Qualitative Analysis
Reminder – Goggles must be worn at all times in the lab!

PRE-LAB DISCUSSION:
Qualitative analysis is the process by which a scheme of simple physical tests is used to identify an unknown. In this case, we will use tests such as solubility and pH, as well as reactions with reagents such as acids, bases, Lugol’s iodine and Benedicts’ solution.

This series of tests, when performed in the proper order, is capable of distinguishing each compound from all of the others. If steps are skipped, or the tests are performed out of sequence, then all bets are off, and mistakes are likely to occur. In addition, these tests are all semi-micro tests, and require only small quantities of each compound in order to complete the identification scheme.

You will first have the opportunity to practice these techniques with each of the ten solids, labeled and identified for you. This will give you confidence that you understand the experimental procedures. You will then be asked to identify a group of unknowns within your lab group.

Because of the number of tests you must perform and the limited amount of glassware that you have available, it is especially important to clean test tubes and glass plates between tests. Compounds or reagents left behind by previous tests are a common cause of poor results in qualitative schemes such as this.

<table>
<thead>
<tr>
<th>Compounds</th>
<th>Reagents</th>
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</thead>
<tbody>
<tr>
<td>Sodium acetate</td>
<td>Magnesium Sulfate</td>
</tr>
<tr>
<td>Sodium carbonate</td>
<td>Calcium carbonate</td>
</tr>
<tr>
<td>Sodium chloride</td>
<td>Boric acid</td>
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<tr>
<td>Sodium bicarbonate</td>
<td>Glucose</td>
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<tr>
<td>Starch</td>
<td>Calcium sulfate</td>
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<tr>
<td>Starch</td>
<td>Lugol’s Iodine</td>
</tr>
<tr>
<td>VInegar</td>
<td>Universal indicator</td>
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<tr>
<td>VInegar</td>
<td>Benedict’s solution</td>
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<tr>
<td>VInegar</td>
<td>Phenolphthalein</td>
</tr>
<tr>
<td>VInegar</td>
<td>Sodium hydroxide</td>
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<tr>
<td>VInegar</td>
<td>Vinegar</td>
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</tbody>
</table>

PURPOSE:
To practice experimental techniques of qualitative analysis and to use those techniques to identify unknown white solids.

PROCEDURE:
Step 1
The first test that you perform is a water solubility test. Take a spatula of the compound and place it in a medium test tube with 1 mL of distilled water. Stir the compound, but do not heat the solution.
- If the compound is not soluble (does not dissolve in water) then proceed to Step 2
- If the compound is soluble (dissolves in water) then proceed to Step 4

Step 2
The three compounds that are not soluble in water are starch, calcium carbonate, and calcium sulfate. Place a spatula of the compound on a glass plate, and place several drops of vinegar on the compound. Vinegar contains acetic acid – acids react with carbonates, producing gaseous carbon dioxide.
- If the compound reacts with vinegar, producing CO₂, it is calcium carbonate
- If the compound does not react with vinegar, then proceed to Step 3

Step 3
Cornstarch and calcium sulfate do not react with vinegar. Starch can be identified using Lugol’s iodine solution as a reagent. Place a spatula full of the compound on a glass plate, and apply several drops of Lugol’s iodine solution.
- If the compound turns black in contact with the iodine solution, you have starch
- If the compound does not turn black with iodine solution, you have calcium sulfate
Step 4
The water soluble compounds are sodium acetate, sodium chloride, sodium bicarbonate, sodium carbonate, boric acid, glucose, and magnesium sulfate.

Place a spatula of the compound on a glass plate, and place several drops of vinegar on the compound. Vinegar contains acetic acid – acids react with carbonates and bicarbonates, producing gaseous carbon dioxide.

- If the substance reacts with vinegar, producing bubbles of CO₂, then proceed to Step 5
- If the substance does not produce CO₂ bubbles with vinegar, then proceed to Step 6

Step 5
Both sodium carbonate and sodium bicarbonate react with vinegar to produce CO₂. Solutions of the two salts can be distinguished by the degree with which they react to phenolphthalein. Bicarbonate ion is slightly basic in solution, while carbonate ion is more strongly basic in solution.

Place a spatula of the solid in a medium test tube, and dissolve in 1 mL of distilled water. Add one drop of phenolphthalein to the test tube.

- If the solution turns a pale pink the solid is sodium bicarbonate
- If the solution turns a bright pink or fuchsia, the solid is sodium carbonate

Step 6
The remaining compounds are sodium acetate, sodium chloride, boric acid, glucose, and magnesium sulfate.

Magnesium hydroxide is insoluble in water. Place a spatula of the solid in a medium test tube, and dissolve in 1 mL of distilled water. Add several drops of NaOH solution to the test tube.

- If a gelatinous precipitate (an insoluble solid) forms (usually at the top of the water layer), the solid is magnesium sulfate
- If no precipitate forms, then proceed to Step 7

Step 7
The remaining compounds are sodium acetate, sodium chloride, boric acid, and glucose

Place a spatula of the solid in a medium test tube, and dissolve in 1 mL of distilled water. Add two drops of Universal Indicator solution, and mix the solution with a clean glass stir rod. Use the colorimetric scale on the side of the Universal Indicator bottle to estimate the pH of the solution.

- The acetate ion is basic (sodium ion is neutral), so if the pH of the solution is basic, the solid is sodium acetate
- If the pH of the solution is acidic, then the solid is boric acid
- If the pH of the solution is neutral, then proceed to Step 8

Step 8
The remaining solids are glucose and sodium chloride. Glucose is a monosaccharide (single sugar), and can be identified by the way in which it reacts with Benedict’s solution.

Place a spatula of the solid in a medium test tube, and dissolve in 1 mL of distilled water. Add ten drops of Benedict’s solution, and heat the test tube briefly over a cool Bunsen burner flame.

- If the solution reacts positively to Benedict’s reagent by turning green, then eventually orange, then the solid is glucose.
- If the solution does not react positively to Benedict’s reagent, then the solid is sodium chloride