 150 SUS naphthenic oil/R-22 mixtures
Fig. 6.9. Inference of oil concentration from viscometer measurements in 150 SUS alkylbenzene oil/R-502 mixtures.
which would install directly in the main flow stream, eliminating the bypass line, were also under development.

Although no operational problems with the viscometer due to contamination were encountered, the very small clearances between the viscometer's moving parts suggest that such problems could occur in field installations. Small solid contaminants could conceivably alter or interrupt the viscometer operation. A simple liquid-line strainer, as typically installed at the inlet to a refrigeration system expansion valve, would eliminate this potential problem. The strainer could be located in the viscometer inlet bypass piping without affecting the operation of the instrument.

The analysis of the performance test data has shown that viscometer measurements can be used to measure oil concentration under a wide variety of liquid-line conditions. The desired accuracy cannot be attained under all test conditions, but no test conditions produced uncertainties greater than ±2 weight-percent. The performance tests have also shown that the viscometer will operate reliably under these conditions. Although some drawbacks to the bypass sensor design were noted, none of these presented a significant obstacle to oil concentration measurement with the viscometer. Even without the improvements which are likely to be incorporated into future models, the viscometer is an accurate, reliable, off-the-shelf instrument for measuring oil concentration.
Although no data were located for the acoustic velocities of oil-refrigerant liquid mixtures, the literature survey revealed large differences between the acoustic velocities of pure oils and refrigerants. These large differences indicated that the mixture acoustic velocity could vary greatly with the oil concentration. The potentially large property gradient was confirmed by the results of the preliminary property measurements. The bench-top experiments also showed that acoustic velocities could be accurately measured with an ultrasonic scheme similar to classical sonar soundings. Because this measurement scheme was also adaptable for flow stream measurements, the construction and performance testing of an acoustic velocity sensor was undertaken. This chapter describes the principles and construction of the acoustic velocity measurement system, results of the performance tests, and an analysis of the accuracy with which oil concentration can be inferred from such a measurement.

**Acoustic Velocity Sensor Description**

A method for measuring the acoustic velocities of liquids with an ultrasonic transducer was discussed in conjunction with the preliminary property measurements in Chapter 3. These experiments made use of an ultrasonic bolt gauge borrowed from research colleagues studying the behavior of bolted pipe flanges. This device was designed for measuring distances in a metallic medium in the same way that sonar soundings
measure distances in water. Although the experimental setup was quite successful, the ultrasonic bolt gage was neither available nor particularly well-suited for adaptation to flow stream measurements in a refrigeration system liquid line.

Ultrasonic pulse-echo instruments are employed in several everyday applications. One example is the use of this technique by sport fishermen for locating lake bottoms and other underwater objects. An inexpensive example of one of these depth-finders was obtained in disassembled form. However, no serious effort was made to adapt components of this system to an acoustic velocity sensor because the ultrasonic transducer was unmanageably large, the pulser/receiver circuitry insufficiently sensitive, and the display device too crude for use in acoustic velocity measurement. Study of this device did, however, provide a valuable primer in ultrasonic measurement fundamentals.

Highly sophisticated ultrasonic measurements are used in the non-destructive evaluation (NDE) of engineering materials. Consultation with scientists from the Ames Laboratory of the U.S. Department of Energy provided a great deal of information useful in constructing an acoustic velocity sensor from industrial NDE components. In addition to confirming the viability of the proposed ultrasonic method of measuring acoustic velocity in a flow stream, these scientists assisted in the selection of an ultrasonic transducer and provided on loan the pulser/receiver unit required to drive the transducer and process the output signals. Additional specifications for the transducer and pulser/receiver are presented in following paragraphs.
Flow stream sensor construction

The flow stream acoustic velocity sensor was to operate in the same manner as the transducer and test cell arrangement used in the preliminary property measurements. However, instead of propagating through a stagnant column of the oil-refrigerant mixture, the ultrasonic signals were directed across a channel filled with the moving fluid. Construction of the flow stream sensor involved selection of a suitable ultrasonic transducer and fabricating a housing to position the transducer perpendicular to the flow stream. Considerations in the sensor housing construction included minimizing the introduction of extra piping volume, providing a good reflecting surface across the channel from the transducer, and providing connections to the test section piping.

Transducer selection  
Ultrasonic NDE transducers can be grouped into two major classifications: contact transducers and immersion transducers. Contact transducers, like the bolt gauge used in the preliminary property measurements, are designed to be attached directly to solid materials. Immersion transducers are typically used for the imaging of submerged surfaces. Although the bolt gauge contact sensor was successfully employed in liquid during the preliminary property measurements, an immersion transducer was selected for adaptation to the flow stream measurements. The smallest immersion transducer readily available from a manufacturer was the Panametrics V310-SU. This transducer was cylindrically shaped with an overall diameter of 0.63 in and an immersion length of 1.25 in. The ultrasonic signal was emitted
from one end of the cylinder, and the signal cable attached to the other end. The actual piezoelectric element had a diameter of 0.125 in and was embedded in a special potting material near the emission end. Although this small element produced comparatively weak ultrasonic pulses, consultation with the manufacturer indicated that adequate power for the simple pulse-echo acoustic velocity measurement would be available. Also upon the advice of the manufacturer, a transducer with a pulse center frequency of 5 MHz was chosen for this application. The transducer and connecting cable were purchased from Panametrics for $242.

**Transducer housing** A convenient housing for the small ultrasonic transducer was constructed from a 1/2 in ball valve, as depicted in Figure 7.1. The ball and stem of the valve were removed and the stem opening bored to the transducer diameter. Figure 7.1 presents an end-view of the completed assembly, with the transducer and reflecting surface cemented in position using refrigeration system epoxy. The reflecting surface was hand-crafted of aluminum and the unmounted transducer was used to align the surface and ensure that the emitting and reflecting surfaces were parallel. After cementing the reflecting surface in place, the loose transducer was raised into position and a gauge block of approximately 0.5 in inserted between the transducer and reflecting surface. The assembly was then firmly clamped and the transducer cemented in place. A safety collar was fabricated over the outside of the valve body to retain the transducer in the unlikely event that the cement should suddenly fail under pressure. To complete the sensor housing, Swagelok tubing connectors were used to adapt the pipe
Fig. 7.1. Diagram of acoustic velocity sensor
thread ends of the valve body to the 3/8 in test section piping. A photograph of the completed sensor is presented in Figure 7.2.

**Additional measurement system components**  The transducer converts an electrical input signal to a burst of ultrasonic energy. The electrical signal was provided by a Panametrics 5052PR pulser/receiver. This device also detected the reflections received by the transducer and provided an oscilloscope connection for displaying the transducer input and output pulses. The pulser/receiver also included adjustments for the pulse repetition rate (200 to 5000 Hz) and pulse energy level. An oscilloscope synchronization pulse was also provided. A Tektronics 2236 100 MHz oscilloscope was used for displaying the pulser/receiver output.

**Measurement system operation**

Measuring acoustic velocities with the system described above was quite simple. When the sensor cavity was filled with liquid, the ultrasonic energy emitted by the transducer propagated across the flow stream, struck the reflecting surface, and returned to the transducer. Both the original pulse and the reflected pulse were displayed on the oscilloscope screen, as indicated in Figure 7.3. Additional pulses were caused by the continued bouncing of the ultrasonic burst between the transducer and reflecting surface, with each successive pulse becoming more attenuated. The output energy of the transducer was adjusted on the pulser/receiver front panel so that all reflections had been completely attenuated before the next ultrasonic burst was emitted. The time between the original pulse and the first reflection was read directly from the oscilloscope, as shown in Figure 7.3. The acoustic velocity was
Fig. 7.2. Acoustic velocity sensor installed in flow loop test section
Fig. 7.3. Example of oscilloscope display during acoustic velocity measurement.
then calculated by dividing twice the known transit distance by the measured transit time.

**Sensor Calibration**

Although the transit distance of the ultrasonic signals was considered a known constant in the above discussion, this length was actually determined by calibration of the sensor. Measuring the distance from the end of the transducer to the reflecting surface would seem the obvious way of determining the transit length. However, as stated in the description of the transducer, the piezoelectric element was surrounded by a potting material and was not visible at the end of the transducer. Because the effect of the intervening length of potting material between the actual sensing element and the fluid was unknown, calibration of the sensor with a fluid of known acoustic velocity was deemed appropriate. In addition, the effect of temperature on the transit length could be assessed through calibration.

The sensor was calibrated using distilled water as a reference standard. With the sensor’s piping connections capped and distilled water filling the sensor cavity, the entire unit was immersed in the well of a Haake B81 constant temperature bath. After allowing the system to reach equilibrium, the transit time was read from the oscilloscope screen. The acoustic velocity of water at the bath temperature was calculated from Equation 2.9. The transit length was then calculated as half the product of the known acoustic velocity and the measured transit time. These calibration experiments were performed over a temperature range of 77 to 104°F, with no effect on the transit length due to
temperature being observed. Because the sensor was disassembled for cleaning and inspection after each test series, the calibration was performed before each run. In addition, the calibration was checked again after completion of the test series. In each case the transit length was found to be unchanged after the flow loop experiments.

**Experimental Results**

The flow loop performance tests were once again conducted according to the procedure presented in Chapter 4. Immediately after charging the flow loop with refrigerant, the pulser/receiver and oscilloscope were started and allowed to warm up as prescribed by the manufacturers. The pulse repetition rate and ultrasonic energy controls of the pulser/receiver were adjusted to give a clear pulse train on the oscilloscope. The oscilloscope time and voltage ranges were then adjusted so that only the output pulse and the first reflection appeared on the screen. The voltage setting was 1V/division, while the time setting was either 5 or 10 microseconds/division, depending on the fluid in the sensor. The oscilloscope was also set for DC coupling and the external triggering signal from the pulser/receiver was employed. Transit times were then measured over the range of oil concentrations and temperatures desired. Since the sensor contained no internal temperature probe, the mean of the inlet and outlet test section thermocouples was used as the temperature corresponding to the acoustic velocity measurement. The difference between these two thermocouple readings was always less than 0.6 F.

The acoustic velocities measured with the sensor are presented in Figures 7.4 to 7.6. The oil-refrigerant combinations were the same as
Fig. 7.4. Flow stream acoustic velocity sensor measurements in 150 SUS naphthenic oil/R-12 liquid mixtures
Fig. 7.5. Flow stream acoustic velocity sensor measurements in 150 SUS naphthenic oil/R-22 liquid mixtures
Fig. 7.6. Flow stream acoustic velocity sensor measurements in 150 SUS alkylbenzene oil/R-502 liquid mixtures
those used to evaluate the densimeter and viscometer: R-12/naphthenic, R-22/naphthenic, and R-502/alkylbenzene. The solid lines on the data plots represent the values predicted by a regression equation for each of the three data sets. The equations were of the following form:

\[
c_a = b_0 + b_1 c + b_2 \theta + b_3 c^2 + b_4 c \theta + b_5 c^2 \theta
\]

where

\[
\theta = \frac{T}{T_{ref}}
\]

Using the coefficients in Table 7.1, the above equation gives the mixture acoustic velocities in units of ft/s. Absolute temperatures must be used, and the reference temperature is 529.67 R.

**TABLE 7.1 Coefficients for Equation 7.1**

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>R-12/ naphthenic</th>
<th>R-22/ naphthenic</th>
<th>R-502/ alkylbenzene</th>
</tr>
</thead>
<tbody>
<tr>
<td>(b_0)</td>
<td>5823.9</td>
<td>6645.8</td>
<td>5974.7</td>
</tr>
<tr>
<td>(b_1)</td>
<td>845.7</td>
<td>-5566.6</td>
<td>3640.1</td>
</tr>
<tr>
<td>(b_2)</td>
<td>-4065.2</td>
<td>-4819.3</td>
<td>-4537.2</td>
</tr>
<tr>
<td>(b_3)</td>
<td>669.1</td>
<td>16831.6</td>
<td>-7230.3</td>
</tr>
<tr>
<td>(b_4)</td>
<td>2004.8</td>
<td>7100.7</td>
<td>-791.8</td>
</tr>
<tr>
<td>(b_5)</td>
<td>-434.3</td>
<td>-14092.6</td>
<td>7435.7</td>
</tr>
</tbody>
</table>

An analysis of variance for each of the regression equations was performed and the multiple correlation coefficients were all in excess of 0.999. The residual errors in the regressions were also found to be small. These results confirm the visual observation that the regression equations describe the experimental data very well. The forms of the regression equations for acoustic velocity are identical to those used for viscosity in the previous chapter, being second-order in oil
concentration and first-order in temperature.

Three items worthy of further discussion occurred during the performance testing of the acoustic velocity sensor. First, inspection of the transducer after the first series of flow loop experiments revealed that the potting material facing the transducer and protecting the piezoelectric element had become distorted and slightly shrunken during the tests. Possible causes for the distortion were chemical attack by the refrigerant and the high temperatures in the test section. The test section temperatures actually exceeded the recommended maximum for the transducer. The post-test calibration revealed no change in the sensor performance, but fear of further shrinkage and possible damage to the underlying electronics required that the problem be corrected. The transducer was removed from its housing and refrigeration system epoxy was added to the transducer face to compensate for the shrinkage. The face was polished flat after the epoxy had set and the transducer was replaced in its housing. This transducer repair was repeated after each experimental series.

The second operational problem encountered was the sudden and permanent loss of output from the transducer during a series of replication experiments. No cause for the failure was identified, but consultation with the manufacturer indicated that the likely cause was the repeated exposure to temperatures higher than recommended. The manufacturer also indicated that transducers with higher maximum temperature ratings are available by special order.

The final interesting occurrence during the performance tests was
the behavior of the acoustic velocity sensor signals when bubbles were present in the flow stream. At several test conditions, the system pressure was reduced while the test section temperature was held constant, thereby causing bubbles in the flow stream. Even with very bubbly flows passing through the acoustic velocity sensor, the indication of acoustic velocity was unchanged from the previous all-liquid value. The signals on the oscilloscope screen were very attenuated with bubbles in the flow stream, but maintained the same time separation. When the void fraction became sufficiently large, the signals disappeared entirely. However, these results indicate that the acoustic velocity sensor could perform quite well in some two-phase liquid-line situations.

**Inference of Oil Concentration from Acoustic Velocity Measurements**

The performance test data and regression equations were used to develop estimates of the accuracy with which oil concentration can be measured with the acoustic velocity sensor. The same statistical procedures employed for the densimeter and viscometer data, as described in Appendix 2, were used in the preparation of Figures 7.7 to 7.9. These figures present the acoustic velocity measurement as the abscissa independent variable and temperature as an independent cross variable. After locating the coordinates of the measured variables, oil concentration can then be read from the ordinate or, alternatively, calculated from Equation 7.1. Only the R-22/naphthenic oil plot, Figure 7.8, includes more than one accuracy region. For the remaining oil-refrigerant combinations, the estimates indicate that the desired
Fig. 7.7. Inference of oil concentration from acoustic velocity sensor measurements in 150 SUS naphthenic oil/R-12 liquid mixtures.
Fig. 7.8. Inference of oil concentration from acoustic velocity sensor measurements in 150 SUS naphthenic oil/R-22 liquid mixtures
Fig. 7.9. Inference of oil concentration from acoustic velocity sensor measurements in 150 SUS alkylbenzene oil/R-502 liquid mixtures
measurement uncertainty of ±1 weight-percent oil was attainable for all test conditions.

Although the measurement accuracy and simplicity of the measurement principle are desirable, the acoustic velocity measurement system described in this chapter was comparatively expensive. The pulser/receiver and oscilloscope used in these experiments each cost approximately $2000. However, each of these devices could perhaps be replaced with much simpler components without affecting the system performance. The pulser/receiver used was far more elaborate than required for these simple measurements.

Examples of similar low-cost ultrasonic measurement applications include the range-finding devices used in many inexpensive automatic-focus cameras. These devices operate on the same principle as the fisherman's depth-finder, except that the propagating medium is air. Additional development of the acoustic velocity sensor could also greatly reduce the cost of the display device, in this case an oscilloscope. The simple task of measuring the time between pulses is performed in many instruments by digital electronic circuits.

Some of the additional development described above has been undertaken by Paar Scientific, Inc. This company currently offers an ultrasonic instrument that measures the alcohol concentration in flowing beer and spirits. The device is currently being marketed to the European brewing industry. Consultation with Paar engineers revealed that the current device was finely tuned to the acoustic velocity range of the alcoholic beverages, and could not be used in oil-refrigerant mixtures.
without modification. The operational principle of the Paar instrument is quite similar to the system described above. One exception is that instead of using a single transducer and the pulse-echo technique, separate transducers are used for sending and receiving.

The results of the accuracy analysis showed that the acoustic velocity sensor would be an excellent way of measuring oil concentration. The sensor was also noted to perform well under some two-phase conditions, although this testing was not comprehensive. As noted previously, substitution of a transducer with an increased tolerance for the high temperatures encountered in the liquid line of a refrigeration system would be a required modification to the sensor used in these experiments. Also, additional development of the drive, sensing, and display electronics could substantially reduce the cost of such a sensor.
CHAPTER 8
SENSOR COMPARISON AND RECOMMENDATIONS

Although some conclusions concerning oil concentration sensor performance have been included in the discussions of the individual sensors, a comparison of the three sensors tested and recommendations regarding their application are presented in this chapter.

Comparison of Oil Concentration Sensors

Many of the attributes that were considered in selecting measurement methods for development into actual oil concentration sensors were also important in comparing the performance of the three sensors tested. These attributes included accuracy, effects of two-phase flows or contaminants in the liquid line, suitability of the sensor for field usage, and cost. In addition, the performance tests revealed certain unique operational restrictions for each sensor. Comparative discussions of these restrictions and the aforementioned attributes are presented in the following sections.

Accuracy

Statistically derived estimates of the uncertainties expected in measuring oil concentration were prepared for each sensor and presented in graphical form (Figures 5.6 to 5.8, 6.7 to 6.9, and 7.7 to 7.9). In using these plots, the oil concentration is determined by locating coordinates corresponding to the measured mixture property and temperature. In addition to the oil concentration, the uncertainty of the measurement is also indicated on these plots by its location within a
labeled uncertainty region. Since the plots cover essentially the same ranges of oil concentration and temperature conditions, the relative accuracy of the sensors can be determined by comparing the areas of the uncertainty regions. Obviously, a sensor which exhibits only a large area of the +1 weight-percent uncertainty region performs better than a sensor which shows areas of +2 weight-percent or greater uncertainty. Using this basis of comparison, the densimeter is clearly the least accurate of the sensors under the conditions tested. The higher measurement uncertainties with the densimeter are caused by loss of sensitivity of density to oil concentration at higher temperatures, not by deficiencies in the densimeter. The viscometer is significantly more accurate under these conditions, due mainly to the fact that the sensitivity of viscosity to oil concentration is much larger than density. It should be noted that the accuracy of this sensor would most likely improve in applications where the more viscous 300 SUS refrigeration oils are used. Of the three devices, the acoustic velocity probe attained the desired +1 weight-percent accuracy over the broadest range of conditions. The accuracy of this device could be improved even further with a pulse-timing scheme that is more sophisticated than the oscilloscope measurements used in the performance tests.

Two-phase flow effects

Because the flow in a refrigeration system liquid line can sometimes include refrigerant vapor, the effects of the vapor phase on the operation and accuracy of the oil concentration sensors must be considered. The densimeter cannot be used with bubbles in the vibrating
U-tube because even small amounts of vapor will greatly lower the bulk density. To prevent two-phase flow in the densimeter, a bypass arrangement which directs only liquid from the main flow stream could perhaps be designed. However, this would slow the transient response of the sensor and eliminate the dual usage of the sensor as a system flowmeter. The viscometer is intended for just such a bypass arrangement, and the very low flow rate through the bypass sensor also provides substantial additional subcooling to the sampled mixture. Consequently, this device could operate well under some low-quality main flow stream conditions. In addition, the viscometer was not found to give false readings when vapor was present in the sensor, but simply stopped cycling until the vapor condensed or was pushed out using the manual priming circuit. Surprisingly, the acoustic velocity sensor was found to give accurate results in low-quality, bubbly flows, but the pulses were sharply attenuated. In higher quality or stratified two-phase flows, the ultrasonic signals were completely attenuated by the vapor. Instead of the bypass arrangements used with the other sensors, concerns over the effect of bubbles in the flow stream could be eliminated by mounting the acoustic velocity sensor in a small liquid-line receiver.

Contaminants

The effects of contaminants were not evaluated in the performance tests. In fact, extreme care was taken to eliminate contamination. However, some conclusions regarding contamination can be offered. Any contaminant or product of oil-refrigerant reaction which changes the
property being measured will certainly cause errors in the oil concentration measurement. However, the possibility of contamination on a scale large enough to significantly alter bulk properties such as density, viscosity, or acoustic velocity seems unlikely. Small amounts of solid or sticky contaminants could impair the operation of the viscosity sensor, however. The densimeter and acoustic velocity sensor should not be permanently affected by such contaminants.

Field usage

The project scope specified that the ability to use the oil concentration sensors in the field as well as in the laboratory was desirable. Densimeters of the type tested are very rugged and widely used under conditions far more severe than refrigeration systems. All parts of the densimeter/flowmeter, including the electronics package and displays, were of very durable construction. The viscometer, because of its small internal moving parts and need for periodic internal inspection, is slightly less suited to use in the field. The sensor is, however, very durable and intended for harsh environments. The manufacturer also offers a selection of rugged enclosures for the viscometer electronics unit and display. The acoustic velocity probe tested in these experiments was only a prototype, and as such can only be recommended for use in the laboratory. However, at least one similar acoustic velocity sensor intended for industrial use is currently available, as mentioned in Chapter 7.
Cost

Cost is a significant consideration in any engineering decision. The densimeter/flowmeter used in these experiments cost approximately $4500. Although instruments which perform only the density measurement are available from other manufacturers, the costs are comparable. The viscosity sensor and accompanying board-level electronics unit can be purchased for $650 and $950, respectively. An enclosure for the electronics and a power supply are also needed, as well as a digital voltmeter for display. None of these additional components need be particularly sophisticated or costly, however. Because of errors experienced with the viscometer's internal temperature sensor, significant cost would be added to the viscometer system by the need for additional temperature sensors. As noted previously, the temperature measurement problem is currently being addressed by the manufacturer and will likely be corrected on future sensors. The components of the acoustic velocity sensor cost approximately $2500, with the oscilloscope display adding an additional $2000. As discussed previously, however, the cost of this system could perhaps be greatly reduced through further development.

Operational restrictions

Particular characteristics which would restrict applicability were noted for each of the sensors. In the case of the densimeter, significant pressure drop is an inherent consequence of the sensor design. Because the liquid-line flow is only slightly subcooled, great care must be taken to avoid flashing of the mixture through the
densimeter. Under the conditions tested, the pressure drop was estimated from manufacturer's application charts to be approximately 1 psi. Pressure drops of this magnitude may cause flashing in some applications and therefore restrict the use of the densimeter. In testing the viscometer, it was noted that the bypass arrangement of the sensor, intermittent measurement cycle, and low flow rate make the response of this sensor to transient changes in main flow stream conditions quite slow. Consequently, this sensor should only be used under relatively steady refrigeration system conditions. The only operational restriction noted with the prototype acoustic velocity sensor was its apparent inability to tolerate the test section temperatures. This problem could be corrected, however, by choosing a more appropriate transducer.

Recommendations

All three of the above sensors can be recommended for oil concentration measurement within certain limitations. The limits of the recommendations for these sensors are discussed below.

Densimeter

The densimeter cannot be used for accurate measurements at higher test temperatures. In addition, great care must be taken to ensure that no bubbles are entrained in the flow through the densimeter. However, a great number of practical situations exist with liquid-line temperatures in the range where the densimeter would perform well. In addition to air conditioning systems with air-cooled condensers below approximately 90°F, the densimeter could also be used in heat pumps, air conditioning units
with water-cooled condensers, and refrigerators. It should also be noted that because it was designed for very harsh industrial environments, the densimeter is also the sensor best-suited for field installations.

**Viscometer**

The viscometer offers better accuracy over a wider range of test conditions than the densimeter. This device is also quite durable and well-suited to most refrigeration system field applications. Some operational problems due to contaminants could occur, but the bypass arrangement of the sensor also simplifies the isolation and removal of the sensor for repair. The device would be slow to respond to transient oil concentration changes, but fast transient changes in oil concentration are not a characteristic of normal refrigeration system operation. Problems noted with the viscometer's internal temperature sensor are currently being addressed by the manufacturer. Because of the excellent accuracy of the device over a wide range of conditions, the viscometer is the best ready-to-use sensor for oil concentration measurement.

**Acoustic velocity sensor**

The acoustic velocity probe displayed the best accuracy over the widest range of conditions. The operational problems encountered with this device could be corrected by simply choosing an ultrasonic transducer that is better suited to the refrigeration system environment. In addition, this sensor could be easily adapted for use under two-phase liquid-line conditions. Unfortunately, complete ultrasonic acoustic
velocity sensors suitable for oil concentration measurement are not commercially available. The prototype system used in these experiments was also quite costly, due mainly to the excessively sophisticated pulser/receiver and the oscilloscope display method. Additional development could simplify the system, improve the performance, and reduce the cost.
CHAPTER 9
CONCLUSIONS AND SUGGESTIONS
FOR FUTURE RESEARCH

In the introductory chapter of this dissertation, four major tasks were planned for the research project:

1. Identification of oil concentration measurement methods
2. Selection of methods for continued development
3. Development and testing of oil concentration sensors
4. Comparison of sensor test results and recommendations

The preceding chapters have presented the results of these tasks. An extensive literature survey of oil refrigerant mixture properties and a wide variety of bench-top experiments were used to identify and select four property measurement methods for development into flow stream oil concentration sensors. Three of these methods were developed into working oil concentration sensors, and much was learned from work on the promising but ultimately unsuccessful refractive index probes (see Appendix 3). A unique facility for testing and calibrating the sensors was designed and constructed, and results from performance tests of the three successful sensors have been presented. Included in these results are estimates of the oil concentration measurement accuracies that could be expected with each of the three sensors. A comparison of these accuracy estimates and other operational characteristics of the sensors has resulted in recommendations regarding the use of these sensors for oil concentration measurement in refrigeration systems. These recommendations, as summarized below, constitute the major conclusions from this research project.
A commercially available vibrating U-tube densimeter was shown to be an accurate method of measuring oil concentration in liquid oil-refrigerant mixtures below approximately 90 F. The highest temperature for which acceptable accuracy can be maintained depends on the oil-refrigerant combination.

Excellent results were obtained in using a recently introduced process viscometer for oil concentration measurement. The viscometer attained the desired ±1 weight-percent oil concentration measurement uncertainty over a wide range of liquid-line conditions. The viscometer was recommended as the best off-the-shelf means of measuring oil concentration in a refrigeration system.

A prototype acoustic velocity sensor was demonstrated as a highly accurate method of measuring flow stream oil concentration in a refrigeration system liquid line. Although it displayed the best measurement accuracy, additional development is needed to reduce the cost and improve the data display elements of the acoustic velocity sensor.

**Future Research**

Although three successful oil concentration sensors have been demonstrated, additional work is suggested by the results of the project. First, the effects of contamination on the oil concentration sensors can only be determined by field testing. Such testing could also be useful in discovering unknown operational restrictions overlooked by this study. Second, continued development of the ultrasonic acoustic velocity probe could result in a highly accurate, portable, low-cost oil concentration sensor. The ability to make such a device portable has been demonstrated
by the manufacturer of the ultrasonic bolt gage used in the preliminary property measurements. In addition, applications beyond oil concentration measurement are possible, as witnessed by the alcohol concentration instrument mentioned in Chapter 7. Because of the potential for improved performance, reduced cost and wider applications, development of the ultrasonic acoustic velocity sensor should continue.

Cambridge Applied Systems has indicated that future models of the viscometer will include improved temperature sensing. In addition, some new models may eliminate the need for the bypass arrangement. This latter type of sensor would simply screw into a threaded port in the liquid line. Because of the excellent performance of the current model, testing of these new viscometers as oil concentration sensors is also recommended.

Appendix 3, concerning the development of refractive index sensors, concludes that the development of a low-cost optical fiber sensor for oil concentration measurement is not feasible. However, the fiber optics industry has grown substantially during the course of this project. Over the course of just the past year, the cost of high-performance fiber optic components and instruments has been greatly reduced. In addition, components that would make assembling an optical fiber reflectometer relatively simple have been introduced. Using a portable fiber optic power meter and a new type of directional coupler, a prototype optical fiber reflectometer could be assembled for as little as $1500. Because these recent developments present the possibility of constructing a successful refractive index sensor at a reasonable cost, development of
such a sensor should also be pursued.

Although the performance tests covered a wide range of actual liquid-line conditions, many more possible conditions could be tested. In order to provide a broad base of data for oil concentration sensor calibration, a wider range of test temperatures and many additional oil-refrigerant combinations should be included in these future experiments. It should be noted that the results presented herein are sufficient to prove that the sensors tested will operate in a flow stream. Therefore, future calibration data could be generated using a stagnant volume of the test fluid. Experiments of this type should be considerably faster and less expensive than using a flow loop.
REFERENCES


APPENDIX 1
PREDICTION LIMITS FOR
PRELIMINARY PROPERTY MEASUREMENTS

A prediction limit comparison plot was presented in conjunction with each of the preliminary property investigations in Chapter 3. The objective of these plots was to provide a visual means of evaluating the potential accuracy of a proposed oil concentration sensor with respect to the performance requirements specified in the project scope. Each plot was constructed using the experimentally determined variation of the mixture property, an estimate of the measurement uncertainty for the proposed sensor, and the performance requirements specified by ASHRAE. This appendix presents an example of the prediction limit comparison plot preparation.

Figure A1.1 depicts the variation of an arbitrary oil-refrigerant mixture property with oil concentration. Actual oil concentration measurements in the liquid line of a refrigeration system would occur in a slightly subcooled liquid state. Therefore, the data collected at 113°F were used in these comparisons because this temperature represents a slightly subcooled state for R-113 at the atmospheric pressure of the bench-top experiments. In addition to the hypothetical data, Figure A1.1 presents a best-fit curve describing the variation of the property with oil concentration.

Figure A1.2 shows the construction of the ASHRAE prediction limits. These limits were obtained by plotting points to the right and left of the property variation curve at distances corresponding to one weight-percent oil concentration. Connecting the points resulted in the upper
Fig. 11.1. Example of a property that varies with oil concentration
Fig. A1.2. Construction of ASHRAE prediction limits
and lower ASHRAE prediction limits. These curves bound the region in which the prediction limits for an actual oil concentration sensor must fall in order to provide the desired one weight-percent accuracy.

The prediction limits for a proposed oil concentration sensor were added to the plot as shown in Figure A1.3. For this example, it was assumed that the manufacturer of an instrument that measures the arbitrary property has stated an uncertainty of $\pm 1$ arbitrary property unit at a confidence level of 95%. The projected prediction limits were then constructed by plotting points above and below the property variation curve at distances corresponding to one arbitrary property unit. Connection of the points produced the 95% prediction limits for the sensor under consideration. In this example, the sensor is seen to perform adequately at the lower concentration levels, but not at high oil concentrations because of the decreasing sensitivity of the property to oil concentration.

It should be pointed out that the above step in this example represents a very simple case. In general, such convenient specifications for the measurement uncertainties of the proposed oil concentration sensors, complete with statistical levels of confidence, were not available. Many times the specifications were given as percentages of full-scale output and without any statement of statistical confidence. The prediction limit has been assumed to be 95% in these cases, which is a conservative assumption. Most instruments are rated at a confidence level of 99%. In the case of proposed oil concentration sensors that are not commercially available, the measurement
Fig. A1.3. Construction of sensor prediction limits and comparison with ASHRAE prediction limits
uncertainties were conservatively estimated based on information from technical papers describing similar instruments.
Estimates of the oil concentration measurement accuracy for each of the three sensors tested have been presented in Chapters 5 through 7. This appendix contains descriptions of the statistical and mathematical procedures used in developing these estimates. The procedures included multiple non-linear regression analysis of the performance test data, calculation of prediction limits for the regression equation, and a method of displaying the prediction limits as oil concentration measurement uncertainties on two-dimensional plots. An example of each of these steps is presented for one of the data sets.

Regression Analysis

The regression equations describe the variation of the measured properties (specific gravity, viscosity, or acoustic velocity) as functions of two independent variables, oil concentration and temperature. Simple polynomial forms were selected for these regression equations. The viscosity and acoustic velocity data sets were described well by equations which were quadratic in oil concentration and linear in temperature. All cross-product terms were retained, and the resulting form of the equations for these two properties was presented in Equations 6.1 and 7.1. For the specific gravity data sets, the assumed forms were quadratic in both independent variables. All cross-product terms were once again retained, resulting in Equation 5.1. For the purpose of the example, this equation is repeated below:
\[ SG = b_0 + b_1 C + b_2 \theta \]
\[ + b_3 C^2 + b_4 \theta + b_5 \theta^2 \]
\[ + b_6 C^2 \theta + b_7 \theta^2 + b_8 C^2 \theta^2 \]  

where \( \theta = \frac{T}{T_{\text{ref}}} \)

The reference temperature in the above equation is 529.67 R. Although this is a non-linear equation, the coefficients were determined using multiple linear regression analysis. The following variable substitutions were used to linearize the equation:

\[ x_1 = C \]
\[ x_2 = \theta \]
\[ x_3 = C^2 \]
\[ x_4 = \theta^2 \]
\[ x_5 = \theta^2 \]
\[ x_6 = C^2 \theta \]
\[ x_7 = \theta^2 \]
\[ x_8 = C^2 \theta^2 \]

Making these substitutions, the regression equation can be written as an eight-variable linear equation:

\[ SG = b_0 + b_1 x_1 + b_2 x_2 \]
\[ + b_3 x_3 + b_4 x_4 + b_5 x_5 \]
\[ + b_6 x_6 + b_7 x_7 + b_8 x_8 \]

The values of the new variables were calculated for each data point in the data sets. The completion of this very large number of calculations was facilitated by using RS/1, a data analysis computer program specifically designed for engineers and scientists [40]. The program calculated the values of the new variables in a convenient tabular form very similar to many popular spreadsheet programs. The values of the new variables were then transferred to a multilinear
regression analysis procedure which was also contained in the RS/1 package. This procedure returned the values for the coefficients of Equation A2.1, which are identical to the desired coefficients of the regression equation, 5.1. The multilinear regression routine also calculated the residual error (also known as the standard error of estimate) and performed an analysis of variance for the regression. The multiple regression coefficients (which are a useful indication of the goodness-of-fit) quoted in earlier chapters were calculated by the analysis of variance. The regression analysis was performed for each of the data sets presented in Chapters 5 to 7.

The data collected with the densimeter in mixtures of R-22 and naphthenic oil are presented as an example in Figure A2.1. The solid lines once again correspond to the predictions of the regression equation. The regression equation has also been used to generate Figure A2.2. This plot reverses the positions of the independent variables, with oil concentration now being the abscissa variable and temperature the cross-variable. With the coordinates rearranged in this manner, prediction limits which estimate the uncertainty in predicting the oil concentration from a densimeter measurement can be displayed in a meaningful manner.

**Prediction Limits**

The regression lines plotted in Figure A2.2 can best be thought of as typical instrument calibration curves. In this example the densimeter has been calibrated to indicate oil concentration by observing the output of the densimeter at known levels of oil concentration and temperature.
Fig. A2.1. Sample data set: densimeter measurements in 150 SUS naphthenic oil/R-22 mixtures
Fig. A2.2. Best-fit curves for sample data on transposed coordinates
Consider next the use of the densimeter as an oil concentration sensor. At a fixed temperature, the roles of the dependent variable (specific gravity) and the remaining independent variable (oil concentration) are reversed; oil concentration is estimated from the measured value of the instrument output, specific gravity. This estimate can be obtained either from the calibration plot (Figure A2.2) or by solving the regression equation for the oil concentration. As pointed out by Graham [41], the appropriate uncertainty in such an estimate can be obtained from the confidence intervals about the individual values of the calibration inputs. Alternate names for these confidence intervals are the single-valued confidence intervals, estimate intervals, or prediction intervals. In keeping with previous terminology, these intervals will be referred to for the remainder of this discussion as the prediction intervals. The prediction intervals about the individual calibration input values are given by the following equation:

\[ A_1 = \frac{t_{\alpha/2, (n-2)}}{\sqrt{n - 1 + \frac{(C - \bar{C})^2}{(n - 1)s_C^2}}} \]  

(A2.2)

where

- \( t_{\alpha/2, (n-2)} \) = t-statistic evaluated at \((1-\alpha)\) confidence level and \((n-2)\) degrees of freedom
- \( n \) = number of observations in data set
- \( s_e \) = standard error of estimate
- \( \bar{C} \) = grand mean of all \( C \) values in the data set
- \( s_C^2 \) = variance in \( C \) for the data sets

The upper and lower densimeter prediction limits for the regression equation are thus given at a fixed temperature by the following
In these equations, $SG(C)$ represents the calculated value of the regression equation (Equation 5.1) at all values of $C$ in the calibration range. These prediction limits have been plotted along three isotherms in Figure A2.3 using a confidence level of 95%.

The prediction limits for the sensors were then compared to the +1 weight-percent prediction limit bands desired by ASHRAE. Graphical construction of these prediction limit bands was discussed in Appendix 1. These bands can also be mathematically described in the following manner:

$$PL_{\text{upper}} = SG(C) + A_1 \quad (A2.3)$$

$$PL_{\text{lower}} = SG(C) - A_1 \quad (A2.4)$$

These prediction bands have been designated right and left because, as described in Appendix 1, they are constructed by simply shifting the property curve right and left by one weight-percent on property vs. oil concentration coordinates. These prediction limit bands have also been plotted along the three isotherms in Figure A2.3.

**Prediction Limit Intersections and Uncertainty Regions**

As can be seen in Figure A2.3, the prediction limits for the sensor intersect and exceed the ASHRAE prediction limits. The intersections have been connected by a spline curve in Figure A2.3. The spline curve is actually based on many more points than shown. The intersections were calculated at 1 F intervals across the test temperature range. The calculation procedure is presented in a later section. The meaning of
Fig. A2.3. Prediction limits for sample data set
this locus of the intersection points can be understood by noting that points above this curve represent conditions where the estimates of oil concentration from densimeter measurements are within the desired \( \pm 1 \) weight-percent uncertainty. Points below the curve represent conditions where the uncertainty is greater than \( \pm 1 \) weight-percent.

These intersection points were calculated by setting the right-hand sides of the appropriate prediction limit equations equal and solving explicitly for \( C \), the oil concentration value at which the intersection occurs. In the example problem, the upper prediction limits for the densimeter and the right prediction limit band were a corresponding pair. For the viscometer and acoustic velocity probe, the upper prediction intersected the left prediction limit band, due to the reversed curvature of the property curve. For this example, however, the right-hand sides of Equations A2.3 and A2.5 were equated:

\[
SG(C) + A_1 = SG(C-0.01) \quad (A2.7)
\]

Substitutions for \( SG(C) \) and \( A_1 \) come directly from Equations 5.1 and A2.2. The right-hand side of Equation A2.7 is calculated by first substituting \( C-0.01 \) for \( C \) in Equation 5.1 and then substituting the result into Equation A2.7. Even after simplification, the final equation is algebraically complex but contains only one unknown: the value of oil concentration, \( C \), corresponding to the point of intersection of the two prediction limits. The resulting equation was solved explicitly for \( C \) using Macsyma, a symbolic math processing computer program [42]. The equation is quadratic in \( C \), but only a single root was positive and, therefore, meaningful. This equation was then evaluated for isotherms at
Intervals for each of the data sets. The intersections of the lower confidence limits were also evaluated, but the upper intersections were always found to give the more conservative estimates of the uncertainty regions. For the densimeter data sets, the analysis was repeated in its entirety to find the boundary of the $\pm 2$ weight-percent uncertainty region as well.

The final manipulation with the regression equations and calculated uncertainty regions was to invert the plotting coordinates as shown in Figure A2.4. This plot depicts how the sensor would actually be used to determine oil concentration. The specific gravity and temperature are the outputs of the instrument, and by locating their coordinates on this plot the oil concentration can be read from the ordinate. An estimate of the uncertainty in the oil concentration measurement can also be obtained from the plot by noting the uncertainty region in which the oil concentration/temperature coordinates are located. Region I represents the conditions under which an uncertainty of $\pm 1$ weight-percent or less can be expected. Oil concentrations inferred from points located in Region II would have uncertainties between $\pm 1$ and $\pm 2$ weight-percent. Region III, which does not appear on most of the plots, represents conditions under which the uncertainty would be greater that $\pm 2$ weight-percent. Estimating the oil concentration from points in Region III is not recommended.

Two important assumptions have been made in this uncertainty analysis. First, it has been assumed that the oil concentrations were calibration standards and, as such, were very precise. As explained in
Fig. A2.4. Inference of oil concentration for sample data
Chapter 4, the oil concentration in the flow loop was set by weighing the amounts of refrigerant and oil which were introduced into the loop. Propagation-of-error analysis was used to estimate the standard error in the oil concentration levels obtained by these successive weighings. Because the balances used were very precise and each individual oil addition was a rather small fraction of the total mixture in the system, the maximum error in oil concentration estimated by this analysis was +0.02 weight-percent. Thus, the assumption that the oil concentration values contained only small errors appears to be valid.

The second assumption was that errors in the measurement of the other independent variable, temperature, were small. No method of including the effect of errors in the temperature measurements could be developed. Multiple calibrated thermocouples were used in the test section, as pointed out in a previous chapter, to ensure that the temperature measurements in these experiments were quite accurate. As stated in Chapter 4 the uncertainty in the test section thermocouples was only +0.4 F. The fact that errors in the temperature measurements were not included in the analysis of oil concentration measurement uncertainties requires that accurate temperature measurement must also be made by those incorporating these uncertainty estimates in their own oil concentration measurements.
Significant differences were found between the refractive indices of pure oils and refrigerants, as discussed in Chapter 2. The literature review also produced references to a relatively new type of instrument, an optical fiber refractometer. Other researchers have reported that this device was capable of determining the composition of two-component solutions by measurement of the refractive index [32,33]. Bench-top measurements of the refractive indices of naphthenic oil/R-113 mixtures disclosed a sufficiently large oil concentration gradient in the mixture refractive indices to justify development of a refractive index sensor for oil concentration measurement. This appendix reports the results of the research efforts dedicated to developing refractive index sensors for oil concentration measurement. Included are discussions of the refractive index measurement principles, descriptions of the construction of several probe types, and sample results from simulation experiments. The problems which led to discontinuing the development work are also discussed.

Background on Optical Fiber Refractive Index Sensors

Several types of optical fiber sensors have been demonstrated for the measurement of liquid refractive indices. The different configurations include U-bend refractometers [32,33,43], multiple-bend refractometers [44], straight-fiber refractometers [45], directionally-coupled reflectometers with both conical [43] and flat [46,47] sensor tips, and linked-prism reflectometers [43,48]. Although literature
concerning the performance of all of these probe types was examined, only the two most common types were evaluated in the laboratory: the U-bend refractometer and the flat-tip reflectometer. The operating principles and basic components used in assembling these sensors are discussed below. In order to discuss the operating principles, however, some basics of light propagation in an optical fiber must first be reviewed.

**Fundamentals of light transmission in optical fibers**

In Figure A3.1, several light rays are shown being launched into the end of a glass rod. The rod has a uniform refractive index, $n_1$, and is surrounded by a medium of lower refractive index, $n_2$. The rays propagate until striking the outside surface of the core material, where they are either reflected back into the core and continue to propagate or are refracted out into the surrounding medium. The critical angle of incidence at which the rays striking the interface will begin to be refracted out depends on the refractive indices of the core and the surrounding media, and is given by Snell's law of refraction:

$$\sin \theta_c = \frac{n_2}{n_1} \quad (A3.1)$$

The angle of incidence is defined as the angle between the incident ray and a normal to the interface surface. Rays that strike at less than the critical angle will be refracted out, while rays striking at angles greater than the critical will continue to propagate. This method of confinement is called total internal reflection.

An optical fiber that propagates light by total internal reflection is shown in Figure A3.2. The core of the fiber, which can be glass, plastic, or silica, is clad with a material of lower refractive index.
Fig. A3.1. Propagation of light in a glass rod
Fig. A3.2. Propagation of light in a step-index, multimode optical fiber
This buffer coating allows the fiber to be jacketed for abrasion protection. Without the intermediate low-index cladding, most of the light would be refracted out into the protective jacket. The cladding can also be constructed of glass, plastic, or silica.

As shown in Figure A3.2, the propagating light rays include some which pass straight through the fiber with no reflections. These are called axial rays or fast modes. Slow modes are those rays which bounce along the fiber path by striking the core-cladding interface at angles near the critical angle. Thus, the fiber contains a distribution of modes from fast to slow. Later sections explain how this modal distribution leads to refractive index sensing in the U-bend refractometer. However, it should be noted at this point that the need for a distribution of modes in the propagating light eliminates the use of some optical fiber types for U-bend refractometers. The fibers discussed above, which have a uniform core refractive index, are referred to as step-index, multimode fibers. Some fibers do not propagate light by total internal reflection, but instead use a process called continuous refraction. These fibers have a non-uniform, or graded, core refractive index. This propagation technique greatly reduces the modal distribution, which is desirable for optical communication but not for U-bend refractometers. Another fiber type which is unsuitable for U-bend refractometers is the step-index, single-mode fiber. These fibers have very small core diameters and the cladding refractive index is only slightly lower than the core. The result is a fiber which propagates only the fastest modes, with essentially no modal distribution. A simple
comparison of the way light is propagated in these three fiber types is presented in Figure A3.3.

**U-bend refractometer operating principle**

Figure A3.4 describes the basic operating principle of the U-bend refractometer. The sensing area, S, is a region of the fiber in which both the protective jacket and the cladding have been removed. Two light rays, \( I_1 \) and \( I_2 \), are shown striking the stripped sensor area. Ray \( I_1 \) strikes the surface at an angle \( \theta_1 \) greater than the critical angle \( \theta_c \) and, consequently, continues to propagate down the fiber to the photodetector. Ray \( I_2 \), however, is shown striking the surface S at an angle \( \theta_2 \) less than the critical angle. This light ray escapes into the surrounding medium and is not sensed by the photodetector. The critical angle is related to the refractive index of the surrounding medium by the relation \( \theta_c = \arcsin(n_m/n_{co}) \). This is a simplified analysis, but it is sufficient to demonstrate how the presence of a distribution of propagating modes in the fiber leads to refractive index sensing. Because the amount of light which reaches the photodetector is modulated by the refractive index of the liquid at the sensing area, the photodetector output signal can used as an indication of the liquid refractive index.

**Flat-tip reflectometer**

Although the simplified Snell's law analysis was sufficient to demonstrate the operating principle of the U-bend refractometer, explanation of the flat-tip reflectometer operation requires more complex
Fig. A3.3. Comparison of optical fiber confinement techniques
Fig. A3.4. Attenuation of light rays in a U-bend optical fiber refractometer
considerations. When a light ray passes from one medium to another, some of the optical intensity is lost to reflections from surface interfaces. These reflections, which were neglected in the analysis of the U-bend refractometer, are called Fresnel reflections or Fresnel losses.

Figure A3.5 shows the Fresnel reflections which occur as a light ray passes from air through a glass plate and back to air. These reflections are also experienced by light rays striking the terminated end of an optical fiber, as shown in Figure A3.6a. Legendre [46] has shown theoretically and experimentally that the magnitude of the reflected light from such a fiber termination depends on the refractive index of the medium at the tip. Consequently, a measurement of the light reflected from the terminated end of an optical fiber can be used as an indication of the refractive index of the fluid surrounding the termination. A simplified schematic of the components necessary for such a measurement is presented in Figure A3.6b.

The reflectometer used by Dr. Legendre was considerably more complex than the simple probe presented in Figure A3.6b. Refinements in the optical and electronic circuitry enabled Legendre to measure changes in the refractive index of a flowing liquid as small as $2 \times 10^{-5}$ [47]. Because of the excellent measurement resolution reported for this probe, Dr. Legendre was contacted for additional information. As a result, he agreed to assist in evaluating his probe as an oil concentration measurement sensor. A series of experiments using oil-refrigerant mixtures was conducted at Dr. Legendre's laboratory at the Canada National Research Council. Results of these experiments are presented in
Fig. A3.5. Fresnel reflections for a light ray passing through a glass plate
Fig. A3.6. Flat-tip optical fiber reflectometer
(a) Fresnel reflection at the tip
(b) Schematic diagram of reflectometer
Refractive index sensor components

Regardless of the type, some components are common to all of the refractive index sensors. The brief discussions below provide an introduction to the types of fibers, light sources, photodetectors, and interconnecting devices used in assembling an optical fiber refractive index sensor.

Optical fiber materials

As stated previously, only step-index, multimode optical fibers are suitable for use in a U-bend refractometer. Although the reflectometer would work with graded-index or monomode fibers, Legendre's reflectometer also used a step-index, multimode fiber. Such fibers are constructed from a variety of different materials. The least expensive use plastic for both the core and cladding. Plastic-clad silica cables give generally higher performance in data transmission applications, but at increased cost. All-silica optical fibers are the most popular for communications applications, but separation of the core and cladding as required for the U-bend sensing area is not possible. The reflectometer probe of Legendre, however, used a typical all-silica communications-grade fiber. In addition to the increased cost of the fiber itself, the components and procedures for connecting silica-core fibers are much more expensive and complex than those for plastic fibers.

Although all-plastic fibers have performed adequately in previous U-bend refractometers [32,33,44] they are unsuitable for use in refrigeration systems due to chemical attack by the halocarbon refrigerants. Consequently, plastic-clad silica fibers were thought to
be more suited to this application. Plastic-clad silica fiber was used successfully in the refractometers of Spenner et al. [43] and Bostick et al. [45]. Plastic fibers were used extensively, however, in the development and simulation experiments reported herein due to their low cost. A discussion of the construction of U-bend refractometers from these two fiber types appears in a later section.

**Light sources** Several types of light sources have been used in refractive index sensors. Incandescent sources are sometimes used, but these are difficult to couple and do not produce a uniform output over time. The most common, simplest, and least expensive light source for fiber-optic applications is the infrared-emitting diode (IRED). These devices produce light in a narrow, predictable wavelength band which is typically centered somewhere between 800 and 1300nm.

In order to obtain a larger output signal from U-bend refractometers, a He-Ne laser light source was used by some investigators [32,33]. Specialized devices, which are also quite costly, are required to couple the radiation from this type of laser into the fiber. Lasers are sometimes used in optical fiber data transmission, but these are usually solid-state diode-lasers. Since they are constructed primarily for use with optical fibers, laser diodes are much easier to couple than gas lasers. However, the cost of laser diodes is quite high, and the light outputs of these devices are quite temperature dependent.

**Photodetectors** Light from any of the above sources can be detected by a variety of available photodiode or phototransistor detectors, with the detector usually being matched to the emitter
wavelength range for optimum performance. Photodiode detectors offer very fast response times, while phototransistors are slower but have much greater responsivity, i.e., higher output current for the same light input. Other types of photodetectors, including photoconductive cells and photomultiplier tubes, are not typically used with optical fibers.

**Fiber coupling**  
Mechanical couplings are required between the optical fibers and the active devices (sources and detectors) and also between fibers. High-quality couplings are important in maximizing the optical power launched by the source and received by the photodetector. These couplings must also hold the components firmly in order to ensure predictable performance. Suitable couplings are commercially available from several manufacturers. The selection of a coupler is based on the electronic package configuration of the emitter/detector component and the size of the fiber-optic cable. Another important interconnection device in the construction of an optical fiber reflectometer is the directional coupler. This device, as depicted in Figure A3.6b, is used to direct radiation reflected from the reflectometer tip to the photodetector. Because the amount of reflected radiation is quite small, an efficient coupling is required. Directional couplers are also commercially available in a variety of designs.

**Component selection**  
The emphasis of the refractive index sensor development was to construct an economical oil concentration sensor. Many of the problems that were encountered during the development could have been solved with expensive fiber-optic components, tools, and instruments, but such solutions were ignored. The possibility of using a
Development of Refractive Index Sensors

Three types of refractive index sensors were investigated in the laboratory. First, several U-bend optical fiber refractometers were constructed, both for preliminary studies and for use in the oil–refrigerant mixtures. Second, a series of experiments was conducted at the Canada National Research Council to evaluate the performance of an optical fiber reflectometer for use as an oil concentration sensor. Finally, U-bend refractometers constructed of common glass rod, not optical fiber, were developed for testing in the flow loop. Descriptions of the sensors and the experiments conducted are presented below.

Optical fiber U-bend refractometers

Plastic fiber refractometers

Although many companies distribute plastic optical fibers, most of these fibers are manufactured by Mitsubishi Rayon Co., Ltd. These fibers are available in a variety of core sizes and use polystyrene as the core material and a proprietary flourine-bearing resin for the cladding. As stated previously, the halocarbon refrigerants were known to attack the polystyrene core of these plastic optical fibers. However, probes constructed from these plastic fibers proved to be very useful in evaluating refractometer sensing area geometries, sources, detectors, and output amplifier circuits. Instead of using the oil–refrigerant mixtures as test fluids in these developmental experiments, aqueous solutions of sucrose were used. Refractive index measurement is a common method of determining
aqueous sucrose concentration, and thus extensive tables of reference values are available. Inspection of these tables revealed that the refractive index range of the R-113/oil mixtures could be duplicated by aqueous solutions of 12 to 50 weight-percent sucrose composition.

Sample quantities of the fibers used to construct these refractometers were obtained directly from Mitsubishi Rayon America, Inc. Two types of fibers were used, Super Eska SK-40 and SH4001. The SK-40 was an unjacketed fiber of 1 mm outside diameter, and the SH4001 cable contained the same fiber in a 2.2 mm O.D. PVC plastic jacket. Removal of the protective jacket in the sensing area proved to be very difficult, leading to the use of the unprotected SK-40 fibers. However, the connectors used to couple the fiber to the active devices required the presence of the protective jacket. Consequently, the usual method of constructing a sensor was to start with a length of the unjacketed fiber, form the U-bend sensing area, and remove the cladding from the sensing area with methyl acetate. Two lengths of the empty PVC jacket, taken from the SH4001 cable, were then pushed over the ends of the fiber to the sensing junction. A short length of Romex Twin-tube was slipped over the two ends and cemented at the base of the U-bend with a common epoxy. A diagram of a typical sensor is presented in Figure A3.7.

Couplers to attach the active devices to the jacketed ends of the plastic fibers were available from Amphenol, Inc., and AMP, Inc. In addition, connectorized sources and detectors were available from Motorola Optoelectronics Division. Two Motorola devices, the MF0E71 IRED emitter and MF0D72 phototransistor detector, were used in the experiments
Fig. A3.7. Diagram of a typical plastic optical fiber U-bend refractometer
reported in later sections. Connecting the fiber to these devices involved simply cutting the end with a heated blade, removing 1/8 inch of the protective jacket, inserting the fiber into the connectorized component, and securing the fiber with the attached compression nut.

The electronic circuitry required to drive the source and detector in these experiments was relatively simple. The IRED source was driven by the regulated DC supply shown in Figure A3.8a. The current output of the phototransistor was converted to a DC voltage by the transimpedance amplifier shown in Figure A3.8b. The output of this amplifier was then monitored by a Hewlett-Packard 3457A digital multimeter.

Several experimental setups were used to test the performance of the plastic refractometers. The most satisfactory arrangement was a flow loop which circulated the sucrose solutions by means of a parastaltic (tubing) pump. The solutions were circulated through a small glass vessel containing a thermometer and the submerged refractive index sensor. This vessel was connected in series with the pump and a large reservoir flask that contained most of the sucrose volume. The reservoir flask was submerged in the well of a constant temperature bath to control the temperature of the sucrose solution. The sucrose concentration in the loop was increased by simply adding more sucrose to this reservoir flask and mixing with a magnetic stirrer. Each solution was circulated for at least ten minutes to ensure uniform concentration throughout the loop. The refractive index of each solution was determined by withdrawing a sample for measurement with an Abbe's method refractometer. During these measurements, the prisms of the Abbe's method refractometer
Fig. A3.8. Refractometer electronic circuits
(a) IRED current supply
(b) phototransistor amplifier
were maintained at the same temperature as the sucrose solution in the flow loop. The results of a typical series of measurements in flowing sucrose solutions with a U-bend refractometer are presented in Figure A3.9.

The results presented in Figure A3.9 showed that a U-bend refractometer could be constructed to measure refractive indices in the range of interest for oil concentration measurement. In addition, the many simulation experiments which were performed produced two other useful conclusions. First, sensors with smaller bend radii were found to be the most sensitive to changes in the fluid refractive index. For the 1 mm fibers, a U-bend of 5 mm radius was easily constructed and performed well. Second, multiple bend sensors were found to be more difficult to construct but did not offer significantly increased sensitivity.

**Plastic-clad silica U-bend refractometers**  Because of the promising performance of the plastic fiber demonstration probes, construction of a plastic-clad silica U-bend refractometer for measurements in oil-refrigerant mixtures was undertaken. Silica fiber of 140 micron core diameter with a chemically bonded plastic cladding was obtained from General Fiber Optics, Inc., along with appropriate couplers for Honeywell Sweet-Spot IRED source and photodiode detector components. Drive and sensing circuits were similar to those shown in Figure A3.8.

Although a somewhat successful U-bend refractometer was constructed from the above components, many difficulties were encountered. First, the chemically-bonded plastic cladding was extremely difficult to remove. Mechanical methods, such as scraping or sanding, damaged the fiber.
Fig. A3.9. Plastic U-bend refractometer measurements in flowing sucrose solutions at 77 F
Scoring the cladding with a heated rod and then pealing away the heat-damaged cladding was suggested by Bostick et al. [45], but this method embrittled the silica fiber core. The brittle core subsequently broke when attempting to form the U-bend. The only successful method of removing the cladding was the use of a chemical stripper. However, the extent to which the stripper leaked under the cladding into areas where the cladding was to remain was impossible to control and the attack continued even after the sensor was placed in service. Attachment of the electronic light sources and detectors to the fiber ends was also very difficult without a number of expensive tools. The connectors for the IRED source and photodiode components required special tools for cutting and polishing the fiber ends and crimping the connectors in place. These tasks were accomplished with make-shift tools, but only with the investment of a great deal of time. The small size of the silica fibers also rendered the completed sensor very delicate. Several sensors were broken while measurements were being taken in the laboratory. Because of these construction and implementation problems, development of plastic-clad silica U-bend refractometers was discontinued in favor of the glass rod probes discussed in a later section.

Cladding-mode refractometers In the transmission of light by optical fiber, some light is coupled into the fiber cladding as well as the core, particularly if a small-radius bend exists in the fiber. If the cladding is left on the sensing junction of a U-bend refractometer, the device is still sensitive to the refractive index of the surrounding fluid due to attenuation of these cladding modes in the region of the
Because of the difficulties encountered with the plastic-clad silica fibers, the possibility of constructing an all-silica cladding-mode refractometer for use in oil-refrigerant solutions was considered. A plastic-fiber cladding-mode refractometer, similar to those of Harmer [44], was constructed and found to perform well in the sucrose simulation experiments. However, an all-silica version of this refractometer was not successful. The resulting device, constructed of 50 micron communications-grade multimode silica fiber with a 125 micron silica cladding, was not sensitive to changes in fluid refractive index at the U-bend sensor tip.

**Fiber-optic reflectometer probe**

As presented previously, the optical fiber reflectometer designed and constructed by Dr. J. P. Legendre of the Canada National Research Council (CNRC) was evaluated for use as an oil concentration sensor through a series of experiments conducted at CNRC in Ottawa, Ontario. A schematic diagram of Dr. Legendre's reflectometer is presented in Figure A3.10. The reflectometer uses a modulated IRED as the light source and a PIN photodiode as the detector. Modulation of the source requires the use of a lock-in amplifier for phase-sensitive detection and amplification of the photodiode output. The lock-in amplifier tracks and amplifies only those electrical signals which occur at the precise frequency of the source modulation signal, thereby eliminating all extraneous signals of other frequencies and virtually all output signal noise. The intended application for Dr. Legendre's reflectometer was as a medical probe. Consequently, the probe tip is of very small size. The
Fig. A3.10. Diagram of Legendre reflectometer
probe tip is simply the cleaved end of a fused-silica optical fiber with a core diameter of 50 microns and a cladding diameter of 125 microns.

**Reflectometer evaluation experiments**

The evaluation experiments were conducted in a very simple manner. The reflectometer probe tip was dipped into a screw-cap vial containing one of the oil-refrigerant mixtures. The samples were identical to those used in our earlier refractive index measurements reported in Chapter 3. The probe tip remained submerged in the static liquids for approximately 60 seconds while the reflectometer output voltage was continuously monitored by a Hewlett-Packard data acquisition system. The temperature of the solutions was not precisely controlled during these preliminary experiments. However, the room temperature near the solutions was monitored by a thermocouple and found to be $77.0 \pm 0.5$ F.

The experimental results are presented in Figures A3.11 and A3.12. The probe output voltages for each of the oil-refrigerant mixtures are plotted as a function of the oil concentration in Figure A3.11. This plot shows that the output voltage decreased non-linearly with oil concentration. In a recent paper, Dr. Legendre [46] has shown that the amount of light reflected from the probe tip varies with the refractive index of the surrounding medium as follows:

$$ R \sim \Delta n^2 $$

(A3.2)

In this equation, $R$ represents the reflected light and $\Delta n$ is the difference between the refractive indices of the fiber core ($n_{\text{core}} = 1.46$) and the surrounding medium. This relationship is demonstrated by Figure A3.12, which plots the probe output voltage as a
Fig. A3.11. Legendre reflectometer measurements in oil/R-113 liquid mixtures at 77 F
Fig. A3.12. Linearized output of Legendre reflectometer
function of the square of the refractive index difference. Although the relationship given in Equation A3.2 is an approximation, the predicted linear relationship between $R$ and $\Delta n^2$ is evidently quite accurate. In the preparation of this plot, the refractive indices of the oil-refrigerant mixtures at 77 $^\circ$F were taken from our previous measurements.

The results shown in Figure A3.11 indicated that the fiber-optic reflectometer was a promising device for measuring oil concentration. Figure A3.12 shows that the device also has the advantage of a relatively simple mathematical model. Based on these encouraging findings, continued investigation of the optical fiber reflectometer was planned. A highly simplified probe, which eliminated the source modulation, mode stripper, mode mixer, and lock-in amplifier, was constructed. Unfortunately, a critical component of this reflectometer, the bidirectional coupler, was over-simplified. A 2x1 squid coupler was used to join the source and detector components to the single fiber sensor tip. Couplers of this type are not intended for use in bidirectional applications such as the reflectometer, where the light reflected from the tip must return through the coupler to the detector component. The losses in the coupler connections were extremely high, resulting in poor reflectometer sensitivity. The purchase of a proper bidirectional coupler was considered, but problems with temperature drift in the source and detector were discovered during the development of the glass rod refractometers at about the same time. The temperature instability of the source and detector components, which ultimately halted all refractive index sensor development, are discussed in the following
section. Because these temperature instability problems would have affected future reflectometers also, no further reflectometer probes were constructed.

**Glass rod U-bend refractometers**

The very delicate nature of the silica optical fibers used in the plastic-clad silica U-bend refractometers resulted in the desire for a more durable optical probe. Consequently, an attempt was made to construct a U-bend refractometer not from optical fiber, but from 3 mm glass rod of the type typically used in chemistry labs as a stirring rod. The rod was heated and bent into the tightest possible U-bend. The ends of the bend were then cut and polished to a 3 micron finish. An IRED source and phototransistor detector were attached to either end of the U-bend with heat-shrink tubing. After connecting the active components to drive and amplification circuits similar to those shown in Figure A3.8, this device was found to be quite sensitive to changes in fluid refractive index at the sensor tip. After the preliminary experiments, the device was firmly mounted in a plastic holder tube with silicone potting, and a complete series of refractive index measurements in room temperature sucrose solutions were made. The results of these experiments are presented in Figure A3.13. Based on these encouraging results, a version of this simple but durable probe was prepared for experiments in the oil-refrigerant flow loop. Unfortunately, adaptation of this probe to flow stream measurement introduced two problems. First, the integrated circuit source and detector of the probe were found to be very sensitive to temperature. Second, the methods employed for
Fig. A3.13. Glass rod refractometer measurements in flowing sucrose solutions at 77 F
constructing the flowstream pressure boundary caused considerable attenuation of the optical signal. These problems and the efforts to overcome them are discussed below.

**Temperature sensitivity** Figure A3.14a shows the first attempt to adapt the refractometer to flow stream measurement. The probe was simply potted in refrigeration system epoxy at the end of a 3/8 in copper tube with a pre-existing Swagelok connection. This assembly was then attached to the branch connection of a Swagelok tee in the flow loop test section. Mechanically, this design proved to be an excellent adaptation of the refractometer to flowstream measurement. However, this refractometer was found to perform poorly when subjected to large fluid temperature changes.

The infrared source and receiver are semiconductor devices, and as such are sensitive to changes in the temperature of their semiconductor junctions. The radiant output of an IRED source falls sharply as temperature increases, while the electrical output of a phototransistor or PIN photodiode rises slightly with temperature. Because these optoelectronic components were located very close to the fluid stream in this probe design, the temperature effects on the probe output were very pronounced.

Figure A3.14b depicts a probe in which a much longer glass U-bend was used. This arrangement moved the optoelectronic components away from the flow stream and into the ambient air. To eliminate the effect of ambient air temperature changes on the probe output, a small housing was constructed around the optoelectronic components. A thermostatically
Fig. A3.14. Two glass rod refractometers adapted for flow stream measurements.
controlled heater within the housing maintained the optoelectronic components at $90 \pm 0.5$ F. This arrangement was thought to be very promising for eliminating the temperature sensitivity problems exhibited by earlier probes. However, this new design also created a new problem which prevented further testing for temperature sensitivity. This new problem is discussed in the following section.

**Optical attenuation at the pressure boundary** The probe design depicted in Figure A3.14b used a layer of refrigeration system epoxy to seal the glass U-bend in the end of the copper tube. Unfortunately, this epoxy material was of a higher refractive index than the glass, causing much of the desired optical signal to be refracted out into the epoxy. A silicone material which is normally used to restore the cladding on damaged optical fibers was obtained for use as an intermediate coating between the glass and epoxy. This material, purchased from General Fiber Optics, has a refractive index much lower than that of glass. A new probe was constructed which used this silicone material between the epoxy material and the glass rod. Experiments with this new probe revealed that the intermediate coating restored the probe sensitivity and maintained the pressure seal to the flow stream environment.

With the optical attenuation problem solved, the only remaining obstacle to the refractometer's success was the sensitivity to changes in temperature. Although the thermostatically controlled heater effectively eliminated any possibility of ambient temperature influencing the sensor, continued research revealed that much of the sensor thermal drift problem resulted from self-heating of the IRED source. As the IRED self-heated,
the amount of radiation launched decreased. In an attempt to relieve the self-heating problem, the IRED drive current was changed from a DC signal to a low duty-cycle square wave signal. This change also required the use of a true-RMS voltmeter to measure the output voltage of the probe, rather than a simple DC voltmeter. Other changes in the probe design to improve stability included replacing the phototransistor detector with a photodiode. In general, photodiodes are less sensitive to incident light but more temperature-stable than phototransistors. This change required improvements in the design of the amplifier used to convert the photodetector output current to a voltage signal. Although these changes improved the probe performance, the repeatability of the probe over several hours was still quite poor. Consequently, development of the refractometer probe was discontinued.

Summary

Although early development of the refractive index sensors was quite promising, the sensor ultimately proved unsuitable for measurements in the flow stream environment. Several sensor types were attempted, including some novel probes constructed of common glass rod. The thermal sensitivity of the integrated-circuit light sources and detectors used in all of these sensors led to discontinuing the development effort. It should once again be emphasized that an important consideration in the development of these sensors was to keep the cost low. The temperature sensitivity problems discussed above have been solved by the manufacturers of optical power meters used in the testing of optical fiber communications systems. One of these power meters could be used,
together with any of several recently introduced bidirectional fiber couplers, to construct an optical fiber reflectometer. Although these optical power meters were prohibitively expensive at the time of the research reported herein (at least $5000), recently introduced portable optical power meters are available for approximately $1000. With relatively few additional components, one of these new instruments could form the basis of a successful refractive index sensor for oil concentration measurement.