CHEM 213
Chemical Analysis
Practice Exam 1

1  __10__  (of 10)
2  __10__  (of 10)
3  __20__  (of 20)
4  __5__   (of 5)
5  __5__   (of 5)

Σ__50__

KEY

Name:___________________________________________
(please print)
1. For the numbers 116.0, 97.9, 114.2, 106.8, and 108.3, find the mean, standard deviation, range, and 90% confidence interval for the mean (t = 2.132). Using the Q test, decide whether the number 97.9 should be discarded (Qtable = 0.64). (10 points)

mean:
\[
\frac{1}{5}(116.0 + 97.9 + 114.2 + 106.8 = 108.3) = 108.6
\]

standard deviation:
\[
\sqrt{\frac{(116.0 - 108.6)^2 + \ldots + (108.3 - 108.6)^2}{5 - 1}} = 7.14
\]

90% confidence interval:
\[
= 108.6 \pm \frac{(2.132)(7.14)}{\sqrt{5}} = 108.6 \pm 6.81
\]

\[
Q_{\text{calculated}}: \frac{|106.8 - 97.9|}{|116.0 - 97.9|} = 0.49 < Q_{\text{table}} (= 0.64)
\]

Therefore, 97.9 should be retained.
2. Traces of toxic, man-made hexachlorohexanes in North Sea sediments were extracted by a known process and by two new procedures, and measured by chromatography.

<table>
<thead>
<tr>
<th>Method</th>
<th>Concentration found (pg/g)</th>
<th>Standard deviation (pg/g)</th>
<th>Number of replications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional</td>
<td>34.4</td>
<td>3.6</td>
<td>6</td>
</tr>
<tr>
<td>Method A</td>
<td>42.9</td>
<td>1.2</td>
<td>6</td>
</tr>
<tr>
<td>Method B</td>
<td>51.1</td>
<td>4.6</td>
<td>6</td>
</tr>
</tbody>
</table>

a. Are the concentrations parts per million, parts per billion, or something else? (2 points)

pg/g corresponds to $10^{-12}$ g/g, which is parts per trillion

b. Is the standard deviation for procedure B significantly different from that of the conventional procedure? (4 points)

$$F_{\text{calculated}} = \frac{4.6^2}{3.6^2} = 1.63 < F_{\text{table}} = 5.05.$$ Standard deviations are not significantly different at the 95% confidence level.

c. Is the mean concentration found by procedure B significantly different from that of the conventional procedure? (4 points)
Because $F_{\text{calculated}} < F_{\text{table}}$, we can use:

$$s_{\text{pooled}} = \sqrt{\frac{s_1^2(n_1 - 1) + s_2^2(n_2 - 1)}{n_1 + n_2 - 2}}$$

$$= \sqrt{\frac{4.6^2(6 - 1) + 3.6^2(6 - 1)}{6 + 6 - 2}} = 4.13$$

$$t_{\text{calculated}} = \frac{|\bar{x}_1 - \bar{x}_2|}{s_{\text{pooled}} \sqrt{\frac{n_1n_2}{n_1 + n_2}}}$$

$$= \frac{|51.1 - 34.4|}{4.13 \sqrt{\frac{(6)(6)}{6 + 6}}} = 7.00$$

Because $t_{\text{calculated}} > t_{\text{table}}$ (2.228 for 10 degrees of freedom), the difference is significant at the 95% confidence level.
3. In the Bradford protein determination, the color of a dye changes from brown to blue when it binds to protein. The intensity of blue color (measured by absorbance of light at a wavelength of 595 nm) is proportional to protein concentration.

Protein (µg):

<table>
<thead>
<tr>
<th></th>
<th>0.00</th>
<th>9.36</th>
<th>18.72</th>
<th>28.08</th>
<th>37.44</th>
</tr>
</thead>
</table>

Absorbance at 595 nm:

<table>
<thead>
<tr>
<th></th>
<th>0.466</th>
<th>0.676</th>
<th>0.883</th>
<th>1.086</th>
<th>1.280</th>
</tr>
</thead>
</table>

a. Determine the equation of the least-squares straight line through these points in the form \(y = [m(±s_m)]x + [b(±s_b)]\) (15 points)

<table>
<thead>
<tr>
<th>(x_i)</th>
<th>(y_i)</th>
<th>(x_iy_i)</th>
<th>(x_i^2)</th>
<th>(y_i^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>0.466</td>
<td>0</td>
<td>0.217156</td>
<td>0.217156</td>
</tr>
<tr>
<td>9.36</td>
<td>0.676</td>
<td>6.327</td>
<td>87.61</td>
<td>0.456976</td>
</tr>
<tr>
<td>18.72</td>
<td>0.883</td>
<td>16.530</td>
<td>350.44</td>
<td>0.779689</td>
</tr>
<tr>
<td>28.08</td>
<td>1.086</td>
<td>30.495</td>
<td>788.49</td>
<td>1.179396</td>
</tr>
<tr>
<td>37.44</td>
<td>1.280</td>
<td>47.923</td>
<td>1401.75</td>
<td>1.6384</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(\Sigma)</td>
<td>(\Sigma)</td>
</tr>
<tr>
<td>93.60</td>
<td>4.391</td>
<td>101.275</td>
<td>2628.29</td>
<td>4.271617</td>
</tr>
</tbody>
</table>

\(x = 93.60/5 = 18.72, \ y = 4.391/5 = 0.8782\)

\(S_{xx} = \Sigma x_i^2 - (\Sigma x_i)^2/N = 2628.29 - 8760.96/5 = 876.098\)

\(S_{yy} = \Sigma y_i^2 - (\Sigma y_i)^2/N = 4.271617 - 19.280881/5 = 0.4154408\)

\(S_{xy} = \Sigma x_iy_i - (\Sigma x_i)(\Sigma y_i)/N = 101.275 - (93.60)(4.391)/5 = 19.07548\)

\(m = S_{xy}/S_{xx} = 0.0217732\)

\(b = y - mx = 0.8782 - (0.0217732)(18.72) = 0.4706052\)

\(s_r = \sqrt{\frac{S_{yy} - m^2S_{xx}}{N-2}} = \sqrt{0.4154408 - (0.0004741)(876.098)} = 0.0059738\)

\(s_m = \frac{s_r}{\sqrt{S_{xx}}} = \sqrt{\frac{0.0059738^2}{876.098}} = 0.002\)

\(s_b = s_r \sqrt{\frac{\Sigma x_i^2}{N\Sigma x_i^2 - (\Sigma x_i)^2}} = 0.0059736 \sqrt{\frac{2628.29}{(5)(2628.29) - 8760.96}} = 0.0046271\)

Equation of best line:

\(y = [0.218(±0.0002)]x + 0.471(±0.005)\)

b. An unknown protein sample gave an absorbance of 0.973. Calculate the number of micrograms of protein in the unknown and estimate its uncertainty. (5 points)

\(x = (y – b)/m = (0.973 – 0.471)/0.218 = 23.0 \ \mu g\)

If you keep more digits for m and b, \(x = 23.07 \ \mu g\).
Uncertainty in x:

\[ s_c = \frac{s_r}{m} \sqrt{\frac{1}{M} + \frac{1}{N} + \frac{(\bar{y}_c - \bar{y})^2}{m^2S_{xx}}} = \frac{0.00059738}{0.0217732} \sqrt{\frac{1}{1} + \frac{1}{5} + \frac{(0.973 - 0.8782)^2}{(0.0217732)^2876.098}} \]

\[ = 0.30 \, \mu g \]

The final answer is 23.1 ± 0.30 µg.
4. A solid mixture weighing 1.372 g containing only sodium carbonate (105.99 g/mol) and sodium bicarbonate (84.01 g/mol) required 29.11 mL of 0.734 4 M HCl for complete titration:

\[
\text{Na}_2\text{CO}_3(\text{aq}) + 2 \text{HCl}(\text{aq}) \rightarrow 2\text{NaCl} (\text{aq}) + \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g}) \\
\text{NaHCO}_3 + \text{HCl} \rightarrow \text{NaCl}(\text{aq}) + \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g})
\]

Find the mass of each component of the mixture. (5 points)

Let's denote the grams of \(\text{Na}_2\text{CO}_3\) by \(x\) and grams of \(\text{NaHCO}_3\) by \(1.372 - x\). The moles of each component must be:

Moles of \(\text{Na}_2\text{CO}_3\) = \(x\)g/105 g/mol

Moles of \(\text{NaHCO}_3\) = \((1.372 - x)\)g/84.01 g/mol

We know that the total moles of HCl used was:

\((0.02911 \text{ L})(0.7344 \text{ M}) = 0.02138 \text{ mol}\)

From the stoichiometry of the two reactions, we can say that:

\(2(\text{mol } \text{Na}_2\text{CO}_3 = \text{mol } \text{NaHCO}_3 = 0.02138 \text{ mol})\)

\(2(x/105.99) + (1.372 - x)/84.01 = 0.02138\)

\[\text{x} = 0.724 \text{ g}\]

The mixture contains 0.724 g of \(\text{Na}_2\text{CO}_3\) and \(1.372 - 0.724 = 0.648 \text{ g}\) of \(\text{NaHCO}_3\).
5. To measure the nickel content in steel, the alloy is dissolved in 12 M HCl and neutralized in the presence of citrate ion, which maintains iron in solution. The slightly basic solution is warmed, and dimethylglyoxime (DMG) is added to precipitate the red DMG-nickel complex quantitatively. The product is filtered, washed with cold water, and dried at 110 °C.

\[
\text{Ni}^{2+} + 2 \text{DMG} \rightarrow \text{Bis(dimethylglyoximate)nickel(II)} + 2\text{H}^+ \\
\text{FM 58.69} \quad \text{FM 116.12} \quad \text{FM 288.91}
\]

If 1.163 g of steel gave 0.179 g of precipitate, what is the percentage of Ni in the steel. (5 points)

For each mole of Ni in the steel, 1 mol of precipitate will be formed. Therefore, 0.179 g of precipitate corresponds to

\[
\frac{0.179 \text{ g Ni(DMG)\textsubscript{2}}}{288.91 \text{ g Ni(DMG)\textsubscript{2}/mol Ni(DMG)\textsubscript{2}}} = 6.213 \times 10^{-4} \text{ mol Ni(DMG)\textsubscript{2}}
\]

The Ni in the alloy must therefore be:

\[
6.213 \times 10^{-4} \text{ mol Ni(DMG)\textsubscript{2}} (58.69 \text{ g/mol Ni}) = 0.03646 \text{ g}
\]

The mass percent of Ni in steel is:

\[
\frac{0.03646 \text{ g Ni}}{1.1634 \text{ g steel}} \times 100\% = 3.134\%
\]