Introduction to Measurement, Error Analysis, Propagation of Error, and Reporting Experimental Results

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Overview

Experimentation involves the observation and measurement of a physical property or a characteristic of something. A fundamental rule of scientific measurement states that it is never possible to exactly measure the true value of any characteristic, only approximations of the true value. For this reason, it is the responsibility of the engineers and scientists to report all measured values along with an estimation of the uncertainty in the measurement. The process of estimating the true value of the measurement and its associated uncertainty is called *error analysis*.

Furthermore, when a measured value is reported directly, the error analysis is complete when the error associated with that value is estimated and reported. In other cases, the measured value is to be combined mathematically with other measured values and the calculated result is the final reported value. In this case, the errors associated with each measured value must be combined to estimate the uncertainty in the result. This additional calculation is called *propagation of error***.** This paper presents a procedure for error analysis and propagation of error for use in Unit Operations Laboratory reports.

Sources of Measurement Error

1. **Human Error**: This is also referred to as Gross Error. Careful planning and execution of your experiment should prevent "mistakes."

2. **Reading Error**: This is a combination of the instrument's *accuracy* and *precision* and can be found in the manufacturer's specifications for the instrument.
   a. Accuracy refers to an instrument's ability to measure the true value of a characteristic. This describes how close the measurement is to the true value.
   b. Precision refers to the randomness of the measured value due to variation in the measuring device. This describes the repeatability of the measurement.
   c. Reading error is treated under the general categories of *Systematic Error* and *Random Error*.

3. **Systematic Error**: This is sometimes called *determinate* error.
   a. Has the same sign and magnitude for identical conditions; systematic error is predictable.
   b. Sources of systematic error:
      i. Mis-calibration of instruments. This class of systematic error refers to the instrument's *accuracy*. Could be due to a zero offset or improper instrument span.
      ii. Natural phenomena or inherent characteristics of the instrument. Could be due to hysteresis or the linearization of a non-linear response, or could be due to the method used, i.e. measuring surface temperature of a pipe to represent fluid temperature.
      iii. Consistent operator error, i.e. parallax.
   c. Often can be removed or compensation made:
      i. Recalibration, adjusting zero and span

    ii. Correction factors or calibration curves
    iii. Improved procedures
    iv. Comparisons to other methods.
  d. Must be corrected before data are reported or used in subsequent calculations.

4. **Random Error**: This is a combination of the randomness of the measurement process and the randomness of the characteristic you are measuring. It is also called *indeterminate* error.
   a. Can be positive or negative and has varying magnitude, is not predictable.
   b. You can not differentiate the source of the fluctuations caused by the measuring instrument from those of the process itself.
   c. Sources of random error:
      i. Random process fluctuations. i.e. Equipment "goblins", moon phase, miscellaneous
      ii. Random instrument fluctuations (referred to in the instrument manufacturer's data sheet as instrument precision)
      iii. Degree of subdivision of instrument scale and your ability to precisely read the scale
   d. Random error is quantified using Statistical methods.

Uncertainty in Values Obtained from Empirical Relationships

Oftentimes you will be comparing your measured values with values calculated from empirical relationships. These empirical values will also have an error (or uncertainty) associated with them. Theoretical values for friction factors, heat transfer coefficients, mass transfer coefficients, etc. are usually obtained from correlating equations and diagrams and have an often overlooked error referred to as "engineering accuracy". *Unless the specific reference states otherwise,* engineering accuracy can be assumed to be in the range of 10-20%; therefore, using a ±15% uncertainty is recommended.

Experimental Planning and Data Collection Activities

With many of the experiments in Unit Operations Lab you will be asked to measure a unit operation's performance at several different steady state conditions. To report the result at each steady state, you will collect data for two reasons. First, you will verify that the unit operation is at steady state. Second, you will make a number of repeat measurements at this steady state at regular, predetermined time intervals so that you can predict the true value of each measurement and estimate its associated error.

An important first step in planning experimental work is to identify what the experimental results should look like. From there, determine what needs to be measured and how it should be measured. In many cases there are choices in the type of measuring instrument you could use. Quite often, an instrument or method that yields high precision measurements takes more time or effort to use. Using trial calculations, determine the effect of that instrument's precision on the final calculated results and select an appropriate instrument. Instrument precision can usually be obtained from the manufacturer's data sheet. This value is your *reading error* and should be recorded in the laboratory notebook along with the model and serial number of the device long before you start any lab work.

Another step in the experimental planning process is to determine the number of replicates required to characterize each measurement and the measurement's uncertainty. An infinite number of replicates can be averaged together to report the true value of the measurement exactly. Time, resources, and other practical limitations prevent this. So, determine how many replicates you will need in order to characterize the measurement. Minimally, it takes 2 replicates to calculate a standard deviation. However, be aware that a standard deviation calculated around 2 or 3 replicates has little or no meaning and will result in a large associated uncertainty. Five values should be considered as a minimum. Finally, check that the data gathering activities fit within the scheduled laboratory time.

Before starting any experimentation on lab day, it is your responsibility to verify that measuring instruments are properly calibrated. If possible, two-point or three-point calibrations are performed. For example, a temperature device can be placed in an ice bath, checked at room temperature, and in boiling water to verify the calibration; or several standard solutions can be carefully prepared and the sensor range checked at these known points. Record any zero or calibration offsets in your laboratory notebook. Prepare a calibration curve if necessary. Add appropriate columns in your spreadsheet to correct measured values. If a two-point calibration is not possible, then minimally check the zero or "at rest" reading against a known and trusted device. For example, pressure gauges can be checked at atmospheric pressure.

While collecting data, check for gross mistakes and repeat experiments if necessary. Early in the day, check each operator for possible systematic error, i.e. from parallax or improper reading technique and correct immediately. Any remaining variation in replicated measured values is treated as *random error* and must be quantified using statistics

Quantifying Random Error – Statistical Analysis of Replicated Data

When reporting the results of a measured value for Unit Operations Lab you will typically report the mean value of your measured replicates along with an estimated standard error. Unfortunately, there is no single method for calculating the true value of a statistic in all situations. For example: in one case, multiple replicates of steady state data can be recorded at some set frequency. In another situation, one or more representative samples of a larger batch of material are set aside to perform an analytical measurement. These situations are very different and must be treated differently.

In either case, the goal in measurement is to determine the true value of something. If an engineer could take all the possible measurements (replicates) of the characteristic, the mean value of these replicates would be the true value of what is being measured. This would be called the grand average or population mean and is represented by the Greek letter, μ. We could also calculate a standard deviation around this grand average to quantify the dispersion of data around the average.

Since time and resources are limited, it is usually not practical to take all possible measurements. So, for this example suppose that there are N measurements of a quantity y, (i.e.: y_1, y_2, y_3, y_4, ..., y_N). These N measurements represent a subset of all the possible replicates that could have been measured and therefore represent a "sample" of the entire population. The sample average is called the sample mean and is represented by the familiar symbol, $\bar{\mathbf{x}}$. Since the sample mean was not calculated from the entire population, it can be expected that the sample mean will differ slightly from the population mean (the true value of the measurement.) A statistic called the Standard Error of the Means can then be calculated to estimate the difference between the sample mean and the population mean.

A suggested procedure for reporting the sample mean and calculating the uncertainty follows:

1. Calculate the Mean Value of the Data Set

   The mean value ($\bar{\mathbf{x}}$) is defined by:

$$\bar{\mathbf{x}} = \frac{\sum_{i=1}^{N} \mathbf{x_i}}{\mathbf{N}}. \qquad \text{(Eq. 1)}$$

2. Calculate the Sample Variance
   The sample variance is the sum of the squares of the difference between each measured value and the sample's mean value, divided by the number of replicates minus one. Variance (σ^2) is:

$$\sigma^2 = \frac{\sum_{i=1}^{N}\left(\mathbf{x_i} - \bar{\mathbf{x}}\right)^2}{(\mathbf{N}-\mathbf{1})} = \frac{\sum_{i=1}^{N}\mathbf{x_i^2} - \frac{\left(\sum_{i=1}^{N}\mathbf{x_i}\right)^2}{\mathbf{N}}}{(\mathbf{N}-\mathbf{1})}. \qquad \text{(Eq. 2)}$$

3. Calculate Average Standard Deviation of the Sample
   When a data set is small calculate an average standard deviation to describe the magnitude of the spread in the data. Average Standard Deviation is simply called Standard Deviation (σ) and is defined as the square root of the variance, i.e. the square root of the expression labeled Eq. 2.

4. Make an Initial Estimation of the Standard Error (Measure of the deviation of $\bar{\mathbf{x}}$ from the true value) Also called the *Standard Error of the Means* (SEM). SEM is defined statistically by:

$$SEM = \frac{\sigma}{\sqrt{N}}. \qquad \text{(Eq. 3)}$$

5. Compare the SEM to the Reading Error
   A measurement can be no more precise than the measuring instrument. Even if the recorded data shows no scatter (standard deviation of zero) there may still be an uncertainty in the data due to the reading error.
   Sources of reading error (e_R) can be:
      Sensitivity of the instrument (the maximum change required for the instrument to respond)
      Degree of subdivision of the scale of the instrument (generally, one-half the smallest subdivision) or the display's resolution.
   The value used for the reading error (e_R) usually can be found in the manufacturer's data sheet. If none is available, use ±½ the smallest increment of the device.
   Generally, some judgment and familiarity with the instrument are needed to come up with a good estimate of the reading error.

   Some considerations for reading error in UO Lab:
      What are the scale subdivions of the rotometer or pressure gauge?
      How sensitive are the platform scales?
      How precisely can you find the endpoint in titrating, +/- how many ml?
      What is the manufacturer's published accuracy for the instrument?

6. Adjust the Standard Error for a Combined Random Error and Reading Error

Once a value is determined for the reading error (e_R) it is compared to the standard deviation (σ) from (Eq. 2) to obtain the standard error as follows:

If $e_R \ll \sigma$, then:

$$e_S = \mathrm{SEM} = \frac{\sigma}{\sqrt{N}} \quad . \qquad \text{(Eq. 4)}$$

But, if $e_R \gg \sigma$, use:

$$e_S = \frac{e_R}{\sqrt{3}} . \qquad \text{(Eq. 5)}$$

(The origin of the $\sqrt{\mathbf{3}}$ in Eq. 5 is the Poisson Distribution.)

If e_R and σ are of the same order of magnitude then use the average of the two errors:

$$\mathbf{e_S} = \frac{\mathbf{1}}{\mathbf{2}}\left(\frac{\sigma}{\sqrt{N}} + \frac{e_R}{\sqrt{3}}\right). \qquad \text{(Eq. 6)}$$

It can be shown statistically that, for normally distributed data, the true value of x (the individual measurement) lies somewhere between:

$\bar{x}$ - e_S and $\bar{x}$ + e_S (with 68.3% confidence)

$\bar{x}$ - 2e_S and $\bar{x}$ + 2e_S (with 95.0% confidence)

$\bar{x}$ - 3e_S and $\bar{x}$ + 3e_S (with 99.7% confidence).

ESTIMATION OF ERROR IN A CALCULATED RESULT

When measured values are used in calculations, the error associated with each measured value will affect the uncertainty in the final calculated result. The error in each term of the equation must be combined with the error in the other terms. This is called **Propagation of Error**. An estimation of the error in the calculated result must be calculated and reported along with the result.

Method:

If y is the desired quantity and all the individual u, v, w, ... are the raw data needed to calculate y, we can represent the general function as:

$$y = f(u, v, w, ...).$$

You would typically run a set of identical repeated experiments and find the individual values of u, v, w,... Next, calculate the mean value of each u, v, w, ... The mean value of y can be calculated by using the mean values of in the functional relationship:

$$\mathbf{\bar{y} = f(\bar{u}, \bar{v}, \bar{w}, \ldots).}$$

Then, to estimate the error associated with y, use either of the two following methods:

A. Root Means Square Error (e_{RMS})

The Root Mean Square Error has a basis in statistics:

$$\mathbf{e_{RMS,y}} = \sqrt{\left\{\left[\left(\frac{\partial f}{\partial u}\right)_{v,w} * e_{S,u}\right]^2 + \left[\left(\frac{\partial f}{\partial v}\right)_{u,w} * e_{S,v}\right]^2 + \left[\left(\frac{\partial f}{\partial w}\right)_{u,v} * e_{S,w}\right]^2 + \ldots\right\}_{\bar{u},\bar{v},\bar{w}}} \qquad \text{(Eq. 7)}$$

where the mean values ($\bar{u}, \bar{v}, \bar{w}$,...) are used to evaluate the derivatives in the above expression.

The RMS Error is tedious to calculate by hand and is best suited to spreadsheets.

B. Upper Estimate of the Propagated Error

An upper limit to the error can be estimated as follows:

$$\mathbf{e_{UL,y}} = \left|\left(\frac{\partial f}{\partial u}\right)_{v,w}\right| e_{S,u} + \left|\left(\frac{\partial f}{\partial v}\right)_{u,w}\right| e_{S,v} + \left|\left(\frac{\partial f}{\partial w}\right)_{u,v}\right| e_{S,w} + \ldots \qquad \text{(Eq. 8)}$$

where the mean values ($\bar{u}, \bar{v}, \bar{w}$,...) are used to evaluate the derivatives in the above expression. This method is easier to use for hand calculations.

Note that $e_{RMS} < e_{UL}$ always. Thus, using e_{UL} will give a more conservative estimate of the error.

SIGNIFICANT FIGURES

When reporting a value and its associated error use the appropriate number of significant figures (SF). For measured values, the number of SF is a function of the precision of the measuring device. When a calculated result combines more than one measured or estimated value the correct number of SF is the same as that of the least of all the measured values. The correct number of SF for estimated error is typically one less than the number of SF of the calculated result (sometimes two, but oftentimes only one.)

ERROR ANALYSIS OF FLOW RATE BY REPLICATED "PAIL AND SCALE" MEASUREMENTS

One common method of measuring flow rate is to measure the mass of liquid collected in a barrel or pail (w_F-w_0) over a time interval (t). If replicated measurements (N) have been made of the final and initial mass ($w_{F,j}$ and $w_{0,j}$) and the time interval (t_j), it would be incorrect to determine the mean, variance, etc. of (w_F, w_0, and t) and then calculate the mass flow rate (m) and its error. The correct procedure would be as follows:

1. Calculate the mass flow rate for each measurement (m_j):

$$\dot{\mathbf{m}}_{\mathbf{j}} = \frac{(\mathbf{w}_{\mathbf{F,j}} - \mathbf{w}_{\mathbf{0,j}})}{\mathbf{t}_{\mathbf{j}}} \quad (\mathbf{j = 1,2,3,\ldots,N}).$$

2. Calculate the mean value of the flow rate ($\overline{\dot{\mathbf{m}}}$):

$$\overline{\dot{\mathbf{m}}} = \frac{\sum_{j=1}^{N} \dot{\mathbf{m}}_{\mathbf{j}}}{\mathbf{N}}.$$

3. Calculate the standard deviation of $\dot{\mathbf{m}}$:

$$\sigma_{\mathbf{m}} = \sqrt{\frac{\sum_{j=1}^{N} (\dot{\mathbf{m}}_{\mathbf{j}} - \overline{\dot{\mathbf{m}}})^2}{(\mathbf{N-1})}}.$$

4. Determine the reading error associated with each mass flow rate ($\dot{\mathbf{m}}_{\mathbf{j}}$) due to propagation of the reading errors in w_F, w_0, and t:

$$(\mathbf{e}_{\mathbf{R,m}})_{\mathbf{j}} = \frac{[\mathbf{e}_{\mathbf{R,w_F}} + \mathbf{e}_{\mathbf{R,w_0}}]}{\mathbf{t}_{\mathbf{j}}} + \frac{[(\mathbf{w}_{\mathbf{F}} - \mathbf{w}_{\mathbf{0}})_{\mathbf{j}} \mathbf{e}_{\mathbf{R,t}}]}{\mathbf{t}_{\mathbf{j}}^2}.$$

5. Determine the average reading error associated with the mass flow rate:

$$\mathbf{e}_{\mathbf{R,m}} = \frac{\sum_{j=1}^{N} (\mathbf{e}_{\mathbf{R,m}})_{\mathbf{j}}}{\mathbf{N}}.$$

6. Combine the reading error and the standard deviation as before:

If $e_{R,m} << \sigma_m$, then

$$\mathbf{e_{S,m} = \frac{\sigma_m}{\sqrt{N}}}\,.$$

If $e_{R,m} >> \sigma_m$, then

$$\mathbf{e_{S,m} = \frac{e_{R,m}}{\sqrt{3}}}\,.$$

If $e_{R,m}$ and $\boldsymbol{\sigma_m}$ are of the same order of magnitude then

$$\mathbf{e_{S,m} = \frac{1}{2}\left(\frac{\sigma_m}{\sqrt{N}} + \frac{e_{R,m}}{\sqrt{3}}\right)}\,.$$

<u>ERROR ANALYSIS OF FLOW RATE BY REPLICATED MEASUREMENTS OF CHANGE IN LIQUID LEVEL IN A TANK</u>

One common method of measuring volumetric flow rate (Q) is to measure the change in liquid level in a tank (h_F-h_0) over a time interval (t). If replicated measurements (N) have been made of the final and initial liquid levels ($h_{F,j}$ and $h_{0,j}$) and the time interval (t_j), an error analysis can be performed in the same way as for the "pail and scale" method:

1. Calculate the volumetric flow rate for each measurement (Q_j):

$$\mathbf{Q_j = \frac{\frac{\pi D^2}{4}(h_F - h_0)_j}{t_j} \qquad (j = 1,2,3,\ldots,N)}\,.$$

where D is the inside diameter of the tank (assumed to have no error associated with it).

2. Calculate the mean value of the flow rate ($\overline{\mathbf{Q}}$):

$$\mathbf{\overline{Q} = \frac{\sum_{j=1}^{N} Q_j}{N}}\,.$$

3. Calculate the standard deviation of Q:

$$\sigma_Q = \sqrt{\frac{\sum_{j=1}^{N}(Q_j - \overline{Q})^2}{(N-1)}} .$$

4. Determine the reading error associated with each flow rate (Q_j) due to propagation of the reading errors in h_F, h_0, and t:

$$(e_{R,Q})_j = \frac{\pi D^2}{4}\left[\frac{e_{R,h_F} + e_{R,h_0}}{t_j} + \frac{(h_F - h_0)_j e_{R,t}}{t_j^2}\right].$$

5. Determine the average reading error associated with the flow rate:

$$e_{R,Q} = \frac{\sum_{j=1}^{N}(e_{R,Q})_j}{N} .$$

6. Combine the reading error and the standard deviation as before:

If $e_{R,Q} << \sigma_Q$, then

$$e_{S,Q} = \frac{\sigma_Q}{\sqrt{N}} .$$

If $e_{R,Q} >> \sigma_Q$, then

$$e_{S,Q} = \frac{e_{R,Q}}{\sqrt{3}} .$$

If $e_{R,Q}$ and σ_Q are of the same order of magnitude then

$$e_{S,Q} = \frac{1}{2}\left(\frac{\sigma_Q}{\sqrt{N}} + \frac{e_{R,Q}}{\sqrt{3}}\right) .$$

EXAMPLE -- ERROR IN CALCULATED VALUE OF THE OVERALL HEAT TRANSFER COEFFICIENT

The overall heat transfer coefficient (U) is obtained from:

$$\overline{\mathbf{U}} = \frac{\overline{\mathbf{Q}}}{\mathbf{A}\overline{(\mathbf{T_h} - \mathbf{T_c})}_{\mathbf{LM}}}$$

where $\overline{(\mathbf{T_h} - \mathbf{T_c})}_{\mathbf{LM}} = \mathbf{LMTD} = \dfrac{[\overline{(\mathbf{T_h} - \mathbf{T_c})_2} - \overline{(\mathbf{T_h} - \mathbf{T_c})_1}]}{\ln[\dfrac{\overline{(\mathbf{T_h} - \mathbf{T_c})_2}}{\overline{(\mathbf{T_h} - \mathbf{T_c})_1}}]}$.

The error in the calculated value of U due to errors in Q, A, and the temperatures (T_{h2}, T_{h1}, T_{c2}, T_{c1}) is given by:

$$\mathbf{e_{S,U}} = \frac{\mathbf{e_{S,Q}}}{\mathbf{A(LMTD)}} + \frac{\mathbf{Qe_{S,LMTD}}}{\mathbf{A(LMTD)^2}} + \frac{\mathbf{Qe_{S,A}}}{\mathbf{A^2(LMTD)}}$$

where

$$\mathbf{e_{S,LMTD}} = \{\left\{\frac{[\overline{(\mathbf{T_h} - \mathbf{T_c})_2} - \overline{(\mathbf{T_h} - \mathbf{T_c})_1}]}{(\mathbf{T_h} - \mathbf{T_c})_2} - \ln\left[\frac{(\mathbf{T_h} - \mathbf{T_c})_2}{(\mathbf{T_h} - \mathbf{T_c})_1}\right]\right\}[\mathbf{e_{T_{h2}}} + \mathbf{e_{T_{c2}}}]$$

$$+ \left\{\frac{[\overline{(\mathbf{T_h} - \mathbf{T_c})_2} - \overline{(\mathbf{T_h} - \mathbf{T_c})_1}]}{(\mathbf{T_h} - \mathbf{T_c})_1} - \ln\left[\frac{(\mathbf{T_h} - \mathbf{T_c})_2}{(\mathbf{T_h} - \mathbf{T_c})_1}\right]\right\}[\mathbf{e_{T_{h1}}} + \mathbf{e_{T_{c1}}}]\} / \left\{\ln\left[\frac{(\mathbf{T_h} - \mathbf{T_c})_2}{(\mathbf{T_h} - \mathbf{T_c})_1}\right]\right\}^2$$

.

If $(T_h\text{-}T_c)_2$ and $(T_h\text{-}T_c)_1$ are approximately equal then:

$$\mathbf{LMTD} \approx \frac{1}{2}[\overline{(\mathbf{T_h} - \mathbf{T_c})_2} + \overline{(\mathbf{T_h} - \mathbf{T_c})_1}]$$

$$\mathbf{e_{S,LMTD}} \approx \frac{1}{2}[\mathbf{e_{T_{h2}}} + \mathbf{e_{T_{c2}}} + \mathbf{e_{T_{h1}}} + \mathbf{e_{T_{c1}}}].$$

TABLE OF NOMENCLATURE

A	Heat Transfer Area
D	Inside Diameter of Tank
e_S	Standard Error
e_R	Reading Error
h_F, h_0	Final and Initial Liquid Levels, respectively, in Volumetric Flow Rate Measurement
i, j	Refer to a Particular Sample or Data Point
LMTD	Log-Mean Temperature Difference
m	Mass Flow Rate
N	Number of Data (Sample) Points
Q	Volumetric Flow Rate; Heat Transfer Rate
σ	Standard Deviation
σ^2	Variance
T_h, T_c	Temperature of Hot and Cold Fluids, respectively
t	Time Interval for Flow Rate Measurement
U	Overall Heat Transfer Coefficient
u, v, w	Independent Variables Used in a Calculation
w_F, w_0	Final and Initial Mass, respectively, in "Pail and Scale" Method
$\bar{\mathbf{x}}$	Mean Value of x
x_i	Sampled Value of x
y	Dependent Variable Determined in a Calculation

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