PROPAGATION OF UNCERTAINTIES IN HIGH PRESSURE DENSITY MEASUREMENTS THROUGH CALIBRATION MODELS OF A VIBRATING TUBE DENSIMETER.

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Abstract

This work aims at presenting an analysis of the uncertainties and their propagation in the indirect determination of the densities of fluids at high pressures, by means of a vibrating tube densitometer (VTD), along with a specific physical model for the instrument known as the FPMC model. Insight is still needed with respect to the accuracy of the density measurements obtained by means of this commonly used technique in research laboratories. This study is based on data obtained from electronic data acquisition, which allows to analyze significant samples of the measured variables, that can be treated by means of descriptive statistics.

The uncertainty analysis is illustrated on the basis of recent experimental measurements of the densities of water (used as the reference calibration fluid at pressure up to 700 bar), and of acetonitrile. The stability and correlation of the measured variables and their distributions are analyzed at different pressures in the range of (10 – 700) bar around 323 K. From this, budgets of uncertainty for the calibration of the VTD and for common measurements of the densities of compressed liquids, with the current methodology, are established and compared with the results from recently published works [4-6], using the same type of instrument and different measurement methods.

The uncertainties in the variables and parameters of the problem are then propagated through the FPMC "model". The standard GUM propagation techniques are used taking into account the correlations between the measured variables (T, p, τ) and the parameters of the model. Due to the complexity in the inter-dependence of the measured variables with the measurand, the adaptive Monte Carlo Method is more suitable than the standard analytical propagation formula, for this type of analysis. It is shown how the uncertainty in the densities obtained from this model, frequently considered to lay in the range (0.0001 – 0.001) g.cm^{-3}, are actually estimated to be closer to 0.0003 – 0.0004 g.cm^{-3}, with a k=2 coverage factor.

1. Introduction

Vibrating tube densimeters are widely used to perform high pressure measurements of the density of fluids for industrial and scientific purposes. The basic principle is to measure the vibrating period τ, of a hollow u-shaped tube filled with the fluid of interest at the desired conditions of pressure, *p*, and temperature *T*. The vibrating period is related to the mass of the tube, and especially to the density of the fluid, *ρ*, by means of mechanical considerations. What we call "model" here is the mathematical relation that gives *ρ* as a function of τ and the conditions *T* and *p*.

Different types of mathematical models for both the calibration of a vibrating tubes and the density calculations can be used, see [1-8] for a non exhaustive but rather recent overview. A detailed explanation of the different models can be found in [3]. This reference, along with [4] and [5], describes completely the model that is considered here. We shall only repeat some parts of the mathematical framework. All the models require measurements performed on some reference fluid to be properly set or calibrated. The classical procedure commonly uses water and nitrogen as the reference fluids to set the value of two pressure and temperature dependent functions.

The "Forced Path Mechanical Calibration" (FPMC) model [3], which consists of a calibration formula for the VTD and, at the same time, a measurand model for the calculation of the density, is considered in this work. The model is as follows [3]:

$$\rho(T,p)=M(T,p)*[Kr(T,p)\,\tau^2/\tau_0^2-1] \qquad (1)$$

where *M(T,p)* and *Kr(T,p)* are determined from measurements of τ for pressurized water along isotherms and τ_0, the period of vibration of the tube under vacuum, thus employing a single calibration fluid. In the practice, the functions *M* (the fraction of the mass to the internal volume of the tube) and *Kr* (the reduced stiffness of the tube) contain semi-empirical parameters to be adjusted from a calibration procedure:

$$M(T,p)=rml_{00}\, f(T,p)/\exp(-\gamma_1 p-1/2\,\gamma_2 p^2) \qquad (2)$$
$$K_r(T,p)=g(T,p)\,\exp(-3\gamma_1 p-3/2\,\gamma_2 p^2) \qquad (3)$$

The parameters rml_{00} *(the fraction of the material mass of the tube to a reference length under vacuum) and* γ_1, γ_2 *(the coefficients of a linear approximation of the isothermal expansion of the material of the tube with pressure)* are obtained from a non linear least square fit of eq. 1 to the reference data of the density of water at various pressures along near isotherms. The reference equation of state for water (IAPWS-95 formulation [9]) is used to obtain the reference density values for this purpose at each measured *T* and *p*. The functions *f* (the inverse elementary volume of a section of the tube) and *g* (the relative inertial moment of the right section of the tube) are rather complex *T*, *p* dependent expressions that where obtained from mechanical considerations and are not subject to further parametrization [3] . These functions are the so called "forced paths" in this model.

To our knowledge, the propagation of uncertainties in measured densities with the FPMC model, has only been performed by using the differential method or direct Monte Carlo calculations according to [11]. An example of this kind of analysis was recently published in [4], and is the only available study of this kind for the FPMC model. The authors report an expanded uncertainty in density of the order of 0.0002 g.cm^{-3} for the calibration fluids.

When using the differential method, only a subset of the required partial derivatives in the propagation formula can reasonably be obtained analytically due to the complexity of the functions *f* and *g*. Another drawback of this method is that the uncertainty in the "forced path" functions of the model are very hard to estimate since the mechanical properties of the steel from which the tube is made do not have their uncertainties reported. We consider more appropriate to apply the GUM [10] to calculate the propagation of uncertainty of the measured values, and this is the aim of the present work.

For this purpose we shall first briefly explain the experimental framework and the measurement procedure for the calibration of the densimeter with water and vacuum conditions, and for the common measurements on compressed liquids. From this an uncertainty budget can be set. Then the tools that are used to do the propagation of uncertainties as stated in the framework of the "GUM" [10,11] will be presented along with the obtained results.

2. Experimental

A detailed overview of the experimental framework is available in [5, 12], from which we summarize some relevant information for our purpose. The experimental setup is based on a commercial DMA 512-P high pressure vibrating tube cell. The DMA cell is temperature controlled by means of a PID regulator and a has a stability of 0.002 *K* which can be reached within several minutes. The temperature is measured within a thermometric well between the arms of the U-shaped tube by means of a Pt-100 thermometer which is regularly calibrated against a F300 bridge or secondary reference thermometers following the EIT90. The overall uncertainty in *T*, measured by an ASL F250 thermometer, is 0.03 *K*.

Two pressure transducers Druck®-PMP 4060, with respective measurement scales up to 138 bar and up to 689 bar are used to measure the pressure of the fluids within the tube. They are placed within an insulated brass block to avoid fast temperature fluctuations that could affect their stability, and their positions with respect to the DMA cell are set so that the fluid column does not affect the pressure measurements. Both transducer are calibrated against a Desgranges & Huot® dead weight gauge (5304 S2, ± 0.005 % F.S. precision up to 1380 bar). The mass to pressure conversion coefficient of the gauge is corrected to the local gravity (Mexico City). The medium pressure transducer has a standard deviation of 0.013 bar from 1.0 bar to 150 bar, and the high pressure transducer standard deviation is about 0.1 bar from 50 bar to 700 bar.

2.1. Data acquisition

The data acquisition system includes the digital thermometer (ASL-F250), a 6.5 digits voltmeter (HP34401-A) and a 10 digits 225 MHz universal counter (HP53131-A) for the measurement of the period of vibration from a electromechanical converter attached to the tube. The data acquisition is sequential in *p*, *T* and τ. A data point is currently recorded every 3.5 s. Measurements of $\tau(T, p)$ are performed by electronically forcing the digital counter to provide a seven digits stable value at stable *T* and *p* in approximately 3 s with the last digit fluctuating in the range corresponding to period variations from 2*10^{-6} ms to 5*10^{-6} ms. To reach this it is necessary to prefer quasi-static measurement procedures.

2.2. Procedures

The periods of vibration of the tube under vacuum τ_0 , necessary for the FMPC model, are measured first for each isotherm.

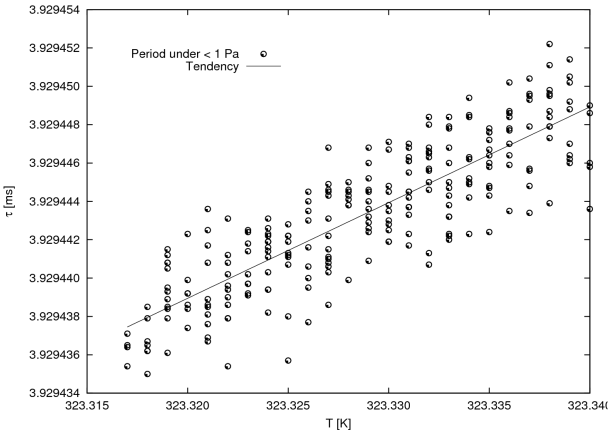

Fig. 1: Measurement of the period of vibration under vacuum around 323.33 K.

Figure 1, presents the measurements of this variable for this study. The mean temperature is 323.329 *K* with a standard deviation of 0.0063 *K*. The period of vibration has a mean value of 3.9294433 *ms* with a standard deviation of $3.7 \cdot 10^{-6}$ *ms*. This example is fully representative of the measurements, with a noticeable correlation of the two variables corr(T,τ_0)=0.8516 in this case. Vacuum is achieved by means of a primary vacuum pump which provides pressures under 1 *Pa*.

For high pressure measurements, the pure fluids contained in a variable volume cell are loaded, free of air (e.g. previously degassed), to the vibrating tube at the highest pressure (700 *bar*), by means of a manual pressure generator (HIP-50-6-15). The measurement procedure consists in successive stabilizations of the state variables (T, p) followed by decompressions of the liquid. Each decompression affects the temperature stability, even with low flow rates (≤0.05 $bar.s^{-1}$). It is necessary to have a high stability in T, to achieve the best stability in τ . The behavior of $\tau(T)$ and $p(T)$ at stable T are usually correlated and the sensitivity of τ is slightly lower than that of p under fluctuations of the temperature.

This is illustrated at two pressures ~700 *bar* and ~20 *bar* in Figures 2 and 3 respectively. The fluctuation of τ is about 4.10^{-6} *ms* around the mean in each case. At 700 *bar* the temperature stability is 0.002 *K* around the mean, and at 20 *bar,* the stability in T is 0.01 *K*. At each measured pressure, a sample region can be defined where all the measured variables are almost simultaneously stable.

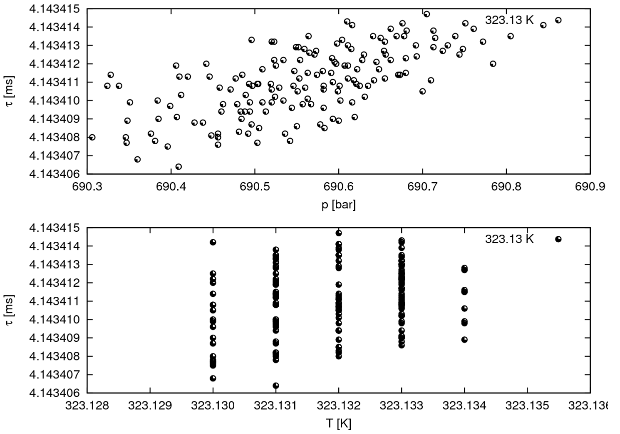

Fig. 2: Stable data point at ~700 bar (water, ~323.15 K).

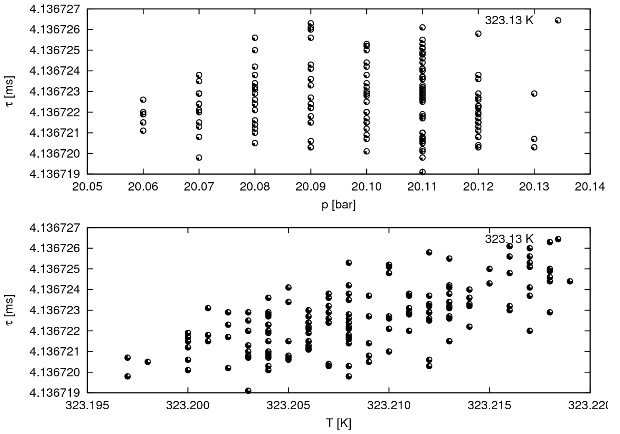

Fig. 3: Stable data point at ~ 20 bar (water, ~323.15 K).

A single representative data point can be extracted from this subset with a definite mean and standard deviation. The correlation between the variables can also be evaluated. In this case at 700 *bar*, corr(T,p) = 0.297, corr(T,τ) = 0.2693 and corr(τ,p) = 0.6554. At 20 *bar*, corr(T,p) = -0.181, corr(T,τ) = 0.6652 and corr(τ,p) = -0.004.

In figure 4 we show the complete isotherm at ~323 *K* measured on water (HPLC grade from Merk Mexico) for the purpose of the calibration of the densimeter. The measurement procedures for other fluids of unknown density are performed in the same way. Measurements on dehydrated and degassed acetonitrile (purity 99.5 % from Merk, Mexico) were performed at about 323 *K* and considered here.

Figure 5 shows the behavior of the measured isotherm in the plane (p, ρ).

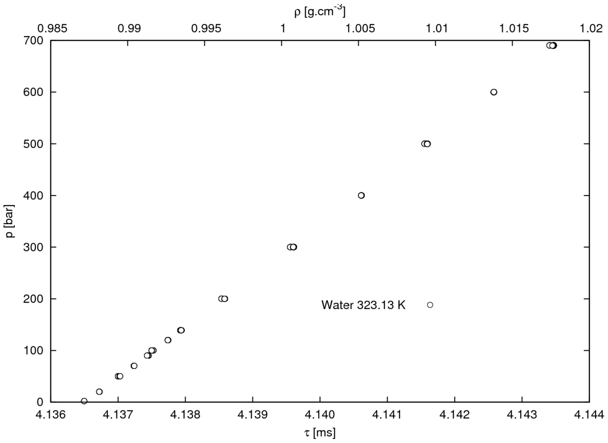

Fig. 4: Measurements on water for calibration purposes. Densities are from the IAPWS-95 EoS.

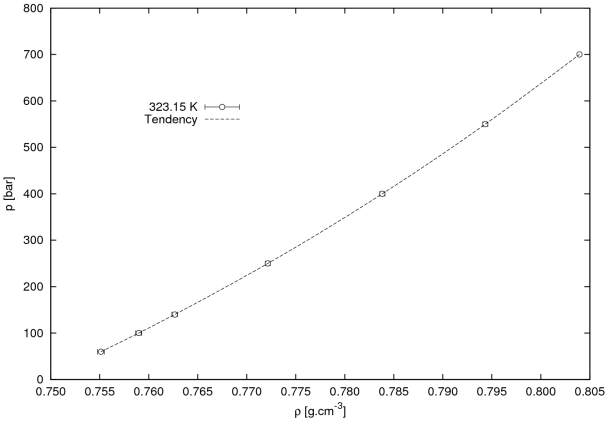

Fig. 5: Measurements on acetonitrile for illustration purposes (densities calculated from the FPMC model.)

The densities of acetonitrile are much lower than that of water and this fluid is slightly more compressible. This is a good opportunity to test the capacity of interpolation of the FPMC model.

3. Uncertainty Budgets

To obtain the propagated uncertainty in densities in the twofold aspects of eq. 1, calibration and measurements, we analyze first the individual uncertainties in the measured variables and then perform the propagation with both the standard GUM formula [10], and the Monte Carlo Method (MCM) [11, 13]. In both cases we take into account the correlations between the measured variables *(T, p, τ)* and between the parameters of the FPMC model (eq. 1). The uncertainty budget we propose is shown in Figure 6.

The measured variables *p* and *T* are affected by three types of uncertainties. The variance (**var.**) and calibration error (**cal.**) are assigned a normal distribution. The resolution (**res.**) of the instruments are assigned uniform distributions. The contributions (**var.**) and (**res.**) are also encountered in variables τ and τ_0. However (**cal.**) is not considered since the periods appear as a ratio in the model. The accuracy of the value is not important, what is important is that it should be the same for both variables. This is fulfilled since they are measured with the same instrument.

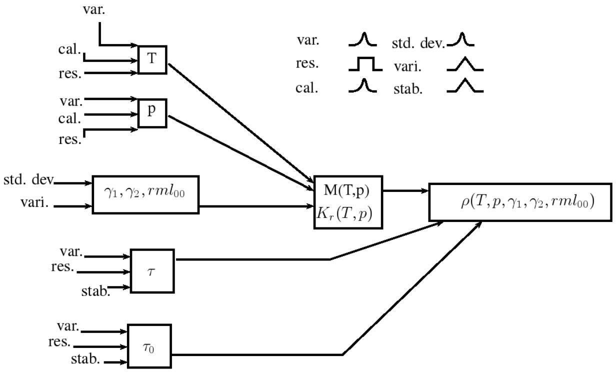

Fig. 6: Tree graph of the uncertainty contributions through the FPMC model.

Another important source of uncertainty is that due to the errors and correlations in the parameters of the model that are obtained by fitting it to the reference densities for water. The standard deviations (std. dev. In Figure 6) of each parameters and their correlation matrix are obtained asymptotically from the non linear Levenberg - Marquardt fitting algorithm, and contribute to the uncertainty in the density. The variability of these parameters due to the error in the reference equation of state for water must also be taken into account. The contribution (**vari.**) is obtained for each parameter, by fitting the model two times to two different set of reference densities. In the region where the IAPWS-95 is used, the uncertainty is less than ϵ = 0.003 % from 1 bar to 1000 bar. So we fitted the FPMC model to $\rho_{ref.}^{up} = \rho_{EoS}(1+\epsilon/100)$ and then to $\rho_{ref.}^{dwn} = \rho_{EoS}(1-\epsilon/100)$. Between these two fits, only the values of the parameters vary. Their standard deviations and correlation are maintained constant. The difference between the values of the parameters can then be used to construct a triangular distribution that will propagate in the uncertainty in density. These are the only contributions that are taken into account for the uncertainty in density during the calibration process.

When doing measurements other than calibration ones, all the previous contributions are involved. The new conditions for the stability and accuracy of *T* and *p* are evaluated again. It is not necessary to consider any uncertainty due to the reproducibility of these variables since the measurements at a given pressure and temperature are not subjected to a calibration at the same conditions (this is an important feature of the FPMC model). When the fluid under study is not water, the periods of vibrations are not the same as during the calibration at the same conditions, so there is no reproducibility problems in the values of the periods. However it is shown from various authors that the values of the periods are not reproducible in time within a given interval. This stability, represented by the triangular distribution (**stab.**) in Figure 6, is an important contribution to the uncertainty in regular measurements. These aspects are actually under study. Under careful measurements of τ_0 in time, we could determine that 12 hours after a change in the temperature of the vibrating tube, the vibrating period reach a new stable value within $2*10^{-5}$ ms from the previous one. This is the contribution to the uncertainty in τ that we consider in this work to propagate the uncertainty in the density of acetonitrile.

4 Results

4.1 Fitting of the FPMC model

The econometrics oriented program Gretl© (validated against NIST reference data set) was used to fit the FPMC model to the reference densities of water calculated at each *(T,p)* conditions for a total of 3627 data points at 14 different nominal pressures (see Figure 4). the results are summarized in Table 1.

Table 1: Parameters of the FPMC model fitted at ~323.15 K. (Reference IAPWS-95 [9])

Parameter	estimate	std. error
rml_{00} (g.cm^{-1})	0.330585	$4.51*10^{-07}$
γ_1 (bar^{-1})	$9.91201*10^{-7}$	$3.39*10^{-10}$
γ_2 (bar^{-2})	$8.58958*10^{-11}$	$9.41*10^{-13}$
Sum of the Squared Residuals: $4.14*10^{-6}$ g.cm^{-3}		
Standard Error of the regression $3.4*10^{-5}$ g.cm^{-3}		

The variability of the parameters with the error in the reference equation of state is summarized in the following Table 2:

Table 2.: Variability of the parameters of the FPMC model and combined uncertainty (~323.15 K).

		Estimate ρ_{ref}^{dwn}	**Estimate** ρ_{ref}	**Estimate** ρ_{ref}^{up}
rml_{00}		0.330575	0.330585	0.330595
γ_1		$9.912*10^{-7}$	$9.912*10^{-7}$	$9.912*10^{-7}$
γ_2		$8.5894*10^{-7}$	$8.5896*10^{-7}$	$8.5893*10^{-11}$
Difference: d		$d/\sqrt{24} = u_1$	Std. Err. $= u_2$	$u_c=\sqrt{(u_1^2+u_2^2)}$
rml_{00}	$2*10^{-5}$	$4.082*10^{-6}$	$4.51*10^{-7}$	$\mathbf{4.11*10^{-6}}$
γ_1	0.0	0.0	$3.39*10^{-10}$	$3.39*10^{-10}$
γ_2	~0.0	~0.0	$9.41*10^{-13}$	$9.41*10^{-13}$

The correlation and covariance matrix between the parameters were obtained, and are given in Table 3:

Table 3.: Correlation and Variance – Covariance Matrix of the FPMC parameters (~323.15 K).

Correlation

rml_{00}	γ_1	γ_2	
1.0	0.85772	-0.75148	rml_{00}
	1.0	-0.96723	γ_1
		1.0	γ_2

Variance - Covariance

rml_{00}	γ_1	γ_2	
$2.04*10^{-13}$	$1.31*10^{-16}$	$-3.19*10^{-19}$	rml_{00}
	$1.15*10^{-19}$	$-3.09*10^{-22}$	γ_1
		$8.86*10^{-25}$	γ_2

4.2 Analytical propagation

At each pressure, a table can be set to evaluate the uncertainties in each of the four measured variables *(T, p, τ, τ_0)*. In Table 4, we present only the data for the two extreme pressures at 20 and 700 bar. Each experimental situation displays its own set of uncertainty values. This makes a systematic treatment difficult. However we show that the variations in the propagated combined uncertainty from one pressure to the other are really low. To do this we used the propagation formula provided by the GUM [10] and calculated the required partial derivatives by means of the symbolic mathematical processor wxMaxima©. The use of such a program is of great help for our purpose, because it easily allows to handle the complex algebra and provides a reliable and quick way to manage the calculation with a minimum errors. The use of Maxima© for our specific purpose is described elsewhere [15, same symposium].

Table 4.: Uncertainty in the variables at two pressures (~323.15 K).

Uncert.		T (K)	P (bar)	τ (ms)	τ_0 (ms)
	Value →	49.982	690.56	4.143411	3.929362
	Distrib.				
Var.	Normal	0.001153	0.10994	$1.83*10^{-6}$	$5.76*10^{-7}$
Cal.	Normal	0.03	0.276	0.0	$3.74*10^{-6}$
Res.	Uniform	0.000288	0.0	$2.88*10^{-8}$	$2.88*10^{-8}$
u_c	Normal	0.030024	0.2973	$1.83*10^{-6}$	$3.78*10^{-6}$
Uncert.		T (K)	p (bar)	τ (ms)	τ_0 (ms)
	Value →	50.059	20.099	4.136722	3.929399
	Distrib.				
Var.	Normal	0.005224	0.0176	$1.56*10^{-6}$	$2.61*10^{-6}$
Cal.	Normal	0.03	0.008	0.0	$3.74*10^{-6}$
Res.	Uniform	0.000288	0.0	$2.88*10^{-8}$	$2.88*10^{-8}$
u_c	Normal	0.03045	0.0193	$1.56*10^{-6}$	$4.56*10^{-6}$

Based on a total of eight experiments as those reported in Table 4, at (~20, ~100, ~200, ~300, ~400, ~500, ~600 and ~700) *bar*, the combined uncertainties were calculated for both the non correlated and correlated variables situations. The results are shown in Figure 7.

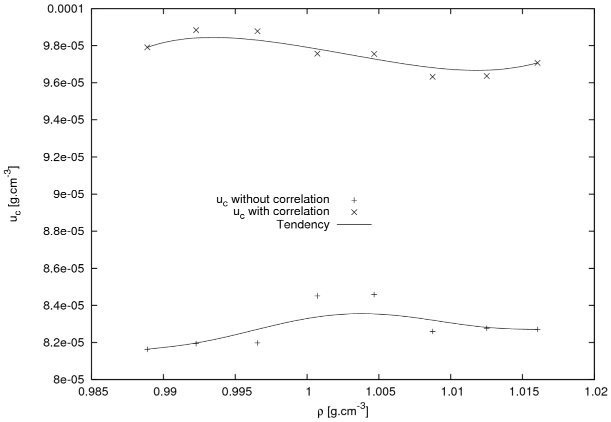

Fig. 7: Combined uncertainty at 8 pressures from the GUM propagation formulas.

The combined uncertainty has no clear tendency with pressure along the near isotherm. The effect of the correlation of the variables is noticeable and changes the value of u_c from about 0.000085 g.cm^{-3} to 0.000098 g.cm^{-3}. As shown in Figure 8, the first value is fully consistent with the amplitude of the residuals. The second value, taking into account the correlations in the variables is closer to the maximum extent of these residuals (e.g. it takes into account the irreproducibility in the calibration measurements).

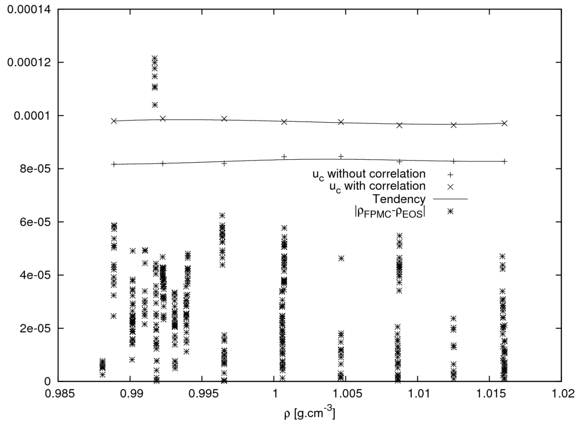

Fig. 8: Absolute residuals of the FPMC fit on water reference densities.

Figure 9 shows the simulated uncertainties obtained from the correlated variables and assigning the (**stab.**) component of the uncertainty in the periods of vibrations.

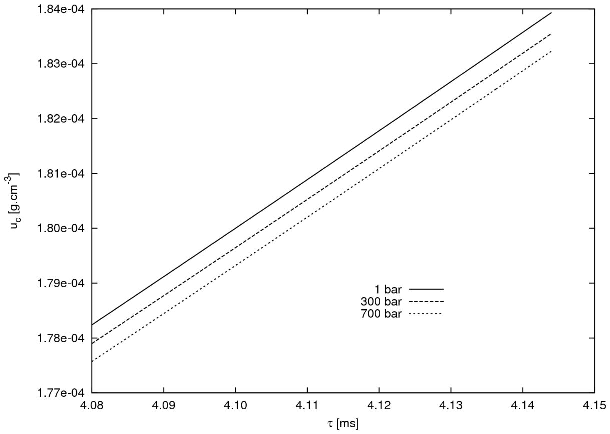

Fig. 9: Combined uncertainty with correlated variables and the influence of the stability in τ_0, as a function of pressure and τ a 323.139 K.

A slight decreasing dependency with pressure is observed at constant values of τ. However, it should be reminded that the period decreases also with pressure. So it is better to read Figure 9 along horizontals for a given fluid. For instance, for a given fluid such as acetonitrile for which the period lay between 4.08 to 4.11 *ms* in the pressure range (60, 700) *bar*, the uncertainty in the density should be in the range (0.000179, 0.000180) $g.cm^{-3}$. We check this hypothesis and compare the densities obtained from our measurements on acetonitrile with the equation of state for this fluid [14]. The results are shown in Figure 10.

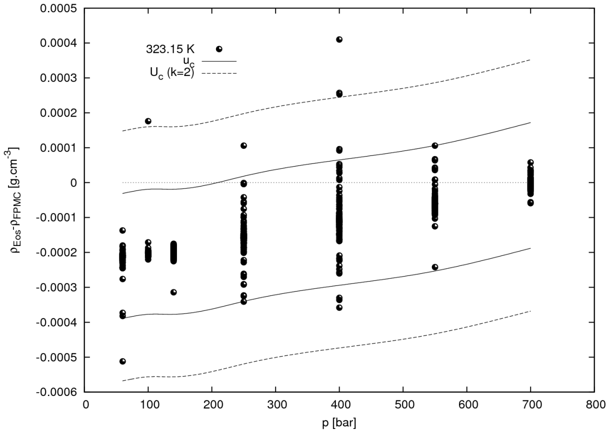

Fig. 10: Comparison of the densities of acetonitrile with an EoS [14], nivel of uncertainty.

4.3 Monte Carlo Simulations

The use of the standard propagation formula from the GUM [10], implies, in this case as in many others, a huge amount of calculations. Tests have been made with Maxima to evaluate the eventual influences of second order corrections in the propagation formula. This involves the calculation of the (7,7) Hessian matrix and another (7,7) subset of the third order tensor of the third order partial derivatives of the model with respect to the seven variables $(T, p, \tau, \tau_0, \gamma_1, \gamma_2, rml_{00})$. At 700 *bar* the result was 6.72*10^{-17} $g.cm^{-3}$ and at 20 *bar*, 2.90*10^{-18} $g.cm^{-3}$. There is a great deal of effort to assess whether the second or higher order of the Taylor series are contributing to the final uncertainty. Fortunately a new approach, using Monte Carlo simulations for the the propagation of uncertainty according has been published in the suplement 1 of the GUM [11]. The code for Matlab® was published recently [13] and can be used directly in Octave© which has been done in this work. To generate distributions of correlated variables we applied the following sequence that can be adapted directly to the existing code. 1) generate normalized distributions for each variable and parameter in the model, 2) The variance – covariance matrix is defined from the previous analysis for the set of variables, 3) obtain the Cholesky decomposition of this matrix, 4) generate the correlated distributions of the variables with desired coefficients of correlation by means of the Cholesky transformation, 5) finally recenter these distributions around the desired mean values.

We performed the simulations at 4 pressures; (20, 300, 500 and 700) *bar*. The results are shown in Figure 11.

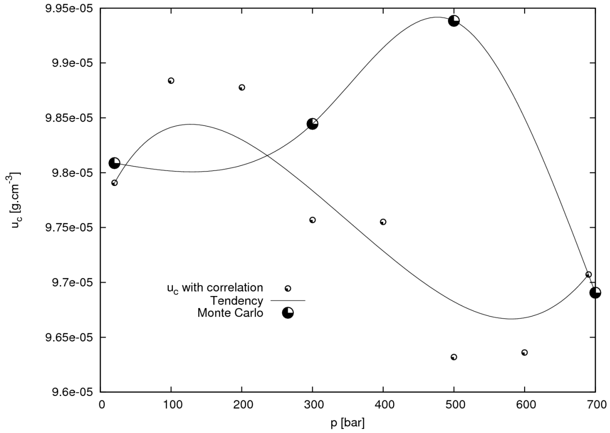

Fig. 11: Comparison between Monte Carlo simulations and analytical calculations for the combines uncertainty in the density at 323 K.

The results obtained from the Monte Carlo simulations compare favorably with the analytical method. They were obtained (with a stable 95 % confidence interval) in little less than 10 *s* occupying the order of 109 draws of 10^5 data, using an AMD-Athlon®X2, 64 bits processor. The advantage of the Monte Carlo method, apart from the fact that it does not require tedious algebraic calculus, is that it also provides the distribution of these uncertainties and the confidence interval. In this case no difference with a normal distribution was observed in the 4 numerical experiments. All of these experiments have terminated with a numerical precision of 10^{-6} which is sufficient to validate the results.

5 Conclusions

This study shows that the FPMC model, for the calibration and measurements of density by means of the widely used vibrating tube densimeters, provides a combined uncertainty in the state region of the compressed liquids (in this case in the range of density (0.75, 1.01) $g.cm^{-3}$) of little less than

0.0001 $g.cm^{-3}$ during calibrations and about 0.000185 $g.cm^{-3}$ in the case of measurements. With a coverage factor of 2 (95.4 % confidence interval), which is commonly used for this kind of measurements, the expanded uncertainty reaches about 0.00037 $g.cm^{-3}$, which is well within the uncertainty reported by various authors. Both the analytical method and Monte Carlo simulations have been used as suggested by the GUM and its supplement. The Monte Carlo simulations perfectly validate the analytical method.

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