Microscale Analysis of Anions Procedure

Objectives

To use microscale and qualitative analysis techniques to test for the presence of various anions in solution.

Background

Although most modern chemical analysis is done spectroscopically, quick tests for specific chemicals that do not require expensive instrumentation are often needed. Situations where this might occur include field testing for various environmental contaminants, home water testing, or when quickly determining if a specific chemical is present in a sample. In these cases various spot tests and qualitative analysis procedures may be used to determine the presence or absence of a particular species in solution. Qualitative analysis means we are only testing for the presence of a particular species; not the amount. After establishing the presence of a particular species we may want to perform quantitative analysis (such as a titration) on the sample to determine the exact quantity of that species in solution. In this experiment we will focus only on qualitative testing, specifically for chemical anions.

The anions that we will be testing for in this experiment are: carbonate (CO$_3^{2-}$), sulfate (SO$_4^{2-}$), phosphate (PO$_4^{3-}$), thiocyanate (SCN$^{-}$), and chloride (Cl$^{-}$).

One of the problems that arises with spot testing for chemicals is interference; another species in solution can sometimes produce "false positive" results. However, it is possible to do follow-up tests or simple separations to distinguish such cases. Although interferences are common, there are still many ions that can be identified in mixtures (or at least determined likely to be present) by simple spot testing.

The procedures we will use in this lab involve microscale techniques that use small amounts of chemicals to facilitate rapid detection and minimize waste. All of the reactions we will do fall into the various classes of reactions that we have studied in chemistry 11 and 12. In each case you should be able to recognize the type of reaction that occurs and to write the overall net ionic equation for the reaction.

In this lab you will compare the results of these tests for:

1. a control (deionized water)
2. a known for each test consisting of the anion in question for the specific test
3. a combined known consisting of all five anions
4. an unknown containing one or more of the anions tested
Procedure

Safety

Goggles MUST be worn during this experiment. Be careful when handling acidic solutions and the molybdate reagent.

All waste must be placed in the appropriate labeled waste container. Please rinse all glassware with about 1mL of deionized water using your squirt bottle and place this rinse in the waste container as well as before rinsing any glassware under the sink.

Materials and Equipment

Obtain the following equipment from the stockroom:

1 well plate
3 small 20-50 mL beakers

Your instructor will set up an electric hotplate in one of the fume hoods.

Instructions

Setting up your well plate:

<table>
<thead>
<tr>
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<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
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</thead>
<tbody>
<tr>
<td>A</td>
<td>A1</td>
<td>A2</td>
<td>A3</td>
<td>A4</td>
<td>A5</td>
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<tr>
<td>B</td>
<td>B1</td>
<td>B2</td>
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<td>C</td>
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<td>C2</td>
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<td>D</td>
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<tr>
<td>E</td>
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<td>E2</td>
<td>E3</td>
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<td>E5</td>
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</tbody>
</table>

1. Your well plate should be oriented so that the vertical columns are numbered and the horizontal rows are lettered as shown above. We will only be using columns 1 through 5 and rows A through E in this experiment. You will need to be careful not to mix up columns and rows in this experiment and to record the results of each test on your data sheet as they occur. Be sure to analyze your well plate over both the black bench top and a white sheet of paper when making observations.

2. Using a clean dropper pipette add 2 drops of deionized water to each of the wells in row A from A1 through A5. These will be your control samples and should show no reaction.
3. Using the droppers from the stock bottles, add 2 drops of each of the solutions listed in the table below to the appropriate well in row B. This row will yield positive tests (without possible interference) for each of the anions we will be testing for. Only add one solution to each well.

<table>
<thead>
<tr>
<th>Well</th>
<th>Solution (2 drops)</th>
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</thead>
<tbody>
<tr>
<td>B1</td>
<td>1 M sodium carbonate</td>
</tr>
<tr>
<td>B2</td>
<td>0.5 M sodium sulfate</td>
</tr>
<tr>
<td>B3</td>
<td>0.5 M sodium hydrogen phosphate</td>
</tr>
<tr>
<td>B4</td>
<td>0.5 M potassium thiocyanate</td>
</tr>
<tr>
<td>B5</td>
<td>0.5 M sodium chloride</td>
</tr>
</tbody>
</table>

4. Using the dropper from the stock bottle labeled "combined knowns" add 2 drops of combined known solution to each of the wells in row C from C1 through C5. The combined known solution contains each of the anions we will be analyzing. It should give a positive test for each of the anions, but may also exhibit some interferences.

5. Finally, using a clean dropper pipette, add two drops of your unknown solution to each of the wells on row D from D1 through D5. By comparing the results of tests in this row with rows A-C you should be able to determine the contents of your unknown solution.

6. Record your unknown number on the last page of the report form.

**Test for the presence of carbonate anions:**

1. To each of the wells you prepared in column 1 (wells 1A through 1D) add 2 drops of 6 M hydrochloric acid solution. If carbonates are present you should observe effervescence (the formation of bubbles). For dilute solutions (such as the combined known in well 1C) you may need to place the well plate against a dark background to see the bubbles formed. Compare your solutions to the control solution in cell 1A (which should not form bubbles).

2. Record all your results for cells 1A through 1D on your data sheet. Write the net ionic equation for the reaction occurring between the carbonate anion and the acidic solution.

**Test for the presence of sulfate anions:**

1. To each of the wells you prepared in column 2 (wells 2A through 2D) add 2 drop of 6 M hydrochloric acid to acidify the test solutions. (You may use this step to confirm the results of your carbonate test if any of the results were ambiguous).

2. Wait for any bubbling to stop. Then add 2 drops of 1M barium chloride solution to each of these wells in column 2. A white precipitate confirms the presence of sulfate anions.

3. Record your results for cells 2A through 2D on your data sheet.

4. Write down the net ionic equation for the reaction confirming the presence of sulfate. Be sure to indicate the physical states.
**Test for the presence of phosphate anions:**

1. To each of the wells you prepared in column 3 (wells 3A through 3D) add 2 drops of 6 M nitric acid to acidify the test solutions. Again, wait for any bubbling to stop.

2. Next add 2 drops of 0.5 M ammonium molybdate, \((\text{NH}_4)_2\text{MoO}_4(\text{aq})\), to each of these wells in column 3. A yellow precipitate of ammonium phosphomolybdate, \((\text{NH}_4)_3\text{PO}_4\cdot12\text{MoO}_3(\text{s})\), establishes the presence of phosphate. Note that in some cases the precipitate may form slowly. You should wait 1-2 minutes before making any final conclusions.

3. Record your results and write the net ionic equation for the formation of this precipitate.

**Test for the presence of thiocyanate anions:**

1. To each of the wells you prepared in column 4 (wells 4A through 4D) add 2 drops of 6 M acetic acid solution, followed by 2 drops of \(2\times10^{-3}\) M iron(III) nitrate solution. A deep red color indicates the formation of an iron thiocyanate complex ion, \(\text{FeSCN}^{2+}(\text{aq})\), which establishes the presence of thiocyanate. You may need to view the well plate against a white background to see the red color.

2. Record your results and write the balanced net ionic equation for the formation of this complex ion.

**Test for the presence of chloride anions:**

(Note: the presence of thiocyanate anions will interfere with this test)

1. To each of the wells you prepared in column 5 (wells 5A through 5D) add 2 drops of 6 M nitric acid followed by 2 drops of 0.1 M silver nitrate.

2. The formation of a white precipitate confirms the presence of chloride anions in the absence of thiocyanate ions. Thiocyanate interferes with this test and will also form a precipitate (even if chloride is not present). (In some cases a pinkish-brown color may develop, however you are only interested in the presence or absence of precipitate).

3. Record your results on your data sheet noting any possible interferences. Write the balanced net ionic equation that occurs.

**Removal of thiocyanate interference for the chloride test:**

Because thiocyanate anions will interfere with this test we will perform the following additional steps to demonstrate how this interfering anion may be removed.

1. Add 2 drops of 0.5 M potassium thiocyanate to the well labeled 5E.

2. Perform the chloride test as outlined above on the contents of well 5E and note how thiocyanate anions may interfere with the test for chloride. What were the results of your test?

3. Obtain 3 small clean 20-mL or 50-mL beakers from the stockroom and label these "#1 thiocyanate", "#2 chloride", and "#3 unknown". Put about 1 mL of 0.5 M potassium
thiocyanate solution into beaker #1, put about 1 mL of 0.5 M sodium chloride solution into beaker #2, and put 1 about mL of your unknown solution into beaker #3.

4. Add about 1 mL of 6 M nitric acid to each of these three beakers.

5. Place the three beakers onto the hotplate in the fume hood and allow the solutions to come to a gentle boil. Watch the solutions closely until each has about half of the original volume but do not allow the solutions to boil down to dryness (in some cases the solution may turn green in color). If your solution does dry out you may add 1 mL of deionized water back to the solid in the beaker after it cools.

6. Remove the beakers from the hot plate using a damp paper towel and allow to cool.

7. Using a clean dropper pipette, place 2 drops of the solution in beaker 1 into well 1E.

8. Rinse your pipette and place 2 drops of the solution in beaker 2 into well 2E. Rinse your pipette and place 2 drops of the solution in beaker 3 into well 3E.

9. Test the solutions in wells 1E, 2E, and 3E for chloride. The solution in well 1E (thiocyanate) should now give a negative chloride test; while the solution in well 2E should give a positive chloride test. Use the results from well 3E to evaluate your unknown solution.

Clean up

Empty the contents of your well plate into the waste container and rinse the plate using your squirt bottle, collecting the rinse in the waste container as well.