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# Uncertainty comparison of viscosity measurements of $CO_2$ loaded MEA and water mixtures in a coaxial rheometer using Monte Carlo simulation and GUM method

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**Abstract**
Evaluation of measurement uncertainty is vital in the measurement of physicochemical properties. The uncertainty of viscosity measurement of a mixture of monoethanol amine (MEA), water and $CO_2$ is evaluated according to the Guide to the expression of Uncertainty in Measurement (GUM) and validated using Monte Carlo Simulation (MCS) method. This helps to estimate the truncation error due to the first order approximation of Taylor series on nonlinear models in GUM. In literature, only one method is normally used. Calculated uncertainty according to GUM for $CO_2$ loaded aqueous MEA is 0.035 mPa·s. For the uncertainty of viscosity in unloaded aqueous MEA solutions, the confidence interval calculated by GUM deviates from calculated confidence interval according to MCS. This deviation is beyond the numerical tolerance defined for the comparison. The probability distributions of the uncertainty sources influence the distribution of the model output in the MCS method. For the uncertainty of viscosity in $CO_2$ loaded aqueous MEA solutions, the confidence interval calculated by GUM is within the defined numerical tolerance and closer to the calculated confidence interval according to MCS. Combining GUM and MCS will improve confidence in the uncertainty evaluation.


**Keywords:** Viscosity; Uncertainty; monoethanol amine; GUM; MCS.

## 1. Introduction
Viscosity measurements of alkanolamines are intensively carried out in the field of amine based post combustion $CO_2$ capture. Various alkanolamines are tested for their performance to capture $CO_2$ in the form of rate of mass transfer and absorption capacity. Viscosity data in both $CO_2$ loaded and unloaded alkanolamines are also significant since it is needed in designing process equipment like absorption and desorption columns, heat exchangers, pumps and useful for correlating mass transfer.
The accuracy of viscosity measurements depends on many factors starting from sample preparation to the measuring instrument. Many mathematical models that have been developed to determine mass transfer coefficients and interfacial area use viscosity data in their correlations. Thus, the accuracy of the design parameters such as packed bed height and pressure drop depends on the accuracy of viscosity measurements. The information about measurement uncertainty of physical properties influences the safety margins in such a system [1].

The uncertainty of viscosity measurement characterizes a range that the measured viscosity could occupy in a considered measuring technique. Currently, different types of rheometers are available to measure liquid viscosities; the uncertainty of each method should be evaluated separately. Evaluating measurement uncertainty is considered a difficult task [2]. Defining uncertainty for the viscosity measurements allows making various decisions in different phases such as plant design and mathematical modelling and simulations. Consequently, it is vital to evaluate measurement uncertainty precisely to evaluate possible fluctuations in the results [3, 4]. For analytical chemistry, a separate document was published called QUAM (Quantifying uncertainty in analytical measurement) [5] following the principles given from GUM (Guide to the expression of uncertainty in measurement). In the GUM uncertainty framework, propagation of uncertainty is concerned with [6] and output is characterized by a Gaussian distribution or scaled and shifted t-distribution to define an appropriate coverage interval. Monte Carlo simulation (MCS) is an alternative approach for the uncertainty evaluation in which the propagation of distributions is estimated by performing random sampling from probability distributions [7].
MCS is a useful technique to validate the results obtained through the GUM. There are circumstances that GUM is not applicable where the linearized model does not give sufficient information and the probability density function (PDF) of the output quantity deviate from a Gaussian distribution or a scaled and shifted t-distribution. Several attempts have been made to compare the output of both the GUM and MCS methods to evaluate measurement uncertainty of various physical parameters. Jalid et al [8] compared both the GUM and the MCS method to estimate measurement uncertainty associated with the flatness error. A study on measurement uncertainty of indirect measurements was done by Sediva and Havlikova [9]; uncertainty was compared according to both GUM and MCS. Uncertainty evaluation and comparison on perspiration measurement system were done by Andrew and Chiachung [10]. Evaluated uncertainty by GUM is smaller than MCS and no significant difference observed considering the precision at two decimal points.
Sumudu et al. [11] discussed a detailed measurement uncertainty analysis of viscosity for unloaded aqueous MEA solutions using the GUM framework. This study extended the uncertainty analysis for the $CO_2$ loaded aqueous MEA mixtures. There, sources that contribute to the measurement uncertainty in a mixture of monoethanol amine, water (both unloaded and loaded with $CO_2$) using a coaxial cylinder type rheometer is discussed. Further, a comparison of both GUM and MCS methods on viscosity evaluation was studied for the best estimate. All the simulations were performed in the MATLAB R2017a environment and inbuilt random number generators were used for the sampling from PDF.

## 2. Methods of uncertainty evaluation

### *2.1 Uncertainty evaluation in GUM*

The International Organization for Standardization (ISO) made the first publication of GUM in 1993. The Joint Committee for Guides in Metrology (JCGM) republished the GUM in 2008 with several additional documents including supplements related to measurement uncertainty [3]. GUM describes two types (Type A and Type B) of uncertainty evaluations. In type A, uncertainty is evaluated from the statistical distribution obtained through results of series of measurements while type B evaluate uncertainty through probability density functions (PDF) based on experience or other information [7].
The propagation of uncertainty based on the first order Taylor series approximation is the main aspect in GUM uncertainty evaluation. In a measuring system, inputs and outputs are combined through a functional relationship.

$$y = f(x_1, x_2, \dots, x_N) \tag{1}$$

Where $y$ is the measurand and $x_1, x_2, \dots, x_N$ are input quantities. The propagation of uncertainty according to the Taylor series expansion of $y$,

$$u^2(y) = \sum_{i=1}^{N} \left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i) + 2\sum_{i=1}^{N-1}\sum_{j=i+1}^{N} \frac{\partial f}{\partial x_i}\frac{\partial f}{\partial x_j} u(x_i, x_j) \tag{2}$$

In Eq (2), $\left(\frac{\partial f}{\partial x_i}\right)$ gives the partial derivatives (sensitivity coefficients) [12, 13], $u^2(y)$ is the variance of the measuring result, the variance of the input quantity $x_i$ is given by $u^2(x_i)$ and the covariance between $x_i$ and $x_j$ is given by $u(x_i, x_j)$ [13].

 

The functional relation (*f*) represent the uncertainty sources involved with the measuring system. It defines both physical laws and the measurement process in which it provides correlations for both systematic effects and other variable sources like instruments, laboratories, samples, different observers and times that the observations are made [12].
Measurement uncertainty is presented as a confidence interval, which explains what percentage (%) of measured data lies within the considered range. The relation of expanded and standard uncertainties are correlated by a factor $k$ in such a way that

$$U(y) = ku(y) \tag{3}$$

$k$ is known as the coverage factor; $k$=1.96 for 95% confidence level for normally distributed measurements [4, 14].

*2.2 Uncertainty evaluation in MCS*
MCS is a numerical approach with a random sampling technique and is applied in many scientific and engineering applications. Sampling is done from the PDFs for inputs ($x_i$) to evaluate the output (y) quantity. MCS discuss the propagation of distribution in which probability distributions of input quantities propagate through a model to provide the distribution of the output [6]. Figure 1 illustrates the propagation of density functions $g_{x_i}(\xi_i)$, $i$=1,…,$N$, of inputs through a model to provide the propagation of density function $g(\eta)$ for the output quantity.

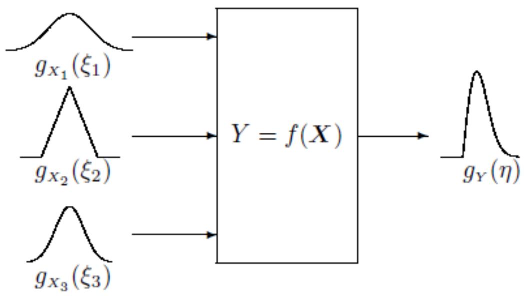


Figure 1. Propagation of distribution for three independent input variables [7].

*2.3 Comparison of GUM and MCS methods in measurement uncertainty estimation*
The evaluation of partial derivation in the GUM method of a complex model can be a difficult task. The truncation error due to the first order approximation of Taylor series on nonlinear models is a major limitation in this framework. The GUM approach assumes that the probability distribution of the output quantity is approximately a normal distribution and can be characterized by a t-distribution. The use of Welch-Satterthwaite formula to determine the effective degree of freedom, which is necessary to calculate expanded uncertainty is an unsolved problem [6]. The MCS method is capable of giving the probability distribution of the output [15], which is not given in the GUM method. In some scenarios, it is useful to have knowledge about probability distributions to understand the characteristics of the output. MCS can deal with both small and large uncertainties in the input quantities and there is no need for performing partial differentiation to evaluate sensitivity coefficients [12]. Even though it is difficult, the sensitivity coefficient derived in GUM framework conveys valuable facts to enhance the measurement performance [10]. Consequently, MCS is a good validation approach to compare the results obtained through the propagation of uncertainty through GUM.

**3. Viscosity measurements of MEA and water mixtures with uncertainty evaluation**
*3.1 Viscosity measurement in a coaxial cylindrical rheometer*
The viscosity measurements of MEA and water mixtures were carried in a coaxial cylindrical rheometer manufactured by Anton Paar. It has a double gap geometry that provide two fluid compartments with a rotating cup. It is good for the measurement of low viscous fluid with high accuracy since the probe provided high surface area between fluid and probe [16]. Figure 2 shows a schematic of double gap geometry of a coaxial cylindrical rheometer.

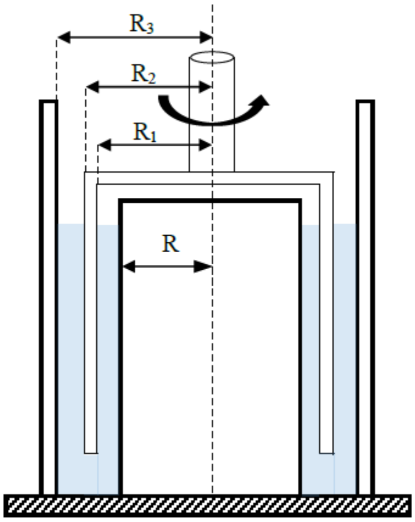


Figure 2. Schematic of double gap geometry of a coaxial cylindrical rheometer.

*3.2 Measurement model*

A mathematical relation was derived to correlate the parameters involved in the measuring system, which is also useful in the identification of uncertainty sources in viscosity measurements. Considering the conservation of momentum under cylindrical coordinates, the following expression can be obtained for the dynamic viscosity for the rheometer arrangement shown in Figure 2.

$$\mu = \frac{T}{4\pi L\omega R^2\left(\frac{k_1^2}{k_1^2-1}+\frac{k_2^2k_3^2}{k_3^2-k_2^2}\right)} \tag{4}$$

Here, $T$ is torque, $\mu$ is dynamic viscosity, $L$ is the liquid height, $R$ is the radius of the inner fixed cylinder, $\omega$ is angular velocity, $R_1 = K_1R$ , $R_2 = K_2R$ and $R_3 = K_3R$.
The schematic of the velocity profile in the liquid compartment is shown in Figure 3.

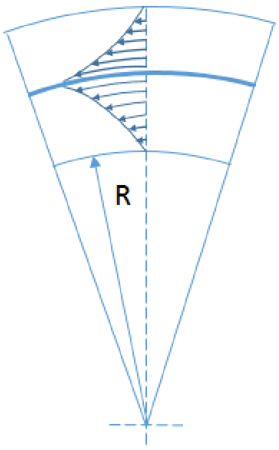


Figure 3. Velocity profile of fluid in the coaxial cylinder [11].

*3.3 Cause and effect analysis*

A cause and effect diagram is a graphical method to represent uncertainty sources in a measuring system. It describes how the uncertainty of individual sources is connected to propagate into a final measurement uncertainty. The cause and effect analysis performed for the viscosity measurement of aqueous MEA solutions was published elsewhere [11]. Figure 4 shows the cause and effect analysis performed for the viscosity measurements of aqueous MEA solutions.

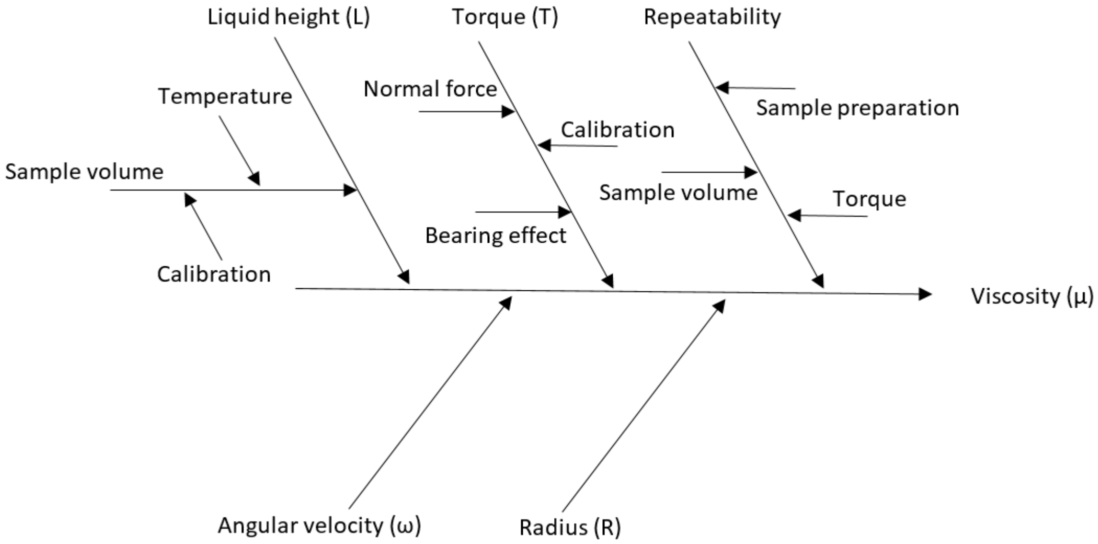


Figure 4. Cause and effect diagram for uncertainties in viscosity measurements of a MEA / water mixture [11].

*3.4 Uncertainty calculation using GUM*

The combined uncertainty of viscosity of aqueous MEA solutions was calculated according to the proposed mathematical model using GUM. The calculated expanded uncertainty for aqueous MEA solutions is 0.0162 mPa·s at $k$=2 [11]. The modified Eq (1) for the uncertainty analysis of viscosity measurement in aqueous MEA according to QUAM is shown as

$$\mu = \frac{T}{4\pi L\omega R^2\left(\frac{k_1^2}{k_1^2-1}+\frac{k_2^2k_3^2}{k_3^2-k_2^2}\right)} f_p f_t f_w f_{rep} \tag{5}$$

Where the $f_p$ is purity of MEA, $f_t$ is temperature, $f_w$ is weight measurement and $f_{rep}$ is repeatability. Those factors are added to the original viscosity expression to consider uncertainty sources, which are not shown in Eq (1).

For the viscosity of $CO_2$ loaded solutions, another factor of $f_{CO_2}$ is introduced into Eq (5) to account for the effect of $CO_2$ loading in the solution. There are various uncertainty sources involved in $CO_2$ loading in which a detailed analysis can be found in Jayarathna et al [17]. Finally, the GUM guidelines were followed to evaluate the uncertainty as described in section 2.1.

*3.5 Uncertainty calculation using MCS method*

The numerical values for the uncertainty sources and factors in the model shown in Eq (5) are considered as the random output of a PDF$gX_i(\xi_i)$. It is assumed that the input quantities are uncorrelated for both viscosity measurement of $CO_2$ loaded and unloaded scenarios. Many sources are available in literature that explains the necessary steps to follow in order to perform MCS. The Adaptive Monte Carlo Method (AMCM) describes that the number of Monte Carlo trials $M$ needs to be selected as $M = max(J, 10^4)$ where $J$ is the smallest integer greater than or equal to $100/(1-p)$ and $p$ is a coverage probability and M is the selected adaptively until the various results of interest have established in a statistical sense [18]. There, the numerical tolerance was set in such a way that $\delta = (1/2) \cdot 10^l$. The MCS method discussed here was performed considering the non-adaptive approach as described in JCGM 101:2008 [7].

The validation of the GUM uncertainty framework using MCS was performed to verify that both methods provide results to agree within a stipulated numerical tolerance. The comparison of coverage intervals obtained by both methods is performed as shown in Eq (6).

$$d_{low} = [y - U_p - y_{low}]$$
$$d_{high} = [y + U_p - y_{high}] \tag{6}$$

When both absolute differences $d_{low}$ and $d_{high}$ no greater than $\delta$, the comparison is considered to be favorable and the GUM uncertainty framework is validated in this instance [7].

## 4. Results and discussion

The uncertainty evaluation of viscosity for unloaded aqueous MEA solutions was presented in a previous study [11]. It was found that 0.0159 mPa·s under $k$ =1.96 for combined expanded uncertainty, as it is the most appropriate coverage factor for the 95% confidence interval. A similar methodology was applied for the $CO_2$ loaded aqueous MEA solutions in which Eq (7) was used for the uncertainty analysis in GUM.

$$\mu = \frac{T}{4\pi L\omega R^2\left(\frac{k_1^2}{k_1^2-1}+\frac{k_2^2k_3^2}{k_3^2-k_2^2}\right)} f_p f_t f_w f_{rep} f_{CO_2} \tag{7}$$

The combined standard uncertainty can now be found through the Tayler expansion as shown in section 2.1. The partial derivatives $\partial\mu/\partial x_i$ were obtained and listed as follows. Based on this the combined standard uncertainty of viscosity for the $CO_2$ loaded aqueous MEA solutions was calculated using the expression shown in Eq (17).

For better overview $k = \frac{k_1^2}{k_1^2-1} + \frac{k_2^2k_3^2}{k_3^2-k_2^2}$

$$\frac{\partial\mu}{\partial T} = \frac{1}{4\pi L\omega R^2 k} f_p f_t f_w f_{rep} f_{CO_2} \tag{8}$$

$$\frac{\partial\mu}{\partial L} = \frac{T}{4\pi\omega R^2 k} f_p f_t f_w f_{rep} f_{CO_2}\left(\frac{-1}{L^2}\right) \tag{9}$$

$$\frac{\partial\mu}{\partial \omega} = \frac{T}{4\pi L R^2 k} f_p f_t f_w f_{rep} f_{CO_2}\left(\frac{-1}{\omega^2}\right) \tag{10}$$

$$\frac{\partial\mu}{\partial R} = \frac{T}{4\pi L\omega k} f_p f_t f_w f_{rep} f_{CO_2}\left(\frac{-2}{R^3}\right) \tag{11}$$

$$\frac{\partial\mu}{\partial f_p} = \frac{T}{4\pi L\omega R^2 k} f_t f_w f_{rep} f_{CO_2} \tag{12}$$

$$\frac{\partial\mu}{\partial f_t} = \frac{T}{4\pi L\omega R^2 k} f_p f_w f_{rep} f_{CO_2} \tag{13}$$

$$\frac{\partial\mu}{\partial f_w} = \frac{T}{4\pi L\omega R^2 k} f_p f_t f_{rep} f_{CO_2} \tag{14}$$

$$\frac{\partial\mu}{\partial f_{rep}} = \frac{T}{4\pi L\omega R^2 k} f_p f_t f_w f_{CO_2} \tag{15}$$

$$\frac{\partial\mu}{\partial f_{CO_2}} = \frac{T}{4\pi L\omega R^2 k} f_p f_t f_w f_{rep} \tag{16}$$

$$u_c(\mu)_{CO_2\,loaded} = \sqrt{\begin{matrix}\left[\frac{\partial\mu}{\partial T}u(T)\right]^2 + \left[\frac{\partial\mu}{\partial\omega}u(\omega)\right]^2 + \left[\frac{\partial\mu}{\partial L}u(L)\right]^2 + \left[\frac{\partial\mu}{\partial R}u(R)\right]^2 + \left[\frac{\partial\mu}{\partial f_p}u(f_p)\right]^2 \\ + \left[\frac{\partial\mu}{\partial f_t}u(f_t)\right]^2 + \left[\frac{\partial\mu}{\partial f_w}u(f_w)\right]^2 + \left[\frac{\partial\mu}{\partial f_{rep}}u(f_{rep})\right]^2 + \left[\frac{\partial\mu}{\partial f_{CO_2}}u(f_{CO_2})\right]^2\end{matrix}} \tag{17}$$

The calculated uncertainty sources and probability distributions are summarized in Table 1. Most of the distributions are selected according to the guidelines provided in QUAM. The uncertainty of $CO_2$ loading was considered as 1.3% according to the study carried by Jayarathna et al. [17]. The cause and effect diagram shown in Figure 4 illustrates how the uncertainty sources contribute to the combined uncertainty. For $CO_2$ loaded MEA solutions, the uncertainty of $CO_2$ concentration measurements conveys a significant impact on the uncertainty of viscosity compared to unloaded solutions.

 

Table 1. Uncertainty sources and probability distributions.

| Input quantity $X_i$ | Probability Distribution | Uncertainty $U(x_i)$ |
|---|---|---|
| Torque $(T)$ | Triangular | 0.082 $\mu Nm$ |
| Level $(L)$ | Gaussian | 0.45 $mm$ |
| Angular velocity $(\omega)$ | Triangular | 0.01 $rad \cdot s^{-1}$ |
| Radius $(R)$ | Triangular | 4.1 $\mu m$ |
| Purity | Rectangular | 2.886x$10^{-3}$ |
| Temperature | Triangular | 2.45 x$10^{-4}$ |
| Weight measurement | Rectangular | 8 x$10^{-6}$ |
| $CO_2$ loading | Gaussian | 0.013 |
| Repeatability | Gaussian | 0.00348 |

The Kragten's approach [5] described a way to perform uncertainty calculations according to the GUM uncertainty framework without evaluating partial derivatives. The expression to estimate standard uncertainty according to GUM is shown in Eq (17). Calculated expanded uncertainty for a $CO_2$ loaded viscosity measurement, $U(\mu)_{CO_2\,loaded}$ at $k = 1.96$ is 0.0346 mPa·s.

In the MCS method, the uncertainty of a viscosity measurement in $CO_2$ unloaded aqueous MEA solutions was evaluated according to the method illustrated in section 3.5. There, $n_{dig}$=2 and $u_c(\mu)$can be expressed as $81 \times 10^{-7}$, and so $c = 81$ and $l = -7$. In the application of the MCS method, a coverage probability $p$ is set to 0.95. It is often considered a value of $M = 10^6$ for providing a confidence interval of 95% and $M$ at least $10^4$ times greater than$1/(1-p)$ [7, 19]. The estimated $y$ values were sorted in non-descending order to determine the boundaries of the confidence intervals. A Gaussian distribution was assumed for the GUM uncertainty evaluation and the PDF from both the GUM and MCS method were compared in Figure 5. The dashed and vertical full line illustrates the 95% coverage intervals determined by MCS and GUM respectively.

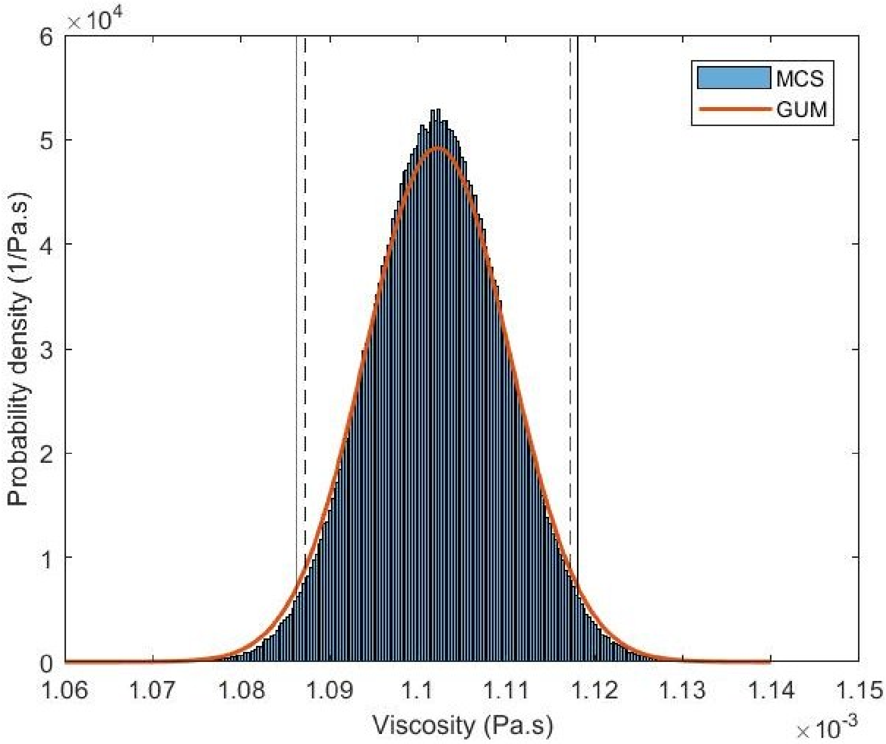


Figure 5. Probability density distribution of unloaded aqueous MEA viscosity from GUM and MCS method, ' __' GUM, '---' MCS.

$d_{low}$ and $d_{high}$ were determined to validate the GUM according to the expression shown in Eq (6). The calculated $y_{low}$ and $y_{high}$ from cumulated probability distribution is shown in Table 2. In this scenario, GUM is not validated since both endpoints of the coverage interval does not satisfy the condition with numerical tolerance $\delta$.

Table 2. Uncertainty evaluation results for unloaded solutions.

| Method | $M$ | $\mu_{unloaded}$ (mPa·s) | $U(\mu)_{unloaded}$ (mPa·s) | Probabilistically symmetric 95% coverage interval | $d_{low}$ | $d_{high}$ |
|---|---|---|---|---|---|---|
| GUM | | 1.1022 | 0.0081 (0.0159) | (0.0010863, 0.0011181) | - | - |
| MCS | $10^6$ | 1.1022 | 0.0077 (0.0150) | (0.0010872, 0.0011173) | $9.89 \times 10^{-07}$ | $7.16 \times 10^{-07}$ |

The MCS method for $CO_2$ loaded aqueous MEA solutions considered the uncertainty of $CO_2$ loadings as proposed by Jayarathna et al. [17]. In the simulation, $u(f_{CO_2})$ = 0.013 considered with Gaussian distribution and all other uncertainty sources were considered to be the same as in the previous scenario. The relevant parameters considered in the simulation are listed in Table 3.

Table 3. Numerical parameters in MCS for $CO_2$ loaded solutions.

| Parameter | Value |
|---|---|
| $n_{dig}$ | 2 |
| $c$ | -6 |
| $l$ | 17 |
| $M$ | $10^6$ |

In order to validate GUM, $d_{low}$ and $d_{high}$ were determined as shown in Eq (6) and PDF from both GUM and MCS method were compared in Figure 6. A Gaussian distribution was assumed for the GUM uncertainty evaluation as considered in the previous scenario. All the required parameters for the validation of GUM for the uncertainty of viscosity measurement of $CO_2$ loaded MEA solutions are listed in Table 4.

Table 4. Uncertainty evaluation results for $CO_2$ loaded solutions.

| Method | $M$ | $\mu_{CO_2\ loaded}$ (mPa·s) | $U(\mu)_{CO_2\ loaded}$ (mPa·s) | Probabilistically symmetric 95% coverage interval | $d_{low}$ | $d_{high}$ |
|---|---|---|---|---|---|---|
| GUM | | 1.1814 | 0.0176 (0.0345) | (0.0011468, 0.0012160) | - | - |
| MCS | $10^6$ | 1.1813 | 0.0174 (0.0341) | (0.0011472, 0.0012155) | $4.75 \times 10^{-07}$ | $2.79 \times 10^{-07}$ |

As in the previous scenario, the validation was done by analyzing $d_{low}$ and $d_{high}$ for the uncertainty of $CO_2$ loaded solution. The calculated $y_{low}$ and $y_{high}$ from cumulated probability distribution is shown in Table 4. The calculated numerical tolerance $\delta$ for this scenario satisfies the condition shown in Eq (6) for both endpoints of the confidence intervals. Consequently, GUM is validated for the uncertainty of viscosity measurement of $CO_2$ loaded solutions.

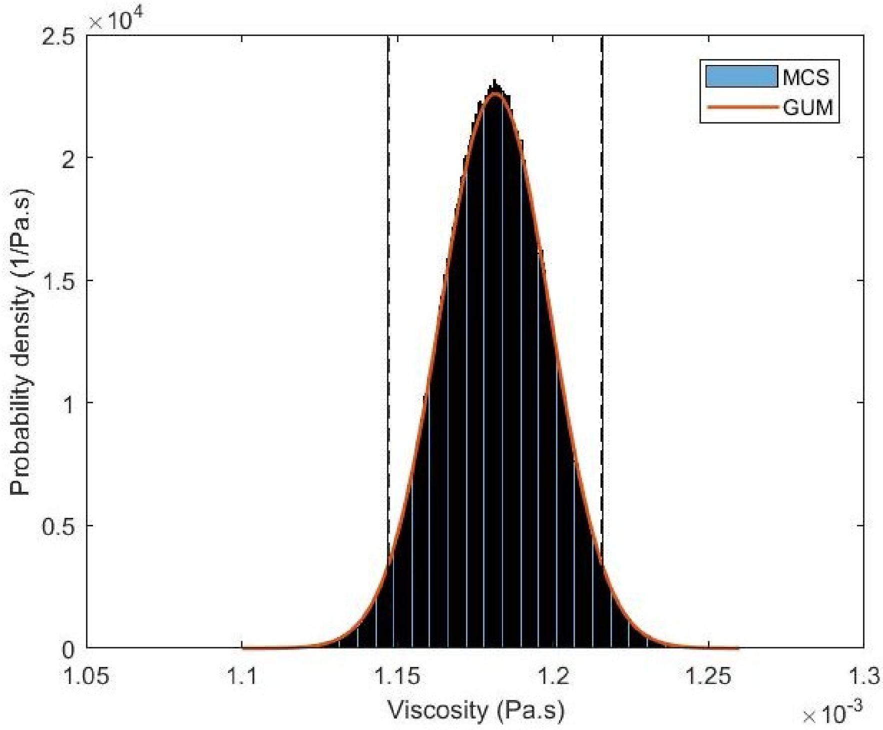


Figure 6. Probability density distribution of $CO_2$ loaded aqueous MEA viscosity from GUM and MCS method, ‘ __’ GUM, ‘---’ MCS.

## 5. Conclusion

This study performs an uncertainty evaluation of viscosity measurement in a coaxial rheometer of $CO_2$ loaded MEA solutions according to GUM. The uncertainty of a viscosity measurement performed for unloaded solutions has been discussed in a previous study was used by modifying the model equation according to QUAM guidelines for the new scenario. The calculated uncertainty for $CO_2$ loaded MEA solutions is in good agreement with uncertainties reported in the literature.

For the $CO_2$ unloaded solutions, the comparison of GUM by the MCS method reveals that endpoints of the two coverage intervals do not satisfy the condition with numerical tolerance $\delta$. As a result, the GUM is not validated for this scenario. This can be due to various reasons including model nonlinearity, covariation of parameters involved in the model and nature of selected uncertainty distributions for the uncertainty sources. For the $CO_2$ loaded solutions, the numerical tolerance satisfied the conditions for the GUM validation. The numerical tolerance fulfilled the condition defined in the JCGM 101:2008.

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