Overview

**Basic Tools**
- Good Laboratory Practice (GLP)
- Labware/Equipment
- Methods

**Experimental Error**
- Types of Error
- Significant Figures
- Propagation of Error in Analysis

**Good Laboratory Practice (GLP)**

"embody a set of principles that provides a framework within which laboratory studies are planned, performed, monitored, recorded, reported and archived...GLP helps assure regulatory authorities that the data submitted are true reflection of the results obtained during the study and can therefore be relied upon when making assessments."
1. Hazard Labeling

**Health Hazard**
0 = No unusual hazard
1 = CAUTION: May cause irritation
2 = WARNING: May be harmful if inhaled or swallowed
3 = WARNING: Corrosive or toxic: Avoid skin contact/inhalation
4 = DANGER: May be fatal on short exposure. Special protective equipment required

**Fire Hazard**
0 = Not combustible
1 = Combustible if heated
2 = CAUTION: Combustible liquid (flashpoint of 100-200°F)
3 = WARNING: Flammable liquid (flashpoint < 100°F)
4 = Flammable gas or extremely flammable liquid

**Instability**
0 = Stable
1 = CAUTION: May react if heated or mixed with water
2 = WARNING: Unstable or may react if mixed with water
3 = DANGER: May be explosive if shocked, heated, confined or mixed with water
4 = DANGER: Explosive material at room temperature

**Specific Hazard**
W = Avoid use of water
ACID = Acid
ALK = Alkali
COR. = Corrosive
OXY = Oxidizer

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2. Material Safety Data Sheet (MSDS)

Physical Data, Toxicity, Health Effects, First Aid, Reactivity, Storage, Disposal, Protective Equipment, Spill Handling
1. Hazard Labeling
2. Material Safety Data Sheet (MSDS)
3. Protective Wear
   - Proper Clothing (no sandals, shorts, etc.)
   - Safety Glasses/Goggles
   - Lab Coats (flame- and spill-resistant)
   - Gloves

GLP: Laboratory Safety

1. Hazard Labeling
2. Material Safety Data Sheet (MSDS)
3. Protective Wear
4. Fume Hoods
5. Waste Disposal
6. Spills and Accidents
GLP: Laboratory Safety

1. Hazard Labeling
2. Material Safety Data Sheet (MSDS)
3. Protective Wear
4. Fume Hoods
5. Waste Disposal
6. Spills and Accidents
7. Label ALL Containers!!

GLP: Laboratory Notebooks

- A laboratory notebook should state what you did and what you observed, and should be understandable to a stranger. The measure of scientific truth is the ability to reproduce an experiment.
- Never erase information; cross-out with a single line to make corrections.
- Basic Elements: Purpose, Materials, Method, Results, Conclusions
Measuring Mass: Gravimetric Analysis

Typically sensitive to 0.01 to 0.1 mg

Use weighing vessel (e.g. pan, weighing paper) NOT to touch user after use!!

Tare = "zero" the balance

Shielding doors prevent drafts

NOTE: Brush clean after use!!

Measuring Mass: Gravimetric Analysis

Filtration and Gravimetric Analysis I

Gooch Crucible

Adapter

Glass Funnel

Rubber Stopper

Filtrate

Trap

Measuring Mass: Gravimetric Analysis

Filtration and Gravimetric Analysis II

Ashless Filter Paper
“Weighing By Difference”

\[ \text{Weight}_{\text{analyte}} = \text{Weight}_{\text{final}} - \text{Weight}_{\text{initial}} \]

Where “Weight\text{\textsubscript{initial}}” is the weight of the vessel (e.g. crucible), filter paper, etc., and “Weight\text{\textsubscript{final}}” is the weight of the vessel (e.g. crucible), filter paper, etc. containing the analyte.

Moisture and Gravimetric Analysis

Hygroscopic = “rapidly absorb moisture (water)"

Methods/Tools for Hygroscopic Substances
1. Drying Ovens
2. (Vacuum) Dessicators
3. Dessicants
4. Repeated “Weighing by Difference”

Preventing Weighing Errors
1. Water Absorption
2. Contact with Vessel/Sample
3. Effects of Temperature/Drafts
4. Vibrations
5. Level
6. Spills
7. Calibration
Measuring Volume: Volumetric Analysis

Burets

Volume = Final - Initial

Example:
24.16 mL - 9.63 mL = 14.53 mL

Meniscus

Measuring Volume: Volumetric Analysis

Digital Burets

Preparing Solutions

Preparing Dilutions

Measuring Volume: Volumetric Flask

TC = To Contain
Measuring Volume: Pipets and Syringes

- Transfer Pipet

TD = To Deliver

- Rinse repeatedly to remove air bubbles
- Steel needle sensitive to strong acids
Good Laboratory Practice (GLP)

“embodies a set of principles that provides a framework within which laboratory studies are planned, performed, monitored, recorded, reported and archived...GLP helps assure regulatory authorities that the data submitted are true reflection of the results obtained during the study and can therefore be relied upon when making...assessments.”

Experimental Error

“Every measurement has some uncertainty which is called experimental error”

...a measurement is an ESTIMATE.

Types of Error

Systematic (Determinate) = “flaw in equipment or design of an experiment”

Random (Indeterminate) = “uncontrolled (or maybe uncontrollable) variables in the measurement”

Experimental Error

Precision = “reproducibility of a result [= measurement]”

Accuracy = “how close a measured value is to the ‘true’ value”

Accurate
NOT Precise

Precise
NOT Accurate

Accurate and Precise
Experimental Error

**Significant Figures**

"The minimum number of digits needed to write a given value in scientific notation without loss of accuracy" is expressed in powers of 10.

Examples:

- $1.234 \times 10^{-5}$ → Four (4) significant figures
- $0.00001234$ → Four (4) significant figures
- $123400$ → Four (4) significant figures
- $1.2340 x 10^5$, $1.2340 x 10^5$, $1.23400 x 10^5$
- $456$

**Experimental Error**

"The Rules for Zeros"

- Zeros are significant when they occur in the middle of a number

Example: $1.2034 x 10^5$

- Zeros are significant when they occur at the end of a number on the right-hand side of a decimal point

Example: $1.2340 x 10^5$

**NOTE:** Assumes that zero is accurate.

**Experimental Error**

**Significant Figures**

"The last significant digit in a measured quantity always has associated uncertainty"

**KNOW**

e.g. 5.5 mL

"Interpolation = Estimating of readings to the nearest tenth of distance between scale readings"
Experimental Error

Significant Figures

"Some numbers are exact - with an infinite number of unwritten significant digits"

Example: 5 people
= 5.0 people
= 5.00 people
= 5.000 people

Experimental Error

Arithmetic and Significant Figures

1. Addition/Subtraction

Express numbers with the same exponent

Significant figures are limited to the least-certain number

\[
\begin{align*}
1.234 \times 10^{-5} + 6.78 \times 10^{-5} &= 1.234 \times 10^{-5} + 0.000678 \times 10^{-5} \\
&= 1.234678 \times 10^{-5}
\end{align*}
\]

“Rules for Rounding”

If a number is less than “halfway” (< 5), then round DOWN.

e.g. 1.234 rounds to 1.23 (with 3 significant digits)

If a number is more than “halfway” (> 5), then round UP.

e.g. 1.23456 rounds to 1.2346 (with 5 significant digits)

If a number is exactly “halfway” (5), then round to the nearest EVEN number.

e.g. 1.2345 rounds to 1.234 (with 4 significant digits)
Experimental Error

Arithmetic and Significant Figures

1. Addition/Subtraction
    Express numbers with the same exponent
    Significant figures are limited to the least-certain number
    \[
    \begin{array}{c}
    1.234 \times 10^{-5} \\
    + 6.78 \times 10^{-5} \\
    \end{array}
    \]
    Significant figures are limited to the least-certain number
    \[
    1.234 \times 10^{-5} \\
    + 0.000678 \times 10^{-5} \\
    \]
    \[
    1.234678 \times 10^{-5} = 1.23 \times 10^{-5}
    \]

Experimental Error

Significant Figures and “Rounding-Off”

“Rounding should only be done on final answers (not intermediate results), to avoid accumulating round-off errors”

24.16 mL - 9.63 mL = 14.5 mL
    = 14.5 mL

24.2 mL - 9.6 mL = 14.6 mL

Experimental Error

Arithmetic and Significant Figures

1. Addition/Subtraction
2. Multiplication/Division
    Significant figures are limited to the least-certain number
    \[
    \begin{array}{c}
    1.234 \times 10^{-5} \\
    \times 6.78 \times 10^{-5} \\
    \end{array}
    \]
    \[
    \begin{array}{c}
    8.3652 \times 10^{-14} \\
    \end{array}
    \]
    \[
    = 8.37
    \]
Experimental Error

Arithmetic and Significant Figures

1. Addition/Subtraction
2. Multiplication/Division
   Significant figures are limited to the least-certain number
   Example: Measure blood glucose level for three people, and calculate average level.
   \[ 4.5 \text{ mM} + 6.78 \text{ mM} + 9.10 \text{ mM} = 6.7933333 \]
   Exact number has infinite significant figures

3. Logarithms and Antilogarithms
   \[ \log n = a \]
   \[ \text{"logarithm of } n \text{ is } a\" \]
   \[ a = \text{characteristic and mantissa} \]
   \[ \text{e.g. } a = 1.234 \]
   \[ \text{characteristic} \]
   \[ \frac{1}{\text{mantissa}} \]
   \[ n = 10^a \]
   \[ \text{"} n \text{ is the antilogarithm of } a \text{"} \]

   * Number of digits in the mantissa of the log of a number should equal the number of significant figures in that number
   * Number of significant figures in the antilog should equal the number of digits in the mantissa
Experimental Error

Arithmetic and Significant Figures

1. Addition/Subtraction
2. Multiplication/Division
3. Logarithms and Antilogarithms

Example:

\[
\log 1.234 \times 10^{-5} = \begin{align*}
&\text{4 digits} \\
&\text{4 significant} \\
&= -4.9087
\end{align*}
\]

Experimental Error

Arithmetic and Significant Figures

1. Addition/Subtraction
2. Multiplication/Division
3. Logarithms and Antilogarithms

Example:

\[
antilog 1.234 = \begin{align*}
&\text{3 significant} \\
&\text{3 digits} \\
&= 17.1
\end{align*}
\]

Experimental Error

Propagation of Uncertainty from Random Error

“We can usually estimate or measure the random error associated with a measurement”

(more next week!)
Experimental Error

Absolute Uncertainty

\[ \pm 0.0001 \text{ g} \]

\[ 9.63 \pm 0.01 \text{ mL} \]

\[ \pm 0.0001 \text{ g} \]

Experimental Error

**Absolute Uncertainty**

**Relative Uncertainty**

\[ \text{Relative Uncertainty} = \frac{\text{absolute uncertainty}}{\text{magnitude of the measurement}} \]

% Relative Uncertainty

\[ \% \text{ Relative Uncertainty} = \frac{\text{absolute uncertainty}}{\text{magnitude of the measurement}} \times 100\% \]

*Note: No Units*

**Experimental Error**

**Propagation of Uncertainty from Random Error**

1. Addition/Subtraction

\[ e_\text{final} = \sqrt{\sum (e_n)^2} \]

Where \( e_\text{final} \) is the final calculated error, and \( \sum (e_n)^2 \) is sum (\( \sum \)) of the squared errors (\( e_n^2 \)).
Experimental Error

Propagation of Uncertainty from Random Error

1. Addition/Subtraction

\[ e_{\text{final}} = \sqrt{\sum (e_n)^2} \]

\[
\begin{align*}
1.23 \pm 0.01 & \quad \Rightarrow (e_1) = 0.01 \\
4.56 \pm 0.02 & \quad \Rightarrow (e_2) = 0.02 \\
+ 7.89 \pm 0.03 & \quad \Rightarrow (e_3) = 0.03 \\
\end{align*}
\]

\[
\begin{align*}
\sum (e_n)^2 &= 0.01^2 + 0.02^2 + 0.03^2 \\
&= 0.0001 + 0.0004 + 0.0009 \\
&= 0.0014 \\
e_{\text{final}} &= \sqrt{0.0014} \\
&= 0.037 \\
\end{align*}
\]

\[ \Rightarrow 13.68 \pm 0.03 \]

Percent Relative Uncertainty?

Relative Uncertainty = \[ \frac{0.03}{13.68} = 0.002 \]

\[
\begin{align*}
\text{Percent Relative Uncertainty} &= 0.002 \times 100\% \\
&= 0.2\% \\
\end{align*}
\]

Experimental Error

Propagation of Uncertainty from Random Error

1. Addition/Subtraction

2. Multiplication/Division

\[ \%e_{\text{final}} = \sqrt{\sum (\%e_n)^2} \]

Where \( \%e_{\text{final}} \) is the final calculated percent error, and \( \sum (\%e_n)^2 \) is sum (2) of the squared percent error (\( e_n^2 \)).
Experimental Error

**Propogation of Uncertainty from Random Error**

1. Addition/Subtraction
2. Multiplication/Division
3. Exponents and Logarithms

\[
\%e_{\text{final}} = \sqrt{\sum (%e_x)^2} \\
1.23 (\pm 0.01) \times 4.56 (\pm 0.02) \\
\begin{align*}
1.23 (\pm 0.01) = \frac{0.01}{12.3x100} = 0.8 \% \\
4.56 (\pm 0.02) = \frac{0.02}{45.6x100} = 0.4 \%
\end{align*}
\]

\[
\%e_{\text{final}} = \sqrt{(0.8)^2 + (0.4)^2} = \sqrt{0.64 + 0.16} = \sqrt{0.8} = 0.9 \%
\]

\[
y = x^n \Rightarrow \%e_y = n(\%e_x) \\
y = \log x \Rightarrow \%e_y = \frac{1}{y} \%e_x \\
y = 10^x \Rightarrow \%e_y = (\ln 10) \%e_x \\
y = \ln x \Rightarrow \%e_y = \frac{1}{x} \%e_x \\
y = e^x \Rightarrow \%e_y = \%e_x
\]

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**Example 1: Uncertainty in Molecular Mass**

Uncertainty in molecular mass of O₂?

Atomic Mass of Oxygen = 15.9994 ± 0.0003

*Note: Difference due to number of neutrons in atoms = isotope variation

**Systematic Error**

We simply ADD systematic error...

\[
O_2 = O + O = (0.0003) + (0.0003) = 0.0006
\]

**NOT** \[
\sqrt{(0.0003)^2 + (0.0003)^2}
\]
Experimental Error

Propagation of Uncertainty from Systematic Error

Example 1: Uncertainty in Molecular Mass

Uncertainty in molecular mass of $C_2H_4$?

- $C_2 = C + C = 2 \times 0.0008 = 0.0016$ Systematic Error
- $H_4 = H + H + H + H = 4 \times 0.00007 = 0.00028$

$C_2 + H_4 = \sqrt{0.0016^2 + 0.00028^2}$

= 0.0016 Random Error (error in C and H independent)

Experimental Error

Propagation of Uncertainty from Systematic Error

Example 2: Multiple Deliveries from One Pipet.

Uncalibrated 25-mL Class A Volumetric Pipet:

25.00 ± 0.03 mL Systematic Error

Four (4) deliveries…

$(0.03) + (0.03) + (0.03) + (0.03) = ± 0.12$

Experimental Error

Propagation of Uncertainty from Systematic Error

Example 2: Multiple Deliveries from One Pipet.

*Calibrate* a 25-mL Class A Volumetric Pipet

*Weigh the water it delivers, calibrate based on mass, density and temperature

25.991 ± 0.006 mL Random Error

Four (4) deliveries…

$\sqrt{(0.006)^2 + (0.006)^2 + (0.006)^2 + (0.006)^2}$

= ± 0.012