

ONLINE MEASUREMENT OF OIL CONCENTRATIONS OF  
R134a-OIL MIXTURES WITH A DENSITY FLOWMETER

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**Abstract:** A very high accuracy, straight vibrating tube type of density flowmeter has been used online to measure oil concentrations of flowing R-134a/oil mixtures. The calibrations covered oil concentrations from 0-6 wt.% oil over the temperature range from -9.4 to 5.9°C. The oil concentrations were correlated statistically as a function of density, temperature and liquid compressibility to an average error of 0.09 wt.% oil with a 95% confidence limit of 0.21 wt.% oil. In addition, a simplified method not requiring calibration tests was developed for general industrial application of the density flowmeter to any refrigerant-oil mixture combination, with an average error of 0.22 wt.% oil and a 95% confidence limit of 0.67 wt.% oil for the present data set.

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## INTRODUCTION

Improving the integrity of thermal design methods and performance tests for refrigeration, air-conditioning and heat pump equipment is dependent on accurate knowledge of the oil concentration in the refrigerant charge. In particular, the heat duty of an evaporator is significantly affected by the presence of oil, where the boiling heat transfer coefficient and the mean temperature difference (MTD) are sensitive to its concentration. Recently, a new Thermodynamic Approach for modelling of refrigerant-oil mixtures has been developed by Thome (1995), which allows the local bubble point temperature, local oil concentration, local heat transfer coefficient, change in enthalpy and (potentially) local physical properties to be calculated for an evaporator from knowledge of the subcooled liquid-phase oil concentration. To implement this method and thus develop new, more efficient refrigeration units requires an accurate method for online measurement of the oil concentration.

Previously, oil concentrations have either been inferred from (i) measuring the masses of refrigerant and oil charged into the system, (ii) measuring the separate oil and refrigerant flow rates leaving an oil separator, or (iii) by withdrawing samples. The first method is unreliable since essentially no oil enters the vapor-phase (its vapor pressure is on the order of one-ten-millionth that of refrigerants) and hence the oil concentration in the liquid increases as an undetermined amount of refrigerant flashes to occupy the vapor space in the unit or test loop. The second method is also not accurate since an oil separator actually acts as a liquid-vapor separator and thus some refrigerant remains in the oil leaving the separator as liquid.

The third method, on the other hand, uses ASHRAE Standard

41.4-1994 to determine oil concentrations from three liquid samples withdrawn from the flow circuit. Each sample is weighed together with its bottle and then the refrigerant is allowed to slowly evaporate off through a valve. To be accurate, the sample must also be evacuated with a vacuum pump and heated up to 150°C to drive off the refrigerant, then weighed again with a top loading balance accurate to 0.01 g or better. The oil concentration is then the average of the three values so obtained. While this method is appropriate as a standard, it is not very convenient for practice. Shortcuts to the standard method, i.e. dispensing with the heating and/or evacuation procedures, result in overstating the oil concentration by as much as 1.5 wt.% oil or more. Consequently, an online method is imperative in order to measure the oil concentration conveniently or during the cyclic operation of a vapor-compression system, where taking numerous samples is too cumbersome to contemplate.

Various methods for determining oil concentrations online have been proposed and several have been tested. Baustian, Pate and Bergles (1988a, 1988b, 1988c) have used a Coriolis density meter, an acoustic velocity sensor and a viscosity meter, respectively, to measure oil concentrations with accuracies in the range of 1-2 wt.% oil. More recently, Meyer and Saiz Jabardo (1994) have built an ultrasonic acoustic velocity device and accurately calibrated it for measuring concentrations for R-12 and R-134a with various oils (95% confidence level accuracy of 0.23-0.26 wt.% oil). Suzuki, Fujisawa, Nakarazawa and Matsuoka (1993) instead have used a light absorption method for measuring oil concentrations online.

## TEST FLUIDS

The new refrigerant R-134a was tested with a polyol-ester type of lubricating oil compatible with R-134a. It was an ISO Grade 68 oil typical of heat pump applications with a specific gravity of 0.971 at 15°C and kinematic viscosities of 62.5 and 8.3 cSt at 40°C and 100°C, respectively [the oil's density was rechecked using a buoyancy type of gauge, obtaining a value of 970 kg/m<sup>3</sup> at 15°C, i.e. confirming the manufacturer's value].

Refrigerant and lubricating oil mixtures often exhibit an immiscible region as a function of temperature and oil concentration. The highest point of the immiscible region for the present R-134a/oil system, cited in the oil manufacturer's literature, was a temperature of -40°C, well below the lowest operating temperature in the present test flow loop.

## TEST FACILITIES AND EXPERIMENTAL PROCEDURES

The experimental work was conducted using the oil concentration test facility at the Laboratoire d'Energétique Industrielle (LENI) at the Swiss Federal Institute of Technology (EPFL) in Lausanne, Switzerland. The unit is part of a large flow boiling test facility described in Kattan, Thome and Favrat (1995).

Figure 1 depicts a simplified flow diagram of the oil calibration flow circuit. The subcooled liquid refrigerant-oil mixture is pumped around the loop by a stainless steel pump with a magnetically driven rotor, the latter which operates without any lubricating oil and eliminates any possibility of a different oil (or additional oil) from entering the flow circuit. The refrigerant-oil mixture then passes through a volumetric float-type of flow meter (utilized as a sight glass to see the flowing fluid and verify that no gas or vapor bubbles are present in the

flow). The test fluid then passes through a calibrated Coriolis meter to measure the mass flow rate. After passing through a filter, the test fluid enters a piston-and-cylinder arrangement that uses nitrogen backpressure on the opposite side of the piston (the nitrogen is not in contact with the refrigerant-oil mixture because a double PTFE O-ring seal is used) to pressurize the loop and maintain the desired pressure during variation of the test temperature, hence absorbing the change in fluid volume during heating and cooling to new test conditions. The refrigerant-oil mixture then passes through the very high accuracy density flowmeter and on to the heater. After the heater, the test fluid goes through a refrigerated cooler and back to the pump. The heater or cooler is used maintain the test fluid at the desired test temperature or change the test temperature.

A speed control on the pump was used to modify the refrigerant-oil flow rate to ascertain any effects of flow rate on the density meter. A wide range of flow rates (0.75-5.5 kg/s) was tested without any significant effect on the integrity of the measurements.

Other important features of the flow circuit are a sampling point for attaching stainless steel sample bottles to the flow circuit. The nitrogen backpressure in the piston-and-cylinder arrangement is used to compensate for the removal of fluid from the circuit into the evacuated sample cylinders. A separate temperature-controlled cooling coil wrapped on the sample bottle maintains its temperature below that of the test fluid to prevent back flashing of vapor into the flow circuit, which would change the oil concentration of the sample. A vacuum pump is also connected to the flow circuit for leak detection purposes and for

evacuating the circuit before introduction of the pure refrigerant. Leak tests are run with the vacuum pump to ensure the loop is completely leakproof, such that oil concentration cannot change with time during tests. The circuit is also heavily insulated to reduce heat gains from the ambient air.

The refrigerated water-glycol solution for the system's coiled tube-in-tube cooler is maintained at a fixed inlet temperature by a low-temperature refrigeration system with a large water-glycol receiver in its flow circuit. This configuration eliminates any cyclic temperature variations in the test fluid that can be caused by the refrigeration system's on-off operating cycle. The hot water circuit for the heater also has a large temperature-controlled receiver for the same purpose.

A high quality data acquisition system linked to a personal computer is used to acquire, analyze and store all test data. All sensors are carefully calibrated in the laboratory. The thermocouples are calibrated using the double precision method available in the data acquisition system. The computerized control system maintains the system at steady-state during tests and then is used to bring the facility to the next test condition. Subroutines calculate and display test variables in order to run the experiments at specific conditions. The outputs of the density, temperatures, pressures and mass flow rate are read and recorded by the data acquisition system.

After charging a measured mass of refrigerant R-134a to the circuit, it is subcooled to remove all vapor. After the pure R-134a calibration tests, the oil concentration is increased by injecting oil into the flow loop using a carefully manufactured and finely calibrated syringe with a threaded rod fitted to its

piston. Turning a nob on the rod forces a calibrated amount of oil into the flow circuit. This arrangement allows a known amount of oil to be added to a known mass of refrigerant, such that the desired oil concentration in the loop can be attained.

#### MEASUREMENTS AND ACCURACIES

The thermocouples are calibrated to obtain the highest accuracy possible, 0.03°C. For adiabatic conditions after leaving the rig off all night, all temperatures are within a maximum deviation of 0.18°F (0.10°C) and most are within 0.09°F (0.05°C) of the average. The refrigerant flow rate is measured with an estimated accuracy of 0.2%, based on our calibrations of the Coriolis flow meter. Absolute pressures are measured using calibrated sensors with an accuracy of 0.2% error.

The oil concentration measurements were done utilizing the ANSI/ASHRAE Standard 41.4-1994. In the present tests, 150 ml stainless steel sample bottles were used. A high accuracy mass balance (accurate to 0.01 g) with a measurement range from 0-4000 g was utilized for weighing the sample bottles. Heating and evacuation of the bottles as called for in the procedure were used. The concentrations determined using the weighed mass of R-134a and calibrated mass of oil injected using the syringe compared very closely to those determined with the ANSI/ASHRAE method.

#### DENSITY FLOWMETER

In the present study, a very high accuracy, commercially available density flowmeter was used as the measuring device. It operates with the entire fluid flowing through the meter such that measurements are continuous and no bypass circuit is required. The meter is a vibrating tube type, where the tube is

a 25.4 mm diameter, stainless steel corrugated tube about 1.0 meter long. The meter has a calibrated accuracy of  $0.1 \text{ kg/m}^3$  over a wide range of density and temperature. The electronics include compensating equations to account for nonlinear temperature effects on the instrument's operation. The device provides a digital display of the density and temperature and also a voltage signal for the density, which was read into our data acquisition system.

Figure 2 depicts the density meter installation. The meter is solidly mounted on an I-beam support in a vertical upflow position as recommended by the manufacturer. A nearby Coriolis (vibrating U-tube) flow meter did not affect operation (determined by shutting either one off). Static flow mixers are located in the inlet and outlet pipes to ensure the fluid is well-mixed and without radial temperature gradients. Calibrated thermocouples (600, 601, 602 and 603) are used to monitor the refrigerant-oil temperatures entering and leaving the meter (a small quantity of heat passes from the meter to the fluid). The average value of the temperatures measured by 601 and 603 is used for the refrigerant-oil temperature. Since the density of R-134a changes by  $3.3 \text{ kg/m}^3$  per  $^{\circ}\text{C}$  at the test conditions, temperature must be measured very accurately. The Pt100 resistance thermometer of the meter is normally used to correct for its small nonlinearity effects with temperature. In the present setup, this temperature correction was done by programming the correction correlation into the PC and using the more accurate thermocouple readings. The system pressure is measured nearby in the flow circuit.

An important operating limitation is the subcooling at the



exit of the meter. To avoid cavitation in the vibrating corrugated tube (apparently caused by its high vibration frequency), a minimum of 8°C of subcooling was required at its outlet. A further consideration is the sensitivity of the density flowmeter. The density difference between pure R-134a and the oil is approximately 300 kg/m<sup>3</sup> at the test conditions. Thus, in order to determine oil concentrations accurate to 0.1 wt.% oil, one must measure density to an accuracy of 0.30 kg/m<sup>3</sup>, including temperature effects.

#### ONLINE DENSITY MEASUREMENTS

Figures 3 and 4 depict the measured density data. Oil concentrations ranged from 0 to 6.0 wt.% while temperatures varied from -9.4°C to 5.9°C. The measured pressure was held from 5 to 6 bar. All together 246 data points were obtained. Repeatability of experimental data was very good and very little scatter was observed.

#### OIL CONCENTRATION CORRELATION

The measured liquid densities in Figures 3 and 4 are not only a function of temperature and oil concentration but also liquid compressibility. For example, the density of compressed R-134a increases by about 0.3-0.4 kg/m<sup>3</sup> for each increase of 1 bar with respect to the saturation pressure at the measured temperature. The oil concentration calibration using a density meter (or other types of meters such ultrasonic velocity or viscosity devices) should thus take the liquid compressibility into account. The liquid compressibility of the mixtures in the range from 0-6 wt.% oil can reasonably be assumed to match that of the pure refrigerant, R-134a.

Compressed liquid densities for R-134a have been measured by

Hou et al. (1992) over a wide range of temperature and pressure. They fit the Tait equation to their data with separate empirical parameters for each isotherm they tested. The Tait equation is given as

$$D_{\text{comp}} = [c - d \ln(P + e)]^{-1} \quad (1)$$

where  $D_{\text{comp}}$  is the compressed density in  $\text{kg/m}^3$ ,  $P$  is the pressure in MPa and  $c$ ,  $d$  and  $e$  are empirical constants. This equation extrapolates very accurately (about 0.3% error) to saturation densities measured in other studies or compared to accurate saturation density equations. Using their published table of empirical constants, these were statistically curvefit here as a function of temperature with the following expressions ( $T$  in  $^{\circ}\text{K}$ ):

$$c = 0.001649 - 1.02105 \times 10^{-5} T + 4.14318 \times 10^{-8} T^2 - 4.70121 \times 10^{-11} T^3 \quad (2)$$

$$d = 0.000204 - 1.90911 \times 10^{-6} T + 7.41708 \times 10^{-9} T^2 - 8.34015 \times 10^{-12} T^3 \quad (3)$$

$$e = 709.286 - 6.20421 T + 0.019443 T^2 - 2.15073 \times 10^{-5} T^3 \quad (4)$$

Figure 5 shows the empirical fit to the tabular values. Thus, the compressed densities and saturation densities can now be calculated directly from Eqs. (1-4) and significantly the density difference due to compressibility can be determined by subtracting the former from the latter.

The oil weight concentrations in mass fraction (not %),  $w_{\text{oil}}$ , were correlated with statistical regression methods to the following polynomial expression as a function of saturation density ( $D$  in  $\text{kg/m}^3$ ) and temperature ( $T$  in  $^{\circ}\text{C}$ ):

$$w_{\text{oil}} = b_1 + b_2 T + b_3 D + b_4 DT + b_5 DT^2 + b_6 D^2 T + b_7 D^2 T^2 + b_8 T^2 + b_9 D^2 \quad (5)$$

where  $b_1$  to  $b_9$  are empirical constants and  $D$  is the measured compressed density minus the density difference due to compressibility at the same temperature. This equation avoids the problem of quadratic equations used in some previous oil concentration studies that can produce multiple positive roots. The subcooled liquid flowing through the density meter is a compressed liquid; hence, the compressed liquid density and saturated liquid density are calculated at this temperature using Eqs. (1-4) for the measured pressure and for the saturation pressure (determined from an accurate vapor pressure equation), respectively, and the difference between these two values (the compressibility) is subtracted from the measured density to obtain the saturated density to use in Eq. (5). This method provides a general approach based to the saturation density curve for calibrating a density flowmeter that can potentially be applied to any refrigerant and oil combination.

The values of the empirical constants  $b_1$  to  $b_9$  are given in Table 1. The calibration correlation has an average error of 0.09 wt.% oil for all 246 data points measured, a maximum error of 0.69 wt.% oil and a 95% confidence limit of 0.21 wt.% oil.

#### INDUSTRIAL APPLICATION OF A DENSITY FLOWMETER

It is important to note that the density differences between refrigerants and their lubricating oils are about 200-400  $\text{kg/m}^3$  over the temperature range of industrial interest. A very high quality Coriolis flow meter can measure density with an accuracy of 2  $\text{kg/m}^3$ ; thus its best concentration accuracy would be 0.5-1.0 wt.% oil, i.e.  $2/200 \times 100\% = 1.0\%$ , which is not satisfactory for low oil concentrations. The present density flowmeter is accurate to 0.1  $\text{kg/m}^3$  with a potential accuracy 20 times that of

the best Coriolis meters, but this is only partially attainable since the temperature measurement error in the measured density becomes significant in this accuracy range.

As a shortcut "industrial" method, the saturation density of the refrigerant-oil mixture can be determined using a linear mixing law of the specific volume, i.e. that cited for the ideal density in ASHRAE (1994) with the correction factor, A, set equal to 1.0. This less accurate approach avoids the necessity of constructing an oil concentration teststand and performing the time-consuming calibrations, and it is applicable to any refrigerant-oil combination. The refrigerant-oil saturation density is defined by this ideal mixing law as:

$$D = D_{Oil}/[1 + (1-w_{Oil})(D_{Oil}/D_{ref} - 1)] \quad (6)$$

Solving this expression to obtain the oil weight fraction  $w_{Oil}$  (in mass fraction not wt.%) gives

$$w_{Oil} = [(D_{Oil}/D_{mixt})(D_{ref}-D_{mixt})]/(D_{ref}-D_{Oil}) \quad (7)$$

The refrigerant saturation density  $D_{ref}$  in this equation is calculated from an accurate equation for the refrigerant (such as ASHRAE methods, but not the less accurate Tait Equation). The lubricating oil density  $D_{Oil}$  can either be measured as a function of temperature or obtained from data supplied by the manufacturer. Otherwise, using a method adapted from Thome (1992), it can be estimated from the following equation for lubricating oils:

$$D_{Oil} = D_{man} [(T_{crit}-T)/(T_{crit}-(T_{man}+273.15))]^{0.29} \quad (8)$$

where  $D_{man}$  is the oil density ( $\text{kg}/\text{m}^3$ ) at the temperature provided by the manufacturer  $T_{man}$  (normally  $15^\circ\text{C}$ ),  $T_{crit}$  is the critical temperature of the oil (assume equal to 760 K for all oils as an approximation), and  $T$  is the measured temperature (K). Subtracting the density difference due to compressibility

determined from Eqs. (1-4) from the measured density and using the pure refrigerant and oil densities determined as discussed, the oil concentration can be determined using Eq. (7). This simplified method has been successfully compared to all the present test data in Figures 3 and 4 with a mean error of 0.22 wt.% oil. Because of nonlinear variations in density with composition, the accuracy ranges from about 0.1 wt.% mean error for the 0.5 wt.% mixture up to about 0.5 wt.% mean error for the 6.0 wt.% mixture, which is satisfactory for most applications.

Hence this simplified method can be used for industrial teststands with installation of a high accuracy density flowmeter. It is suggested that the meter be calibrated against the pure refrigerant first, i.e. measure the pure refrigerant's density with the meter, correct this value for liquid compressibility and then compare it against the calculated saturated refrigerant density, to get the best results.

The accuracy for conditions and refrigerant-oil combinations other than the present ones will be dependent on the nonlinearity in the density with oil concentration; as the correction factor A deviates farther from the value of 1.0, the less the accuracy will be. If, on the other hand, the value of A is known or can be correlated as a function of temperature from published data, the method will provide even more accuracy than reported for the present tests.

## CONCLUSIONS

A high accuracy density flowmeter has been accurately calibrated to measure online oil concentrations of R-134a/ester oil mixtures. The calibrations covered liquid oil concentrations from 0-6 wt.% oil for temperatures from -9.4 to 5.9°C (those typically confronted in flow boiling tests with oil). The oil concentration was statistically correlated using a new method that now includes the effect of liquid compressibility together with the functional dependency on saturation density and temperature. The mean error was 0.09 wt.% oil and the 95% confidence limit error was 0.21 wt.% oil. In addition, an easy to use method not requiring calibration tests was developed for general industrial application of the density flowmeter to any refrigerant-oil mixture combination, with an average error of 0.22 wt.% oil for the present data with a 95% confidence limit of 0.67 wt.% oil.

## ACKNOWLEDGEMENT

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Table 1. Constants for Eq. (5).

$b_1$	=	0.000429355
$b_2$	=	0.000013673
$b_3$	=	0.275218460
$b_4$	=	0.006379454
$b_5$	=	0.000298102
$b_6$	=	-0.000005479
$b_7$	=	-0.000000240
$b_8$	=	0.000038854
$b_9$	=	-0.000212907

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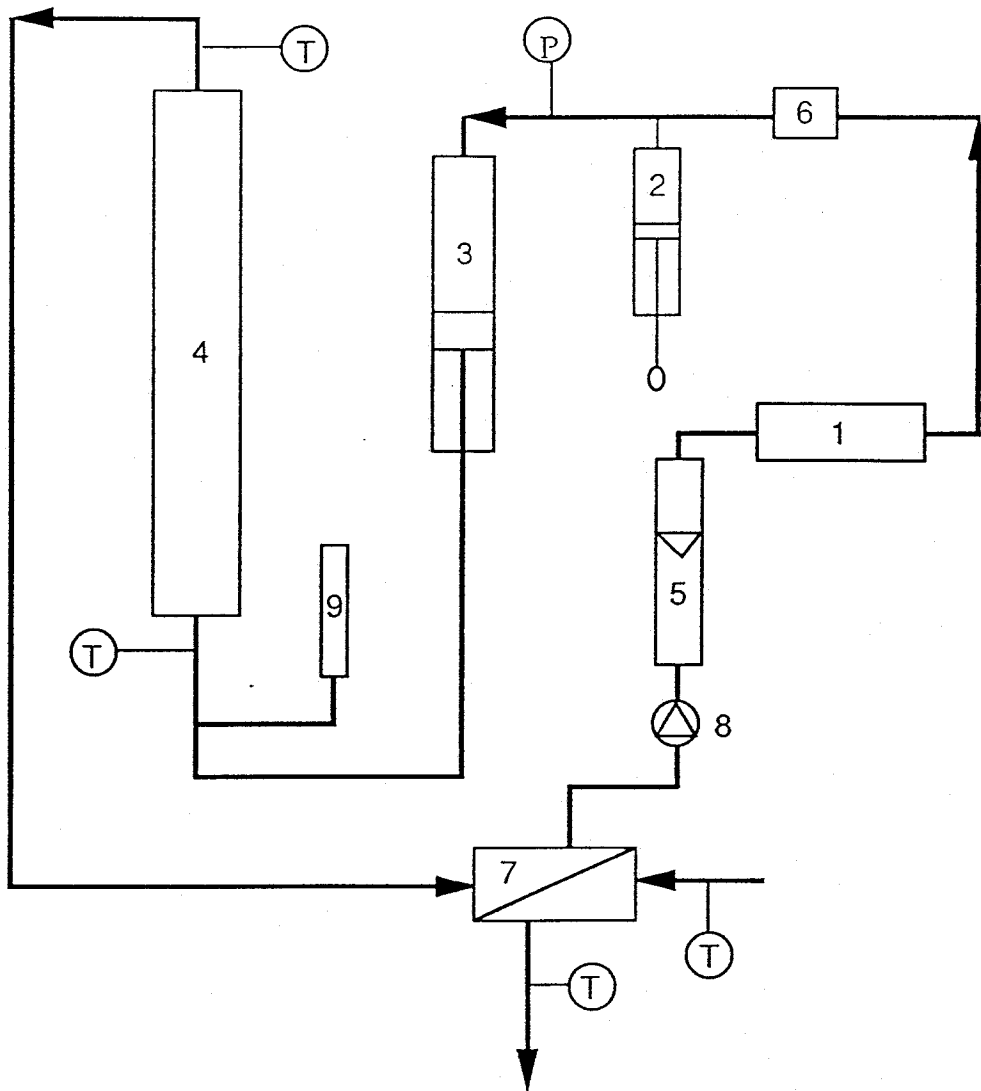


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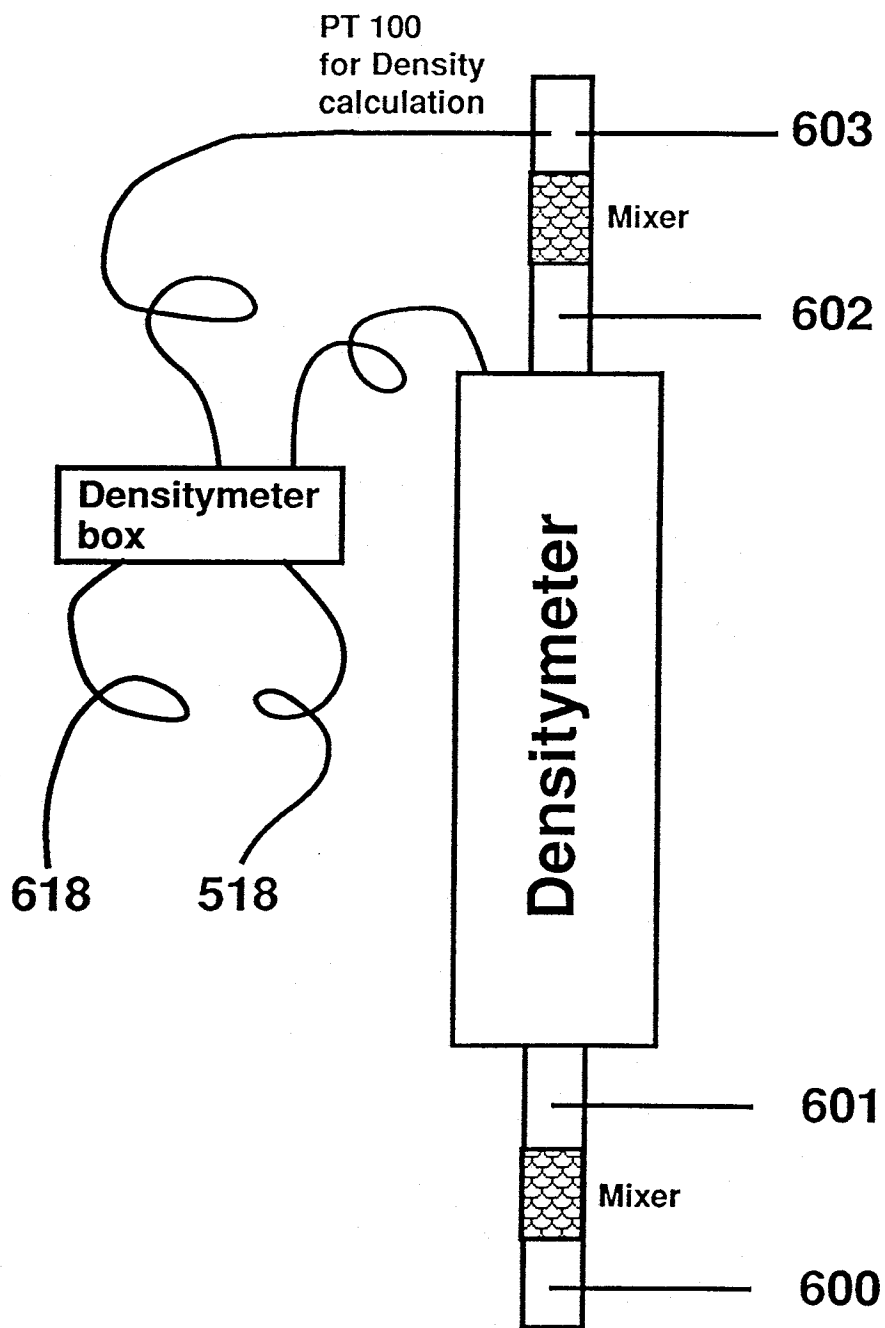


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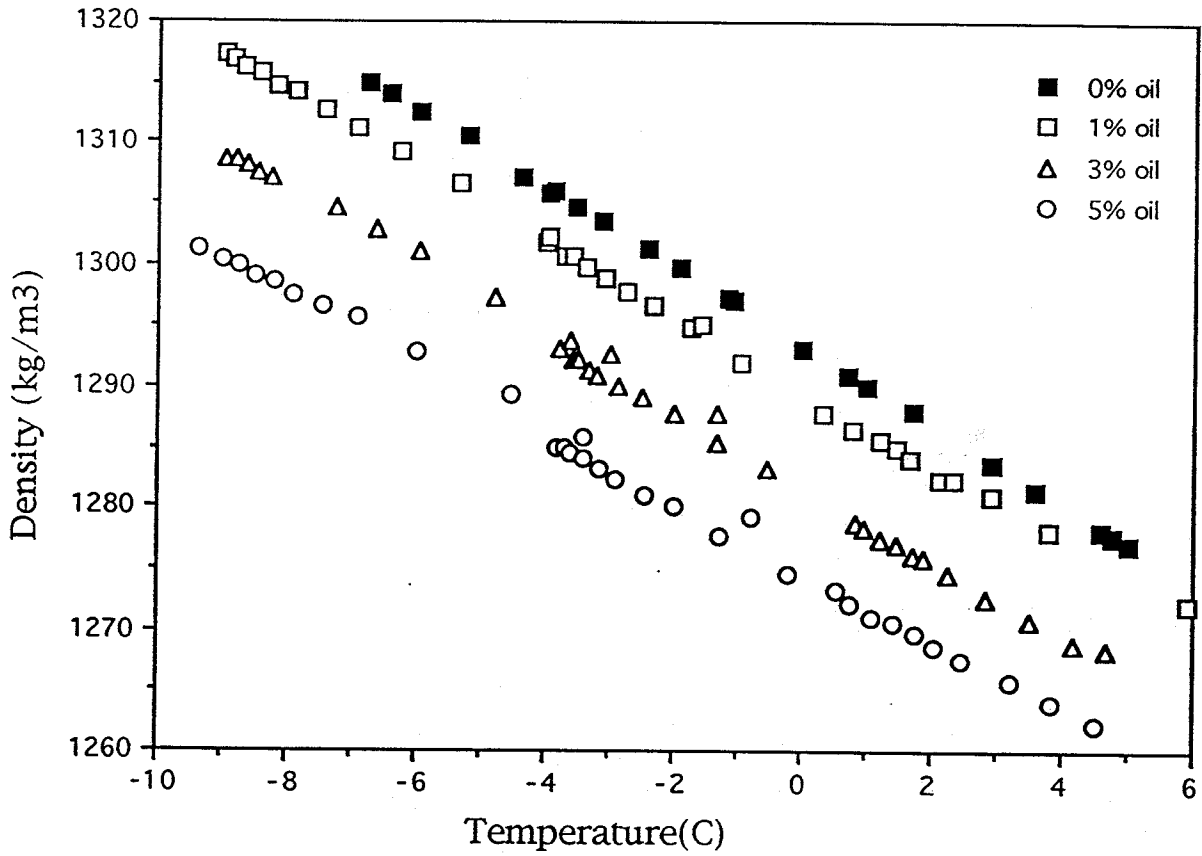


Figure 3. Density measurements for 0, 1, 3 and 5 wt.% oil concentrations in R-134a.

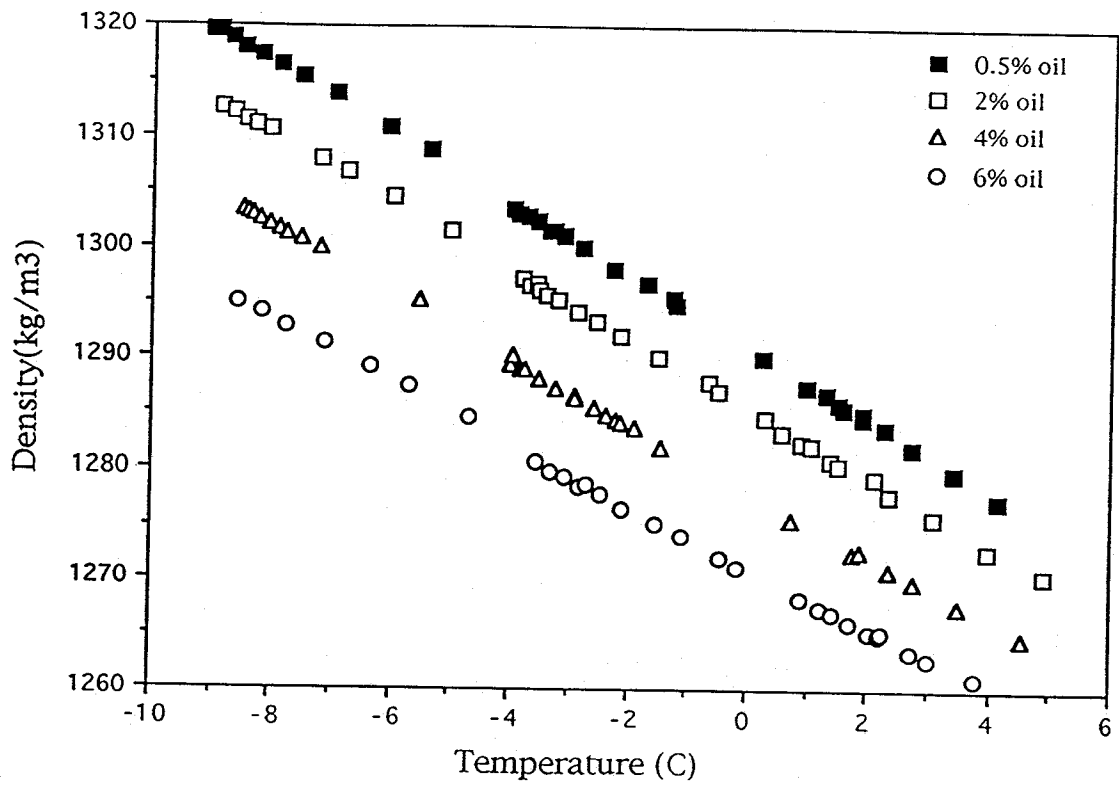


Figure 4. Density measurements for 0.5, 2, 4 and 6 wt.% oil concentrations in R-134a.

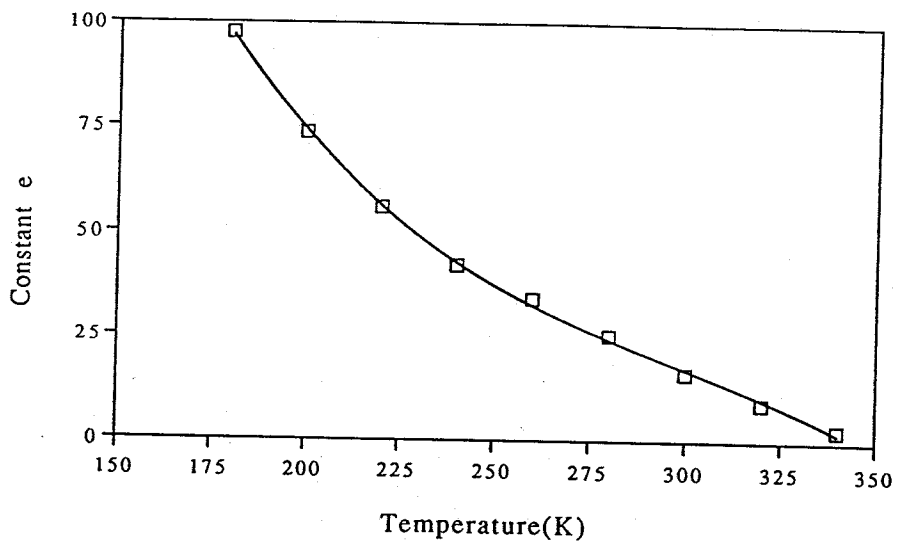
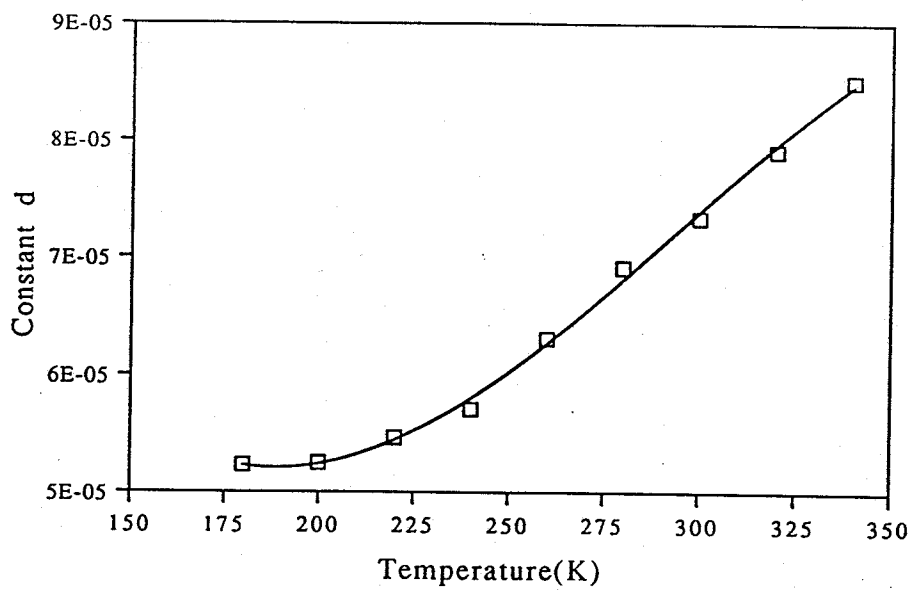
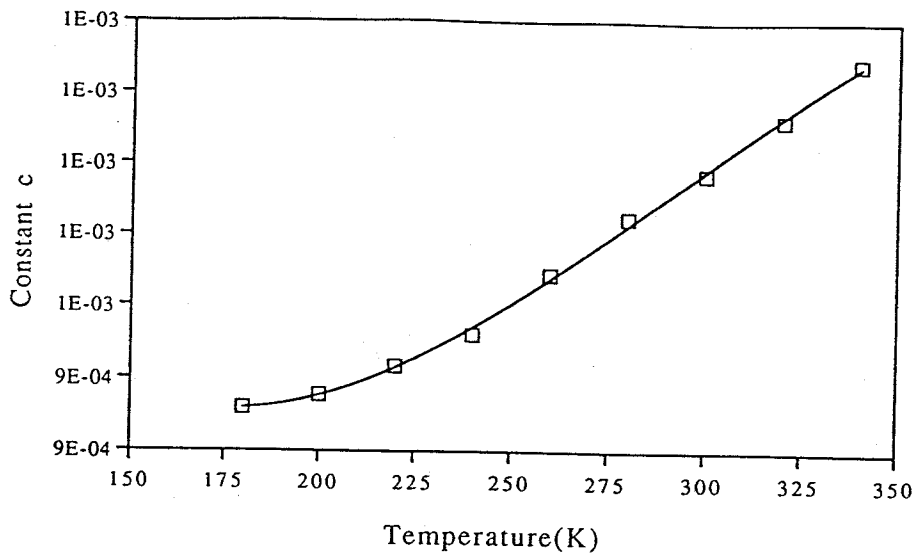


Figure 5. Constants for Tait Equation.