**White Powder Lab**

**Introductions**

In this lab we will identify 10 unknown substances by following a qualitative analysis flow chart. We will also use a variety of chemical and physical tests to identify 10 common household substances, all of which are white solids.

**Background**

The process of determining the identities of unknown substances is called qualitative analysis. Qualitative analysis schemes are generally summarized in a flow diagram, like the one shown below. A flow diagram is designed with procedural steps on the vertical lines, the possible test results on the horizontal lines, and the resulting identifications in the boxes.

Qualitative analysis procedures include physical tests as well as chemical tests. The physical tests in this lab are melting point determination and solubility in water or in alcohol. The chemical reactions or tests in this lab are with iodine, vinegar, sodium hydroxide, and phenolphthalein. All of the chemical tests involve either formation of a precipitate (solid), color change, or evolution of gas bubbles.

**Materials Needed**

**Unknown Samples (in alphabetical order)**

|  |  |  |
| --- | --- | --- |
| Boric acid, H3BO3 | Magnesium sulfate, MgSO4 | Sodium Chloride, NaCl |
| Calcium carbonate, CaCO3 | Sodium bicarbonate, NaHCO3 | Sodium hydroxide, NaOH |
| Calcium sulfate, CaSO4 | Sodium carbonate, Na2CO3 | Sucrose, C12H22O11 |
| Cornstarch |  |  |

**Chemicals/Test Reagents**

|  |  |  |
| --- | --- | --- |
| Iodine tincture, 6 drops | Sodium hydroxide solution, 0.2 M, 18 drops | Isopropyl alcohol solution, 12mL |
| White vinegar, 4mL | Phenolphthalein solution,1%, 1mL | Deionized water |

**Equipment:**

|  |  |  |
| --- | --- | --- |
| Test tubes, 13x100mm, 10 | Stirring rod | Ring stand and ring |
| Test tube rack | Graduated cylinder, 10mL | 250mL beaker |
| Marking pen | Bunsen burner |  |

**Pre-Lab Notes**

The steps of the procedure written below correspond to the qualitative analysis flow chart provided. As each step is followed, record detailed observations of your results in the data table you have created in your lab book.

Examine the flow chart that has been provided. The numbers 1 – 13 are provided next to some of the possible results. Write the corresponding number in your data table as you go through the flow chart. For example, if unknowns A, B, And C are water-insoluble (which is result #1) and unknowns D – J are water-soluble then write a "1" by A, B, and C and record "Insoluble in H2O," and for D – J record "Soluble" in H2O" on your data sheet.

**Procedure**

* 1. Label 10 test tubes A – J with a marking pen and place the tubes in a test tube rack.
  2. Place a **small scoop** of each of the 10 unknown substances, A – J, into the appropriate test tube (*Note*: the results will be affected if you use too much of a sample).
  3. Add approximately 5mL of deionized water to each tube (*Note*: this can be efficiently accomplished by measuring 5mL once using a 10mL graduated cylinder. Pour the 5mL of water into test tube A and then add water to each of the 9 remaining tubes to the same height of the liquid in tube A).

1. a. Stir the contents of each tube with the stirring rod. Be sure to rinse the stirring rod with deionized water between tubes.
   * 1. Record observations of which substances are soluble and which are insoluble in water. Remember to record both the result (#1) as well as the written observation (*Note*: some soluble solids may take longer to dissolve than others). Only three of the unknowns – cornstarch, calcium sulfate, and calcium carbonate – will not readily dissolve in water and are considered insoluble.
   1. Following the flow chart, take the three tubes containing the insoluble substances from step 4, and add 2 drops of iodine tincture to each. Two of the tubes will show no reaction with iodine and will be an orange-brown color. The contents of one tube will turn a deep blue color. The deep blue color is a starch-iodine complex which positively indicates ***cornstarch***.
2. a. Dispose of the contents of the two tubes that did not react with iodine. Rinse out the tubes. Prepare fresh tubes of these two unknowns by placing a small scoop of the solid into the appropriate tube. **Do not add** **water**.
   * 1. Add approximately 10 drops of vinegar to these two tubes and note whether gas bubbles are produced. The evolution of carbon dioxide gas positively identifies ***calcium carbonate***. The remaining solid must be ***calcium sulfate***. Record the numbers and observations.
3. The other seven solids are water soluble. To each of the seven tubes from step 4, add 3 – 4 drops of phenolphthalein solution. Two of the unknowns, sodium hydroxide and sodium carbonate dissolve in water to produce alkaline solutions basic enough to give a bright pink color upon addition of phenolphthalein. Do not be concerned with precipitate (solid) formation or a faint pink color at this point.
4. a. Dispose of the contents of the two tubes that tested positively in step 7. Rinse out the tubes. Prepare fresh tubes of these two unknowns by placing a small scoop into the appropriate tube. Do not add water.
   * 1. Add approximately 10 drops of vinegar to each tube and note whether gas bubbles are produced. The evolution of carbon dioxide gas positively identifies ***sodium carbonate***. The remaining solid must be ***sodium hydroxide***.
5. a. Dispose of the contents of the tubes containing the five solids that remain to be identified. Rinse out the tubes. Prepare fresh tubes of these five unknowns by placing a small scoop into the appropriate tube.
   * 1. Add 5mL of distilled or deionized water to the five tubes and stir as in step 4 to dissolve the solids.
6. Add 3 drops of 0.2M NaOH to each tube. All of the tubes should remain clear except one which gives a white precipitate. This white precipitate positively identifies ***magnesium sulfate***, which forms an insoluble hydroxide upon addition of sodium hydroxide.
7. a. Dispose of the contents of the tubes containing the four solids that remain to be identified. Rinse out the tubes. Prepare fresh tubes of these four unknowns. Do not add water.
   1. Add approximately 10 drops of vinegar to each tube and note whether gas bubbles are produced. The evolution of carbon dioxide gas positively identifies ***sodium bicarbonate***.
8. a. Dispose of the contents of the three remaining tubes. Rinse out the tubes and prepare fresh tubes of these three unknowns. Do not add water.
   1. Add approximately 5mL of isopropyl alcohol to each tube. Stir the contents of each tube to attempt to dissolve the solids. Of the three solids, only boric acid dissolves readily in alcohol; thus, this test is a positive identification for ***boric acid***.
9. a. Dispose of the contents of the two remaining tubes. Rinse out the tubes. Prepare fresh tubes of these two unknowns. Do not add water.
   1. Hold each tube with a test tube holder and heat it gently with a Bunsen burner. The solid in one tube will turn brown, smell sweet, and begin to melt in 1–2 minutes. **Cease heating the tube once you have** **determined that the contents are changing**. Place the hot test tube on the lab top. Do not place it in theplastic test tube rack. This change is an indication that the material has a low melting point and that it is ***sucrose***. The other solid will not change as it is heated. This indicates that the solid has a high meltingpoint and is ***sodium chloride***.