

Design and testing of the supporting setup for the high-pressure vibrating tube densimeter

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Abstract. Vibrating tube densimeters (VTDs) have become relatively popular instruments for measuring the density of a large variety of gases and liquids since their development about fifty years ago. The apparatuses measure the characteristic frequencies of vibrating U-shaped tube filled with a fluid sample, when the fluid fill shifts the dynamic characteristics of the U-tube. This study describes design and testing of the in-house developed supporting setup for a commercial high-pressure VTD Anton Paar DMA HP. The instrument covers density range from 0 to 3000 kg/m³ at pressures up to 700 bar and temperatures from -10 to 200 °C. Whereas the temperature of density measurement is fully controlled by the apparatus using a thermal block with Peltier unit, pressure has to be generated and monitored externally. Therefore, a high-pressure sampling system was designed and assembled. Special care is taken when measuring temperature with an external resistance thermometer connected to precise thermometry bridge. In order to avoid unfavorable condensation of air moisture during the measurement, VTD apparatus is protected by a dry box of own design. Technical details together with the calibrations of pressure and temperature probes are described in detail. A preliminary data for temperature dependence of density of the selected liquids (water, ethanol or ethylene glycol) obtained at the barometric pressure are also provided and compared to the reference density correlations.

1 Introduction

Vibrating tube densimetry (VTD) is a well - established experimental method often used for systematic studies of fluid density. Since its development about 50 years ago, the VTD method has been adopted by many scientists mainly for its relatively straightforward and fast measurements with good accuracy over wide ranges of temperatures and pressures. A commercial design of VTD offers reliability, small amount of sample required and convenient operation, when the density data can be obtained in the order of minutes or even seconds. The principle of VTD is based on measurement of resonance period τ of a vibrating, usually U-shaped, tube filled with the fluid sample, which can be related to the fluid density by means of relation derived from the spring mass model:

$$\rho = A \cdot \tau^2 - B \quad (1)$$

where

$$A = \frac{k}{4 \pi^2 V}, \quad (2)$$

$$B = \frac{m}{V} \quad (3)$$

In equations (1) and (2), m stands for the mass of the cell, k denotes the spring constant, V is the inner volume of the cell, and ρ is the fluid density.

This work focuses on high pressure VTD equipped with a U-tube made of metal alloy and allowing measurements over a wide range of temperatures and pressures. In our laboratory, a commercial high-pressure VTD Anton Paar DMA HP is available. The device itself allows for temperature dependent measurements but for high pressure application it has to be equipped with an external filling and pressurizing system. Design, construction, technical details and testing of such in-house developed setup is described here.

Since most VTDs are operated in a relative measurement mode, it follows that in order to obtain accurate experimental density data a great attention must be paid to the instrument calibration including a suitable selection of the reference fluids. The calibration process is based on determination of parameters A and B , when the period of oscillation can be expressed as:

$$\tau = \sqrt{\frac{\rho + B}{A}} \quad (4)$$

The relation between the density and the period of oscillation is influenced by the temperature of the U-tube and the internal pressure. From this point of view, the calibration of high pressure VTDs becomes relatively complicated [1-9] compared to common atmospheric instruments. There have been developed several different approaches to obtain A and B parameters as described further.

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In this work, a preliminary density data of selected liquids (water, ethanol and ethylene glycol) for temperature dependence at atmospheric pressure compared to the reference density correlations are presented. The preliminary data were evaluated based on a polynomial model similar to that employed on a barometric VTD in our previous studies [10,11].

2 Temperature and pressure functions – Evaluation of parameters A, B

There are two main approaches to obtain instrument's parameters A and B . Measurements of two reference fluids made at almost exactly the same pressures and temperatures as the sample density required is one of the reliable way of calibration. Some authors call it a "classical" model. Using equation (1) at the measured points the parameters A, B are determined. To realize this linear calibration procedure, the following conditions must be fulfilled: (i) the densities of standards cover the whole range of pressures and temperatures, (ii) the density uncertainty of the standards is in full range sufficiently low, (iii) the standard densities do not differ much from each other and the sample density lies between the standard densities, (iv) viscosities of reference fluids are similar to that of the sample, (v) the reference substances under demanded temperatures and pressures are chemically stable. The uncertainty of density depends in this case on the uncertainties associated to each calibrating fluid densities. The main problem of such calibration lays in the finding of suitable calibration fluids because there is a lack of fluids with known densities as a function of temperature and pressure with sufficiently low uncertainty.

To solve the problem of unavailable reference fluids, alternative methods have been developed using just one calibration fluid (usually water) together with vacuum [3,4]. Vacuum can be realized by coupling a vacuum cylinder to a Hastelloy vibrating cell. When the air density is in the order of 1 kg/m^3 at 1 bar then the density of the vacuum at pressure below 100 Pa is lower than 0.001 kg/m^3 , which is far below the resolution of most high-pressure VTDs and as such can be assumed as 0 kg/m^3 . Using vacuum as the second standard solved the problem of its density uncertainty but arose a new one with the evaluation of the pressure dependence of parameters A and B . Bouchot et al. [5] showed that the assumption which parameters depend on the pressure (determination of boundary conditions) determines the shape of the calibration equation and subsequently relativizes the values of the constants A and B . This kind of problem does not occur when utilizing the classical calibration using two reference fluids along with eq. (4).

To figure out the way how to conserve the advantage of vacuum as one reference and remove the uncertainty arose from the classical discrete formula the physical signification of terms m , V and k has been evaluated independently [5,8,9]. To do this the equation (1) must be considered not as a discrete conversion tool but as a continuous transform [5]. Thus, calibration measurements can be made over a similar (not identical) range of

conditions as the range of target conditions and parameters A and B can be expressed continuously as functions. This can be done either as a pure empirical function [1,8] or as a function in which correlation parameters have a physical meaning [5,9]. While maintaining the linear formalism of eq. (1) and preserving the mechanical spring-mass model, the constants A and B can be modeled as pressure functions using rigorous considerations on the stress and thermal behavior of the vibrating tubes.

Outcalt and McLinden [8] introduced a polynomial model, where 13 constants need to be evaluated from a calibration on a series of reference fluids at various temperatures and pressures.

$$\rho(T, p) = \left(\begin{aligned} &A_1 + A_2T + A_3T^2 + A_4T^3 + \\ &+ A_5p + A_6p^2 + A_7Tp \end{aligned} \right) \frac{\tau^2}{\tau_0^2} - \left(B_1 + B_2T + B_3T^3 + B_4p + B_5p^2 + B_6Tp \right) \quad (5)$$

In addition, a second-order polynomial for the fundamental period of an evacuated cell τ_0 , has to be correlated to the experimental data for τ at varying temperature T .

$$\tau_0 = c_0 + c_1T + c_2T^2 \quad (6)$$

May et al. [9] developed a physically-based model having a general form similar to that of equation for atmospheric VTD.

$$\rho(T, p) = A(T, p)\tau^2 - B(T, p) \quad (7)$$

where coefficients A and B are given as follows

$$A(T, p) = \frac{(1 + \beta_\tau p)(\rho_M / S_{00} \tau_{00}^2)}{(1 + \varepsilon_{\tau 1} T + \varepsilon_{\tau 2} T^2)^2 (1 + \alpha_V T + \beta_V p)}, \quad (8)$$

$$B(T, p) = \frac{\rho_M}{S_{00} (1 + \alpha_V T + \beta_V p)}. \quad (9)$$

Quantities τ_{00} , $\varepsilon_{\tau 1}$, and $\varepsilon_{\tau 2}$ are determined from measurements of the evacuated cell at various temperatures. Other parameters S_{00} , α_V , β_V and β_τ are determined from measurements of a reference fluid. ρ_M is the density of the U-tube material, e.g., for Hastelloy $\rho_M = 8890 \text{ kg/m}^3$.

3 Experimental setup

3.1 High-pressure vibrating tube densimeter

A commercial high-pressure vibration tube densimeter Anton Paar DMA HP was used for the measurements presented in this work. The instrument is designed to measure the density of liquids and gases at pressures up to 700 bar in the temperature range from $-10 \text{ }^\circ\text{C}$ to $+200 \text{ }^\circ\text{C}$. A simplified scheme of the experimental setup with DMA HP is shown in Fig. 1. Operation of the high-pressure cell

in DMA HP is controlled via the DMA 5000 M densimeter designed for the atmospheric measurements. Both units are connected to a cooling water circuit with thermostatic bath for the measurements at temperatures below the ambient temperature. The DMA HP instrument has to be enclosed in a dry box connected to a drying

circuit with air pump and a moisture absorber (silica gel column). The dry box allows measurements at temperatures below the dew point of the laboratory. A special filling and pressurizing system (FPS) for filling a liquid sample into the DMA HP U-tube had to be developed as described further.

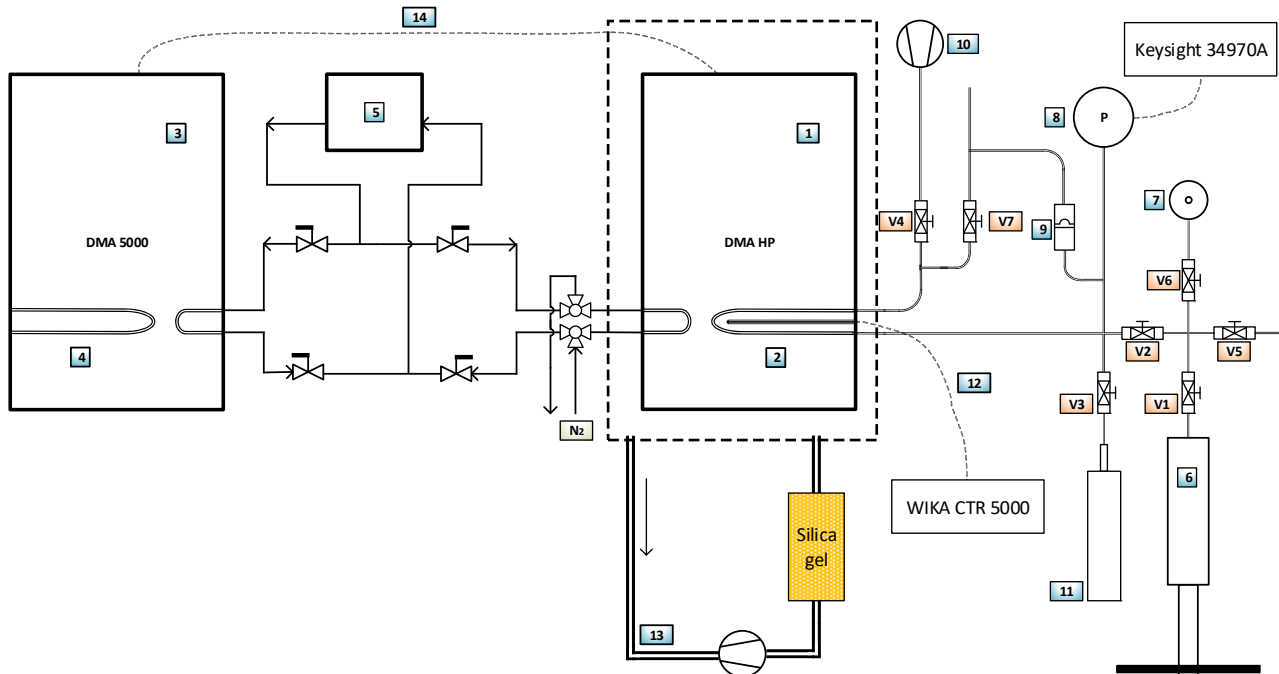


Fig. 1. Scheme of the high-pressure vibrating tube densimeter setup. 1 – DMA HP module, 2 – Hastelloy vibrating tube, 3 – DMA 5000 M atmospheric module, 4 – borosilicate glass vibrating tube, 5 – thermostat, 6 – hand piston pump, 7 – filling cylinder with sample, 8 – pressure transducer Omega, 9 – rupture disc, 10 – vacuum pump, 11 – 250 ml vacuum buffer or gas vessel (N₂), 12 – Pt100 with bridge WIKA CTR 5000, 13 – dry air circulation, 14 – data cable.

3.2 Filling and pressurizing system

A high pressure vibrating-tube densimeter measuring system was described in the literature [8,9,12]. A similar filling and pressurizing system (FPS) to the one used by Sampson et al. [12] was developed. Next to the DMA HP U-tube, FPS represents the core part of the apparatus as can be seen in Fig. 1. The FPS consists of high pressure needle valves (Parker 10V2081, USA), various fittings and adapters, safety rupture disc (Parker Autoclave Engineers, USA) and tubing (stainless steel 316L capillary 1/8" x .036" ~ 3.18 x 0.91mm), high pressure generator (manual pump HiP, USA, pressure range 0 to 10 000 PSI ~ 689 bar), pressure transducer (OMEGA, PX01S1 10KGI, USA), stainless steel sample vessel and a membrane vacuum pump (Vacuubrand MD1, max 1.2/1.4 m³/h, 1.5 bar, Germany). FPS is operated in the following manner:

- Before the density measurement, FPS is cleaned and evacuated via valve V4 which is connected to the membrane vacuum pump.
- A sample is delivered into the first part of the system from the sample vessel via valve V6 while valves V2 and V5 are closed and sample enters via valve V1

also volume of the high pressure generator, which is open to its maximum.

- Then valve V6 is closed and the second part of the FPS is filled by opening valve V2, while valves V3, V4, V5, and V7 are closed.
- Whole system is then pressurized by the pressure generator to the required pressure and then the valve V1 is closed.
- Sample can be removed via valves V5 and V7 using either simple water vacuum pump or expelled by nitrogen via valve V3.
- For the calibration measurement under vacuum, a buffer volume of approximately 150 ml was connected via valve V3.

3.3 Dry box for VTD

A circuit for the humidity reduction in the dry box in which DMA HP is enclosed was developed. The drying box constructed from PMMA (Polymethyl methacrylate, plexiglass) is placed on an aluminum base. All necessary outputs and inputs were equipped with rubber grommets. During the operation, air is circulating in the closed circuit consisting of the inner space of the box, absorption column with inner diameter of 20 mm and a length of

65 cm filled with activated silica gel connected by polyurethane tubes. The level of humidity content was checked by thermohygrometer Testo 625 with its sensor inserted inside the dry box.

3.4 Pressure measurement

Pressure inside the system is measured using a transducer Omega PX01S1-10KG with the operating pressure range from 0 to 10 000 PSI (0 to 689 bar). DC output of the transducer corresponding to 4 to 20 mA is measured with data acquisition unit Keysight 34970A. According to manufacturer specification the linearity and hysteresis count $\pm 0.05\%$ of the transducer full scale.

Prior to the installation of transducer in the system, its accuracy was tested against a highly-accurate pressure transducer Paroscientific 415K-101. During the test measurement both transducers were installed simultaneously in the apparatus with gaseous nitrogen capable of stable pressure setting. Due to the fact that the transducer zero-point has a tendency to drift over time, the transducer is equipped with an adjusting screw. However, in our measurements we decided to adjust the zero-point numerically from the recorded data based on the reading at ambient pressure in the following manner

$$p_{\text{offset}} = -I_0 p_{\text{sl}} \quad (10)$$

where p_{offset} is pressure offset, I_0 is DC current reading at ambient pressure and p_{sl} denotes slope of pressure vs current I .

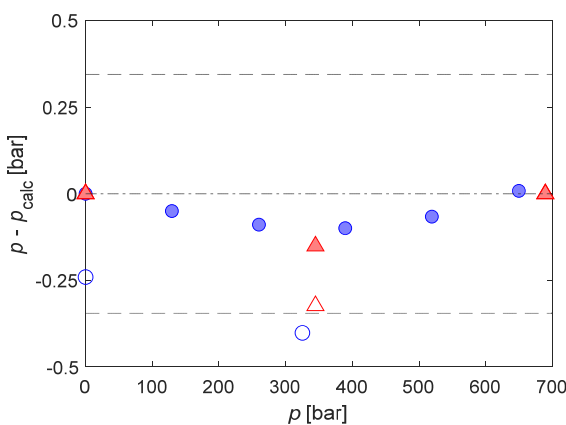


Fig. 2. Test of Omega PX01S1-10KG pressure transducer; deviations of the reference pressure measured with Paroscientific 415K-101 from the calculated values using linear pressure-current dependence. Triangles stand for calibration from the manufacturer, circles mark points from own measurements, when filled symbols denote measurements in the sense of increasing pressure, and empty symbols correspond to measurements with the descending pressure.

Comparison test was carried out at several points from ambient pressure up to 650 bar. Subsequently transducer was tested for hysteresis at two points in the sense of decreasing pressure. Test results are shown in Fig. 2 together with the data from original calibration from the

manufacturer. Dashed lines correspond to $\pm 0.05\%$ tolerances. Value of transducer sensitivity, i.e. pressure vs DC current slope p_{sl} , stated in the calibration certificate from the manufacturer was confirmed within test measurements. Individual calibration points are compared in the figure with the values calculated using original slope p_{sl} and respective pressure offset calculated from eq. (10).

As can be seen, transducer exhibits some non-linearity, which can be better described in future, e.g., by quadratic equation. The transducer hysteresis was found lying within the tolerance level. For a reliable pressure measurement, it is recommended to carry out density measurements in the sense of increasing pressure. Also the initial point should be at ambient pressure to enable determination of pressure offset.

3.5 Temperature measurement

The DMA HPM instrument contains built-in Pt100 temperature sensor with an error $< 0.1\text{ }^\circ\text{C}$ stated by the manufacturer. In order to achieve more accurate temperature measurement and inspect its stability over time, an external Pt100 probe (Omega PR-10-3-100-M30-50-E-2M) with a length of 60 mm and a 3 mm diameter was used. The external thermometer can be placed in a hole located in the center of the DMA HP U-tube. Originally, the Pt100 probe had a specified length of 50 mm but approximately 10 mm of a plastic ring between the probe and the wiring had to be removed as it hindered inserting the probe deep enough. Probe is equipped with 2 m PFA cable resistant to the temperatures up to $200\text{ }^\circ\text{C}$. The calibration was done in the Czech Metrology Institute using a secondary temperature standard at 6 points in the range from $-20\text{ }^\circ\text{C}$ to $200\text{ }^\circ\text{C}$. In order to imitate condition in the hole for the external temperature probe in the DMA HPM instrument, the Pt100 thermometer was calibrated while placed inside a stainless steel tube $1/4" \times 0.036"$ ($6.35 \times 0.91\text{ mm}$) with a length of 400 mm. Expanded uncertainty based on the calibration was estimated to be below $0.048\text{ }^\circ\text{C}$ over the whole temperature range. For the measurement of the resistance of the Pt100 probe a WIKA CTR 5000 thermometry bridge is used in our measurements, when the Callendar-Van Dusen equation correlated to the calibration data is used.

Both instruments WIKA CTR 5000 bridge and the Keysight 34970A data acquisition unit are connected to PC and data are collected by the LabVIEW program.

4 Water and dry air calibration

In order to prevent eventual temperature oscillations, the instrument and other accessories (pressure gauge and external resistance thermometer) were stabilized for 2 hours before each measurement campaign. The stabilization included (i) water circulation through the DMA HP cooling kit, (ii) drying the dry box below a required dew point value to prevent humidity condensation in the cell block inside the instrument, (iii) the air conditioning in the laboratory set to the constant temperature of $22\text{ }^\circ\text{C}$.

A preliminary high-pressure VTD calibration at atmospheric pressure was based on accurate measurements of water + air pairs over wide temperature range. Water from 2 °C to 90 °C and air from -5 °C to 90 °C with 5 °C step. In the calibration measurements, an ambient air applied into the U-tube was dried using a silica gel cartridge. To determine its density, the humidity was checked using a portable hygrometer (Testo 625, Testo AG, Germany). The relative humidity of dried air varied typically from 3.5 % to 6.0 %. The laboratory pressure was recorded at each temperature point by a high-precision external pressure gauge (Druck DPI 142, GE, USA). The pressure gauge was repeatedly compared to the stationary mercury barometer B 3 type from the VEB Werk für Technisches Glas Ilmenau manufacturer. The agreement of the measured pressures was typically better than 0.2 hPa. Reference density values of dry air were obtained from IAPWS guideline G10-8 for humid air [14].

Ultrapure water obtained from a purification system consisting of a reverse osmosis unit Rowapur purifying Prague tap water and of Purite Neptune Analytical unit was used in all measurements. The ultrapure water had a resistivity from 17.4 to 18.2 MΩ·cm, was free of particles larger than 0.2 μm and had an organic carbon content below $5 \cdot 10^{-9}$.

The different isotopic composition between the laboratory tap water and the Vienna Standard Mean Ocean Water (VSMOW) [15,16], to which the IAPWS-95 equation of state is correlated, was considered. Nevertheless, a slight correction of the molar mass of water that has been applied was not in the case of Hastelloy U-tube perceptible as it is one order of magnitude below the actual resolution of the DMA HP instrument.

4.1 VTD output quantities

VTD Anton Paar DMA HP with the Hastelloy C-276 cell measures the fluid density in the range from 0 to 3000 kg/m³ with a resolution of the period of oscillation 0.01 μs. The density error is declared to be ± 1.0 kg/m³ for higher pressures and even ± 0.1 kg/m³ for the optimum conditions. However, the final uncertainty of the measured density can be quite different as it depends on the considered temperature and pressure ranges, quality of the instrument calibration and the employed reference fluids, viscosity of the samples and other effects. We note that careful uncertainty analysis of the determined densities will be in the interest of our future research and is beyond the scope of this preliminary work.

Figures 3 and 4 show period of oscillation τ for water and dry air measured from 2 to 90 °C and from -5 to 90 °C, respectively, during three individual measurement campaigns. As it can be seen, the period is gradually increasing with increasing temperature. Periods measured at the same temperature on different days do not differ from each other by more than 0.01 μs both for water and dry air. These results confirm good reproducibility of the period of oscillation, i.e. a primary experimental output quantity. Any slight systematic offset between the

individual air measurements caused by a different atmospheric pressure during individual measurements was not observed unlike in case of highly sensitive atmospheric VTD Anton Paar DMA 5000 M with a borosilicate glass U-tube [10,11]. This follows from the technical capabilities of the device. While the DMA 5000 M instrument has density resolution of 0.001 kg/m³ (per 0.001 μs oscillation period), the DMA HP cell can distinguish the difference in density of 0.05 kg/m³ (per 0.01 μs – device resolution). DMA 5000 M was found to achieve the uncertainty of water density better than 0.03 kg/m³ for the 2 to 90 °C temperature range [11].

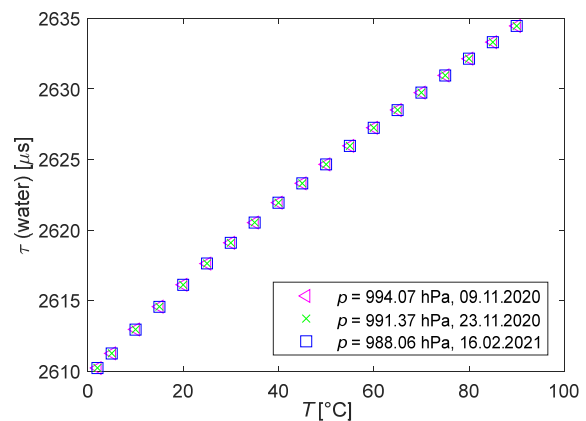


Fig. 3. Temperature dependence of period of oscillation τ for water at atmospheric pressure.

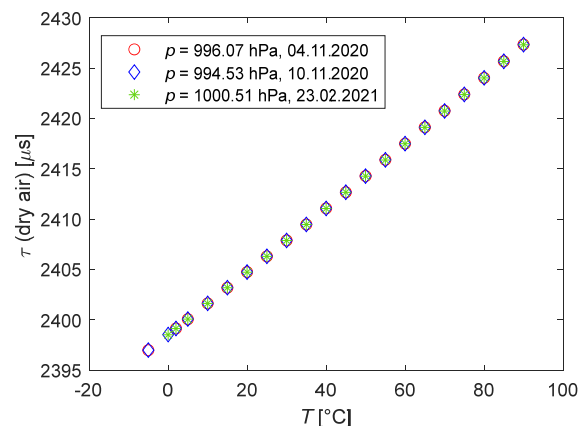


Fig. 4. Temperature dependence of dry air period of oscillation at atmospheric pressure.

4.2 Water and air measurements

The densities of water and dry air were determined from eq. (1). Temperature dependencies of instrument parameters A and B were fitted with cubic polynomials providing a sufficient accuracy. Figs. 5 and 6 show temperature dependencies of A and B reproduced with the following correlations:

$$A(T) = -1.5584 \cdot 10^{-5} T_r^3 + 3.582 \cdot 10^{-5} T_r^2 - 1.3915 \cdot 10^{-4} T_r + 0.001, \quad (11)$$

$$B(T) = -117.9996T_r^3 + 311.8838T_r^2 - 345.149T_r + 5563.2, \quad (12)$$

where T_r is the reduced temperature defined as $T_r = T/400$ with T in Kelvin.

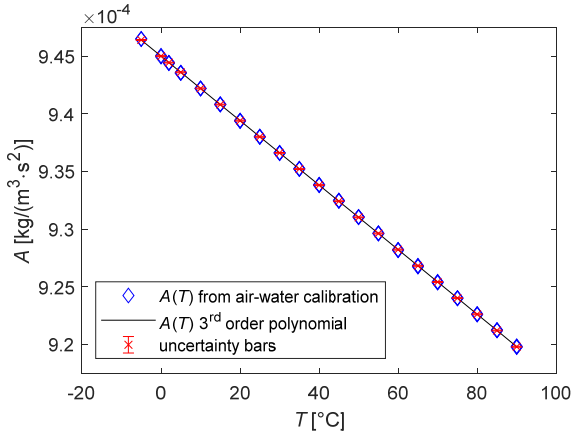


Fig. 5. Calibration parameter $A(T)$ correlated with 3rd order polynomial at atmospheric pressure.

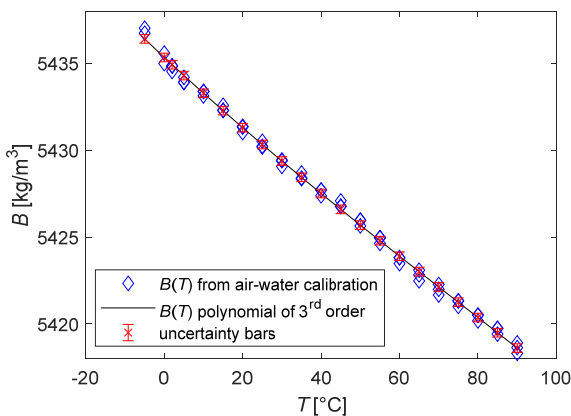


Fig. 6. Calibration parameter $B(T)$ correlated with 3rd order polynomial at atmospheric pressure.

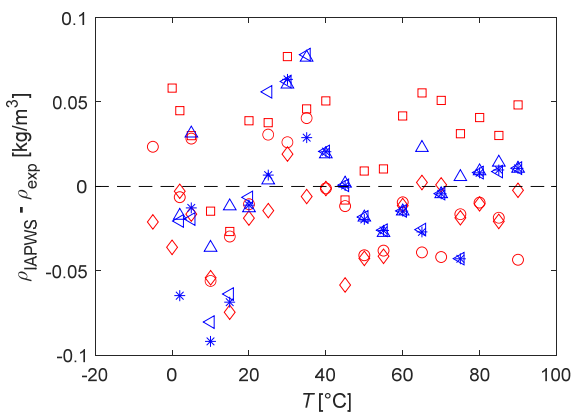


Fig. 7. Density of water (blue symbols) and air (red symbols) exp measured at atmospheric pressure compared to the reference models IAPWS – IAPWS-95 equation of state for water [16] and IAPWS guideline G10-8 for humid air [14].

Fig. 7 shows differences between the density of air and water from the reference values depending on temperature. The reference values were obtained from the models approved by IAPWS [14,16]. The preliminary density data determined from eq. (1) considering the polynomial correlations (11) and (12) led to the deviation from the reference values lying within $\pm 0.1 \text{ kg/m}^3$.

4.3 Density of ethanol and ethylene glycol

In order to test the accuracy of the DMA HP Hastelloy vibrating tube, two commercially available chemicals, one with the density lower and one with the density higher than water, were selected as the test fluids. Ethanol from Supelco Merck batch number I1055390948 with the declared purity $\geq 99.90\%$ and ethylene glycol from Sigma Aldrich batch number SHBL0616 with the declared purity $\geq 99.96\%$ were used. Before and after each density measurement, the water content in the sample was determined using the coulometric Karl Fisher titrator Mettler Toledo C30. No considerable increase of the water content in the samples was detected during our experiments.

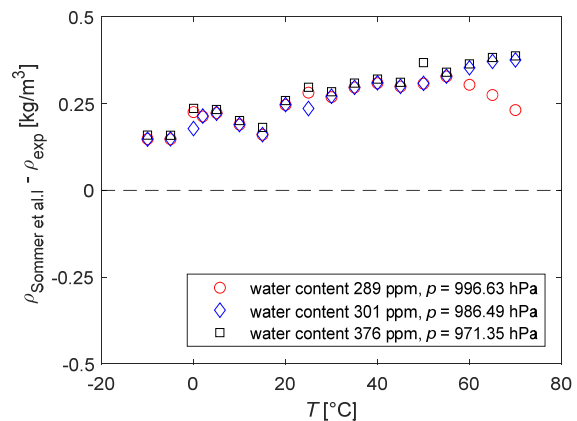


Fig. 8. Difference of the experimental density of three independent measurements of ethanol with similar amount of water impurities from the correlation by Sommer et al. [17].

Three individual measurements of ethanol and two individual measurements of ethylene glycol were carried out. In Fig. 8, it can be seen that the deviations from the reference correlation for liquid density of ethanol by Sommer et al. [17] does not exceed 0.40 kg/m^3 . In the case of ethylene glycol shown in Fig. 9, the mutual difference between the experimental density and the VDI correlation [18] taken as a reference lies in the interval from -1.1 to 0.37 kg/m^3 in the temperature range from $-10 \text{ }^\circ\text{C}$ to $+140 \text{ }^\circ\text{C}$. It should be noted that the ethylene glycol densities for temperatures below $2 \text{ }^\circ\text{C}$ and above $90 \text{ }^\circ\text{C}$ were determined based on an extrapolation of A and B polynomials given by eqs. (11) and (12). The standard uncertainty of the ethanol and ethylene glycol densities determined from the calibration are 0.36 and 0.37 kg/m^3 , respectively.

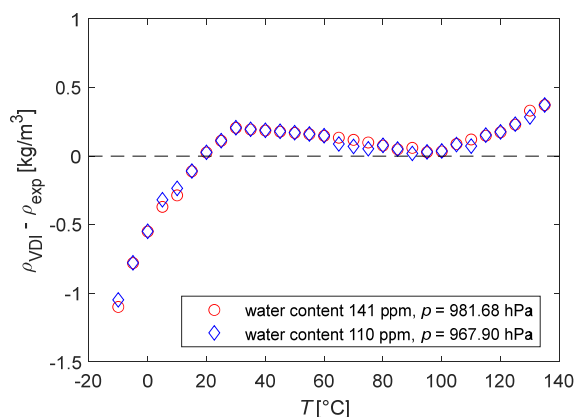


Fig. 9. Difference of experimental density of two independent measurements of ethylene glycol with similar amount of water impurities from the VDI [18] correlation.

5 Conclusions

Team of our laboratory is operating an atmospheric VTD Anton Paar DMA 5000 M with a borosilicate glass cell for more than three years. When properly treated and calibrated, the instrument provides high-accuracy density data at barometric pressure over the temperature range from 0 to 100 °C. The instrument can be extended with an independent high pressure vibrating cell DMA HP made of Hastelloy allowing the density measurements at pressures up to 700 bar. Our intention is to use these systems for accurate density measurements of large variety of technically interesting fluids over wide pressure ranges at temperatures from -10 °C to +150 °C.

The effective use of DMA HP VTD linked with the atmospheric instrument DMA 5000 M required development of several supporting systems. In this work, the design, manufacture and operation of the new setup consisting of the filling and pressurizing system (FPS), special dry box, and the external pressure transducer and temperature probe with a thermal bridge were described. Technical details together with the preliminary calibration measurements of the DMA HP unit at atmospheric pressure were presented.

A classical formula between the density and the period of oscillation based on a spring-mass model along with the polynomial correlations of instrument parameters A and B were considered. For the preliminary measurements, the dry air and ultrapure water were used as two reference fluids, similarly as in case of the atmospheric VTD with the glass U-tube. Temperature dependent parameters A and B were evaluated over a temperature range from 2 to 90 °C. The initial barometric calibration was inspected on a series of testing measurements with four reference fluids; namely dry air, water, ethanol, and ethylene glycol. The collected data helped to carry out the uncertainty analysis of the determined density. For less viscous fluids, the standard uncertainty was found to be of around 0.37 kg/m³ over the temperature range from -5 to +140 °C. Good reproducibility of the measurements carried out with selected

fluids proved sufficient purity of the U-tube cell and correctness of the measurement procedure.

In the subsequent research, measurements with a closed filling system under various pressures will be performed. A physically-based model developed May et al. [9] will be tested on our instrument by using the vacuum and one reference liquid calibration.

Acknowledgement

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