SCMB Chemistry Lab Workshop
Orientation
Designing a Chemistry EEI: Do you start with a context or the tools?

Sugar
Content
- Colorimetric
- Density

Vitamin
C Content
- Redox Titration
- HPLC Assay

Alcohol
Content
- Redox Titration
- Specific gravity
Designing a Chemistry EEI: Do you start with a context or the tools?

Sugar Content
- Compare foods
- Compare processes

Alcohol Content
- Compare fuels
- Compare wines

Heavy Metal Ion Content
- Compare water sources
- Compare foods
EEIs: Most Accessible Techniques

- **TITRATIONS**
  - Acid/Base
  - REDOX

- **CALORIMETRY**
  - Fuels
  - Foods
  - Salts

- **SEPARATION**
  - Biofuels
  - Pigments
What chemistry tools/technique that underpins your EEI’s?
 Conductivity

- Corrosion
- Ion Depletion
- Drinks
- Water Chemistry
- Conductivity
Colorimetry

- Antioxidants
- Molecules in Food
- Corrosion
- Heavy metals
Risk Assessment

Risk assessment is context specific – appropriate controls that should be implemented relate to amounts of each substance and environment.

At UQ, we require students to complete an institutional safety form and pre-laboratory activities each week including seeking MSDS information.
Safety in the laboratory

- Wear gloves (change gloves if chemicals spilt on them)
- Appropriate footwear
- Laboratory coat, buttoned up.
- Safety glasses / goggles.
- Long hair tied back
- No food and drink, including water and chewing gum.
- Lab coats and gloves are not allowed outside of the lab. Bathrooms and water drink fountains are in the hallway.

- Mobile phones in bag and on silent
- No headphones
- No food and drink, including chewing gum
- Remove labcoat and gloves before you leave the lab
Thirsty Work!
To determine the concentration of ions in popular soft drinks by Conductivity.
Conducting Electricity
Additional safety notes

- Acetic acid can trigger asthmatic attacks in some people.
- All solutions can be disposed of down the sink.
The 3-way toggle switch on the conductivity probe switch should be on the 0-20 000 μS cm\(^{-1}\).

The area circled red needs to be fully submersed in the solution for the conductivity probe to work properly. The silver tip measures temperature and there are two graphite electrodes either side.
Plug in your USB drive at the side of the Labquest. When you finish your titration, choose File, then Export. Click on the USB drive icon. This will save a text file that you can open in Excel on the computer and plot. (If you choose Save, the file can only be opened in Vernier Logger Lite software.)
Sweetness & Light!
DNSA is a highly corrosive chemical

- Avoid contact with skin and eyes. If skin contact occurs wash immediately with water. Wash eyes with water for 15 minutes if DNSA contact occurs. You will be taken immediately to a medical centre or hospital after you have washed your eyes.
- Change your gloves if you get DNSA on them. Never touch your face or eyes while wearing laboratory gloves.
- Do not invert pipettes when pipetting solutions, (especially if using DNSA) as the barrel becomes contaminated.
- Dispose of DNSA in waste containers provided.
Aim

To determine the amount of simple reducing sugars present in cereals through an oxidation-reduction reaction that results in a product that can be analysed by UV-VIS spectrophotometry.
The linear form of D-glucose (for example) is oxidised to D-gluconate:

\[
\begin{align*}
\beta\text{-D-glucopyranose} &\quad \overset{\text{oxidising agent}}{\xrightarrow{\text{basic solution}}} &\quad \text{D-gluconate} \\
\text{(\textit{\beta-D-glucose})} &\quad &\quad
\end{align*}
\]

N.B. An equilibrium exists between the cyclic and linear forms of glucose
The 3,5-dinitrosalicylic acid (DNSA) reacts with the reduced sugar. 3,5-dinitrosalicylic acid is produced and it absorbs light at 540 nm:

3,5-dinitrosalicylic acid (DNSA) + glucose → 3-amino-5-nitrosalicylic acid (red-brown) + oxidised glucose
UV/Vis Spectrophotometry

Place solution in cuvette: wipe the outside clean with a Kimwipe.

Spectrophotometer has 4 sample positions – make sure your sample is in the light beam.

Present clear surface of cuvette to light beam (don’t touch either of the clear surfaces with your gloves as any smudges will absorb the light).
the absorbance of a solution is directly proportional to its concentration

The extent of the linear region depends on the analyte and an ideal Beer-Lambert curve is linear over a wide range of concentrations.

$$A = \varepsilon \cdot c \cdot d$$

Where:  
- $A$ = Absorbance  
- $\varepsilon$ = molar absorptivity  
- $c$ = concentration  
- $d$ = distance
Y = mx + c

Must pass through origin

Absorbance 540 nm

Concentration
Hydrolysis of Sucrose

Disaccharide $\rightarrow$ Reducing sugar

Glycosidic bond

Error: p 4-3: add CH$_2$OH group here
Micropipettes

The accuracy of the experiment relies on accurately measuring all volumes with the auto pipettes provided.

- Place a tip on the end with a gentle slight twisting motion to ensure a tight seal.
- Depress the push button to the FIRST stop position.
- Insert the tip just below the surface of the liquid.
- Release the button SLOWLY to draw up the liquid.
- Dispense the sample by SLOWLY depressing the push button to the SECOND stop position.

**NEVER TURN THE PIPETTE UPSIDE DOWN OR LAY IT ON ITS SIDE WHEN THERE IS LIQUID IN THE TIP**
Experiment Flow Chart

Use autopipettes to measure volumes. Make sure you check with your tutor which pipette to use. Remember to change tips between different samples!

For Sucrose Standard

<table>
<thead>
<tr>
<th>1 mL</th>
<th>2 mL</th>
<th>3 mL</th>
<th>4 mL</th>
</tr>
</thead>
<tbody>
<tr>
<td>SU1</td>
<td>SU2</td>
<td>SU3</td>
<td>SU4</td>
</tr>
<tr>
<td>1 mL</td>
<td>2 mL</td>
<td>3 mL</td>
<td>4 mL</td>
</tr>
<tr>
<td>SH1</td>
<td>SH2</td>
<td>SH3</td>
<td>SH4</td>
</tr>
</tbody>
</table>

of 0.02 M sucrose solution

Unknown Cereal Extract

5 mL

Soft Drink

250 μL

X

make up each tube to 8 mL with dist. H₂O & mix

<table>
<thead>
<tr>
<th>SU1</th>
<th>SU2</th>
<th>SU3</th>
<th>SU4</th>
</tr>
</thead>
<tbody>
<tr>
<td>SH1</td>
<td>SH2</td>
<td>SH3</td>
<td>SH4</td>
</tr>
<tr>
<td>5H</td>
<td>5U</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| 0.25 H | 0.25 U |

X

Add 0.1 mL of 2 M HCl to

Hydrolysed (H) samples only & mix

Heat in boiling water bath for 25 min

Add 1.0 mL of 2.0 M NaOH, make up the volume to 10 mL with dist. H₂O & mix

FOR ALL TUBES

Transfer 1 mL of solution into a new tube & add 0.5 mL of DNSA then mix

Heat in a water bath for 5 min

Dilute to 10 mL with dist. H₂O & mix

Transfer aliquot to spectrophotometric cell & read Absorbance at 540 nm

0.25 H | 0.25 U
Label your sample tubes!

Prepare the sucrose standards and unknown samples simultaneously!

Only add HCl to the samples labelled ‘H’
IMPORTANT - SAFETY

Do not screw the lids on the tubes tightly before they are heated. Pressure will build up while they are heated and they could explode!!!

They only need to be loosely covered. Be careful though when you remove them that you do not spill any of the hot liquid.
Dilutions!

**Known volume pippetted**

**Known concentration**

Sucrose Stock Solution

**Diluted to 10 mL**

**Dilution 1**

**Dilution 2**

**Diluted to 10 mL**

**Measured Absorbance**

Transfer to cuvette
Data Analysis: Day 2

- Calculations
- Critical Appraisal of Data
- Error Analysis & Propogation
- Common Student Errors