Measuring Lubricant Concentration in the Mixture with Refrigerant: A Comprehensive Review

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https://doi.org/10.5109/7363498

出版情報：Evergreen. 12 (2), pp.1106-1130, 2025-06. Transdiscilinary Research and Education Center for Green Technologies, Kyushu University
バージョン：

Measuring Lubricant Concentration in the Mixture with Refrigerant: A Comprehensive Review

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(Received December 09, 2024; Revised March 14, 2025; Accepted April 10, 2025)

Abstract: Lubricants in mechanical vapor compression systems play an important role in lubrication and thermal insulation. During system operation, small quantities of oil are observed to dissolve with working fluid. Oil migration from the compressor results in refrigerant lubricant mixture, which effects the thermophysical properties of working fluid. A prediction of oil composition can help in determining properties in refrigerant/lubricant mixture. A lot of reviews have been published regarding to lubricants, their interaction with refrigerants, their thermophysical properties and effect on overall system. However, there is a need for systematic database for oil concentration evaluation methods. This review paper focuses on an overview of various techniques to measure oil concentration in refrigerant/lubricant mixtures. This review provides an overview of oil concentration evaluation methods based on various properties of the refrigerant lubricant mixture.

Keywords: compressor; lubricant; refrigerant/lubricant mixture; thermophysical properties

1. Introduction

In a mechanical vapor compression (MVC) system, the compressor circulates the refrigerant across the evaporator and condenser. The compressor drives the system using electricity. Being prime movers, compressors need lubrication. It is reported that properly designed lubricants for compressors can decrease the energy consumption up to 15 %[1] increasing the overall efficiency of the system. Lubricants help in avoiding overheating and prevent corrosion[2]. The lubricant, mainly responsible for lubricating and providing thermal insulation to moving parts, is usually fed during compressor manufacturing in many applications[3]. Although initially charged concentration is expected to lubricate compressor for the entire life of system, small quantities of lubricant often migrate to other parts of the system including condenser, evaporator and connecting pipes. To manage this problem, oil separators are usually installed, however, it is not possible to completely eliminate oil migration, hence oil returned to the compressor needs to be ensured to avoid oil starvation and early system failure. On the other hand, properties of pure refrigerant are important for designing HVAC (Heating, Ventilation and Air Conditioning) system as they effect the overall efficiency of system[4] as well as environment[5]. Next generation low GWP (Global Warming Potential) refrigerants[6] are also being extensively researched[7] to mitigate global warming and meet new regulations. Since lubricants interact with the circulating working fluid, the resulting mixture has properties that are different from those of the pure refrigerant[8] and it effects performance of refrigeration system[9,10]. Therefore, it is very important to understand the nature of lubricants, refrigerant lubricant interaction and mixing ratio of refrigerant and lubricants in various parts of the system. Lubricants are mainly divided into two main categories, (1) mineral oils which are obtained from hydrocarbons such as petroleum and (2) synthetic

Cite: I. Sameen et al., "Measuring Lubricant Concentration in the Mixture with Refrigerant: A Comprehensive Review". Evergreen, 12 (02) 1106-1130 (2025). https://doi.org/10.5109/7363498.

lubricants. Mineral oils have high miscibility with the first generation refrigerants; however, they have limited miscibility and solubility with hydrofluorocarbon (HFC) and hydrofluoroolefin (HFO) refrigerants. For example, mineral oils have poor miscibility with R-32[11], but synthetic lubricants offer as excellent replacements. Common types of synthetic oils include Polyalkylene glycol (PAG), Polyolester (POE), Alkyl benzene (AB) and Polyalphaolefin (PAO) oils and Polyvinyl ether–(PVE). Table 1 provides common lubricants and refrigerants. More details on the lubricants used in refrigeration system is provided elsewhere[12].

Understanding the interaction between the refrigerant and the lubricant helps evaluating compatible pairs that can offer better solubility and miscibility ranges. Replacement of high GWP refrigerants, reinforced by various protocols, requires suitable pairs of new low GWP refrigerants and lubricant oils[13], which are compatible with each other. Lubricant presence alters thermophysical properties of refrigerant, impacting the overall heat transfer[14,15]. Literature reports various properties of refrigerant lubricant mixtures, including speed of sound, viscosity, density, capacitance, refractive index, and thermal conductivity. These properties not only help in better understanding of refrigerant lubricant interaction but can also facilitate predicting the oil composition. Moreover, oil composition in a system is a good indicator of the overall health and failure monitoring of the system. It is also an indicator of oil quantity in the compressor and oil retention in various parts of the system. Higher oil concentration in various parts of system can result in decreased heat transfer in the evaporator[16] and a decrease capacity and coefficient of performance (COP)[17]. Few researchers have also reported positive effect on heat transfer in certain conditions and at low oil quantities[18]. Since the presence of excessive oil has mostly negative impact on the system, compressors with low oil circulation rate are becoming a trend, which makes accurate determination of the oil concentration highly important[19]. Several techniques including speed of sound measurement, viscosity measurement and density measurement have been used to characterize oil refrigerant mixing ratio in working fluid. These techniques involve both offline and inline measurements. The central idea is that there is usually a huge difference between the properties (speed of sound, viscosity etc.) of pure lubricant and pure refrigerant, which helps in relating mixing ratio with these properties. A lot of reviews have been published regarding to lubricants, their interaction with refrigerants, their thermophysical properties and effect on overall system[15,16]. However, there is a need for systematic database for oil concentration evaluation methods. This review paper focuses on an overview of various techniques to measure oil concentration in refrigerant/lubricant mixtures, as summarized in Fig 1. A comparison of accuracy and limitations is also discussed.

Table 1: Various types of lubricants in refrigeration system

Lubricant	Refrigerant	Comments
Mineral Oils	CFCs (R-12) HCFCs (R-22) HCs (R-600a)	Low Cost Insoluble with HFCs and HFOs. Inadequate pour point
Polyalkylene Glycols	HFCs (R-134a) HFO (R-1234yf) HC (R-290) CO_2 (R-744)	High viscosity index, excellent lubricity, and low pour point Susceptible to depolymerization and hygroscopic
Polyvinyl Ether	HFCs (R-134a, R-404A, R-410A, R-407C, R-32) Pure HFOs and blends (R-1234yf, R-1234ze(E)), CO_2	More hygroscopic than POE, but resistant to hydrolysis High electrical resistivity Higher miscibility than PAOs with HFC refrigerants.
Polyolester	HFC (R-744) Pure HFOs and blends ((R-1234yf, R-1234ze(E))	Thermal stability Can be finely optimized for a particular refrigerant and application.
Polyalphaolefin	Ammonia (R-717) CO_2 (R-744) CFCs (R-12, R-114)	High Viscosity Index Low pour point Thermal Stability
Alkyl Benzene	HCFC refrigerants (R-22, R-502) HFC (R-32)	Suitable for low temperature application in HCFC refrigerants due to lower pour point High temperature stability Oxidation stability

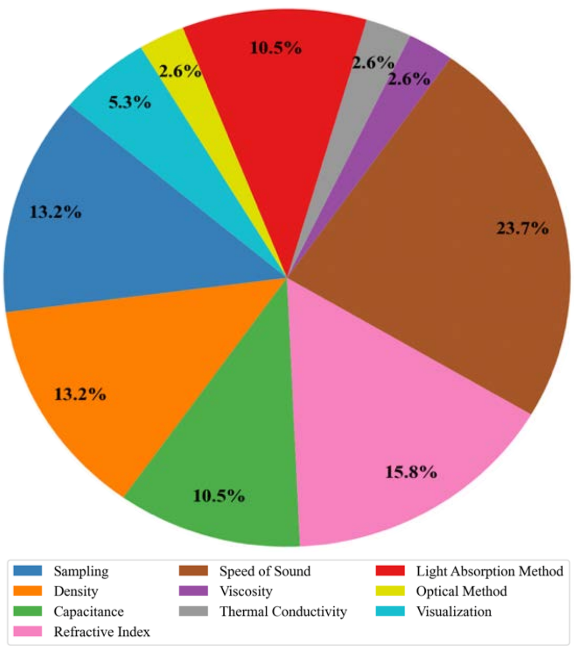


Fig. 1: Methods described in the literature for oil composition evaluation in the refrigerant/oil mixture based on different properties

2. Oil composition evaluation in Lubricant/ Refrigerant Mixtures using Speed of Sound

The speed of sound in a refrigerant is usually significantly lesser than that in the oil. Observing the speed of sound in the mixtures at various compositions, relations for predicting the oil concentration can be established.

Speed of sound is an important thermodynamic property that can be related to other thermodynamic properties of a liquid[20)21)]. Sound velocity, speed of sound, and ultrasonic speed of sound are largely the terms used to describe speed of ultrasound waves in a liquid[22)]. Thermodynamic speed of sound evaluation is based on the length of travelling path and time of flight and hence there are less error sources. Newton Laplace equation relates speed of sound to density and isentropic compressibility[23)]. Furthermore, speed of sound provides a direct approach to determine isentropic compressibility which can be used to get other properties at both ambient and high-pressure conditions. Isentropic heat compressibility provides the most accurate and convenient approach to calculate isothermal compressibility. Moreover, experimentally determined speed of sound alongside isobaric heat capacity and isobaric expansibility can be used to calculate isochoric heat capacity[22)]. Speed of sound measurements, owing to their simple approach and lower overall uncertainty, are widely used in developing the Equation of State (EoS)[24)25)26)].

Speed of sound can be a beneficial approach to study thermodynamic properties of refrigerant lubricant mixtures as well as determining oil concentration in various parts of the system, using theoretical models. This prediction method has generally no effect on flow rate, viscosity, and pressure of refrigerant lubricant system, which makes it quite useful.

Speed of sound can be related to other thermodynamic properties of fluid. For the ideal gas, it can be written as,

$$w_0 = \sqrt{\frac{\kappa_0 RT}{M}} \qquad (1)$$

Where w is speed of sound and $\kappa 0$ is the ideal gas heat capacity ratio. Speed of sound is related to specific heat capacity using the following,

$$w^2 = \left(\frac{\partial p}{\partial \rho}\right)_T + \frac{T}{\rho^2 C_V}\left[\left(\frac{\partial p}{\partial T}\right)_\rho\right]^2 \qquad (2)$$

Using speed of sound, it is possible to get the specific heat capacity, which can be used to develop the EoS.

2.1. Experimental measurement of Speed of Sound

Sound waves are longitudinal waves that oscillate along the direction of propagation of waves and have regions of compression (increased pressure) and rarefaction (decreased pressure). The nature of sound wave propagation depends on the thermodynamic properties of medium and dissipation of sound energy[27)].

Speed of sound can be mainly evaluated using steady-state methods and transient methods. Steady state methods involve measurement of speed of sound by analyzing non-transient behavior of sound waves. These methods are mainly used for gases and outside the scope of this review. Transient methods, more specifically, pulse methods are extensively used for speed of sound measurement in liquids.

Thermodynamic speed of sound evaluation using pulse methods is mainly based on length of travelling path in a test fluid and time of flight taken by sound wave to travel from one side of fluid to other.

Pulse methods involve single pulse and multiple pulse techniques. Single-pulse techniques involve two transducers (Time-of-fight technique)[28,29)] or one reflector and transducer (pulse echo technique)[30–34)]. Time of flight method involves two ultrasonic transducers connected to opposite sides of an acoustic cell. The transducer can be either separated from liquid surrounded a wall or can be directly in contact with testing fluid. The time difference between sent and received signals is measured to calculate the speed of sound. In pulse echo technique, there is mainly one ultrasonic transducer and one reflector arrangement. In this case, half of the time difference between sent and received signals is used to calculate speed of sound. An overview of various devices used for speed of sound measurement in liquids is shown in Table 2.

Azevedo et al28). proposed non-intrusive time-of-flight (ToF) using apparatus as shown in Figure 2. It consists of

two transducers attached to opposite sides of a thick stainless-steel cylinder containing a test fluid. This apparatus provided an additional benefit since transducers were not in direct contact with the fluid thus avoiding any damage to transducers in the case of non-compatible or corrosive fluids.
Speed of sound is measured as follows:

$$w(p,T) = \frac{l(p,T)}{(t_{tof} - t_{steel})} \qquad (3)$$

where t_{tof} ,w , t_{steel} and l denotes ToF, speed of sound, delay time and acoustic length, respectively. Delay time is the additional time taken by sound waves due to presence of steel walls before the transmitting and receiving transducers. t_{steel} and l are dependent on temperature and pressure.
Institute of Fundamental Technological Research group35) also proposed a ToF based approach to measure speed of sound. This apparatus was mainly proposed for speed of sound measurement in edible oils as shown in Figure 3. Ultrasonic transducers were immersed in a test fluid, of which pressure was controlled using a piston cylinder assembly working with a hydraulic press. Speed of sound is measured using the basic formula:

$$w = \frac{l}{\Delta t} \qquad (1)$$

where Δt is the time required between transmitted and received sound signal and l is length of travelling path in the fluid between transducers.

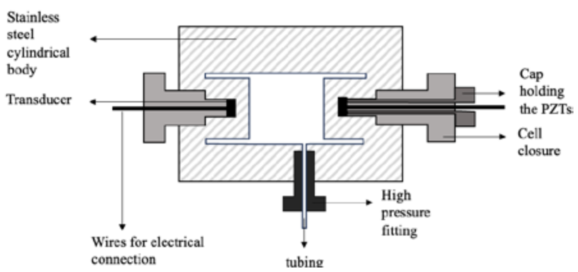


Fig. 2: Non-intrusive time-of-flight apparatus

Javed et al36), used dual path pulse-echo setup proposed originally by Kortbeek30), in which a quartz crystal was used as a transducer as shown in Figure 4. The arrangement consists of single transducers with two reflectors on the opposite sides at different distances. When the quartz crystal was excited using a sinusoidal burst signal, two sound waves were propagated in both directions and were received by the quartz crystal transducer after passing through the fluid and reflecting from the reflectors.

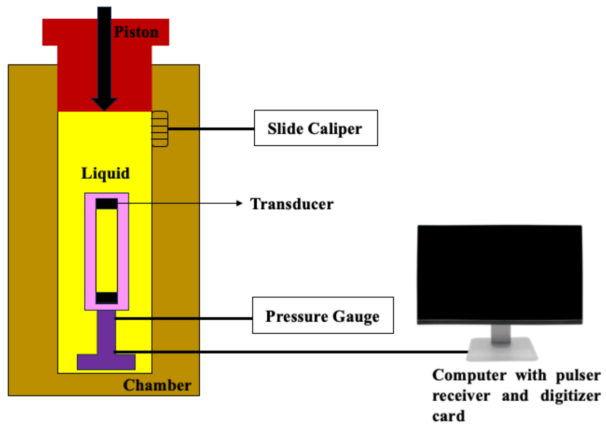


Fig. 3: A schematic of speed of sound apparatus, redrawn by the author based on the setup by IFTR [35)]

Since both reflectors were placed at different distances, the reflected sound waves are received at different time instances by the quartz crystal transducer. Speed of sound was measured as:

$$\boldsymbol{w = \frac{2\Delta L}{\Delta t}} \qquad (5)$$

Where ΔL is the difference in path length of two reflectors and Δt is the delay in time of flight for the length $2\Delta L$.

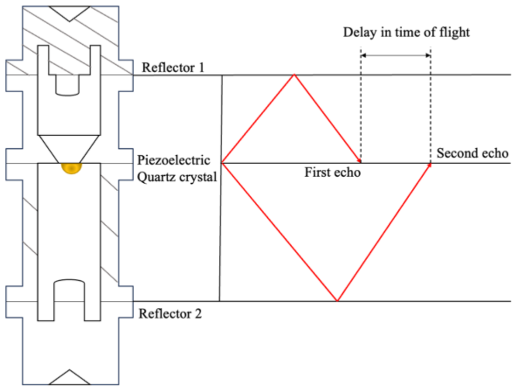


Fig. 4: A schematic reproduced by the author based on the dual path pulse-echo method[36)]

Trusler et al37,38), also used dual path pulse echo method for speed of sound measurement. A similar setup was also used by McLinden et al. for measurement of speed of sound in liquids mainly refrigerants and refrigerant mixtures as shown in Figure 5. Furthermore, Fortin et al.39) used a commercial speed of sound analyzer in pure polyester lubricants. Three transducer array method uses pressure fluctuation to estimate speed of sound mainly for two phase mixture40,41) but can also be used for pure liquids as shown in Figure 6.

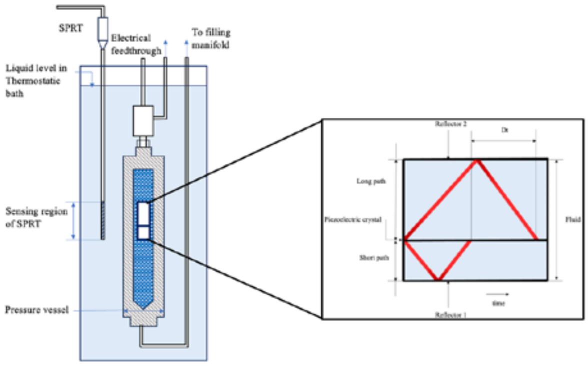


Fig. 5: A schematic recreated by the author, based on the setup used by National Institute of Standards and Technology. [20]

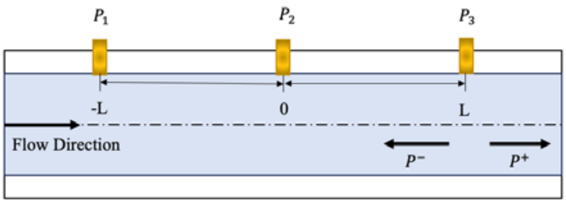


Fig. 6: A schematic of the transducer array method, reproduced by the author based on the setup described in literature. [40]

This method assumes that in tubes of common systems, for example refrigeration systems, the acoustic waves travel as one-dimensional plane waves.

The pressure pulsation has the following form:

$$\boldsymbol{P = ae^{-j\frac{2\pi f X}{w}} + be^{-j\frac{2\pi f X}{w}}} \qquad (6)$$

where f is the frequency, w is the speed of sound and X is axial distance along the tube. The speed with which fluid is moving is ignored when testing liquids.

The pressure from each pressure transducer can be written as following:

$$\boldsymbol{P_1 = ae^{-j\frac{2\pi f X}{w}} + be^{-j\frac{2\pi f X}{w}}} \qquad (7)$$

$$P_2 = a + b \qquad (8)$$

$$P_3 = \ ae^{-j\frac{2\pi f X}{w}} + be^{-j\frac{2\pi f X}{w}} \qquad (9)$$

Hence,

$$\boldsymbol{\frac{P_1 + P_3}{2P_2} = \cos(\frac{2\pi f d}{w})} \qquad (10)$$

The value of w is adjusted so that the minimization function becomes minimum.

$$E^* = \sum_{i=1}^{n} \left(\frac{P_1 + P_3}{2P_2} - \cos\left(\frac{2\pi f d}{w}\right) \right)^2 \qquad (11)$$

This speed of sound in fluid is generally compared with speed of sound of pure fluid to account for impurities such as oil mixed in fluid.

Single-pulse methods are useful for measurement of variation in speed of sound in liquids. However, these methods have limitations in measurement of absolute speed of sound[42]. Accuracy of Single-pulse methods is mainly dependent on measurement of time delays between transmitting and received signals and pulse shape. Distortion in pulse shape or electronic time-delays can induce high error. This limitation is overcome by using multiple reflections of pulse between transmitter and receiver. Transducer array method is also useful and provides reliable results however it was found that at low speed of sound conditions, the effect of noise can cause additional error[40]. Although speed of sound is obtained using different methods, the experimental setup for such measurement mainly consists of the ultrasonic transducers with housings, an acoustic cell, an oscilloscope and a pulser receiver device. Oscilloscope is used to visualize and acquire the sound signal. A pulser receiver device is used to trigger the signal. Once the signal is triggered and received, an oscilloscope is used. Then using wave analysis, the speed of sound is measured. Either a system simulating refrigeration system is used and sound sensor is installed in the subcooled region, or a mixture tank with controlled temperature and pressure is used with a speed of sound sensor.

Table 2: Various devices used for speed of sound measurement in liquids

Author	Type	Summary
Marek et al.[35,43–45]	ToF*	Temperature range: 293 – 303 K Pressure range: up to 650 MPa Fluids: Vegetable Oil, Biofuels, Olive Oil and Camelina Sativa Oil Uncertainty: 0.3%
Azevedo et al.[28,46–51]	ToF	Temperature range: 265 – 343.15 K Pressure range: up to 160 MPa Fluids Tested: methanol, hexane, heptane, octane, toluene, ethanol, 1-propanol, Ionic liquids, and Propane derivatives. Uncertainty: 0.1%
Mclinden et al. NIST group[52–55]	Dual Path PE**	Temperature range: 230 – 420 K Pressure range: 2.1 to 70 MPa Fluids Tested: R-32, R-1234yf, R-1234ze(E), R-1233zd(E) and R-1336mzz(Z), Mixtures of R-32, R-1234yf and R-1234ze(E) and Mixtures of R-134a, R-1234yf and R-1234ze(E). Uncertainty: 0.08%
Javed et al,2020.[36,56]	Dual Path PE	Temperature range: 208 – 500 K Pressure range: up to 151 MPa Fluids Tested: alcohols, i.e., methanol, propanol, 1-propanol, butanol, hydrocarbons, i.e., heptane, octane, 1-hexene,1-heptene, siloxanes, xenon, vinyl chloride and Novec 649, Glycols. Uncertainty: 0.1%
Trusler[37,42,57,58]	Dual path PE	Temperature range: 248 – 473 K Pressure range: up to 200 MPa Fluids Tested: n-hexane, n-hexadecane, N-methyl-2-pyrrolidinone, methanol , R-32, Toluene and Para-xylene. Mixtures: $CO_2 + H_2O$, $NaCl + H_2O$ as well as the ternary $CO_2 + H_2O + NaCl$ mixtures. Uncertainty: ±0.1%

*Time of flight
** Dual Path Pulse echo

2.2. Oil composition prediction in Oil Refrigerant Mixtures using speed of sound

Speed of sound in pure liquids can be related to the speed of sound in liquid mixtures using a mixing rule as discussed by several authors[59–62]. These models relate the speed of sound in the mixture to the composition of each component and vice versa. However, in case of refrigerant and lubricant mixtures, special assumptions are required. Oil composition prediction using speed of sound in literature is mainly based on polynomial equations and general model as summarized in Table 3. The polynomial equations are first order and second order equations with fitting coefficients depending on specific experimental data. Various models relating the speed of sound in mixtures to their constituent composition are provided in literature as shown in the table. These models can be used to calculate speed of sound for mixtures using the concentration of each component and vice versa. Andrade developed a theoretical model to relate speed of sound in the mixture with oil concentration using following,

$$\boldsymbol{w_{mix}} = \frac{[\boldsymbol{m_{oil}} + \left(\frac{\boldsymbol{\rho_{oil}}}{\boldsymbol{\rho_{ref}}}\right)\boldsymbol{m_{ref}}}{\boldsymbol{m_{oil}}\left(\frac{\mathbf{1}}{\boldsymbol{w_{oil}}}\right) + \left(\frac{\boldsymbol{\rho_{oil}}}{\boldsymbol{\rho_{ref}}}\right)\boldsymbol{m_{ref}}(\frac{\mathbf{1}}{\boldsymbol{w_{ref}}})} \quad (12)$$

It was observed that error is prediction increased with increase in oil concentration[63]. Later, Fukuta et al[64] utilized this model for the oil composition prediction. However, great prediction inaccuracy was observed. Andrade model was used to formulate a relation between oil concentration and speed of sound. While Andrade Model provides a good estimation for oil concentration (and mixture speed of sound), it has few limitations. This model assumes that the lubricant and refrigerant mixture has two separate layers of lubricant and refrigerant. However, practically, a homogenous mixture of lubricant and refrigerant is obtained upon mixing both components (if they are miscible in each other). Furthermore, since miscibility of different lubricant and refrigerant pairs changes with temperature, generally small amount of refrigerant exists in vapor form above the liquid surface.

Table 3: Available model for oil composition prediction in oil/refrigerant mixtures

Model	Mixtures Tested	Comments
Andrade Model	R-22/AB[63], R-410/PVE, CO2/PAG[64]	Theoretical model based on assumptions. Low accuracy at higher oil concentration.
Meyer correlation	R-12 with 2GS and 5GS oils R-134a/POE[65]	Polynomial Equation Requires experimental data.
Baustian Model correlation	R-12/naphthenic oil. R-22/naphthenic oil, R-502/AB[67]	Polynomial Equation Requires experimental data.

There is lack of theoretical models for speed of sound in refrigerant lubricant mixtures and further improvement of Andrade model can help in avoiding the time tedious calibrations required in speed of sound measurement. It was estimated that incase of mixtures with higher oil concentration, quadratic/polynomial approximation curves can provide a better accuracy compared to Andrade model. Following equations show the relation used by Baustian, Meyer, and Lebreton et al.[65–67]:

$$\boldsymbol{x_{oil} = A_0 + A_1 w_{sat} + A_2 T} \qquad (13)$$

and

$$\boldsymbol{w - w_{sat} = (A_3 + A_4 T)(P - P_{sat})} \qquad (14)$$

where A_{0-2} are constant coefficients, T is temperature and w_{sat} is the speed of sound in saturated liquid and w is speed of sound in subcooled state. Equation 2 is used to understand the effect of subcooling on sound speed and requires experimental data obtained from NIST for pure refrigerants. While the equation offers simplicity, accuracy is limited.

Another relation proposed by Baustian is as follow [67]:

$$\boldsymbol{w = b_0 + b_1 x_0 + b_2\theta + b_3 x_o^2 + b_4 x_o \theta + b_5 x_o^2 \theta} \qquad (15)$$

where,

$$\boldsymbol{\theta = T/T_{ref}} \qquad (16)$$

A significant limitation of Bastian relation is that oil concentration term in squared which makes correct prediction difficult in case of negative roots.

Due to lack of theoretical models specifically for oil refrigerant mixtures, fittings which require experimental data are used. Experimental measurement of sound speed in oil refrigerants mixture and prediction of oil composition is discussed below in Table 4. It is important to note that overall accuracy is determined by considering uncertainty in speed of sound measurement, error in charged oil composition and the error caused by specific fitting polynomial chosen for experimental data. In case of theoretical model, it also involves errors due to assumptions considered in the theoretical model development.

Speed of sound measurement methods include time of flight[63,68] and pulse echo technique[65,66]. Another technique[41] is utilizing an array of three pressure transducer and measuring speed of sound from pressure fluctuation. The speed of sound data in mixture is used to predict its oil concentration. Furthermore, in immiscibility regions, emulsion of oil and refrigerant is formed by mixing. In this case, other oil prediction methods like refractive index-based technique cannot be used, however, speed of sound provides a promising solution.

Baustian et al compared oil prediction using acoustic measurements in refrigerant/lubricant mixtures with methods based on density, refractive index, and viscosity. It was concluded that acoustic methods provide better accuracy. Further improvements can be made by using a more efficient measurement setup, since in current setup, transducers were used outside recommended temperature range. This also led to their early failure and error in repeatability experiments. Meyer et al 65), used time of flight method to determine speed of sound in refrigerant lubricant mixture as shown in Figure 7. The testing fluid is in the subcooled state, however, for reference value of speed of sound, saturation speed of sound of refrigerant is used. Since, it was difficult to maintain subcooling, Meyer provided correction for pressure effect on subcooling with relation to saturation state. Speed of sound was related to oil concentration and effect of pressure on speed of sound was also considered.

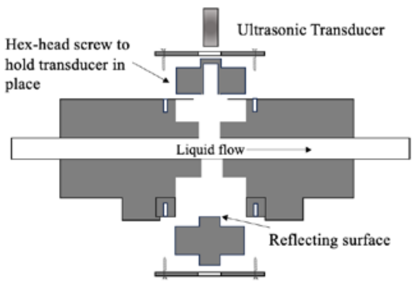


Fig. 7: A schematic of the speed of sound apparatus, reproduced by the author based on the setup by Meyer et al. [65]

Andrade et al63) measured speed of sound using time-of-flight method, transducers were immersed in the liquid, thus reducing any additional uncertainty related to fluid path length in calculations as shown in Figure 8. Moreover, the oil refrigerant mixture was formed for specific composition based on their masses. This reduces any uncertainty which can arise in due to excess volume because of non-ideal mixing of lubricant and refrigerant. Neither accuracy of speed of sound measurement nor information about lubricant is provided by the author, which makes it difficult to discuss the overall accuracy.

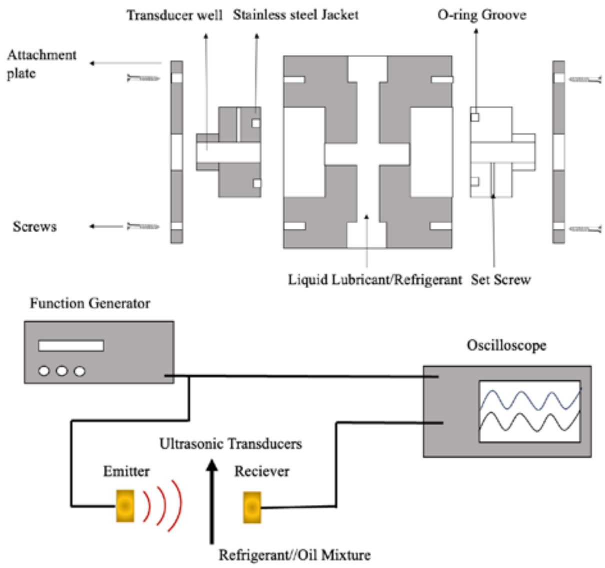


Fig. 8: A conceptual schematic, reproduced by the author based on the setup used by Andrade et al. [63)]

Lebreton et al[66)], investigated online prediction of oil concentration in oil refrigerant mixture using speed of sound. The experimental setup is shown in Fig. 9. Pulse echo technique was employed, and effect of flow rate of liquid was assumed to be negligible. LeBreton assumed that density of oil and refrigerant mixture can be calculated accurately by ideal mixing rule. With this assumption, oil concentration x was calculated from the total volume of calibration bench $V_{mix}$to make specific composition of oil refrigerant mixture as follows,

$$V_{mix} = \frac{m_{m,l}}{\rho_{m,l}} + \frac{m_{r,v}}{\rho_v} \qquad (2)$$

where,

$$\boldsymbol{m_{m,l} = \frac{m_{r,l}}{1 - x_{oil}}} \qquad (18)$$

where $\rho_{m,l}$, ρ_v , $m_{m,l}$ and $m_{r,l}$ are the density of the mixture, the density of refrigerant, mass of the mixture and mass of liquid refrigerant, respectively.

Later, theoretical correlation between the oil concentration and speed of sound in the mixture was developed. This creates a possible error in data provided by Lebreton, since it is evident from literature that oil refrigerant mixtures do not follow ideal mixing rule for density[69)]. Calibration using sampling technique also showed a difference of 1.7 mass% oil concentration between intended oil ratio and actual oil ratio in oil refrigerant mixture.

Lebreton work can be improved by better estimation of mixture density. Furthermore, the correlation provided by Baustian for oil concentration prediction was used. This correlation contains oil concentration in squared term which makes oil prediction invalid in case of negative value from the expression. Koji et al68), and Fukuta et al64), also used an ultrasonic sensor based on time-of-flight measurement technique as shown in Figure 10, however, effect of pressure on speed of sound measurement was not considered in the prediction of oil concentration.

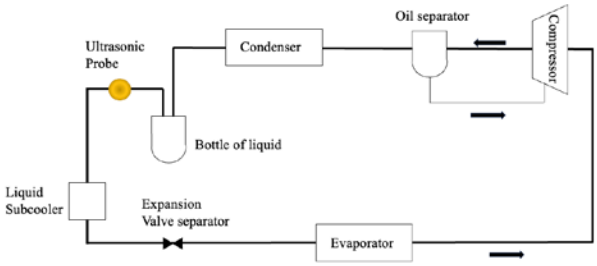


Fig. 9: A schematic of the experimental setup reproduced by the author based on the setup used by Lebreton et al. [66)]

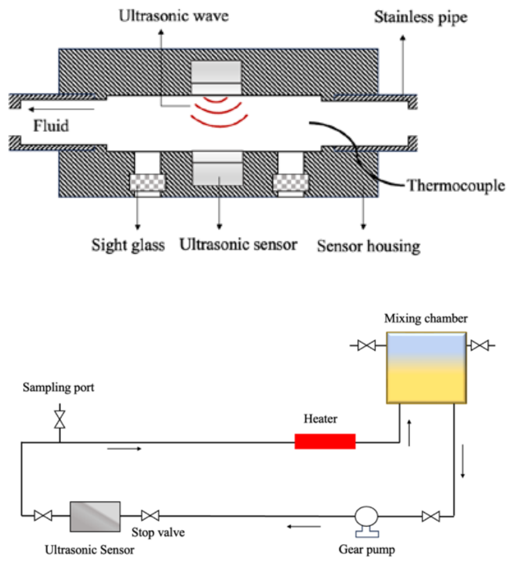


Fig. 10: A schematic of the experimental setup, redrawn by the author based on the setup used by Fukuta et al. [64)]

In addition, the overall accuracy for speed of sound measurements or oil prediction is not provided. Nevertheless, author concludes that higher accuracy setup needs to be developed for accurate prediction of oil concentration. Ultrasonic measurement was inaccurate in the presence of bubbles, which is a limitation of this setup. Lei Gao70) used a speed of sound sensor to measure oil circulation ratio in refrigerant lubricant mixtures. He tested R-1234yf with PAG oil for 0 to 3 masses % concentration of oil. It was concluded that the speed of sound in the mixture was dependent not only on the oil concentration in the mixture but also on temperature and pressure. Speed of sound was measured in subcooled region. Oil circulation ratio was estimated using droplet and volumetric method by installing sight glass after evaporator. No empirical or theoretical relation for prediction was provided by the author and only a general approximation of 1 m/s increase in speed of sound for 1 mass % increase in OCR was mentioned. The author did not specify the sensor utilized or its working. Due to which, the overall accuracy determination is difficult. Furthermore, since R-1234yf is not miscible with PAG oil in all conditions, it is important to properly mix the fluid mixture before speed of sound measurement as immiscibility can cause error and discrepancy. This is because of the presence of larger oil droplets which disturb the sensor measurement. This work can be further extended to develop a correlation between oil concentration in R-1234yf. The miscibility of R-1234yf with different lubricants have been readily investigated in recent years15). This information can improve the accuracy of speed of sound measurements with the utilization of more precise acoustic sensor and mixing device. One additional approach can be to use conventional method of time of flight or dual path pulse echo for speed of sound measurement at known oil concentration in oil refrigerant mixture.

While these techniques are mainly used for pure liquids, they can be extended to these mixtures. Once speed of sound, temperature and pressure data is obtained, a relation between speed of sound and oil concentration can be developed. This relation can be used for the prediction of speed of sound in mixtures using oil concentration and vice versa using theoretical models as explained in next section. Yvon Goth41) presented the three-transducer array method for measurements of speed of sound in oil refrigerant mixtures as shown in Figure 11. The main theme of experiment is to relate the variation in speed of sound of refrigerant lubricant mixture with the operation of oil separator. It was assumed that variation in speed of sound was purely dependent on oil concentration in the mixture (shown in Figure 2). Although, it was concluded that this method can help in detection of up to 0.5 % variation in oil content, several limitations are presented.

Speed of sound depends on temperature, but the accuracy of temperature sensors is not described, making it difficult to estimate the accuracy of experimental results. Moreover, in oil prediction experiments, since many factors can impact an accurate prediction, it is very important to calibrate the system using sampling technique (or any other technique). However, no additional calibration was performed in this case which significantly reduces experiment accuracy. Furthermore, a general variation in speed of sound of mixture in comparison to experimental speed of sound with oil concentration change was discussed, but no empirical or theoretical correlation was provided.

Table 4: Overview of speed of sound measurement techniques in oil/refrigerant mixtures

Mixtures	Summary	Comments
R-12/naphthenic oil. R-22/naphthenic oil, R-502/AB[67]	Sensor: Pulse Echo Temperature Range: 25 - 49 ℃ Uncertainty*: 1 wt.% (for 0-30wt%)	Transducers were used outside the recommended temperature range. Pressure Effect on speed of sound measurement was not considered.
R-12 with 2GS and 5GS oils R-134a/POE[65]	Sensor: Pulse Echo Temperature Range: -20 - 30 ℃ Uncertainty: 0.26 wt.% (0 to 10wt%)	Proper subcooling was not maintained and a pressure correction was provided.
R-22/AB[63]	Sensor: Time of flight Temperature Range: 16 - 41 ℃ Uncertainty: 5wt% (0 to 25%)	Speed of sound accuracy is not provided. Effect of pressure on speed of sound measurement is not considered.
R-410A/POE[66]	Sensor: Pulse echo method Temperature Range: 30-50℃ Uncertainty: 0.7wt% (0 to 15%) 0.5wt% (for 0 to 5% oil conc)	Baustian correlation was used for oil concentration prediction without any further improvements. Error in charging oil compositions.
R-410A/POE[41]	Sensor: Transducer array Temperature Range: 19-23.5 ℃ Uncertainty: up to 0.5% variation can be detected.	Calibration was not performed, which compromises overall accuracy. Lack of any empirical or theoretical correlation for oil concentration prediction.
R-134a/PAG[71]	Sensor: Time of flight Accuracy: 0.5wt% Range: 0 to 7 wt.%	Theoretical correlation cannot be extended to other oil refrigerant mixtures since oil concentration was predicted using a correlation specific for the current mixture.

*Uncertainty includes the uncertainty due to prediction model, measurement device error and other experimental uncertainties. Range specifies the various oil composition in oil refrigerant mixtures for which testing was performed.

Cite: I. Sameen et al., "Measuring Lubricant Concentration in the Mixture with Refrigerant: A Comprehensive Review". Evergreen, 12 (02) 1106-1130 (2025). https://doi.org/10.5109/7363498.

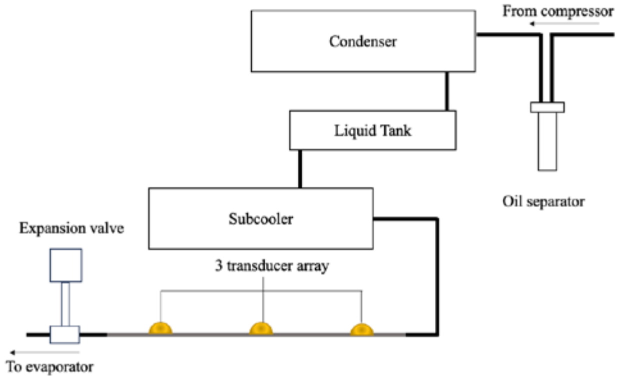


Fig. 11: Schematic reproduced by the author based on Three transducer array method [41)]

Xin et al.[72)], investigated the oil concentration in oil refrigerant mixtures before and after installing an oil-gas separator to know the effectiveness of an oil return system in refrigeration system. The experimental setup is a refrigeration cycle and a commercial speed of sound sensor installed after the subcooling to measure oil concentration. This sensor is based on time of light technique and has one emitter and receiver. The average relative deviation from oil concentration obtained by sampling is around 2.63%. This is lesser than relative deviation between oil concentration obtained by density method and sampling (4.28%)[73)], making speed of sound method more accurate. However, author didn't specify refrigerant lubricant pair used in the experiment,

or its temperature and pressure range which makes it difficult to further comment. Moreover, it is assumed that oil circulating in the system was constant and same as oil discharged from the compressor. This assumption does not consider possible oil retention in the system which can cause error in results and accuracy. The commercial sensor by Anton Parr GmbH was also used in research work by Knipper et al[71)] where the pressure drops characteristics for condensation of R-134a/PAG oil mixtures was investigated. In addition to the effect of channel shapes, the effect of oil concentration on pressure drop was studied. Oil concentration in oil refrigerant mixture was predicted using speed of sound, temperature and pressure of subcooled liquid and correlation provided by MAHLE Behr GmbH & Co KG and Anton Paar GmbH.

In conclusion, Speed of sound can be a promising approach for predicting oil concentration measurement since it is more accurate and reliable comparable to other available techniques. It has some limitation as it requires a homogenous mixture of oil and refrigerant for highly accurate measurement. Moreover, calibration is a tedious process and takes a long time. Special attention needs to be made for time-of-flight measurement and wave analysis since speed of sound is highly sensitive to time loss. Theoretical models generally available for liquids can be extended to refrigerant lubricant mixtures to get acceptable range of accuracy for oil concentration prediction.

3. Oil Composition Prediction Using Other Properties of Oil/Refrigerant Mixtures

3.1. Sampling

The standard approach of oil composition evaluation involves connecting an evacuated cylinder to the liquid line which is filled once a measurement is required. This cylinder is disconnected from the main setup and refrigerant is slowly allowed to evaporate. The remaining oil is measured. Additional amount of refrigerant and oil is charge into system to maintain overall concentration. Another sampling technique is to take only few milliliters of liquid from the system. This amount is reduced in comparison to standard sampling method by venting the sample to the compressor once its volume is measured. The remaining oil value is measured. Moreover, flush and measurement technique is also used for sampling. In this technique, a solvent or refrigerant is flushed through a section and the oil concentration is measured in the solvent. Additionally, a liquid level probe can be used to measure oil flow rate. This requires separating oil from the refrigerant prior to the measurement as shown in Figure 12. This approach is recommended as it can also measure oil circulation ratio (OCR) when oil and refrigerant are immiscible74,75). This is because oil is collected from suction line where it is separated from refrigerant vapor using a separator. Another technique is weight measuring method, which utilizes a weight measurement method bomb. A sample of mixture is taken from the liquid line and charged to the evacuated bomb. The refrigerant inside the bomb is removed and remaining oil is measured. This technique is useful for compressor manufacturers. Sampling technique involving oil separator is also used for concentration measurement. Oil is collected in a sight glass and concentration is measured under transient conditions without effecting refrigerant concentration in the system.

A benefit of sampling technique is convenience. However, it is not very reliable for online measurement and transient measurement of oil concentration in the system. Another disadvantage is that it reduces the overall refrigerant present in the system. Table 5 shows few sampling techniques.

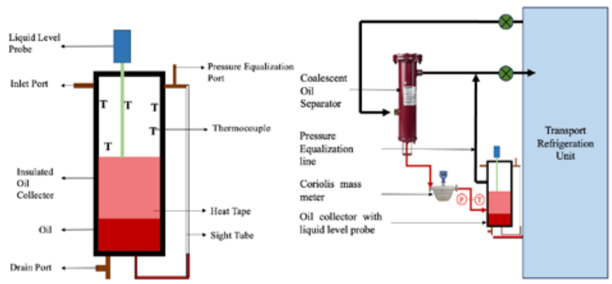


Fig. 12: A schematic of the oil collector, used for oil level measurement. B. OCR measurement loop

Table 5: Technique employed in sampling method for OCR measurement

Technique	Comments
ASHRAE[76]	low cost large sample taken
M C Govern[77]	low cost low amount of sample taken sensitive to low oil concentration
Flushing technique[76]	more uncertainty
Liquid Level Probe[74]	automated measurement with lesser human error
OSM and WMM[78]	Easy to install

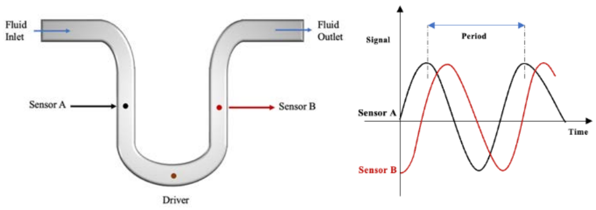
Sampling method is the common method for determining oil concentration in the mixture. However, since it cannot be carried out online, it is usually used to verify oil composition results obtained from other methods. Other than speed of sound, few other properties of oil refrigerant mixtures can be used for determining oil composition. These properties include density, capacitance, refractive index, thermal conductivity, and light absorption. Furthermore, some other techniques including visualization[79) 80)], viscosity[67] ,and optical methods[81] can also be used for oil composition prediction as mentioned in literature. However, these are outside the scope of this review paper.

3.2. Oil composition prediction in Oil Refrigerant Mixtures using density

Density is an important thermodynamic property of refrigerant lubricant mixtures. There is a significant difference in the density of pure refrigerant and lubricants as evident from data available in literature[39,67,82–84]. Due to this reason, change in the concentration of lubricant impacts the density of refrigerant lubricant mixture. This change in density with respect to lubricant concentration can be used to measure the lubricant concentration in the mixture.

A commonly used approach to measure the lubricant concentration is to utilize a densimeter based on Coriolis effect to measure density of the refrigerant lubricant mixture.

Coriolis effect is the basic principle of coriolis densimeter85). When a fluid is passed through a vibrating system, it experiences an inertial force which is perpendicular to the direction of motion, and it induces a curve in the fluid path. This is referred to as Coriolis effect. Such densimeter generally consists of a single or a double flow tube (either curved or straight depending on the design), which is oscillated using an electromagnetic or a mechanical drive system at their natural frequency. Various sensors are used to detect the phase shift and natural frequency of the vibrations at inlet and outlet of tubes of densimeter, as shown in Figure13.



(a) (b)

Fig. 13: (a) U-tube densimeter, (b) Sine waves generated by signals obtained with electromagnetic sensors

Modern Coriolis densimeters consist of two components: a flow sensor and a flow convertor. Flow sensor consists of flow tubes, drive system, motion sensors and supporting structure. An electromechanical driver connected to the center of tubes is used to excite the tubes at a resonant frequency of the tubes. In addition, Motion sensors are attached at the inlet and outlet sides of tubes, which convert motion of tubes to the electric signals (sinusoidal waves). These sensors can be magnetic pickup coil sensors, capacitive sensors or any other type depending on specific design and requirements. Due to Coriolis effect, the fluid mass induces a phase difference in the oscillations at different points of the tubes. Moreover, the resonant frequency of the oscillation also changes. The vibrations of the tubes are read in the form of sine waves and these sine waves are analyzed to see the phase difference and change in the resonant frequency of the oscillations. Phase shift in oscillations is directly proportional to mass flow rate of fluid flowing through the tubes and allows accurate measurement of mass flow rate. It ranges from nanoseconds to microseconds. The resonant frequency depends on the stiffness and mass of the tubes and the fluid flowing through tube, making it easier to calculate fluid density.

An electronic control system provides excitation to the drive system and is responsible for maintaining the tube oscillation at a constant amplitude by continuously tracking its resonant frequency. In addition, it processes signals from the flow sensor and calculates mass flow rate and density using phase difference and resonant frequency respectively. This electronic control system is also referred to as flow convertor.

Moving forward, the fundamental density calculations which are being performed inside flow convertors of Coriolis densimeter are explained. As we know, the density of the fluid can be obtained mainly by the natural frequency of the oscillating tube filled with the tested fluid. Since the natural frequency of tube decreases with the increase in mass flow rate of fluid flowing through the tube, it is important to consider the effect of mass flow rate of the fluid. In addition, changes in temperature also impact the density estimation as they change material properties of vibrating tubes[86]. The vibrating tube can be considered as a mass spring system vibrating at resonant frequency denoted as f:

$$\boldsymbol{2\pi f} = \sqrt{\frac{\boldsymbol{K}}{\boldsymbol{M}}} \quad (19)$$

where K is spring constant of system and M is mass of system, including the mass of fluid and tube. Mass of fluid can be written in terms of density and volume as follows:

$$\boldsymbol{m_{fluid}} = (\boldsymbol{D_m} * \boldsymbol{V}) \quad (20)$$

Substituting the value for m_{fluid} and rearranging:

$$\boldsymbol{D_m} = \frac{\boldsymbol{K}}{\boldsymbol{4\pi^2 V}}\boldsymbol{T^2} - \frac{\boldsymbol{m_{tube}}}{\boldsymbol{V}} \quad (21)$$

where T is the period of tube oscillations. Since, spring constant is dependent on temperature, calibrations are performed using air and water. Various corrections are introduced in density measurement. Generally commercial Coriolis meter have density calculation as follows:

$$\boldsymbol{D_m} = \boldsymbol{K_2 T_m^2}(\boldsymbol{1} - \boldsymbol{t_c t_m}) - \boldsymbol{K_1} \quad (22)$$

where, K_1, K_2 are given as follows:

$$\boldsymbol{K_1} = \boldsymbol{K_2 T_a^2} - \boldsymbol{D_a} \quad (23)$$

$$K_2 = (Dw - D_a)/(T_w^2 - T_a^2) \quad (24)$$

Where, T_m, T_a and T_w is the tube period in seconds for testing fluid, air, and water respectively. T_a and T_w are obtained using air and water as calibration fluids in no flow conditions and corrected to 0 ºC. This is done to eliminate any additional complexities introduced by mass flow rate. Temperature compensation factor t_c is in % and is obtained by testing tube period at multiple temperatures. Since this is obtained for specific calibration fluid, further correction is introduced. D_c indicates the density of fluid with mass flow rate and temperature corrections, initially presented by Buttler et al.[86],

$$\boldsymbol{D_c} = \boldsymbol{D_m} - \boldsymbol{K_3}\left(\boldsymbol{1} + \boldsymbol{K_4}(\boldsymbol{t_m} - \boldsymbol{t_{K_3}})\right)(\boldsymbol{M_m})^{\boldsymbol{2}} \quad (25)$$

where, M_m is the measured mass flow rate and t_m is the measured temperature. The correction factors K_3 and K_4 account for the density errors observed during calibration mainly due to change in mass flow rate and temperature of the fluid.

$$\boldsymbol{K_3} = \boldsymbol{D_{K_3}}/(\boldsymbol{M_{K_3}^2}) \quad (26)$$

$$K_4 = \left[\frac{D_{K_4}}{K_3 \times M_{K_4}^2} - 1\right]/(t_{K_4} - t_{K_3}) \quad (27)$$

D_{K_3} and D_{K_4} are the error is measured density at temperatures t_{K_3} , t_{K_4} and mass flow rate M_{K_3}, M_{K_4} respectively. K_3 introduces the correction for mass flow rate of fluid and K_4 introduces correction for temperature variation respectively. Additional developments introduced elimination of density-mass flow rate dependency[87].

There are certain uncertainties involved in the measurement mainly due fluid properties, temperature, pressure, and external conditions. Since water/air is mainly used as calibration fluids, in scenarios where the tested fluid has deviation from water properties, the error in density measurement is inevitable and it is important to compensate for these errors[88]. Changes in the temperature and pressure system can affect Young's Modulus of the flow tube, which ultimately impacts the tube period[88]. Young's Modulus is related to tube dimensions as follows:

$$\boldsymbol{E} = \frac{\boldsymbol{FL_o}}{\boldsymbol{A}(\boldsymbol{L_n} - \boldsymbol{L_o})} \quad (28)$$

where E, is Young's Modulus, F is the applied force at each end of material, L_o and L_n denotes original and new length respectively and A is cross sectional area of material. This results in the variation in tube stiffness and phase shift of the coriolis densimeter. Moreover, changes in surrounding air temperature of densimeter also induce additional errors in the density measurements[89]. Since, the properties of refrigerant and oil mixtures can have significant deviation from calibration fluid properties such as water, uncertainties are introduced. Furthermore, a temperature sensor is installed within a Coriolis meter, which allows proper temperature correction. However, this sensor(s) is attached on the flow meter and not in direct contact with the fluid. This can cause additional uncertainty in measurements if fluid temperature fluctuates rapidly due to lag in temperature measurement[90]. Usually there is no sensor attached for the fluid pressure compensation and user must provide pressure measurement for online correction. This is achieved using calibration at multiple pressure conditions[90,91]. A detailed description of the impact of various external factors on the density measurement has been provided by Cheesewright et al.[92]. Other uncertainties involve uncertainties due to corrosion and any external vibrations which might impact the Coriolis densimeter. Single tube densimeter are more prone to external vibrations compared to double tube densimeter, hence double tube densimeter are preferred for higher accuracy[93]. Coriolis meters have various correction factors information stored in flow convertors. It is provided by the manufacturer and densimeter shows the output density with all these corrections. An important consideration in Coriolis densimeters is the shift in zero-point, which is the baseline frequency measurement when there is no flow and mass in the tubes. Coriolis meters are tested and zero error in the device is considered while performing density measurements. Often an additional in-house calibration is performed before using the densimeter to account for any error. A rigid housing is used to protect the densimeter from any external influence.

3.2.1. Density measurement in Oil Refrigerant Mixtures

To use the densimeter as an oil concentration sensor, a flow loop simulating a refrigerant liquid line is designed where proper subcooling is ensured by controlling pressure and temperature to avoid any gas in the densimeter. Pressure is usually controlled by a pump, which pumps fluid at a specific discharge pressure. Temperature and pressure sensors are also installed in the system as shown in Figure 14. The Coriolis meter is attached vertically to the system and flow direction is upwards. This orientation allows proper draining of tube from any possible gas and solid impurities when liquid is not flowing. Once, density is measured at varying oil concentrations, a theoretical relation between the density of mixture and oil concentration is developed. It mostly involves fitting constants which are obtained via experimental data. Later, these theoretical relations are used to predict oil concentration in oil refrigerant mixture. In some cases, ideal mixing rule/Jensen equation is also used to predict oil concentration in the mixture however significant errors are observed.

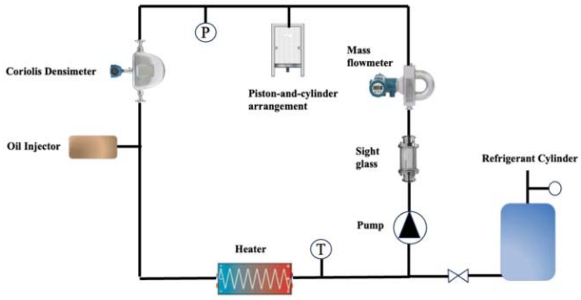


Fig. 14: A schematic diagram of the general experimental setup used in density measurement

Baustian[67] conducted density measurements using a commercial densimeter and developed a correlation for oil concentration and mixture density. A pump and a pressure vessel were used to maintain the overall pressure in the system. Refrigerant is weighed and charged into this vessel and using the pump, it is circulated through the system. Small quantities of oil are injected through hydraulic cylinder which is also later used for sampling. Various temperature and pressure sensor allows temperature and pressure measurements. Densimeter was calibrated using air and pure refrigerant, which provides additional benefits over calibrating only with water. Calibration with refrigerant provides more accurate estimation for the measurement of refrigerant lubricant mixtures. The correlation developed is as follow:

$$\boldsymbol{SG = b_o + b_1 c + b_2\theta + b_3 c^2 + b_4 c\theta + b_5\theta^2 + b_6 c^2\theta + b_7 c\theta^2 + b_8 c^2\theta^2} \qquad (29)$$

where $\theta = T/T_{ref}$ and T_{ref} is 529.67K. Deviation from Jensen equation was up to 6% in terms of mixture density. Although Baustian provided data with claimed accuracy of 0.01 specific gravity units, there are several additional uncertainties involved. Since, the equation provides oil concentration in terms of square, if both roots are positive, this equation can't be used to infer oil concentration.

Pressure is controlled using positive displacement pump and simple Marsh gauges were used to read pressure on the test loop which induces uncertainty in pressure. Furthermore, Baustian assumed that the natural frequency of Coriolis densimeter was only related to density of fluid flowing through it. This assumption neglects the effect of mass flow rate on density measurement. The temperature measurements obtained using densimeter were also different (slightly lesser) than those of test section. Since density is significantly dependent on temperature, this discrepancy can cause additional error. Another limitation in Baustian work is that it was assumed that oil quantity/mass in the oil refrigerant mixture flowing through the system is same as the injected amount of oil. However, since solubility is affected by temperature leading to oil retention in various parts of system, this assumption can be a source of error as it can affect the concentration of the flowing oil.

Bayini et al.[94] setup is based on a straight vibrating tube type densimeter used to measure density. The subcooled oil refrigerant mixture is pumped in the system through stainless steel pump which is magnetically driven. Accuracy of mixture composition was confirmed by comparing injected amount with the oil concentration obtained from ASHRAE sampling method which has a mass accuracy of 0.01g. Desired pressure is maintained in the system by flowing oil refrigerant mixture through a piston and a cylinder arrangement with nitrogen at the back of it. It allows properly subcooled mixture to pass through densimeter. Bayini introduced compressibility as another parameter impacting density of the oil refrigerant mixture as mentioned in the equation, however for 0 to 6wt% oil concentrations, compressibility was considered as same of pure refrigerant, which can be inaccurate.

$$\boldsymbol{w_{oil} = b_1 + b_2T + b_3D + b_4DT + b_5DT^2 + b_6D^2T + b_7D^2T^2 + b_8T^2 + b_9D^2} \quad (30)$$

where b is an empirical constant, T is temperature in ^oC and D is the saturation density in kg/m^3. D is the measured compressed density minus the density difference due to compressibility at the same temperature. This work accounted for the effect of mass flow rate variation on densimeter making it accurate compared to earlier research. Bayini used densimeter which also accounted for effect of nonlinear temperature on the measurement making it more accurate compared to Baustian. Additional flow mixers are installed in inlet and after outlet pipes which allow proper mixing of fluids. Four different thermocouples are attached to the system allowing a better/ an improved temperature measurement accuracy.

Although Bayini improved the theoretical correlation, however, for pure refrigerant saturation density, he merged calibration of Coriolis meter with the curve fitting of the EoS. This work can be further improved by choosing more accurate values of pure fluid density. Since Bayini used a straight tube densimeter, these densimeters are more sensitive to mass flow rate compared to U-tube densimeters, causing additional uncertainty in the measurement.

Later, Chang Nyeun Kim et al.[95] proposed a correlation for oil concentration prediction. This correlation was also based on fitting coefficients obtained from experimental data [95].

$$\boldsymbol{C = a + b \times t + c \times t^2 + (d + e \times t + f \times t^2) \times SG} \quad (31)$$

where SG is the specific gravity in g/cc, t is temperature in ℃ and C is oil concentration in wt.%. a, b, c, d, e, f are fitting parameters for the specific mixtures tested i.e., R-12/ VG 68 Naphthenic Oil and R-134a/ VG 68 POE Oil. The pressure accuracy is 1kPa, whereas it is 0.1 ^oC for temperature. Nevertheless, the overall inaccuracy is not mentioned. Refrigerant is initially added to the system by an accuracy of 0.1 grams. Initially pure refrigerant is circulated, and later small quantities of oil are injected. To ensure the correct oil refrigerant mixture compositions, once data for each composition at various temperatures and pressure is obtained, sampling technique is used to analyze the mixture composition. Overall system is calibrated using pure refrigerant for combined error due to densimeter, thermometer and pressure gauge. Pressure is maintained using a pressure vessel and temperature using isothermal bath. While Baustian considered the injected oil amount to be same as flowing amount, Kim et al.[95] work provides better accuracy by retesting the mixture composition. The author did not mention overall accuracy of oil concentration prediction which makes it difficult to compare with other work. While densimeter is calibrated using water and air, it isn't calibrated with refrigerant. Since, refrigerant lubricant mixture deviates from water behavior, it can induce error in the measurement. Moreover, only overall system calibration was performed using pure refrigerant which makes it difficult to account for error caused by densimeter. Oil prediction using Coriolis densimeter can also be applied to immiscible combinations of oil and refrigerants if it is assumed that the liquid phase of both oil and refrigerant travel homogenously in the liquid line. Wujek et al.[96] developed a theoretical equation to predict oil circulation rate for an automotive air conditioning system. Density was calculated using Coriolis meter by applying 1.8 MPa pressure (by compressing hydraulic cylinder using nitrogen gas), at test temperature. The pressure was gradually reduced by releasing nitrogen gas until bubble point was reached. Same process was repeated at all temperatures up to 45^{o}C. Flow rate of working fluid was adjusted to 30g/s and temperature was controlled by placing experimental setup in an environmental chamber. Measured oil concentration with an accuracy of 0.001 (verified using ASHRAE sampling technique) was gradually added in the system using high pressure liquid chromatography pump and data points were taken. Refrigerant was also added gravimetrically using charging port with accuracy within 0.2 grams. Temperature and pressure accuracy are 0.3C and 10.5kPa. The following equation was developed to obtain OCR,

$$\boldsymbol{OCR = A + BT + CT^2 + D(\Delta\rho) + E(\Delta\rho)^2 + FT(\Delta\rho)} \quad (32)$$

where A, B, C, D, E, F are fitting coefficients specifically obtained for this experiment, ρ is difference of density of mixture from pure refrigerant density, and T is the temperature respectively. It is important to note that the volume of coriolis meter used in the experimental setup was lesser than the actual internal volume of the automotive liquid line. To properly simulate the automotive air-conditioning system with actual refrigerant charge, volume needs to be modified. Moreover, since the difference between densities of pure oil and refrigerant becomes lesser above 30 ^oC, the error in the prediction significantly increases. Although, above relation provides a better approximation compared to the ideal mixing rule, however it is only limited for 20 – 30 ^oC while desired range is usually 20 – 45 ^oC. Further corrections for higher temperatures are required.

Under steady conditions, individual component of refrigeration system i.e., compressor, condenser etc. is considered to have a constant value of oil retention. In this case, oil discharge rate from the compressor can be assumed to be the same as the oil circulation rate in system.

Based on this assumption, Oil Discharge Rate (ODR) from compressor is obtained by determining oil concentration in oil refrigerant mixture in the subcooled region of the system. An approach to measure oil discharge rate from compressor [73)] is to use a U-tube Coriolis densimeter for determining the density of oil refrigerant mixtures. This setup used actual compressors at three test operating conditions. Tests were conducted after charging the exact amount of R-410A and POE oil into the compressor. Experimental setup is based on refrigeration cycle and Coriolis meter is connected in the subcooled region where both oil and refrigerant are in liquid phase. Once the density of mixture is measured, Jensen equation[97)] is used to predict the oil discharge rate:

$$\boldsymbol{x_{oil}} = \left(\frac{\boldsymbol{\rho_{ref}}}{\boldsymbol{\rho_{mix}}} - \mathbf{1}\right)\left[\frac{\mathbf{1}}{\left(\frac{\boldsymbol{\rho_{ref}}}{\boldsymbol{\rho_{oil}}} - \mathbf{1}\right)}\right] \qquad (33)$$

The above equation provides a higher accuracy at low oil concentrations for this system and vice versa. Compared to literature, the error in prediction is about 0.63%. Both sampling technique and light absorption technique were also used in the subcooled region, to validate and compare the density measured using densimeter. Oil prediction using density measurement is accurate as compared to light absorption method in this case. There is a possibility that oil retention value might change with temperature or other factors[98)]. This can result in a difference between oil discharge rate from the compressor and oil circulation ratio obtained in the sub cool region, causing an error. In addition, no details regarding pressure and temperature sensors are provided which makes it difficult to analyze overall accuracy. Average relative deviation is 4.28% which makes it less accurate than sampling technique employed in the same setup. Calibration is only performed using water. It is usually recommended to calibrate the densimeter with similar fluid as testing fluid to ensure proper calibration. Jensen equation is used for mixture density. This equation is only suitable for limited mixtures with less overall accuracy. Developing a novel correlation for relating density, temperature and oil concentration can be a significant improvement.

Since the densimeter method is based on significant difference between lubricant and refrigerant density, it cannot be accurately used once this difference becomes smaller e.g., in case of heat pump cycles, the liquid temperatures are between 20 ºC and 50 ºC, and density difference between lubricant and refrigerants e.g., R-134a and R-407 becomes insignificant. This makes it a less accurate method [99)]. Furthermore, there is no theoretical relation found in literature which can be used for all mixtures. Mostly, empirical correlations which needs experimental data are developed. This method can be significantly improved if a theoretical relation more accurate than Jensen Equation is proposed.

Coriolis basis densimeters provides an ideal choice for simplicity and accuracy. They are also easily installed and offer in-line measurement. In addition, since they don't have moving parts except the tube vibration, there is no wear down over time. In addition to density, they can also measure mass flow rate, viscosity and temperature if required, which makes them versatile. However, they are quite expensive. Moreover, depending on the line size, the physical size of Coriolis densimeter increases which makes it difficult to handle.

Recently, more accurate densimeters have been provided, which can lead to better prediction of oil concentration in oil refrigerant mixtures. It should also be noted that the oil circulation rate/ratio is ratio of mass flow rate of oil to mass flowrate of oil and refrigerant flowing through system. It is not equal to local concentration of oil (oil concentration at specific point) or overall oil concentration in the system. Table 6 shows various mixture tested using densimeter and accuracy for oil concentration prediction. Most published data is based on refrigerants that have been already phased out due to high global warming potential. Recently, new low GWP refrigerants have been introduced which is beneficial not only for oil concentration prediction but for analyzing overall efficiency of the system.

3.3. Capacitance Basis Method

Dielectric constants and relative permittivity for oil refrigerant mixture can be related to oil concentration in the mixture. The basic principle of this measurement method is based on the considerable difference between dielectric constant of pure refrigerant and oil/refrigerant mixture. Sensors are used to measure changing dielectric constant with varying oil concentration. An extensive literature review shows mainly three types of sensors: cylinder type, needle type and parallel plate electrodes sensor. Baustian[67)] provided a preliminary oil concentration measurement in R-113/Naphthenic Oil using a capacitance sensor. However, capacitance-based method was not extensively explored for oil concentration measurement. This is due the fact that slight impurities in mixture and ionization of oil can produce great error. Later, Fukuta[100)] used following relation to predict oil concentration from capacitance measurement in various mixtures, where A is a fitting parameter, x is oil composition and Ɛ is relative permittivity:

$$\varepsilon_{\boldsymbol{mix}} = \varepsilon_{\boldsymbol{oil}}(\boldsymbol{x}) + \varepsilon_{\boldsymbol{ref}}(\mathbf{1}-\boldsymbol{x})\boldsymbol{x} + \boldsymbol{A}(\mathbf{1}-\boldsymbol{x})\boldsymbol{x} \qquad (34)$$

A capacitance sensor was also developed, and while its accuracy/working is not provided by the author, it is claimed to work accurately even during bubbles presence. It is important to note that since mixed refrigerants have almost similar dielectric constant and pure oil, it can be difficult to predict composition in some mixtures. Pierre101), also tested various theoretical models for

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relating the mass fraction with the relative permittivity and found above linear mixing relation to be the best. A few studies for oil concentration measurement in CO2/oil systems were found in the literature. Yunho Hwang et al.102) used a capacitance sensor as shown in Figure 15 to measure oil concentration in CO2/PAG mixture. A polynomial fitting equation relating oil mass fraction to dielectric constant, and the reduced density of CO2 was provided.

A major limitation is that the experimental data for specific mixtures is required to predict oil composition. Moreover, dielectric constant is affected by small impurities in the mixture and extra care needs to be taken. Furthermore, the difference in dielectric constant of oil and refrigerant is usually significant however in case of refrigerant blends it is not true. The difference in dielectric constant of refrigerant blends and oil is less significant which makes this method less accurate.

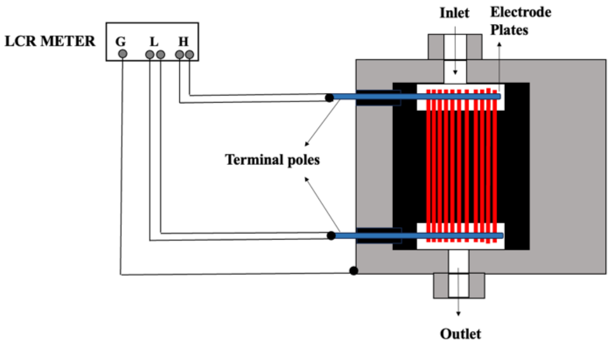

Fig. 15: Schematic of Capacitance sensor[102)] for lubricant/ refrigerant mixture, reproduced by the author

Table 6: Methods for determining oil composition in oil/refrigerant mixtures

Method	Mixture	Sensor	Maximum Overall Accuracy	Comments
Density based method	R-113 and R-12 with VG 32 naphthenic oil, R-502/ 150 SUS alkyl benzene oil[67)]	MicroMotion DT7 attached with D25mass flow meter. Accuracy: ±0.01 sg	+/-1 wt. % ≥ +/-2 wt. % ≥+/-2 wt. % 0 – 30 wt.% 21 - 48.8 ℃	It is assumed that the mass flow rate has no effect on density measurement. Effect of oil retention was not considered. Oil composition prediction needs experimental data.
	R-134a /POE[94)]	Straight vibrating tube densimeter with an accuracy of 0.1 kg/m^3	0.09 wt. % 0 to 6 wt.% -9.4 - 5.9 ℃	Straight tube densimeter is used which has more uncertainties compared to a U tube densimeter. Oil composition prediction needs experimental data.
	R-134a/ 46 cSt PAG oil[96)]	U tube CMF-25 connected to Micromotion RFT-9739 transmitter. With an accuracy of 0.2 kg/m^3	Standard deviation of 0.004 up to 30 ℃ 0-16%oil by mass for the range 20 – 45 ℃	The error in prediction significantly increases with temperature. Oil composition prediction needs experimental data.
	R-410A/POE[73)]	U-tube densimeter with an accuracy of 0.2 kg/ m^3	Standard deviation of 0.004 up to 30 ℃. 0-16%oil by mass. 20 - 45℃	Jensen equation is used for the mixture density which has less accuracy in case of oil refrigerant mixtures.

Capacitance based Method	R-22 / MO (MO, VG32, VG56, VG10), R-134a /ester oil (POE, VG56), ether oil (PVE, VG68), and polyalkylene glycol oil (PAG, VG56), R-410A / POE and PVE[100]	Commercial Variable condenser type sensor Maximum capacitance: 50 pF	0.63 wt.% (Overall) 60 to 85%oil by mass. -10 - 60 ℃	Experimental data is required for oil composition prediction. Impurities can create huge uncertainties. Some oil and Refrigerant blends don't have a significant difference in dielectric constant. This leads to difficulty in the prediction of oil composition.
	CO_2/PAG[102]	Capacitance Sensor combined with LCR meter. Measuring Frequency: 100kHz	0.5 mass % 0 to 7 masses% oil 20-50℃	
Refractive Index based method	R-134a, R-32, R-125, R-410A and R-600a, with PAG, PVE and a paraffinic mineral oil.[103] R-410A, its components, R-134a and R-600a. (PVE) for R-410A, R-32 and R-125, (PAG) for R-134a and paraffinic mineral oil for R-600a.[104]	Laser displacement sensor Resolution: 1 μm	0.2wt % 0 to 1 wt.% 30-50 ℃ 0.2wt % with 0 to 100% ref conc 30 – 50 ℃	Existence of bubbles can disrupt sensor functioning. Ideal volumetric mixing is assumed to develop theoretical correlation which can be inaccurate for oil refrigerant mixtures. Accuracy is compromised due to several factors including the effect of temperature and pressure
Thermal Conductivity based method	R-410/PVE oil[105]	Micro Heater (based on 3ω method) Resistance: 8.44Ω (at 30 ℃	Frequency scanning method: 5.8 wt.% 7.3 wt.% in SFM 0-1 mass fraction 30-60 ℃	Effect of liquid flow on oil concentration is neglected. Can be used only for measurement in small fluid quantities.
Light absorption-based Method	R-134a/Mobil 68 (R-407C/Castrol SW32[106]	JASCO UV Oil concentration meter	0.1wt % 16.5°C to 18.8°C	Oil composition prediction is Heavily dependent on UV absorbance level in specific refrigerant lubricant mixtures and cannot work for all refrigerant lubricant mixtures

3.4. Refractive Index

The difference in refractive index of oil and refrigerant is large enough to detect oil concentration as refractive index of mixture changes linearly with the mixing ratio. Moreover, refractive index measurement can also be taken at high pressure without any additional error.

Theoretical relation suggested by Fukuta[104] between refractive index and oil ratio is as follow:

$$\boldsymbol{n_{mix}} = \boldsymbol{\varphi n_{ref}} + (\mathbf{1} - \boldsymbol{\varphi})\boldsymbol{n_{oil}} + \boldsymbol{c\varphi}(\mathbf{1} - \boldsymbol{\varphi}) \quad (35)$$

where φ is the volume fraction and c determine the curvature of the correlation curve as a function of temperature. This relation is mainly used for the prediction of oil composition in the mixture. However, since this relation is based on ideal volumetric mixing, it can cause some additional uncertainties. By making correction for oil refrigerant mixtures, this relation can be further improved. Several authors including Newell[107] and Bastian[67] provided some preliminary experimentation for refractive index measurement in refrigerant lubricant mixtures using different types of refractive index sensors. However, in case of Bastian due to several limitation mainly thermal sensitivity of the sensors, no progress was made.

Fukuta103,104,108) employed a novel technique for the refractive index measurement in several refrigerant lubricant mixtures. The basic idea is to use a laser displacement sensor to detect the change of optical path. Refrigerant lubricant mixture is placed in a higher-pressure chamber with a glass window. The incident light is passed through the air and glass interface and glass and test medium interface respectively. Depending on the refractive index of testing fluid, the optical path changes due to refraction, and this change is detected by the sensor. Main principle of refractive index sensor is showed in Figure 16.

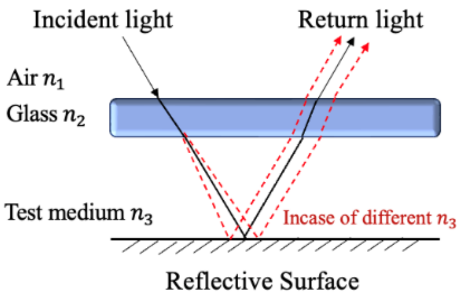

Fig. 16: A schematic representing the basic principle of the refractive index method

The experimental unit is shown in Figure 17. While flow rate of mixture and oil degradation had almost no effect on oil composition prediction, presence of bubbles disrupt the sensor functioning and hence limitation in this method. While accuracy up to 1% oil composition is achieved, it couldn't be further improved because of several factors including temperature and pressure. More accurate sensors with better calibration can be developed for accurate prediction. Later, Yoon et al, also used a correlation provided by Fukuta to calculate the oil discharge rate from the compressor105). Refractive index method can be more useful than density-based prediction since densimeter is affected by flow rate of mixture leading to additional uncertainties.

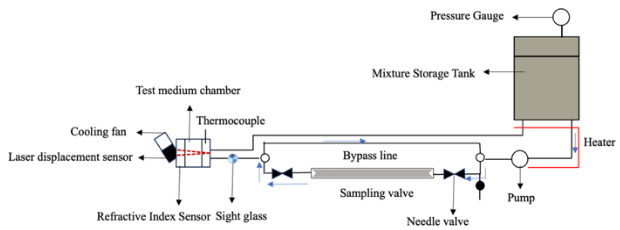

Fig. 17: A schematic of the refractive index sensor unit

3.5. Thermal Conductivity

Thermal conductivity of refrigerant lubricant mixture can be used to measure oil concentration. A detail discussion on measurement of thermal conductivity in liquids can be found elsewhere109). Limited literature was found for oil composition prediction using thermal conductivity of oil/refrigerant mixtures. One approach is to develop a micro thermal sensor110) and adopting either frequency scanning method or single frequency method. Sensor consists of metallic strip which is deposited on a glass surface as shown in Figure 18. In the frequency scanning method, voltage signals at different heating frequencies are used to measure thermal conductivity and oil concentration is measured by the sampling method. A relation between thermal conductivity and oil concentration is obtained. Another technique is the single frequency method which involves using relative change of sensor signal at certain heating frequency to detect oil concentration.

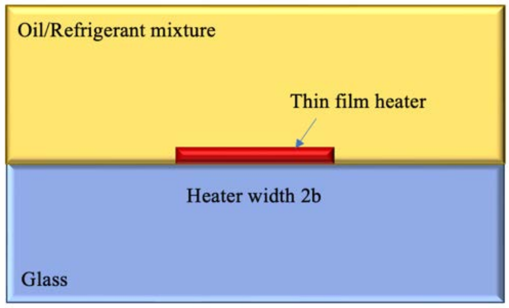

Fig. 18: A schematic of the micro thermal sensor

A common setup used to measure thermal conductivity is shown in Figure 19. This method is not widely used for oil composition prediction. Effect of liquid flow on oil concentration is neglected throughout the measurement. Furthermore, small temperature changes can affect the accuracy of thermal conductivity measurement which can cause additional uncertainties in oil prediction. In addition, the micro thermal sensor can be used only for small quantities of the mixture.

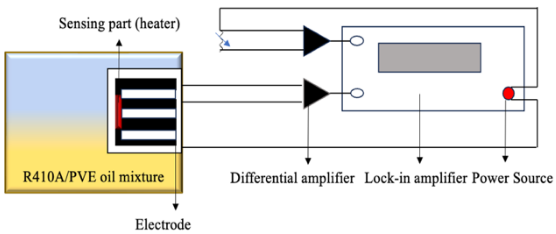

Fig. 19: Thermal conductivity measurement in oil refrigerant mixture

3.6. Light absorption method

Oil circulating with refrigerant illustrates a unique behavior of absorbing UV light in comparison to pure refrigerant[99)], which shows no absorption. This behavior can assist in predicting oil concentration in the mixture. Lambert-Beer[111)] gave the following theoretical approximation for oil concentration,

$$\boldsymbol{A}^* = \log\left(\frac{\boldsymbol{I}_i}{\boldsymbol{I}_t}\right) = \in \boldsymbol{cL} \qquad (36)$$

where A* is the absorbance. I_o is the intensity of incident light, and I is the intensity of transmitted light. ∈ is the molecular extinction coefficient, L is optical path length and c is concentration (mol/V) that helps in determining oil concentration in weight %. One approach is to use ultraviolet light of specific wavelength range, which is readily oil and hardly absorbed by refrigerant112). This technique can be used for transient and real time monitoring of oil concentration. Later, a technique was developed by measuring absorption at specific wavelength using spectrometer. An UV oil concentration meter which monitors the absorption of light in refrigerant lubricant mixtures was utilized. One limitation is that oil concentration must be kept below 10% since there is signal loss for higher oil concentrations. This phenomenon is also heavily dependent on UV absorbance level in specific refrigerant lubricant mixtures and cannot work for all refrigerant lubricant mixtures106). Another approach is to use infrared light absorption; however, this technique becomes highly inaccurate in case of water presence113). A simple experiment setup is shown in Figure20

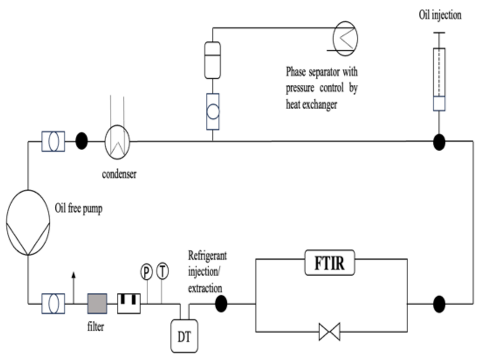

Fig. 20: Light absorption method for oil composition measurement

4. Outlook and Conclusion

The study of role of lubricants and prediction of their concentration in HVAC industry is very crucial. Further research is required on measurement of thermophysical properties of new low-GWP refrigerants and oil mixtures. Moreover, there is a need for extensive development of theoretical models for the prediction of oil concentration in these mixtures. There is limited research on the fundamental interactions between lubricant and refrigerants based on their molecular structures. Advance molecular dynamics and Monte Carlo simulation techniques can give valuable insights about these interactions on molecular levels. Various additives are added to the lubricants, and it is important to characterize system performance in presence of these additives. Nano lubricants are an interesting improvement in lubrication technology. Extensive research on these fluids for oil concentration prediction as well as efficiency analysis can be a useful research direction.

This paper presents a detailed analysis of the literature on the measurement of oil concentration in oil/refrigerant mixtures. This overview highlights several properties of oil refrigerant mixtures that can be used to obtain oil concentration. The experimental and theoretical models on the density, viscosity, speed of sound and various other properties is discussed. A comparison of accuracy achieved in various measurement techniques is made and it is concluded that oil concentration measurement using density and refractive index gives a maximum accuracy up to 0.1 %. In addition, speed of sound can also provide a good estimate for oil concentration in the system. Although, generally, there is a lot of research literature available on properties of oil-refrigerant mixture, these properties are not well explored to be used as prediction parameters for oil concentration measurement. This indicates a significant gap in research area. Further research can be done to predict oil evaluation using techniques like density, refractive index, and thermal conductivity etc. The measurement of these properties is not only useful for oil composition prediction but can also be extended to develop other thermodynamic and heat transport properties of refrigerant lubricant mixtures. Industrial sensors have compromised accuracy, relatively more expensive and have limited range of pressure and temperature measurement. Extensive research is required for development of sensors to measure these properties in complete range for refrigerant lubricant mixtures.

Nomenclature

w	Speed of sound (m/s)
κ	Heat capacity ratio
R	Ideal gas constant ($J/K.mol$)
T	Temperature
C_V	Isochoric Heat Capacity ($J/mol * K$)
t	Time (s)
l	Fluid length (m)
X	Axial distance along pressure tube (m)
E^*	Minimization Function
m	Mass (g)
V	Volume (cc)
K	Springs constant
T	Time period (1/s)
E	Young's Modulus
SG	Specific gravity (g/cc)
D	Saturation density (kg/m3)
x	Oil composition
n	Refractive index
I	Intensity of light (lux)
A-F, a-e	Fitting parameters (unless specified)
c	Concentration (mol/V)

Greek symbols

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ρ	Density (kg/m3)
Ɛ	Capacitance
φ	Volume fraction (-)
ϵ	Molecular extinction coefficient (–)

Subscripts

sat	Saturated liquid
0	Ideal Gas
tof	Time of flight
steel	Steel
m,l	Refrigerant/Oil mixture
r,l	Refrigerant in liquid phase
r,v	Refrigerant in vapor phase
x	Oil composition
i	Incident light
mix	Refrigerant/Oil mixture
ref	Refrigerant
oil	Oil

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