Name: _____ Section: _____

Experiment #12 – Qualitative Analysis of Common Anions

The principles that are employed in the identification of cations can also be applied to the analysis of anions. The qualitative detection of anions in a sample depends on the distinctive solubility properties of particular salts of the ions and specific chemical reactions that are (ideally) unique to a particular ion. In this experiment, we will explore ways to detect the presence of CO_3^{-2}, SO_4^{-2}, PO_4^{-3}, Cl^-, and I^-. You will be testing both known and unknown solutions.

Procedure

1. Test for the Sulfate Ion

   To 1 mL of the test solution, add 6 M HNO_3 drop by drop until the solution is acidic. Then add 1 mL of 0.1 M $BaCl_2$ solution in order to produce a white precipitate of $BaSO_4$.

   $$SO_4^{-2}(aq) + Ba^{+2}(aq) \rightarrow BaSO_4(s) \qquad (1)$$

2. Test for the Carbonate Ion

   To 1 ml of a new test solution, observe to see if any gas bubbles form while adding 20 drops of 6 M HNO_3. Verify it is acidic. This gas formation is a strong indication of the presence of CO_3^{2-}.

   $$CO_3^{2-}(aq) + 2H^+(aq) \rightarrow CO_2(g) + H_2O(l) \qquad (2a)$$

3. Test for the Chloride Ion

   To 1 mL of a new test solution, add a couple drops of 6 M HNO_3 as needed to make the solution slightly acidic. Add 10 drops of 0.1 M $AgNO_3$. No precipitate proves the absence of Cl^-, Br^-, or I^-. Centrifuge the mixture. Test the clear filtrate with 1 drop of 0.1 M $AgNO_3$ for complete precipitation. If necessary, centrifuge again. Discard the filtrate. To this precipitate, add 1 mL of D.I. water, 2 drops of 6 M NH_3, and 6 drops of 0.1 M $AgNO_3$. The proportions are important, since we want to dissolve ONLY AgCl.

   $$AgCl(s) + 2NH_3(aq) \rightarrow Ag(NH_3)_2^+(aq) + Cl^-(aq) \qquad (3a)$$

   Shake the mixture well and centrifuge. Transfer the clear solution to a clean test tube, and acidify once again with 6 M HNO_3. A white precipitate of AgCl confirms the presence of Cl^-.

   $$Ag(NH_3)_2^+(aq) + Cl^-(aq) + 2H^+ \rightarrow AgCl(s) + 2NH_4^+(aq) \qquad (3b)$$

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4. Test for the Iodide Ion

   Acidify a 2 mL sample of a new test solution by adding 6 M HCl. Add 1 mL of 0.1 M $FeCl_3$ to oxidize any I^- to I_2. Add 1 mL of hexane and agitate the mixture. A purple color of I_2 in the hexane layer indicates I^- was present in the original sample.

   $$2\ I^-(aq) + 2\ Fe^{+3}(aq) \rightarrow I_2(aq) + 2Fe^{+2}(aq) \quad (4)$$

5. Test for the Phosphate Ion

   (A) If no I^- was present, mix about 2 drops of 0.5 M $(NH_4)_6Mo_7O_{24}$ reagent with 5 drops of 6 M HNO_3 to 1 mL of a new test solution.

   (B) If I^- was present, add 5 drops of 6 M HNO_3 to 1 mL of a new test solution and boil the test tube for 5 to 10 minutes to remove the iodide. Then add 2 drops of the ammonium molybdate reagent to the test solution.

   A yellow precipitate of ammonium phosphomolybdate, $(NH_4)_3PO_4 \cdot 12MoO_3$, appearing at once or after the mixture has been warmed a few minutes to 40 °C indicates the presence of PO_4^{-3}.

   $$21NH_4^+(aq) + 12[Mo_7O_{24}^{-6}](aq) + 7H_3PO_4(aq) + 51H^+(aq) \rightarrow 7(NH_4)_3PO_4 \cdot 12MoO_3(aq) + 36\ H_2O(l) \quad (5)$$

Record your observations for your known and unknown solutions below. Determine the identity of your unknown.

Experiment Observations and Results:

UNKNOWN_____ IONS PRESENT_____

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Pre-Lab Assignment:

1. Construct separate flow charts for the identification of the various five anions in a known sample. Refer to Experiment #10 for guidelines on preparing your flow charts.

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Post-Lab Assignment: Anion Analysis

1. A solution may contain Cl^-, CO_3^{-2}, PO_4^{-3}, and/or SO_4^{-2}. No effect is observed upon addition of 6 M HNO_3; this resulting mixture will be referred to as solution 1. No effect is observed on addition of 0.1 M $AgNO_3$ to solution 1. A white precipitate is reported on addition of 1 M $BaCl_2$ to solution 1. Finally, a yellow precipitate is observed on addition of 0.5 M $(NH_4)_6Mo_7O_{24}$ to solution 1. Which of the ions are present, which are absent, and which remain undetermined? State your reasoning below. NOTE: simply listing ions below without the appropriate reasoning will NOT earn you any credit!

Present _____

Absent _____

In Doubt _____