Determination of Ca\textsuperscript{2+} and Mg\textsuperscript{2+} in an Aqueous Sample

Mary Jane

October 10, 2011

UCI ID number: XXXXXXX

TA: XXX
Lab section: XXX
Unknown code: XX

Comment [G1]: this lab makes me have faith in reporting again! If you keep on writing your reports like this, you will get an A in the class. I emailed Sergey about showing other people this lab, it's really that good. 89/90 for lab report. 5/5 for notebook pages. 4.5/5 for prelab

98.5/100 GOOD JOB!

Comment [G2]: 1/1 title page

Comment [SN3]: This is a fake name. The actual student is enrolled in the 2011.
Abstract
An ethylenediaminetetraacetic acid (EDTA) titrant solution was made by diluting it with nanopure water. The concentration of the EDTA solution was calculated to be 0.001619 M. A blank containing some Ca$^{2+}$ and Mg$^{2+}$ was made and titrated three times in order to obtain data used to calculate corrected volume. The unknown samples were prepared by diluting the fraction of the unknown by a factor of 5 and adding spike, water, buffer, and Eriochrome Black T indicator to the samples. Three titrations of the unknown were carried out to determine the concentration of the mixture. The amount of Ca$^{2+}$ and Mg$^{2+}$ in the unknown was calculated and found to be 0.049 ±0.004 M. The concentration of Ca$^{2+}$ in the mixture was then determined. Three blank and three unknown titrations were also performed, but Hydroxynaphthol Blue indicator and NaOH was added instead of Eriochrome Black T and buffer, respectively. The undiluted unknown was also used in this case. The concentration of Ca$^{2+}$ was calculated to be 0.020±0.002 M, giving the concentration of Mg$^{2+}$ to be 0.029±0.004 M.

Experimental Section
Deviations from the above procedure or any unusual circumstances pertaining to the experiment are:

- The number of drops of Eriochrome Black T indicator differed in the titrations because after adding specific drops of indicator described below, the color of the solution turned pink enough. If more drops were added, the color of the solution would be too dark. Therefore, some samples contain fewer drops of indicators.
  - For the first blank, 4 drops were added
  - For the second blank, 3 drops were added
  - For the last blank and other 3 unknown samples, only 2 drops of indicator were added.

Results
The concentration of the standard EDTA solution, 0.3022 g of the dried chelating agent was weighed and diluted in a 500.0mL of nanopure water. By assuming that dehydrate is contaminated by 0.3% wt with water, the actual mass of the EDTA is 0.997 of the total mass.
Therefore, in order to calculate the concentration of the EDTA solution, the following formulas were used:

\[
\text{Mass}_{\text{EDTA}} = M_{\text{total}} - M_{\text{total}} \times 0.3/100 = 0.997M_{\text{total}} \tag{1}
\]

\[
\text{Moles of EDTA} = \frac{\text{Mass}_{\text{EDTA}}}{\text{Molar Weight}_{\text{EDTA}} (MW372.25)} \tag{2}
\]

\[
\text{Concentration of EDTA} = \frac{\text{Moles of EDTA}}{\text{Volume of nanopure water}} \tag{3}
\]

After performing the calculation with Excel, the 0.001619M of EDTA was obtained. The mass of EDTA was .3013g, giving the mole of 8.094E-4. In addition, the uncertainty of the concentration of the standard EDTA solution can be calculated by propagation of errors. Once it was assumed that the uncertainty for the mass of EDTA is ±0.01g and for the volume of nanopure water is ±0.03mL, the following formula can be used to calculate the uncertainty of the concentration. The uncertainty was determined to be ±0.000054 M.

\[
\Delta C_{\text{EDTA}} = \text{data}
\]

### Data

<table>
<thead>
<tr>
<th>#</th>
<th>Titration</th>
<th>Indicator</th>
<th>Endpoint volume observed</th>
<th>Volume of EDTA added</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Blank for Ca(^{2+}) and Mg(^{2+})</td>
<td>Eriochrome Black T</td>
<td>6.7 mL</td>
<td>6.7-0 = 6.7 mL</td>
</tr>
<tr>
<td>2</td>
<td>Blank for Ca(^{2+}) and Mg(^{2+})</td>
<td>Eriochrome Black T</td>
<td>12.9 mL</td>
<td>12.9-6.7= 6.2 mL</td>
</tr>
<tr>
<td>3</td>
<td>Blank for Ca(^{2+}) and Mg(^{2+})</td>
<td>Eriochrome Black T</td>
<td>18.7 mL</td>
<td>18.7-12.9= 5.8 mL</td>
</tr>
<tr>
<td>4</td>
<td>Unknown for Ca(^{2+}) and Mg(^{2+})</td>
<td>Eriochrome Black T</td>
<td>31.8 mL</td>
<td>31.8-19.5= 12.3 mL</td>
</tr>
<tr>
<td>5</td>
<td>Unknown for Ca(^{2+}) and Mg(^{2+})</td>
<td>Eriochrome Black T</td>
<td>45.3 mL</td>
<td>45.3-32.8= 12.5 mL</td>
</tr>
<tr>
<td>6</td>
<td>Unknown for Ca(^{2+}) and Mg(^{2+})</td>
<td>Eriochrome Black T</td>
<td>14.4 mL</td>
<td>14.4-2.3= 12.1 mL</td>
</tr>
<tr>
<td>7</td>
<td>Blank for Ca(^{2+})</td>
<td>Hydroxynaphthol Blue</td>
<td>22.0 mL</td>
<td>22.0-15.2= 6.8 mL</td>
</tr>
<tr>
<td>8</td>
<td>Blank for Ca(^{2+})</td>
<td>Hydroxynaphthol Blue</td>
<td>28.7 mL</td>
<td>28.7-22.9= 5.8 mL</td>
</tr>
<tr>
<td>9</td>
<td>Blank for Ca(^{2+})</td>
<td>Hydroxynaphthol Blue</td>
<td>33.5 mL</td>
<td>33.5-28.7= 4.8 mL</td>
</tr>
<tr>
<td>10</td>
<td>Unknown for Ca(^{2+})</td>
<td>Hydroxynaphthol Blue</td>
<td>23.9 mL</td>
<td>23.9-5.5= 18.4 mL</td>
</tr>
<tr>
<td>11</td>
<td>Unknown for Ca(^{2+})</td>
<td>Hydroxynaphthol Blue</td>
<td>42.7 mL</td>
<td>42.7-23.9= 18.8 mL</td>
</tr>
<tr>
<td>12</td>
<td>Unknown for Ca(^{2+})</td>
<td>Hydroxynaphthol Blue</td>
<td>36.45 mL</td>
<td>36.45-18.65= 17.8 mL</td>
</tr>
</tbody>
</table>
**Discussion**

**Determination of Total Ca(II) and Mg(II) in a Water Sample.** Three unknown titrations were performed in order to obtain the data required to determine the concentration of Ca\(^{2+}\) and Mg\(^{2+}\) in the sample. Meanwhile, three blank titrations were carried out prior to the unknown titrations in order to obtain the data used to calculate corrected volume. The titration of the blank revealed that a 6.2 mL correction was necessary. This number represents the volume of EDTA needed to titrate the blank. In order to obtain this number, the volumes of EDTA used to titrate blank samples were averaged out. The average number, \(V_1^{\text{blank}}\) was then used to subtract from the volume of EDTA required to titrate the solutions contain unknown and Ca/Mg spike (\(V_1^{\text{unknown}}\)) in order to calculate the volume of EDTA needed to bind to all the metals. The formula for correct volume is shown below.

\[
V_1^{\text{corrected}} = V_1^{\text{unknown}} - V_1^{\text{blank}}
\]  

\[
\Delta V_1^{\text{corrected}} = \sqrt{\left(\Delta V_1^{\text{unknown}}\right)^2 + \left(\Delta V_1^{\text{blank}}\right)^2}
\]

Here, \(\Delta V_1^{\text{corrected}}\) represents the uncertainty of the correct volume which was calculated by propagating errors. \(\Delta V_1^{\text{unknown}}\) and \(\Delta V_1^{\text{blank}}\) stand for the standard deviations of the \(V_1^{\text{unknown}}\) and \(V_1^{\text{blank}},\) respectively. By taking the square root of the sum of the squares of the individual absolute standard deviation, the standard deviation of a difference can be obtained.

All the data reported below have been corrected. The average of the corrected volume as well as its uncertainty is shown below.

<table>
<thead>
<tr>
<th>Trial</th>
<th>Volume of EDTA added</th>
<th>Corrected Volume</th>
<th>The average of the Corrected Volume</th>
<th>Uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12.3 mL</td>
<td>6.1 mL</td>
<td>(6.1+6.3+5.9)/3=6.1 mL=0.0061L</td>
<td>(\Delta V_1^{\text{corrected}}) = 0.49mL</td>
</tr>
<tr>
<td>2</td>
<td>12.5 mL</td>
<td>6.3 mL</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>12.1 mL</td>
<td>5.9 mL</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The (average) corrected volume of EDTA used to titrate the unknown is 0.0061±0.0005 L.

Once the corrected volume was calculated, the combined concentration of both Ca\(^{2+}\) and Mg\(^{2+}\) can be calculated by multiplying the number of moles of EDTA used to bind to the metals...
to the number of moles of metals in $V_{\text{aliquot}}$ (volume of diluted solution) and accounting for the 5 fold dilution. By multiplying the corrected volume with the concentration of EDTA, the moles of EDTA can be obtained:

$$V_1^{\text{corrected}} \times C_{\text{EDTA}} \times$$

The error of the concentration of Ca$^{2+}$ and Mg$^{2+}$ can be calculated by multiplying the standard deviation of the corrected volume to the concentration of the EDTA and to dilution factor of divided by the volume of the diluted solution. The uncertainties associated with preparation of the standard EDTA solution were neglected because the number of uncertainties was small. It would have very small to no effect on the uncertainty of the concentration of Ca$^{2+}$ and Mg$^{2+}$.

$$\Delta V_1^{\text{corrected}} \times C_{\text{EDTA}} \times$$

The results of these calculations, which were done in Excel are as followed. The concentration of Ca$^{2+}$ and Mg$^{2+}$ is 0.049 M, while its error is ±0.004 M.

### Determination of Total Ca(II) Content in the Mixture.

Similarly to the Determination of Total Ca(II) and Mg(II) in a Water Sample part, three blank titrations and three unknown titrations were performed in order to determine the concentration of Ca$^{2+}$ in the mixture. However, Hydroxynaphtol Blue was used as the indicator instead because it worked better at pH. In addition, undiluted unknown and 50wt% NaOH were added to the solution instead of diluted unknown and buffer. In this case, three blank titrations reveal that 5.8mL correction was required. This number would be used to subtract from the volume used to titrate unknown samples. The equations below were used to calculate the corrected volume and the uncertainty.

$$V_2^{\text{corrected}} = V_2^{\text{unknown}} - V_2^{\text{blank}}$$

$$\Delta V_2^{\text{corrected}} = \frac{\Delta V_1^{\text{corrected}}}{\sqrt{3}}$$

<table>
<thead>
<tr>
<th>Trial</th>
<th>Volume of EDTA added</th>
<th>Corrected Volume</th>
<th>The average of the Corrected Volume</th>
<th>Uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>18.4 mL</td>
<td>18.5-12.7 mL</td>
<td>(12.7+13.0+12.0)/3= 12.6 mL=0.0125</td>
<td>$\Delta V_1^{\text{corrected}}$</td>
</tr>
</tbody>
</table>
The (average) corrected volume of EDTA used to titrate the unknown is 0.0125±0.0011 L.

Because undiluted unknown was used in these unknown titrations, there is no dilution factor in the formula used to calculate the concentration of Ca\(^{2+}\) and the error of the concentration. Moreover, like the previous, the uncertainties associated with preparation of the standard EDTA solution were neglected because it was assumed that the error was very small.

\[
\text{Corrected volume} = V_2 \text{corrected} \times C_{\text{EDTA}} \times \\
\Delta V_2 \text{corrected} \times C_{\text{EDTA}}
\]

After doing the calculation in the Excel, the concentration of Ca\(^{2+}\) was determined to be 0.020±0.002 M.

**Concentration of Mg\(^{2+}\).** Once the sum of Ca\(^{2+}\) and Mg\(^{2+}\) concentration and concentration of Ca\(^{2+}\) were determined from the first set of titration and second set of titrations, respectively. The concentration of Mg\(^{2+}\) can be calculated by subtracting the concentration of Ca\(^{2+}\) from the concentration of both Ca\(^{2+}\) and Mg\(^{2+}\). The uncertainty can also be calculated by propagating errors.

The concentration of Mg\(^{2+}\) was found to be 0.029±0.004 M.

<table>
<thead>
<tr>
<th>Metal</th>
<th>Concentration</th>
<th>Uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca(^{2+}) and Mg(^{2+})</td>
<td>0.049 M</td>
<td>±0.004 M</td>
</tr>
<tr>
<td>Ca(^{2+})</td>
<td>0.020 M</td>
<td>±0.002 M</td>
</tr>
<tr>
<td>Mg(^{2+})</td>
<td>0.029 M</td>
<td>±0.004 M</td>
</tr>
</tbody>
</table>

The molar ratio of Mg\(^{2+}\) to Ca\(^{2+}\)

Molar ratio = mole of ————
By using the formula above, the molar ratio of Mg$^{2+}$ to Ca$^{2+}$ was determined to be 1.45. Because the ratio is greater than 0.1, it can be concluded that this sample has been significantly contaminated by Dr. Evil.