

Experiment 10

Analysis of Phosphorus in Plant Food

Adapted by **Ross S. Nord**, Eastern Michigan University, from "Analysis of Phosphorous in Fertilizer" found at <http://chem.lapeer.org/Chem2Docs/PhosphateAnal.html>

PURPOSE

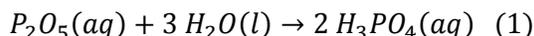
To introduce students to developing their own data analysis methodology, rather than being told exactly what calculations to perform. Also, to introduce students to solution preparation using mass percent (rather than the more common unit of molarity).

PLANT FOOD

Plant foods and fertilizers are often labeled with three numbers corresponding to the mass (weight) percents of the various nutrients present. These nutrients are nitrogen, phosphorus (present as P_2O_5), and potassium (present as K_2O). Therefore, a bag of lawn fertilizer labeled 28-3-4 would contain 28% nitrogen, 3% P_2O_5 , and 4% K_2O , by weight. In this experiment we will analyze a sample of plant food to determine the value of the second number (% P_2O_5). [Note the second number in plant foods is typically considerably higher than in lawn fertilizers.]

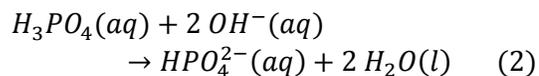
PHOSPHORUS ANALYSIS

When plant food is added to water, *each mole of P_2O_5 in it reacts to form two moles of phosphoric acid:*

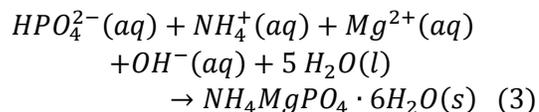


An excess of Epsom salts ($MgSO_4$) and aqueous ammonia (NH_3 , also written NH_4OH) is added to this solution. Ammonia is a weak base that generates hydroxide ions, OH^- , which

neutralize the phosphoric acid, converting it into monohydrogen phosphate ions:



The magnesium ions from the Epsom salts react with the HPO_4^- and NH_4OH to form a compound that precipitates from solution as follows:



Note that a weak base (ammonia) is used to generate the hydroxide ions needed for reaction to occur. If you recall the solubility rules, you may remember that $Mg(OH)_2$ is insoluble and so using a strong base to rapidly add hydroxide ions could precipitate out the Mg^{2+} ions as $Mg(OH)_2(s)$ rather than the desired phosphorus-containing compound. This is also why the ammonia will be added slowly during the experiment.

The compound that is produced, $NH_4MgPO_4 \cdot 6H_2O$, is called ammonium magnesium phosphate hexahydrate. As a solid mineral it has another name, **struvite**. It is what

we call a double salt as it contains two different cations (NH_4^+ and Mg^{2+}). The hexahydrate indicates that when this ionic compound crystallizes it incorporates 6 moles of water into the structure for each mole of NH_4MgPO_4 . *This is important since the mass of the water must be included when determining its molar mass.*

NONESSENTIAL, BUT INTERESTING, BACKGROUND ON STRUVITE

This mineral was first isolated from medieval sewer lines. Not surprisingly, it can be used to produce fertilizer and research is ongoing to viably extract it from wastewater, or animal wastes, and process it into commercially useful forms.

Some bladder (or kidney) stones, particularly those caused by certain bacterial infections, are composed of struvite. This is quite common in housecats, as well as humans. [Author's note: on average, women rate the pain of kidney stones as similar to that of childbirth. Having experienced a bout of kidney stones, the author has an improved

understanding and respect for women who have given birth.] Recent research has shown that struvite begins to decompose (dehydrate) at temperatures as low as 40°C and it has been suggested that prolonged warming of the kidney area may be able to shrink kidney stones in patients who experience this type of stone.

MASS PERCENT REVIEW

Mass percent is defined as follows:

$$\% \text{ component} = \frac{\text{mass component}}{\text{total sample mass}} \times 100\%$$

IN THIS EXPERIMENT

A measured quantity of plant food will be reacted with excess magnesium sulfate and ammonia to precipitate ammonium magnesium phosphate. From the amount of precipitate recovered, the mass percent of diphosphorus pentoxide in the plant food will be determined.

PRE-LABORATORY PREPARATION

- 1. Read the procedure and data analysis sections of this experiment.**
- 2. Complete the computer-generated PRELAB assignment.** The prelab questions for this experiment provide practice with the types of calculations you will need to complete the data analysis section.

EXPERIMENTAL SECTION

REAGENTS PROVIDED

Plant Food, solid.
Epsom Salts ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), solid.
Aqueous Ammonia (NH_4OH), 1 M.

SPECIAL EQUIPMENT

Buret stands and suction filtration apparatuses should be available in the lab.

WASTE DISPOSAL

All liquid wastes can go down the drain. Your final solid product should be put in the appropriate waste container. Used filter paper can go in the trash cans.

PROCEDURE

You will do the first solution preparation in a group of two or three. However, each individual is responsible for preparing his/her own ammonium magnesium phosphate hexahydrate product and turning in her/his own individual report.

SOLUTION PREPARATION

This part should be done as a group.

1. Each group of three needs approximately 50 mL of a 10% by mass, aqueous solution of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (Epsom salts). Get together with your lab partners and develop a simple recipe for preparing this solution. When you have your recipe, obtain the approval of the lab assistant or instructor before proceeding.

Hint: You may assume that the density of water is approximately 1 g/mL.

2. Prepare the solution in a 100-mL beaker according to your approved plan.

Write a brief description of how it was prepared on your data sheet.

PRECIPITATION REACTION

Each person should do this part individually.

3. Weigh out 1.0 +/- 0.1 g of plant food in a clean 250-mL beaker. Record the mass to the nearest 0.001 g.

Any mass between 0.9 g and 1.1 g will be fine. However, you need to record the exact mass measured.

4. Using your 50-mL graduated cylinder, add 15 mL of distilled water to the beaker to dissolve the plant food.

This can take several minutes of vigorous stirring. You can speed up the dissolution by using a glass stirring rod to crush the solid and stir the mixture.

Note: once you put the stirring rod in the solution you should leave it in the solution. If you remove it, any liquid (containing phosphate ions) clinging to the rod will be lost.

Depending upon the specific type of plant food, the solution may still appear cloudy after it has dissolved. There may also be a few small particles that do not dissolve. However, *in this experiment*, more error would be introduced by trying to filter them out than to leave them. So, do your best to get them dissolve, but continue even if they don't completely dissolve.

5. Measure out 15 mL of the 10% $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}(\text{aq})$ solution you prepared earlier into a 50-mL graduated cylinder. Add this to the 250-mL beaker.

It's okay to add a couple of mL too much, as it is being added in excess. Just make sure there is enough for your two partners because you don't want to run out so that someone adds too little.

6. Measure approximately 15 mL of 1.0 M $\text{NH}_4\text{OH}(\text{aq})$ into a 50-mL graduated cylinder. Slowly (over the course of about a minute) pour this into your reaction mixture while stirring continuously.

The exact amount of ammonia is not crucial as it is being added in excess. However, slightly too much is better than not quite enough.

If no solid forms, something is not right. You can try adding more ammonia or see if the instructor has any wisdom to share. However, most likely you will need to start over.

7. Let the reaction mixture sit undisturbed in the beaker for a full 10 minutes in order to ensure that precipitation is complete.

8. While waiting for precipitation to finish, you should set up a suction filtration apparatus to collect the product.

See the *Analysis of Calcium Carbonate Tablets* experiment if you don't remember how to use a suction filtration apparatus.

9. Take a one-inch piece of labeling tape and attach it to the bottom of a clean watch glass. Write your name on the tape. Place a dry piece of 42.5 mm (small) filter paper on

the watch glass and weigh them together. Record the mass on your data sheet.

10. Place the (just-weighed) filter paper in the funnel of a suction-filtration apparatus, wet it down with 70% isopropyl alcohol from a spray bottle, and collect the solid precipitate by suction filtration.

To ensure complete transfer of the solid onto the filter paper, you should rinse the 250-mL beaker using a spray bottle containing 70% isopropyl alcohol. Pour the rinse liquid into the filter funnel and repeat rinsing as necessary.

11. With the suction still on, spray the liquid in the filter funnel using several mL of the 70% isopropyl alcohol solution.

This will help remove the excess water from the solid so it will dry more quickly.

12. Leave the suction on for an additional 5 minutes (or more) to help dry the solid. Then turn off the suction and carefully remove the filter paper and product from the funnel and place it on the watch glass. (A spatula or tweezers can be used to facilitate this transfer.) Be careful not to lose any of your precipitate!

13. Weigh the watch glass containing the filter paper and wet product. Record this mass on your data sheet.

This step is being done so that (1) you have some data to practice your calculations; and (2) you will see how much the weight changes by the next lab period after the product has had a chance to dry.

In general, it is always a good idea to use to the same balance for before and after readings. This eliminates any systematic error in the results due to the calibration of a balance being slightly off.

14. Store the watch glass containing your product in your section's designated drawer until the next lab period, to allow for complete drying.

Your instructor will inform you of where to store your product. You will weigh your product at the beginning of the next lab period.

15. Wash and dry your glassware and your hands.

16. At the start of the next lab period you will weigh the watch glass and product to determine the mass of dry product obtained. Record this mass on the page labeled *Yield Report*.

If the mass of product you obtain is negative (or more than 2 grams) there is a problem. If so, you may want to determine the mass of product by taring a balance with a piece of weighing paper (or an empty watch glass) and then transferring your product (by scraping it off your watch glass and filter paper) to the weighing paper to get the mass of the product by itself. Consult your instructor if you are uncertain of how to proceed.

After weighing, the product will go into a designated waste container and the filter paper can be discarded in the trash cans.

DATA ANALYSIS

Determine the mass percent of P_2O_5 in the plant food sample that you analyzed.

You may attach additional sheets of paper, if necessary. Be sure to:

1. Clearly specify and label any molar masses you determine and use in the calculations.
2. Clearly indicate the relevant stoichiometric relationship between moles of P_2O_5 and moles of $NH_4MgPO_4 \cdot 6H_2O$.

WET Mass Percent P_2O_5 in your plant food sample: _____

(Since the product was still wet when you weighed it, you will not be graded on the accuracy of the mass percent. You will only be graded on whether the calculation was done correctly.)

Turn in your Data Sheet and Data Analysis at the end of lab. You will turn in the Yield Report next lab.

Name

Instructor/Day/Time

YIELD REPORT

This page will be turned in after the product has been reweighed, when it has had a chance to dry overnight.

Brand of Plant Food Analyzed: _____

Mass of Plant Food used: _____

Mass of watch glass plus filter paper: _____

Mass of watch glass, filter paper, and DRY $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$: _____

Determine the mass percent of P_2O_5 in the plant food sample that you analyzed. (This is the same calculation you did for the wet product during the last lab period, only the mass of product has changed.) Show your calculation.

Mass Percent P_2O_5 in your plant food sample: _____