CHEM 109
Introduction to Chemistry
Revision 1.1

Laboratory Exercise: Synthesis of Calcium Oxalate

In this exercise we will synthesize the compound Calcium Oxalate; an ionic compound formed from the Calcium Ion (Ca^{2+}) and the Oxalate Ion ($C_2O_4^{2-}$). This white crystalline compound will be formed by oxidizing elemental Calcium (Ca) to the ion and then combining this with an Oxalate containing compound; Ammonium Oxalate $(NH_4)_2C_2O_4$.

As mentioned, this synthesis occurs in two basic steps. First, metallic Calcium will be treated with aqueous Hydrochloric Acid. The acid will oxidize the Calcium to Calcium Chloride. Hydrogen gas will be given off as a by-product.

$$Ca_{(s)} + 2\ HCl_{(aq)} \longrightarrow CaCl_{2(aq)} + H_{2(g)}$$

This reaction is reasonably violent and so will have to be carried out cautiously.

The resulting chloride salt is then treated with Ammonium Oxalate to form the final product.

$$CaCl_{2(aq)} + (NH_4)_2C_2O_{4(aq)} \longrightarrow CaC_2O_{4(s)} + 2\ NH_4Cl_{(aq)}$$

Aqueous Ammonia is added to this reaction mixture to prevent the excess Hydrochloric Acid present from reacting with the Oxalate Ion. Also, the Calcium Oxalate actually crystallizes out of solution as a hydrate; $CaC_2O_4 \bullet H_2O_{(s)}$.

As noted, Calcium Oxalate is an ionic compound formed from the Calcium and Oxalate ions. Ions can be formed by adding or removing electrons to or from an atom. So, in a compound like Sodium Chloride (NaCl), formed from Sodium and Chloride ions, the Sodium atoms have lost one electron to form Na^+ ions and the Chlorine atoms have gained one electron to form Cl^- ions. As should be apparent, the electron lost by the Na atom is picked-up by the Cl atom. The rationale for this electron transfer can be seen by examining the relevant electron configurations. Sodium atoms have the following configuration:

$$Na = [Ne]\ 3s^1$$

By losing a single electron, the resulting Na^+ achieves a stable Neon electron configuration.

$$Na^+ = [Ne]$$

Likewise, Chlorine atoms have an electron configuration of:

$$Cl = [Ne]\ 3s^2\ 3p^5$$

And, by picking-up an extra electron, the resulting Cl^- ions will achieve a stable Argon configuration:

$$Cl^- = [Ne]\ 3s^2\ 3p^6 = [Ar]$$

Using similar reasoning, the Calcium atom will want to lose two electrons to form the Ca^{2+} ion, which has an Argon configuration:

$$Ca = [Ar]\ 4s^2$$

$$Ca^{2+} = [Ar]$$

In this particular case, the two electrons are picked up by a grouping of Carbon and Oxygen atoms to form the Oxalate Ion; $C_2O_4^{2-}$. This polyatomic ion contains 6 Carbon and Oxygen atoms that are bound together and they collectively pick-up the two electrons lost by the Calcium.

Procedure

All your measurements must be made to the correct number of significant figures and must have the unit of measurement indicated.

1. Weigh out about 0.5g of Calcium pieces in a weighing boat and then transfer the pieces to a 250 mL dry beaker. Cover the beaker with a watch glass.

2. Very, very slowly add 10 mL of 6M HCl.

3. When the Calcium has dissolved, dilute the sample to 150 mL with deionized Water. Heat this aqueous solution to incipient boiling on a hot plate in the fume hood.

4. At the first sign of boiling, remove the beaker from the hot plate.

5. Weigh out 5g of Ammonium Oxalate and dissolve it in 50 mL of Water. It may be necessary to heat the mixture to dissolve the salt completely.

6. Add the Ammonium Oxalate solution to the hot Calcium Chloride solution and 3-4 drops of Methyl Orange. The Methyl Orange is an acid-base indicator that turns Orange in an acidic solution and Yellow if basic. (If the indicator indicates Yellow, insufficient HCl was added initially. Add one or two milliliters of additional 6M HCl.)

7. Add 6M NH_3 solution 1 mL at a time until the indicator is intermediate between Orange and Yellow, or until a persistent precipitate appears.

8. Continue adding NH_3a few drops at a time until the Yellow color persists. Allow the system to stand for about 20 minutes.

9. Set-up a gravity funnel in a Ring and using a fine filter paper filter the bulk of the liquid from the solution. Once this filtration is complete, transfer the remaining liquid and all the solid to the funnel. Use a few squirts of Water to wash the solid and the beaker. Use a rubber policeman to complete the transfer the remaining solid to the funnel. Wash the solid with 4 portions of 7 mL cold Water. Use the wash Water to flush the solid into the apex of the filter paper's cone.

10. Remove the filter paper from the funnel and spread it out on a watch glass so that the solid can dry.

11. Allow the solid to dry until the next laboratory period.

12. Once dry, weigh the solid on glazed weighing paper.

13. Obtain a 3 dram vial and submit a sample of your product. Label the vial with the following information:
    Name(s)
    Name of Compound
    Formula of Compound
    Date
    Weight of Product

Synthesis of Calcium Oxalate

Observations

Calcium Metal:

Ammonium Oxalate Solid:

Masses

Calcium Metal = _____

Amm. Oxalate = _____

Observations

During Reaction:

Masses

Glazed Paper = _____

Paper & Product = _____

Observations

Calcium Oxalate:

Name: _____ Date: _____

Signature: _____

Post Lab Questions:

1. Write electron configurations for each of the following atoms. In each case, predict the charge of the ion the atom may form. As a general rule, metals tend to lose electrons and non-metals gain electrons.

   K =

   Ion _____

   Al =

   Ion _____

   Ga =

   Ion _____

   Ba =

   Ion _____

   N =

   Ion _____

   Se =

   Ion _____

   Cl =

   Ion _____

2. Many Transition Metal Ions form ions by losing *s* electrons before *d* electrons. So, Co^{2+} forms by losing two 4s electrons:

   Co = [Ar] $4s^2$ $3d^7$

   Co^{2+} = [Ar] $3d^7$

   Additionally, many of these elements form multiple common ions. Write the correct electron configurations for the following ions.

   Fe^{2+} =

   Fe^{3+} =

   Ni^{2+} =

   Cu^{+} =

   Cu^{2+} =