Closed toed shoes and goggles are REQUIRED for the lab

Chem. 111
Experiment 6
October 15th – 19th

Experiment 6. Gravimetric Analysis: The Determination of Phosphorus in Plant Food

Adapted from "Laboratory Experiments", J.H. Nelson and K.C. Kemp, Prentice-Hall, 2000.

Reading: Understand Chapter 3 of "Chemistry, the Central Science"

Background

Gravimetric analysis is a quantitative *(i.e. how much?)* method of classical analysis. The element to be determined is isolated in a solid compound of known identity and definite composition. The mass of the element that was present in the original sample can be determined from the mass of this compound. Plant foods contain three essential nutrients that are not readily available from soils. These are soluble compounds of nitrogen, phosphorus, and potassium. A typical label on a plant food will have a set of numbers such as 15-30-15. These numbers mean that the plant food is guaranteed to contain at least 15% nitrogen, 30% phosphorus (expressed as P_2O_5) and 15 % potassium (expressed as K_2O). The remaining of the product is fillers, dyes and other anions and cations to balance the charge in the chemical compounds. In this experiment, we will illustrate a quality control analysis for the determination of phosphorus in plant food by gravimetric analysis. Phosphorus will be determined by precipitation of the insoluble salt magnesium ammonium phosphate hexahydrate according to the reaction:

$$5H_2O(l) + HPO_4^{2-}(aq) + NH_4^{+}(aq) + Mg^{2+}(aq) + OH^{-}(aq) \rightarrow MgNH_4PO_4.6H_2O(s)$$

The % P and % P_2O_5 in the initial sample can be calculated from the mass of $MgNH_4PO_4 \cdot 6H_2O$ obtained using the following method:

mass of $MgNH_4PO_4 \cdot 6H_2O$ → moles of $MgNH_4PO_4 \cdot 6H_2O$ → moles of P → mass of P → %P

and

mass of $MgNH_4PO_4 \cdot 6H_2O$ → moles of $MgNH_4PO_4 \cdot 6H_2O$ → moles of P → moles of P_2O_5 → mass of P_2O_5 → $\%P_2O_5$

Procedure

Prepare 2 samples per group as directed below. Rinse all your glassware thoroughly with water before using.

1. Weigh by difference, to the second decimal point, 3.00 to 3.50 g of your unknown sample using a weighing dish. Record the sample mass in your notebook. Remember to record all digits on the balance.

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2. Transfer your sample to a 250-mL beaker and add 35 to 40 mL of distilled water and stir the mixture with a glass stirring rod to dissolve the sample. They may be a small amount of insoluble residue. If your sample does not dissolve completely, remove the insoluble material by filtration.

3. Add 45 mL of 10% $MgSO_4 \cdot 7H_2O$ solution to the filtrate. Then add approximately 150 mL of 2*M* NH_3 slowly while stirring. A white precipitate of $MgNH_4PO_4 \cdot 6H_2O$ will form. Allow the mixture to sit at room temperature for 15 minutes to complete the precipitation.

4. Prepare a vacuum filtration apparatus using a buchner funnel. Obtain one filter paper for each one of your samples, weigh them and record their mass in your notebook (label them with a pencil to be able to differentiate them later).

5. Wet the paper with distilled water to hold it in place in the funnel. Transfer **all** the solution and the precipitate from the beaker using a rubber policeman. Wash the precipitate with two or three 5-mL portions of distilled water. Do this by adding each portion to the beaker in which you did the precipitation to transfer any remaining precipitate; then pour over the solid in the funnel. Finally, pour two 10-mL portions of 75% isopropyl alcohol through the filter paper.

6. When all the liquid has gone through, disconnect the hose and turn the water off. Carefully remove the filter paper with the precipitate and place on a marked paper towel. Blot your samples dry with a second paper towel. Discard the filtrate down the sink.

7. When the $MgNH_4PO_4 \cdot 6H_2O$ precipitate is thoroughly dry, weigh the filter papers with precipitate. Be careful not to lose any of the solid. Record their masses. Record your group data on the computer provided by your lab instructor. This class data will be available on-line for use in the discussion section.

Data Prepare the following table:

	Sample 1	**Sample 2**
Mass of Sample		
Mass of filter paper and $MgNH_4PO_4 \cdot 6H_2O$		
Mass of filter paper		
Mass of $MgNH_4PO_4 \cdot 6H_2O$		
Mass of P in original sample		
% P in sample		
% P_2O_5 in sample		
Average % P_2O_5		

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Results

Show your calculations for mass of P, %P and $\%P_2O_5$ in samples.

Discussion

Compare the $\%P_2O_5$ that you calculated for your sample to the value of the label. Are they in good agreement? If they are not, explained what could have gone wrong in your procedure. How can we improve this analysis for future trials. Discuss any trends you see in the class data, how do your results compare?

Questions

1. The label on a plant food reads 23-19-17. What does this mean?
What is the minimum percentage of potassium in this plant food? What is the minimum percentage of phosphorus in this plant food?

2. $MgNH_4PO_4 \cdot 6H_2O$ has a solubility of 0.023g/100 mL in water. Suppose a 5.02- g sample were washed with 20 mL of water. What fraction of the $MgNH_4PO_4 \cdot 6H_2O$ would be lost?

3. $MgNH_4PO_4 \cdot 6H_2O$ loses H_2O stepwise as it is heated. Between 40°C and 60°C the monohydrate ($MgNH_4PO_4 \cdot H_2O$) is formed and above 100°C the anhydrous material ($MgNH_4PO_4$) is formed. What are the phosphorus percentages of the monohydrate and anhydrous material?

4. Ignition of $MgNH_4PO_4 \cdot 6H_2O$ produces NH_3, H_2O and magnesium pyrophosphate, $Mg_2P_2O_7$. Complete and balance the equation for this reaction. If 5.00 g of $MgNH_4PO_4 \cdot 6H_2O$ are ignited, how many grams of $Mg_2P_2O_7$ would be formed?

$2\ MgNH_4PO_4 \cdot 6H_2O\ (g) \xrightarrow{heat}$