General Comments on CHN Analysis and Method Development

1) Equipment & Software
   a) The equipment does what it is suppose to do, but there are very few adjustments to finesse the GC separations (assuming combustion was good) other than GC column temperature (isocratic), flow rate.
   b) The software is Antiquated
      i) Software requires Windows NT operating system (20 year old operating system?)
      ii) The software is designed for the mass spec, not elemental analysis (CHNS).
      iii) To get to the TCD chromatograms is not too hard, but care must be taken on integration, parameters have to be set just right.
      iv) The integrated chromatograms can not be saved, only printed.
      v) The calibration curve (or K factor) must be determined manually using Excel.

2) Weighing
   a) The method weighing of samples is critical. Sample sizes are generally 0.5-1.5 mg. Therefore the balance in Dr. Minter’s lab was used.
      i) The weighing procedure developed must be followed meticulously to get both accuracy and precision.
      ii) The samples need to be weighed while the lab is not very busy. Once the lab gets busy, then you can forget about getting good weights.

3) Logistics
   a) The ability of having access to the EA is a plus but prevents several logistical challenges.
      i) Getting access to both Dr. Kischner’s and Dr. Minter’s lab can be a challenge.
      ii) Getting into Dr. Minter’s lab to weigh samples while the lab is not busy is a challenge.

4) Results
   a) The final results obtained from the instrument were for polymer analysis, therefore the combustion in general was challenging. The results for the polymer, poly-phenylene (estimated 6 ring average), although are not publishable gave reasonable results. A sample of the same polymer was sent to an outside lab for CHBr analysis.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Avg. %C</th>
<th>Avg. % H</th>
<th>Avg. % Br</th>
</tr>
</thead>
<tbody>
<tr>
<td>In House</td>
<td>69.66%</td>
<td>3.98%</td>
<td>N/A</td>
</tr>
<tr>
<td>(10 samples)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Urea calibration standard</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Outside Lab</td>
<td>69.49%</td>
<td>4.65%</td>
<td>21.93%</td>
</tr>
<tr>
<td>(2 samples)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

b) As can be seen, the carbon percentages match very well, but the hydrogen percentages are off. The in-house hydrogen results were very possibly low due to the chromatography of the water separation. The water peak tails very badly in the chromatogram, very possibly causing error in the quantification. With the method in house, the carbon and nitrogen peaks have baseline resolution and do not tail.

c) It should be noted, that more “typical” organic compounds may combust better than the polymer samples and provide more accurate data.

5) Recommendations

a) Using a different standard (acetanilide instead of urea) may give better results (standard routinely used).

b) Packing a new combustion/reduction columns or changing to a single column system may eliminate some of the water tailing due to combustion and/or extra column effects.

c) Adjusting the furnace temperatures may resolve combustion issues as well (if there are any).