World Applied Sciences Journal 33 (11): 1740-1745, 2015
ISSN 1818-4952

DOI: 10.5829/idosi.wasj.2015.33.11.15618

Source of Uncertainties in Uncertainty Estimation of Analytical Balance and Volumetric Glassware Calibration

M.A. Rahman, M. Afroze, R.M. Mazumdar, M. Moniruzzaman, M.T.H. Bhuiyan, M.H. Razu and Mala Khan

Designated Reference Institute for Chemical Measurements (DRiCM),
Bangladesh Council of Scientific and Industrial Research (BCSIR),
Dr. Qudrat-I-Khuda Road, Dhanmondi, Dhaka-1205, Bangladesh

Abstract: Measurement of uncertainty is a major concern in all fields of measurements. Since weighing is a very important in chemical laboratory and analytical methods, it has played an important source of uncertainty in uncertainty measurements in an analysis. In this paper we attempted to present the influencing factors in uncertainty measurement that affect mass and volume determination. Technical specification of an analytical balance such as: readability, repeatability, linearity, off-center loading and hysteresis and for volumetric glassware: repeatability, readability, temperature coefficient of sensitivity, temperature scattering, meniscus reading and environmental conditions (temperature and humidity) were considered. Measurement of uncertainty should be performed in the absence of some disturbing issues for instance: external magnetic field, electrostatic field and evaporation.

Key words: Weighing · Measurement Uncertainty · Linearity · Sensitivity coefficient

INTRODUCTION

All research activities, especially in chemical laboratory, start from weighing of different sorts of samples. If someone starts work with wrong measurement then whatever he/she gain ultimate result will be wrong. So, for every laboratory, the results from analytical balances or other measuring devices (volumetric glassware) are of critically important for the next steps of analysis as it determines the acceptability of products or the outcome of a test.

Weighing data are associated with some uncertainty [1], as this is common with all other procedures and their data. Also all manufacturer specifications are based on idealized conditions. Otherwise, comparisons could not be done between various instruments because the methods actually used in the field often differ from those used by the manufacturer. Hence it is important to have procedures for assuring the quality of these weighing devices in every laboratory conditions.

Many influences affect the accuracy and precision of weighing results. These influences provide a bias, an average value different from zero and contribution to uncertainty. Therefore, estimating the uncertainty in measurements [2] has become a key requirement when aiming to obtain a laboratory accreditation. Uncertainty measurements are very important for quality control (QC), quality assurance (QA) and Inter Laboratory Comparison (ILC) programs.

MATERIALS AND METHODS

Analytical balance (maximum load of 500 g), Calibration weight: E_1, E_2, F_1, F_2 [3] or NIST Standard, Glassware (Class A and Class B), Barometer, Digital Thermometer/ readout (at least two inputs), humidity measuring instrument.

Methods and Discussion: In our present study we have sorted out all possible areas and facts that might affect measurement uncertainty for both balance and volumetric

Corresponding Author: M.A. Rahman, Designated Reference Institute for Chemical Measurements (DRiCM), Bangladesh Council of Scientific and Industrial Research (BCSIR), Dr. Qudrat-I-Khuda Road, Dhanmondi, Dhaka-1205, Bangladesh.

glassware. To calculate uncertainty, a series of repeated observations were carried out and their effect was evaluated. The influences that affect accuracy and precision of weighing results can be divided into three categories. Influences originate from balance (such as readability, repeatability, departure from nominal value, off center loading and hysteresis), influences due to ambient conditions (such as: air humidity, air temperature, air pressure, heat radiation and direct sunlight) and at last but not least, influences from physical condition of weighing object (i.e., air buoyancy).

Static electricity exerts a mechanical force which is readily detectable by analytical and microbalances. An example of static electricity exerting a mechanical force would be lint sticking to clothing. Static will be a problem when it exists on the object being weighed, on the person using the balance, on draft shields, or on weighing vessels. Sources of static are carpets, vibrum shoe soles, plastic draft shields, plastic weighing vessels and melamine (Formica) table tops. Low ambient humidity exacerbates static problems.

All the measurements have error that trouble the accuracy and create uncertainty about the quality of the measured mass. "A parameter associated with the results of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand". The uncertainty reveals the lack of exact knowledge of the value of the measured values owing to random and systematic effects.

Uncertainty designates a general concept; its quantitative measure is called standard uncertainty. The standard uncertainty is an estimate of the standard deviation, the positive square root of the variance, of the probability distribution of the possible values of the measured values. We described possible areas of type-A uncertainties first then type-B uncertainties and calculated all parameters and their interplay to the combined standard uncertainty for an analytical balance and volumetric glassware below [4].

Type A Uncertainty: Type A evaluations of uncertainty are done by calculations from a series of repeated observations, using statistical methods.

A.1 Repeatability of Measurement: Repeatability of measurement is very important because the measurement conditions - power dissipated by electronic circuits, wind draft at the site of the balance, vibrations of the support on which the balance stands, temperature differences, pressure fluctuations of the ambient air could be changed and therefore it could deviates the results when the same object is weighing repeatedly. In addition to the alignment of meniscus level with the mark induces deviation to the measured results for volumetric glassware.

Uncertainty from repeatability measurement is called type A uncertainty. The standard uncertainty due to repeatability (U_{rep}) is normally evaluated as the simple standard deviation of the balance readings for *n* successive loadings of a same weight [5].

$$\sigma = \text{Standard deviation for n repeated readings} = \sqrt{\frac{\sum(X_i - \bar{x})^2}{(n-1)}} \quad (1)$$

where X_i is the ith balance reading and $\bar{x}$ is the average of the n balance readings. To arrive at the random uncertainty of the population, the standard deviation of the mean has to be evaluated. This is obtained by dividing the standard deviation of the sample by $\sqrt{n}$.

$$\text{Standard deviation of the mean} = \frac{\sigma}{\sqrt{n}} \quad (2)$$

This is the type 'A' uncertainty from Balance readings or

$$\text{Random Uncertainty } U_{repB} = \frac{\sigma}{\sqrt{n}} \quad (3)$$

A.2 Temperature Scattering: Sensitivity of balance and volume of volumetric glassware is affected by temperature. It plays major rules during the calibration of volumetric glassware. Because, glass material expand or shrink against small change in temperature and different types of glass material have different expansion coefficient. So, uncertainty due to temperature scattering throughout the complete measurement process should be taken into account. Temperature scattering is calculated by measuring room temperature for every thirty or sixty seconds throughout the whole calibration process and then using equation 1, 2 and 3 uncertainty of temperature scattering (U_{repT}) can be measured.

Type B Uncertainty: While considering the various contributory factors towards type B uncertainties (other than statistical data), one has to analyze the calibration process itself, if necessary by breaking it down into a number of small processes and studying the influences that are likely to affect the process. Type B evaluations of uncertainty are derived from other sources

e.g. from calibration data (Given by the manufacturer). Generally, type B factors may include but are not limited to: the reference standard, the UUC (Unit Under Calibration) itself, other supporting instruments involved in the calibration process, personnel carrying out the calibration and environmental factors such as temperature, pressure, humidity, air density, local gravitational acceleration (g) value, etc. Any possible interaction other then the above factors may also be taken into account.

Generally, the following factors should be included in type 'B' evaluation [5].

Readability: Readability (resolution), important in uncertainty measurement, is a measure of an instrument's ability to display the exact value of the measurand. Since, different types of balances (i.e. microbalance, analytical balance and precession balance) have different technical limitation and most of the cases last digit of balance output is unstable. Therefore, it round up the last digit value inherently and ultimately add some error to the output values. This uncertainty is, measured from the balance resolution, plus-minus half of the balance resolution.

That is, half resolution,

$$R_{1/2} = \frac{\text{Resolution of Balance}}{2} \quad (4)$$

Readability is a bias and thus with rectangular distribution its standard uncertainty can be approximated as:

$$U_{readB} = \pm \frac{R_{1/2}}{\sqrt{3}} \quad (5)$$

B.2 Temperature Coefficient: The temperature coefficient is the relative change of a physical property when the temperature is changed by 1 K. The weighing transducer of the balance may exhibit a temperature dependent characteristic. If a weight is divided by the change in temperature, this will yield the value of the temperature coefficient [6]. This influence is expressed by the (sensitivity) temperature coefficient (TC) of the balance and is defined as the ratio of relative change of sensitivity $\Delta S/S_0$ and the temperature difference ΔT:

$$TC = \left(\frac{\Delta S}{S_0}\right) / \Delta T \quad (6)$$

The TC of sensitivity only states how sensitive the slope of the balance's characteristic curve reacts to temperature. As earlier mentioned S_0 for a correctly adjusted balance is 1. Variance of TC is a rectangular distribution and can be obtained as follows:

$$U_{tc} = \left(\frac{1}{\sqrt{3}}.TC.\Delta T.W_{net}\right) \quad (7)$$

Temperature coefficient (TC) is obtained from balance specification and its unit is ppm/°C or g/g/°C. ΔT is temperature difference in balance room.

B.3 Deviation of Reference Standard: Uncertainty contribution due to deviation of the reference standard from its nominal value is considered as a rectangular distribution and therefore coverage factor is v3.

$$U_{dvrs} = \frac{\text{Deviation from nominal value}}{\sqrt{3}} \quad (8)$$

B.4 Certificate of Reference Standard: Reference standard itself is calibrated from a higher level laboratory or NMI or BIPM declaring combined uncertainty. Distribution function of uncertainty from calibration certificate is considered as normal distribution and coverage factor is usually K = 2 for normal distribution.

$$U_{crs} = \frac{\text{Combined Uncertainty from Calibration Certificate of Reference Standard}}{2} \quad (9)$$

B.5 Resolution of the Temperature Measuring Device: Uncertainty contribution due to resolution of the temperature measuring device (Thermocouple Readout) is, measured from the thermocouple readout resolution, half value of the thermocouple readout (digital thermometer) resolution.

That is, half resolution,

$$R_{1/2} = \frac{\text{Resolution of digital thermometer}}{2} \quad (10)$$

Readability is a bias and thus with rectangular distribution its standard uncertainty can be approximated as:

$$U_{readT} = \frac{R_{1/2}}{\sqrt{3}} \quad (11)$$

B.6 Certificate of Digital Thermometer: Digital thermometer itself is calibrated from a higher level laboratory or NMI or BIPM declaring combined uncertainty. Distribution function of uncertainty from calibration certificate is considered as normal distribution and coverage factor is usually K = 2 for normal distribution.

$$U_{ccdt} = \frac{\text{Combined Uncertainty from Calibration Certificate of Digital Thermometer}}{2} \quad (12)$$

B.7 Sensitivity tolerance: The sensitivity of the balance has some tolerance or uncertainty. The sensitivity tolerance is a permissible deviation of the actual sensitivity from the nominal sensitivity. It is given as a percentage with respect to the nominal sensitivity. This parameter also treated as a rectangular distribution and is proportional to the net weighing value [6]. The maximum deviation is obtained from balance specification.

$$U_{ST} = \left(\frac{1}{\sqrt{3}}.St_{max}.W_{net}\right) \quad (13)$$

B.8 Meniscus Reading: The variability of meniscus settings and scale readings made by a single operator depends upon his/her individual expertise. This reading influences directly the experimental standard deviation; therefore only type B components of meniscus and scale reading uncertainty should be estimated and composed [7]. These components are intended to take into account the unavoidable bias (or average deviations of the positioning of meniscus that is characteristic of a given operator in a given artifact) with reference to the ideal position defined by ISO 4787 ("the meniscus shall be set so that the plane of the upper edge of the graduation line is horizontally tangential to the lowest point of the meniscus, the line of sight being in the same plane") [8].

Sensitivity Coefficients: Before the uncertainty contributions from the input quantities can be combined, they must all be in the same units (one cannot add apples and oranges, or inches and millimeters). Sensitivity coefficients are essentially conversion factors that allow one to convert the units of an input quantity into the units of the measurand. Sensitivity coefficients are also and more importantly, measures of how much change is produced in the measurand by changes in an input quantity. Mathematically, sensitivity coefficients are obtained from partial derivatives of the model function with respect to the input quantities. Sensitivity coefficients may also be evaluated directly by experiment; this is particularly valuable where no reliable mathematical description of the relationship exists.

For Glassware Calibration: The equation to determine the volume at 20°C is given by [8]:

$$V_{20} = (R_L - R_E) \times \left(\frac{1}{\rho_w - \rho_a}\right) \times \left(1 - \frac{\rho_a}{\rho_b}\right) \times \{1 - \gamma(t - 20)\} \quad (14)$$

Let $R_L - R_E = m$

$$\frac{1}{\rho_w - \rho_a} = A$$

$$1 - \frac{\rho_a}{\rho_b} = B$$

$$1 - \gamma(t - t_{20}) = C$$

Therefore, $V_{20} = m \times A \times B \times C$

$$\frac{\partial V_{20}}{\partial m} = A \times B \times C = V_{20}/m$$

$$\frac{\partial V_{20}}{\partial t} = m \times A \times B \times (-\gamma)$$

For Balance Calibration: The equation to determine sensitivity coefficient for temperature is given by

$$y = \left(\frac{1}{\sqrt{3}}.TC.\Delta T.W_{net}\right) + \left(\frac{1}{\sqrt{3}}.ST_{max}.W_{net}\right) \quad (15)$$

Now, $C_{T_C} = \frac{\partial y}{\partial (T_C)} = \frac{1}{\sqrt{3}}.\Delta T.W_{net}$

And, $C_{S_T} = \frac{\partial y}{\partial (S_T)} = \frac{1}{\sqrt{3}}.W_{net}$

Combined Standard Uncertainty: To obtain the combined standard uncertainty of measurement, all individual uncertainties discussed above are compiled and it provides a reasonable estimate of the combined uncertainty of mutually independent contributions. Some of the influences, such as readability, repeatability, non-linearity etc are additive influences and their contribution simply adds to the reading, because they are independent of sample mass. Whereas, some of influences are multiplicative in their effect as they are proportional to the sample mass and therefore can be considered to influence the sensitivity of the transfer characteristic such as temperature coefficient [9].

Table 1: Uncertainty budget (for balance calibration).

Uncert-ainty source	Unit	Type	Probability distribution	Uncertainty (U or S)	Divisor	Sensitivity Coefficient C_i	Standard uncertainty	$(U_c)^2$
U_{CRS}	g	B	Normal	Calibration	2	1		
U_{repB}	g	A	Normal	Std. Dev.	v(n)	1		
$U_{resolation}$	g	B	Rectangular	Half interval	v(3)	1		
U_{TC}	°C	B	Rectangular	$TC.\Delta T.W_{net}$	v(3)	$\frac{\delta y}{\delta T_C}$		
U_{DVRS}	g	B	Rectangular	Deviation from nominal value	v(3)	1		
U_{ST}	°C	B	Rectangular	$ST_{max}.\ W_{net}$	v(3)	$\frac{\delta y}{\delta S_T}$		
							Sum	
							Comb. std. Uncr.√(*sum*)	
							Cov. Fact. k	
							Expanded uncertainty	

Table 2: Uncertainty Budget (for volumetric glassware calibration)

Uncertai-nty	Unit	Type	Probability Distribution	Uncertainty (U or S)	Divi-sor	Standard Uncertai-nty U_C	Sensitivi-ty Coeffici-ent C_I	$U_C \times C_I = U_{I(y)}$	$U^2_{I(y)}$
U_{certB}	g	B	Normal	Cer. value.	2		V_{20}/m		
U_{rep}	g	A	Normal	Std. dev.	$\sqrt{n}$		V_{20}/ m		
U_{resoB}	g	B	Rectangular	Half scale gradua.	$\sqrt{3}$		V_{20}/m		
$U_{minscus}$	ml	B	Rectangular	Half scale gradua.	$\sqrt{3}$		1		
U_{certT}	°C	B	Normal	Cer. value	2		$\frac{\delta V}{\delta t}$		
$U_{scatterT}$	°C	A	Normal	Std. dev.	$\sqrt{n}$		$\frac{\delta V}{\delta t}$		
								Sum:	
								Combined Standard Uncertainty(vSum):	
								Coverage Factor k:	
								Expanded Uncertainty:	

Table 3: Declaration of significant uncertainty value

Measured Value (x) Result	Expanded Uncertainty $U=ku_c(x)$	Reported
0.9372	0.0342	0.937±0.034
0.9372	0.3421	0.94±0.34
0.9372	3.4209	0.9±3.4
0.9372	34.2091	1±34
0.9372	342.0913	0±340

We assume that all individual uncertainties that discussed earlier for both type of additive and multiplicative are independent and statistically uncorrelated.

For combined standard uncertainty due to additive influences we have;

U_{add} = Sum of all uncertainty components =

$$\sqrt{(U^2_{repB} + U^2_{repT} + U^2_{readB} + U^2_{dvrs} + U^2_{redT} + U^2_{ccdt} + \cdots\cdots)} \quad (16)$$

As mentioned earlier multiplicative influences are proportional to the sample mass. Therefore for calculating these influences the sample mass and the condition of balance environment such as temperature and air humidity are needed to be considered.

The combined standard uncertainty of multiplicative influence for our balance in our facility is:

$$U_{multi} = U_{TC} \quad (17)$$

Hence the total combined standard uncertainty will be:

$$U_C = \sqrt{U^2_{add} + U^2_{multi}} \quad (18)$$

All possible uncertainty sources (described above) must be declared in an uncertainty budget. As for example, Table 1 shows uncertainty budget for balance calibration and Table 2 shows uncertainty budget for

volumetric glassware calibration. Finally, result (i.e., expanded uncertainty) should be stated by rounding up two significant values as the following Table 3.

The number of significant figures that should be reported for the result of a measurement depends on the uncertainty of the result. A common convention is to round the uncertainty (standard uncertainty or expanded uncertainty) to either one or two significant figures and to report both the measured value and the uncertainty to the resulting number of decimal places. MARLAP recommends this convention and suggests that uncertainties be rounded to two figures. The following examples demonstrate the application of the rule.

Only final results should be rounded in this manner. Intermediate results in a series of calculation steps should be carried through all steps with additional figures to prevent unnecessary round off errors. Additional figures are also recommended when the data are stored electronically. Rounding should be performed only when the result is reported.

CONCLUSIONS

The present view concerning measurement, as expressed in the VIM [10], is that no measurement can provide a complete state of knowledge, so that any estimated quantity value has an associated uncertainty. This paper explains how uncertainty of measurement can be estimated in balance and volumetric glassware calibrations. By carefully following the procedures outlined above, laboratory personnel will eliminate many errors that might be introduced into measurement procedures. For more accurate and precise measurement it should be used a more sensitive balance and other instruments which have less value of readability, repeatability, non linearity and sensitivity accuracy and for any application all of these influences should be checked. The ambient condition should be maintained according to ISO standards. However, it is important for each instrument to be serviced and calibrated regularly by a specially trained internal or external service person.

REFERENCES

1. Kallner, A., 2006. Uncertainty in Measurement: Introduction and Examples. J. Internat. Fed. Clin. Chem. & Lab. Med., 13: 103-109.
2. Ellison, S.L. and M. Rossieni, 2000. Quantifying Uncertainty in Analytical Measurement. Eurachem/CITAC Guide, Second Edition, pp: 4-5.
3. Weights of classes E1, E2, F1, F2, M1, M1–2, M2, M2–3 and M3, OIML, International recommendation no. 111, Edition 2004(E): 66-70.
4. Salahinejad, M. and F. Aflaki, 2007. Uncertainty Measurement of Weighing Results from an Electronic Analytical Balance, MEAS SCI REV, 7(3): 6.
5. Guide for Evaluation of Uncertainty in calibration, 2008, International Accreditation Service, Inc. USA. www.iasonline.org
6. Schwartz, R., 2000. Determination of mass in practice: Comprehensive mass metrology. Wiley-VCH, Weinheim, pp; 400.
7. Guidelines on the determination of uncertainty in gravimetric volume calibration, 2012. Calibration Guide, EURAMET cg-19, Version 2.1 Germany.
8. ISO 4787 - Laboratory glassware - Volumetric glassware - Methods for use and testing of capacity; 1984, Genève.
9. Reichmuth, A., 2000. Non-Linearity of Laboratory Balances and Its Impact on Uncertainty. NCSL, Workshop & Symposium, Toronto.
10. BIPM, IEC, IFCC, ILAC, ISO, IUPAC, IUPAP and OIML, 2012. International Vocabulary of Metrology—Basic and general concepts and associated terms (VIM) JCGM 200:2012.