Elemental Analysis and Stoichiometry

Background

Elemental analysis is an experiment that determines the amount (typically a percent mass) of an element in a compound. Just as there are many different elements, there are many different experimental methods for determining elemental composition. The most common type of elemental analysis is for carbon, hydrogen, and nitrogen (CHN analysis). This type of analysis is especially useful for organic compounds (compounds containing carbon-carbon bonds).

The elemental analysis of a compound enables one to determine the empirical formula of the compound. The empirical formula is the formula for a compound that contains the smallest set of integer ratios for the elements in the compound that gives the correct elemental composition by mass.

Carbon-Hydrogen (CH) analysis is performed by burning the unknown sample. As a result of the complete combustion of the compound, all of the carbon in the compound is converted to carbon dioxide gas and all of the hydrogen in the compound is converted to water vapor.

\[ C_x H_y A + z O_2 (g) \rightarrow x CO_2 (g) + y/2 H_2O (g) + A \]

The combustion is conducted in the presence of excess oxygen (\( z \gg x + y/2 \)). The symbol A represents other elements in the system, which burn to produce either a solid residue or an unreactive gas (unreactive for the purposes of this analysis).

The sample is typically wrapped in a tin capsule and inserted into a furnace held at 1200°C. A steady stream of an unreactive gas (e.g., helium) is passed through the furnace and a burst of pure oxygen gas is added as the sample capsule is inserted into the oven. As the sample burns, the temperature rises to approximately 1700°C. The furnace contains a catalyst (Cr2O3) that facilitates the complete combustion of the sample. Copper turnings are also present to ensure that any nitrogen in the sample is reduced to N2, which is unreactive.

As the gas leaves the furnace, the carbon dioxide and water vapor produced by the combustion reaction are carried out of the furnace. The gas stream first passes through a desiccant, typically Mg(ClO4)2, which removes all moisture from the gas stream. Next the gas stream passes through a tube containing NaOH, which removes all carbon dioxide from the gas stream (forming NaHCO3).

By weighing the tubes containing Mg(ClO4)2 and NaOH before and after the combustion reaction, it is possible to determine the amounts of water and carbon dioxide produced by the combustion reaction and thus the amounts of hydrogen and carbon in the unknown compound.

The website, [http://www.chm.davidson.edu/vce/stoichiometry/ch.html](http://www.chm.davidson.edu/vce/stoichiometry/ch.html), at the very bottom of the webpage, shows the experimental apparatus. The furnace lies at the left side of the image. The tubes containing Mg(ClO4)2 (H2O Trap) and NaOH (CO2 Trap) rest on the balances, which permits their masses to be determined. (The gas line connecting the two tubes is very light and flexible. For the purposes of this simulation, it is assumed they do not perturb the measurements of the masses of the tubes.)
Experimental procedure:

1. Go to the website: [http://www.chm.davidson.edu/vce/stoichiometry/ch.html](http://www.chm.davidson.edu/vce/stoichiometry/ch.html) and scroll to the bottom of the page.

2. Create your data table in Microsoft Word
   a. Open Microsoft Word
   b. Type your name and class period
   c. Insert a table with 6 columns and 6 rows
   d. Labels the columns:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compound Contains</th>
<th>Mass of Sample (grams)</th>
<th>Molecular Mass</th>
<th>Grams of Water</th>
<th>Grams of Carbon Dioxide</th>
</tr>
</thead>
</table>

e. Label the rows:

   A
   B
   C
   D
   Random

3. Select Sample A.

4. Zero the readings on the balances.
   
   To make calculations easier, click the "Tare" button on each balance to zero the mass reading. Any increase in mass during the experiment will thus be attributable to the adsorption of water or carbon dioxide from the combustion of the sample.

5. Record the elements in the compound, the mass of the sample and the molecular mass in the data table.

6. Start the combustion reaction by selecting the "Burn Sample" button.

7. Wait a few seconds for the combustion reaction to occur, the water and carbon dioxide to be absorbed, and the mass readings to stabilize.

8. Record the masses of water and carbon dioxide produced by the combustion of the sample in your data table.

9. Repeat steps 2-7 for samples B, C, D and Random

10. Save your data table. Print two copies and turn in one copy today.
11. Insert a second table under your data table, with 6 columns and 6 rows. The rows should be labeled the same as for your data table. Label the columns:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Moles of Water</th>
<th>X</th>
<th>Moles of Carbon Dioxide</th>
<th>Y</th>
<th>Formula for Sample</th>
</tr>
</thead>
</table>

12. For each sample, calculate the number of moles of water and carbon dioxide using the mass of water and carbon dioxide. Record the number of moles in your results table.

13. Determine the formula for each sample, using the moles of water and carbon dioxide and the formula below:

\[ C_xH_yA + z \text{ O}_2 \rightarrow x \text{ CO}_2 + \frac{y}{2} \text{ H}_2\text{O} + A \]

The symbol A represents other elements in the system, such as Cl, N or O.

X and Y need to be whole numbers. Your results for the number of moles will be decimals. To convert the decimals to whole numbers, divide by the smaller of the two numbers.

<table>
<thead>
<tr>
<th>Decimal part of number</th>
<th>Multiple both by:</th>
</tr>
</thead>
<tbody>
<tr>
<td>.25, .75</td>
<td>4</td>
</tr>
<tr>
<td>.33 or .66</td>
<td>3</td>
</tr>
<tr>
<td>.5</td>
<td>2</td>
</tr>
</tbody>
</table>
Report Requirements

Title:

Include a title, your name, the date of the activity and the date it is turned in.

Purpose:

1. What topic is currently being studied in class?
2. How does the Elemental Analysis related to this topic?

Procedure:

Write a paragraph describing the simulator and how it was used to generate the data.

Data:

Include the completed data table.

Calculations:

Include a typed table with the calculated values for the moles of both water and carbon dioxide, the coefficients for the combustion equation (X and Y) and the formula for each sample.

Analysis:

1. Describe, in a paragraph, how the mass of the different elements related to the moles of the elements needed for the reaction to occur. Compare how the coefficients relate to the formula of the sample.
2. The simulator assumes that there is an excess of oxygen. Predict what would happen if there was less oxygen.

Conclusion:

1. Describe your results. Which sample generated the most carbon dioxide? Which generated the most water? How does that relate to the sample's formula?
2. Find two careers where Elemental Analysis may be used. How could Elemental Analysis be used in these fields? (Future Application)
3. Describe any errors that might have occurred in the activity. (Review the steps – did you forget to do any of them? Did you check your calculations?) How could you check that your formula was correct?