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# **Density of the Refrigerant Fluids of R365mfc and R152a: Measurement and Prediction**

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#### **ABSTRACT**

This work presents the density of new environmentally friendly refrigerants 1,1-difluoroethane (R152a) and 1,1,1,3,3-Pentafluorbutane (R365mfc) in their pure fluid form as well as a mixture. The density is covered in the temperature range of  $-10^0C$  to  $45^0C$  and the pressure range of p=0.65-10.47 bar. The density is measured by a vibrating-tube densitometer (DMA-HPM) manufactured by Anton Paar. The apparatus supplies data in the temperature range of -10°C to 200 °C and a pressure range of 0 to 1400 bar, with an uncertainty of 0.1%. The experimental data is validated using the 'Volume Translated Peng Robinson Equation of State' and high precision fundamental equations of state by Outcalt and McLinden from the National Institute of Standard and Technology (NIST). Outcalt and McLinden model achieve deviations less than 0.56% for R365mfc and 0.51% for R152a. The deviations of VTPR are within 2.5% and 15% for the pure fluid and the mixture respectively. .

## **1. INTRODUCTION**

The last two decades have witnessed a significant increase in Chlorofluorocarbons (CFCs) and Hydrochlorofluorocarbons (HCFCs) based refrigerants which are harmful to the atmosphere. These materials have been widely used as solvents, foam blowing agents, aerosols and especially as refrigerants due to their preeminent properties such as stability, non-toxicity, non-flammability and availability. However, these substances have a harmful effect on the earth's protective ozone layer. Since the declaration of the Montreal protocol in 1987 the use of these substances has been consequently regulated (Fialho et al.,1996). Moreover the CFCs had been detected as substances contributing towards global warming. The first possible alternatives included some hydro chlorofluorocarbons (HCFCs), however they will also be phased out internationally between 2020-2030 because of their potential to deplete the ozone layer, and its global warming potential is almost as high as that of the CFCs (Fialho et al.,1996). This paper supports more accurate density data for these materials, including the pure fluids and mixtures of R152a and R365mfc. R152a is a HFC type refrigerant and has zero ODP and a GWP value of 120. The boiling point temperature at a pressure of  $p=1.013$  bar is  $\theta^s = -24.0^\circ \text{C}$  and the molecular weight is M=66.1 kg/kmol. R152a is a medium pressure refrigerant for the medium temperature refrigeration range (Meurer et al.,2007). The physical, thermodynamic and refrigeration characteristics are similar to those of the refrigerants R12 and R134a. For all the experiments in this work, the same samples for R152a have been used in accordance to the specifications provided by the manufacturer GHC Gerling Holz & Co. Handels GmbH, the samples had a purity of  $\geq$ 99.5% and were used without further purification (Meurer et al., 2007). R365mfc is a HFC type refrigerant. This fluid has zero ODP and a GWP value of 782. The boiling point temperature at a pressure  $p=1.013$  bar is  $9^{s}=+41.4^{\circ}$ C, the molecular weight is M=148.07 kg/kmol. R365mfc is a new fluid which is mainly used for the production of rigid polyurethane foams used in insulation purposes, where a liquid foaming agent with a low thermal conductivity and a high vapor pressure at low temperatures are needed. Regarding thermodynamic properties and safety requirements, R365mfc is employed as a main component in binary blends with 7% or 13% by mass with 1,1,1,2,3,3,3-Heptafluoropropane (R227ea) in the production of liquid foaming agents (Meurer et al.,2007). R365mfc has

demonstrated that it acquires a high insulation capacity which, enables it to reduce energy consumption and therefore its contribution to global warming is reduced. It is considered to be more eco-efficient than alternative solutions, based for example, on water as a blowing agent (Krähling and Zipfel,2000). Eventually, another field of application is the use of R365mfc as a possible working fluid in high-temperature heat-pump systems at condensation temperatures of about  $9=100^{\circ}$ C. For all the experiments in this work the same samples for R365mfc have been used in accordance to the specifications provided by the manufacturer Solvay Fluor & Derivate GmbH, Hannover, the samples had a purity of  $\geq$ 99.7% and were used without further purification (Meurer et al.,2007). In spite of these practical applications, only a very limited amount of data for the thermodynamics properties of R365mfc is available in the literature, more data were mentioned by (Fröba et al.,2007), (Fröba et al.,2004).

#### **2. EXPERIMENTAL METHODS**

The principle of the densitometer is very simple, so as to represent a unique relation between the natural frequency of the vibrating-tube and the density of fluid confined in this tube. The most recognized progress of the vibratingtube densitometer is the high accuracy in relative density measurement with minimum amount of sample. Most of the commercial densitometers have been produced by an associated manufacturer, Anton Paar, K.G., Austria. There are many types of vibrating-tube densitometer for process engineering purposes. The principle of the vibrating-tube densitometer relies on a fixed relation between the natural frequency of the vibrating-tube and the density of fluid contained in the tube. Most of the theory behind vibrating densitometers can be derived from a simple mass spring model, detailed information for which is found in (Stabinger et al.,1969), (Murer et al.,2003).



**Figure 1:** Vibrating tube densitometers with electro-optical system (Anton Paar, 2013).

There is an electronic vibration circuit to activate and sustain a continuous vibration of the tube with its natural frequency, by means of the electromagnet fixed to the cell and the permanent magnet on the tube. This principle is an electro-optical system as shown in Fig.1. The direction of the vibration should be normal to the U-surface, and no twists are provided to occur (Fröba et al.,2004). The basic equation for the density (ρ) using vibrating sensors is defined as:

$$
\rho = A * \tau^2 - B \tag{1}
$$

Where 'A' and 'B' are unknown constants and  $\tau$  is the period of density. The experimental set up was carried out at the Institute for Thermodynamics, Leibniz University, Germany. As shown in Fig.3, the density meter (DMA HPM, Anton Paar) was used to detect the liquid density in a temperature range of  $9 = -10$  °C to 45°C. Due to the high temperature dependency of density, the measuring cell is accurately thermostatted. The temperature of the U-tube is controlled within ±0.02 °C using an external thermostat (Lauda, ECORE 1050) and is also used to regulate the temperature inside the cell, whereas the whole apparatus was maintained in an isothermal isolation chamber ISOCHAM. Since the apparatus does not have any source for pressure generation, an auxiliary pressure generator was integrated into the apparatus. The pressure was measured by a high accurate pressure sensor (type: PAA35X-C-300, omega) with a precision of  $\pm 0.1$ kPa. The pressure measuring unit was successfully integrated into the density measuring cell via hand spindle pump SP (type Ruska, No 41619). To ensure fine adjustment and recharging of the pressure, an additional hand pump HP was integrated with the spindle pump. After evacuating the apparatus, all side products were accumulated into an external waste vessel WV by using a cold trap CT unit. The apparatus has two entrances for feeding IN1 and IN2, which were regulated manually by valves V4 and V5. The valves V1 and V2 are

responsible of regulating the flow rate of the sample into the measuring cell. Evacuation of the apparatus was achieved by a water jet pump (type Brand, No 159665) with pressure of up to 30 mbar. All valves used were needle valves (type: Autoclave 30VM4081-material of construction 316 stainless steel). The samples were contained in sample glass cylinders which were suited in a high geostatic position to simplify the flowing of the fluid into the cell. The layout of the measuring cell was suited to be in a low geostatic position to minimize flow of gas bubbles into the cell. During the charging process all fluids stacked between the cell and the sample cylinder in line V4 and V1 were considered in the pressure measurement. Since the density has a high dependency on the temperature, the measuring cell must be well thermostatted. The temperature gradients affect the quality of the measuring data. Therefore it is necessary to excite the cell well under isothermal condition. The measuring cell (type DMA HPM) was equipped with an internal heat exchanger which regulates the temperature of the surface of the vibrating U-tube. For this purpose an auxiliary thermostat (type Lauda ECORE 1050) (Lauda Ecore,1998) was integrated and the temperature was controlled up to  $g=\pm 0.005^{\circ}$ C. The temperature gradient between the measuring cell and the environment was also considered. Therefore, a house made isolation chamber was constructed to enhance the isothermal condition of the ambient air. During the operation, the fluid to be measured must have the same temperature as in the vibrating U-tube. Since all parts of the components were sealed, it had been very difficult for the operator to watch the fluid behavior in the vibrating U-tube. Thus, there is no guarantee of the removal of all the bubbles from the samples. The formation of the bubbles normally leads to poor density measurement results. To ensure minimum bubbles at the dead volume zones in the fitting tubes, a sample cylinder with upper and lower entrances regulated by two valves was used. To ensure a precise repeatability of the measuring data produced by the apparatus, the period of oscillation of different fluids were tested at least three times at the same conditions. The repeatability may be expressed with the repeatability standard deviation. This standard deviation was calculated from the measurements carried out under repeatability conditions. For a series of n-measurements of the period of oscillation data, the experimental standard deviation characterizes the dispersion of the results and is given by (Anton Paar,2013):



**Figure 2:** Oscillation profile during the measurement show the thermal equilibrium approach.

A calibration is the comparison of achieved measurement results with a standard reference value. This process was performed to validate the quality of measurements and adjustments (Anton Paar,2013). The calibration was achieved with a precise measurement of the pressure, temperature and the density of two well-known fluids. To determine the density of the sample with a mathematical interpolation, the sample should have a density value between the higher and the lower densities of the two reference fluids. The constant parameters of equation (1) were calculated as a function of the period of oscillation and density which normally expressed as (Murer et al.,2003):





Pressure Measurement
Density Measuring Cell
<b>Isolation Chamber</b>
Vacuum Pump
Cold Trap
<b>Waste Vessel</b>
Inlet
Exit
<b>Hand Pump</b>
Spindle Pump
<b>Inlet Valve</b>

**Table I.** Equipment Abbreviation

$$
A = \frac{\rho_1 - \rho_2}{\tau_1^2 - \tau_2^2} \qquad B = \frac{\tau_2^2 \rho_1 - \tau_1^2 \rho}{\tau_1^2 - \tau_2^2}
$$
 (3)

Where  $\rho_1$  and  $\rho_2$  are the densities of the two standard fluids respectively  $(\rho_1 > \rho_2)$ ,  $\tau_1$  and  $\tau_2$  the period of oscillation of the two standard fluids respectively. The parameter constants  $A$  and  $B$  were supplied to the electronic device mPDS5 continuously after every calculation. An external Matlab program was used to analyze the measuring data. Furthermore, the density of the vibrating U-tube can be expressed in the following form (Moreno,2006),(Hradezky and Sommer,2002):

$$
\rho = (\rho_2 - \rho_1)^* \left( \frac{\tau^2 - \tau_1^2}{\tau_2^2 - \tau_1^2} \right) + \rho_1 \tag{4}
$$

Where  $\rho$  and  $\tau$  are the density and the period of oscillation of the sample fluids respectively. For various applications the apparatus was calibrated using water and ethanol. The samples had a purity of  $\geq$ 99.9% and were

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used without further purification. The period of oscillation was measured under certain conditions ( $p=1.01325$ bar,  $=25\degree$ C) and the density was successfully measured as well as the calibration parameters A and B. Fig.4 illustrated three dimension temperature dependency calibration of the period of oscillation measured for water and ethanol varying with density. Obviously the increase of temperature led to an increase of the period of oscillation.



**Figure 4:** Variation of the density with period of oscillation and temperature.

#### **3. PREDICTION METHOD**

A new group contribution EOS has been developed by Gmehling and his coworker Ahlers (Gmehling et al.,2001,2002). The idea was to develop the model of PSRK and to assert more attention in the prediction of saturated liquid densities. Ahlers and Gmehling (Gmehling et al.,2001,2002), Wang (Gmehling et al.,2003), Ahlers (Gmehling et al.,2004) have improved this model, which finally led to the successful Volume Translated Peng-Robinson group contribution EOS. Especially for any  $g<sup>E</sup>$  mixing rule, the EOS used has had great influence on accuracy of data (Gmehling et al.,2001,2002). The realization of applying a volume translation to the EOS was proposed by Peneloux and Freze (Peneloux and Freze,1982) and directly applied to the Peng-Robinson EOS as follows (Gmehling et al.,2012):

$$
P = \frac{RT}{v + c - b} - \frac{a}{(v + c)(v + c + b) + b(v + c - b)}
$$
(5)

The translation parameter (c) indicates the deviation between the experimental and the calculated liquid volume at a

$$
c = v_{calc} - v_{exp}
$$
 (6)

reduced temperature  $T_r = 0.70$ .

The subscripts 'exp' and 'calc' represent molar volumes *v* obtained from experiment and calculated from the EOS respectively. In the statement of Ahlers and Gmehling (Gmehling et al.,2001,2002), if there is no experimental data for liquid densities, then the'c' term could be estimated from critical data as follows:

$$
c = -0.252 \frac{RT_c}{P_c} (1.5448 Z_c - 0.4024)
$$
\n(7)

In this model the Mathias-Copeman  $\alpha$  function was replaced with the Twu  $\alpha$  function (Twu et al., 1991), (Twu et al.,1995) for the calculation of energy parameter a.

$$
\alpha(T) = T_r^{N(M-1)} \exp[L(1 - T_r^{NM})]
$$
\n(8)

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The Twu  $\alpha$  function improved the results of pure component vapor pressures and provided acceptable temperature extrapolations. The  $\alpha$  function approaches zero at very high temperatures, meanwhile the Mathias-Copeman  $\alpha$ function fails. The parameters N, M, and L have been predicted by fitting the vapor pressure experimental data of pure component, more data is well stored in DDB (Dortmund Data Bank,2006). If the parameters are not available or if  $T > T_c$ , then the generalized  $\alpha$  function for the PR EOS developed by Twu can be used as this work has followed the mentioned method. The  $\alpha$  values for different compounds are presented as a function of the acentric factor  $\omega$  for each temperature and is given by:

$$
\alpha(T) = \alpha^{(0)} + \omega(\alpha^{(1)} - \alpha^{(0)})
$$
\n(9)

To apply equation (9), Ahlers (Gmehling et al.,2001,2002) has used special equation for subcritical conditions. This equation is generalized for aromatics, ketones, alcohols and the refrigerant fluids (Ahlers,2003):

$$
\alpha^{(0)} = T_{r_i}^{\ -0.1883273} \exp[0.1048767(1 - T_{r_i}^{2.1329765})]
$$
\n(10)

$$
\alpha^{(1)} = T_{r_i}^{-0.6029386} \exp[0.5113343(1 - T_{r_i}^{2.2059312})]
$$
\n(11)

In order to tackle problems associated with highly asymmetric systems , Ahlers et al (Gmehling et al.,2001,2002) identified that the development of the VTPR model required the introduction of improved mixing rules for calculation of the  $a_m$  and  $b_m$  parameters (Gmehling et al.,2012). The mixing rule for this model is similar to the Huron-Vidal mixing rule; however the reference pressure is equal to 1 bar and not infinity. The residual part of the activity coefficient is used. The relative van der Waals volumes  $r_i$  was terminated in this mixing rule. The new  $g<sup>E</sup>$ mixing rule in equation (12) has introduced the VTPR-  $g<sup>E</sup>$  mixing rule (Gmehling et al., 2001, 2002).

$$
a_{m} = b_{m} \left( \sum_{i=1}^{n} X_{i} \frac{a_{i}}{b_{i}} + \frac{g_{res}^{E}}{q_{1}} \right)
$$
 (12)

Where  $q_1 = -0.53087$ ,  $p^{ref}=1$  bar and  $g_{res}^E$  is calculated according to the UNIFAC parameters (Gmehling et al., 2012). In this work the  $g_{res}^E$  was obtained according to the UNIFAC method stated by Kleiber (Kleiber, 1995), (Kleiber and Axman, 1998).

#### **4. RESULTS AND DISCUSSIONS**

Experimentally measuring the liquid density of R365mfc in a temperature range of  $9 = -10$  °C to 30°C, with the relative deviation as shown in Fig.5-6, both Outcalt and McLinden (Outcalt and McLinden,1996), (McLinden and Lemmon,2013) and VTPR have delivered accurate results with the experimental data. Minimum positive deviation less than 1.95% was reached by this model, as shown in Fig.7. The mixture consisting of 30% R365mfc for a temperature range of  $9=10.74-10.54$  °C, has been presented in Fig.8, illustrating that the model VTPR reached a relative deviation less than 15% (see Fig.9). Additionally, the liquid densities of R152a and R365mfc were measured in a temperature range of T=262.35-262.45K and T=270-320K, respectively, and a pressure range of p=0.65-10.47 bar as illustrated in Fig.10. The deviation diagram in Fig.11, has shown that both models belong to high precision equation of state by Outcalt and McLinden (Outcalt and McLinden,1996), (McLinden and Lemmon,2013) and VTPR, delivering highly accurate date. The model of Outcalt and McLinden from (NIST) (Outcalt and McLinden, 1996), (McLinden and Lemmon, 2013) has shown a maximum deviation less than 0.56% for R365mfc and 0.51% for R152a. VTPR model delivered a maximum deviation less than 2.5% for both R365mfc and R152a. The Volume Translated Peng-Robinson group contribution EOS has been successful in delivering completely accurate data. Since this model delivers a better reproduction of liquid volumes. Furthermore, the Twu  $\alpha$  function in this model shows a reasonable temperature extrapolation. The mixing rules for the (a) energy and (b) the co-volume parameters allow for a highly accurate prediction of the Equation of State for the asymmetric fluid R152a.



**Figure 5:** Liquid density measurement of R365mfc.



**Figure 6:** Relative deviation between liquid density measurement data and VTPR model for R365mfc.



**Figure 7:** Relative deviation between liquid density measurement data and VTPR model for R152a.



**Figure 8:** Liquid density measurement for 30%R365mfc.



**Figure 9:** Relative deviation between liquid density measurement data and VTPR model for 30%R365mfc.



**Figure 10:** Liquid density measurement of R152a and R365mfc.



**Figure 11:** Relative deviation in liquid density measurement data of R152a and R365mfc.

#### **5. CONCLUSION**

The work has demonstrated the capability of the experimental setup which was used to detect the density of the environmentally friendly system of R152a and R365mfc using the vibrating tube densitometer. Despite some challenges regarding the stability of the phases inside the vibrating U-tube, particularly for the previously mentioned refrigerant fluids, the work provides the necessary application of the new group contribution equation of state, as it executed the VTPR combined with modified UNIFAC method by Kleiber et.al which also improved the results considerably.

# **NOMENCLEATURE**

## *Abbreviation*



## *Latin letters*



#### *Greek letters*



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