

# CHEM 1112: Separation and Identification of Unknown Substances

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In this experiment you will determine the identity of several unknown substances. Each student will have their own unknowns. The first unknown will be a pure compound it will have a cation and an anion or it will be sand. The second unknown will be a mixture of two compounds. One compound of the mixture is soluble in water (hence, it will have a cation and an anion) and the other is not (it may be sand or a cation and an anion).

## Procedure

The method used to identify the unknown substances is to perform a series of tests on small samples of your unknowns and compare the results of those tests with results of the same tests performed on known samples and/or comparison with properties reported for these known substances. More detailed descriptions of these tests are found in the following paragraphs and you may find it useful to create a flow chart to help organize these analyses. A sample flow chart has been included for your reference.

### 1. Solubility

#### a) Soluble Compounds

One of the first tests on an unknown material is a test of its solubility in water. Compounds that dissolve in water dissociate to form ions in solution. Identification of the compound can be achieved by separate analyses of the cations and anions using various chemical tests.

#### b) Insoluble Compounds

Compounds that do not dissolve in water are called insoluble. Generally, however, these materials can be dissolved in acidic or basic solutions. By dissolving these insoluble compounds in acidic or basic solution we are able to proceed with the chemical analysis of cations and anions using similar methods for soluble compounds.

#### c) Mixtures

Mixtures containing a soluble compound with an insoluble compound **must** be separated before further analysis can be done. Separation can easily be achieved by dissolving the soluble compound in water. The insoluble compound will remain at the bottom in solid form and the soluble compound can be removed by decanting the liquid into a separate test tube. The insoluble compound should be washed with water several times to remove all traces of soluble compound before subsequent analysis.

### 2. Cations and Anions

Generally, cations and anions are identified separately using various chemical tests that rely on the differences in solubilities between various materials.

For example, suppose an unknown contains either  $\text{Ca}^{2+}$  or  $\text{Mg}^{2+}$  cations. To identify which cation is present, a solution of  $\text{Na}_2\text{SO}_4$  can be added to the unknown. Since  $\text{CaSO}_4$  and  $\text{MgSO}_4$  have different solubilities we can use the sulfate test to determine the presence of  $\text{Ca}^{2+}$  or  $\text{Mg}^{2+}$ . The solubility of  $\text{CaSO}_4$  is 0.3 g/100 ml whereas, the solubility of  $\text{MgSO}_4$  is 71 g/100 ml.

Therefore, we would expect a precipitate to form if the unknown contained  $\text{Ca}^{2+}$  cations.

Conversely, suppose an unknown contains either  $\text{SO}_4^{2-}$  or  $\text{Cl}^-$  anions. Again, we can use the difference in solubilities of various compounds to distinguish between the two anions. Specifically, we know that  $\text{CaSO}_4$  is insoluble but  $\text{CaCl}_2$  is soluble. Therefore, addition of a solution of  $\text{CaCl}_2$  to our unknown could determine the presence of  $\text{SO}_4^{2-}$  anions (a precipitate,  $\text{CaSO}_4$ , would form).

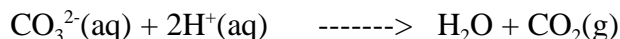
However, if the unknown contained either  $\text{SO}_4^{2-}$  or  $\text{CO}_3^{2-}$  anions, addition of  $\text{CaCl}_2$  solution would cause the precipitation of either  $\text{CaCO}_3$  or  $\text{CaSO}_4$ . Therefore, this test could not be used to distinguish the  $\text{SO}_4^{2-}$  from the  $\text{CO}_3^{2-}$ . A good method to distinguish these two anions is by using a  $\text{MgCl}_2$  solution.  $\text{MgCO}_3$  is insoluble whereas  $\text{MgSO}_4$  is soluble.

### 3. Solution pH

The acidity of a solution can be measured with pH paper. Use a stirring rod to deposit a drop of the solution onto a piece of pH paper. A high pH indicates a basic solution, characteristic of carbonates and bicarbonates. Carbonates are more basic than bicarbonates. Test solutions of known compounds and compare. (Note: Obviously this test can only be used to identify soluble carbonates and bicarbonates since insoluble compounds must be dissolved in acid or base.)

### 4. Addition of Acid or Base

a) Insoluble carbonates and bicarbonates can be identified by adding an acid such as acetic acid. Carbon dioxide gas is formed upon addition of the acid and bubbles are observed coming out of solution.



b) All ammonium compounds are soluble and a good test for the presence of  $\text{NH}_4^+$  is the addition of  $\text{Na}_2\text{CO}_3(\text{aq})$ . This causes the evolution of  $\text{NH}_3(\text{g})$  which can be detected by placing wet pH paper above the solution.  $\text{NH}_3(\text{g})$  will cause pH paper to change color to blue indicating a high pH.

### 5. Flame Tests

Several of the cations in these unknowns can be detected and identified by the color they emit when placed in a flame. Dip a wire attached to a glass rod into a beaker of acid and place in a flame until it glows red-orange. Do this several times until the wire is clean. Dip it into a concentrated solution of either a known or unknown substance. Observe the color of the flame when the liquid is evaporating and compare with the list below.

$\text{Na}^+$  -- persistent yellow

$\text{K}^+$  -- purple-red through blue glass obscured by Na, so must be clean.

$\text{Ca}^{2+}$  -- yellowish red, greenish through blue glass

$\text{NH}_4^+$  -- feeble green

Ba<sup>2+</sup> -- green

Li<sup>+</sup> -- brilliant crimson

Sr<sup>2+</sup> -- blood red

#### 6. Melting Points / Effect of Heat

If you suspect that your unknown is one of the compounds on the attached list with a melting point below 400 °C, you can use the melting point apparatus in the lab to verify the melting temperatures of your unknown and compare with the known compounds.

Other compounds that have relatively high melting points or decomposition temperatures can be placed in a flame to observe any changes. Sand and NaCl will be unchanged. Bicarbonates often "sputter" off the spatula.

#### 7. Some Further Comments

It would be very unusual for any single test to identify an unknown uniquely. Be certain to use other tests to confirm your identification if it is possible. If the tests do not permit you to distinguish between two or more choices, then explain this in your conclusion. This experiment is worth 20 points. You will earn 4 points for each correctly identified unknown and a maximum of 8 for your report in your notebook.

### **Results**

Make a table of the results and observations from your tests on both known and unknown compounds so that you can compare results and eliminate those substances which you know can't be present. It may be helpful to design a flow chart, so that you can eliminate or confirm possibilities to aid you in the decision making process. Each student will have their own unknown.

### **Conclusions**

For your conclusions identify the unknown substances. Explain your conclusions by referring to the data in your Results table. Be certain to include the identification numbers that you get from the stockroom.

TABLE: Data for Unknown Substances\*

name	formula	MP °C	water solubility (g per 100 ml)
ammonium acetate	$(\text{NH}_4)\text{C}_2\text{H}_3\text{O}$	114	148
ammonium bicarbonate	$(\text{NH}_4)\text{HCO}_3$	108	12
ammonium carbonate	$(\text{NH}_4)_2\text{CO}_3$	d58	100
ammonium chloride	$\text{NH}_4\text{Cl}$	335	29.7
ammonium sulfate	$(\text{NH}_4)_2\text{SO}_4$	d280	76
barium carbonate	$\text{BaCO}_3$	--	0.002
barium chloride	$\text{BaCl}_2$	d113	35.7
barium sulfate	$\text{BaSO}_4$	1580	0.00022
calcium carbonate	$\text{CaCO}_3$	d825	0.0015
calcium chloride	$\text{CaCl}_2$	772	60
calcium sulfate	$\text{CaSO}_4 \times 1/2\text{H}_2\text{O}$	-1/2H <sub>2</sub> O, 163	0.3
lithium carbonate	$\text{Li}_2\text{CO}_3$	618	1.5
lithium chloride	$\text{LiCl}$	613	63.7
magnesium carbonate	$\text{MgCO}_3$	d350	0.01
magnesium chloride	$\text{MgCl}_2$	708	54
magnesium sulfate	$\text{MgSO}_4 \times 7\text{H}_2\text{O}$	-6H <sub>2</sub> O, 150	71
potassium bicarbonate	$\text{KHCO}_3$	d100	22
potassium carbonate	$\text{K}_2\text{CO}_3$	891	112
potassium chloride	$\text{KCl}$	776	35
potassium sulfate	$\text{K}_2\text{SO}_4$	588	7
sodium bicarbonate	$\text{NaHCO}_3$	-CO <sub>2</sub> , 270	7
sodium carbonate	$\text{Na}_2\text{CO}_3$	-H <sub>2</sub> O, 32	17
sodium chloride	$\text{NaCl}$	801	209
sodium sulfate	$\text{Na}_2\text{SO}_4$	884	5
strontium carbonate	$\text{SrCO}_3$	1497	0.0011
strontium chloride	$\text{SrCl}_2 \times 6\text{H}_2\text{O}$	d60	106
sand	$\text{SiO}_2$	--	i

\*This data is taken from the HANDBOOK OF CHEMISTRY & PHYSICS. Additional information about these compounds can be found there.