Colorimetric Lab Demonstration by the
Florida Industrial and Phosphate Research Institute

Introduction:

History of Phosphate Chemical Processing and Phosphogypsum in Florida

Until the 1950s, fertilizer manufacturing facilities were relatively small and produced fertilizers tailored to the soil needs of area farmers, commonly within a 100-mile radius. Prior to 1950, only 4 million tons of primary nutrients were produced yearly. In the late 1940s, however, this began to change. Domestic agriculture and industry, as well as European and Western Pacific markets devastated in World War II, increasingly requested these nutrients. The demand to deliver more phosphate to the farmers at lower costs changed the way fertilizers were produced. In the 1960s, phosphoric acid replaced normal superphosphate as the primary fertilizer commodity, turning what had been strictly a mining business into chemical production. This was especially true in Florida, which produces approximately 75% of the phosphate rock mined in the U.S. Phosphate rock mined in Florida is no longer sold. It is exclusively used by the mining companies, primarily to make phosphoric acid, almost all of which is used in the production of phosphate fertilizers.

Overview of Phosphate Chemical Processing and Phosphogypsum in Florida

After the phosphate is separated from the sand and clay at the beneficiation plant, it goes—along with the phosphate pebble from the washer—to the chemical processing plant. There it is reacted with sulfuric acid to create the phosphoric acid needed to make fertilizer. The principal fertilizer product of the industry is diammonium phosphate (DAP), made by reacting ammonia with the phosphoric acid. Chemical processing is necessary because phosphate rock is not soluble in water. The DAP fertilizer is water-soluble and will be available for plants to take up through their roots.

When sulfuric acid is reacted with phosphate rock to produce phosphoric acid, a by-product, calcium sulfate (gypsum) is also produced. This by-product gypsum is called phosphogypsum. Approximately five tons of phosphogypsum are produced for every ton of phosphoric acid product that is produced. Phosphogypsum must be stockpiled in stacks after a 1989 US Environmental Protection Agency (EPA) rule banning its use based upon the trace amount of radioactivity it contains.

Standards:

SC.912.N.1.1f	SC.912.N.1.1b	SC.912.N.1.1	SC.912.L.18.12
SC.912.L.17.16	SC.912.N.4.2		

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Objectives:

- The student will be able to follow step-by-step procedures and conduct systematic observations.
- Identify the components of a solution (solute, solvent, solution). Identify factors that affect the rate of solubility.
- Describe the impact of acids and bases as they relate to environmental concerns and industry.
- Weigh the merits of alternative strategies for solving a specific societal problem by comparing a number of different costs and benefits, such as human, economic, and environmental.

Pre-Lab: Making Reagents

Molybdovanadate Color Reagent

1. By volume: Dissolve 16.5g ammonium molybdate (VI) tetrahydrate $(NH_4)6Mo_7O_{24}\cdot H_2O$) in 400ml hot DI (nano) water and set aside to cool.
2. Dissolve 0.6g ammonium metavanadate NH_4VO_3 in 250ml hot DI (nano) water.
3. Cool and add 58ml concentrated hydrochloric acid (HCl). While stirring, add the molybdate solution to the vanadate solution.
4. Transfer solution to a 2L volumetric flask and dilute to volume with DI (nano) water and mix.

Ascorbic Acid Solution

By volume: Dissolve 0.5 g L-ascorbic acid ($C_8H_8O_8$) in 100 ml DI (nano) water.

Sulfuric Acid Solution

By volume: Add 20 ml concentrated sulfuric acid to 80 ml DI (nano) water. This produces a 20% sulfuric acid solution. **Safety warning: mixing sulfuric acid with water produces heat. Use heat-resistant containers and watch your fingers.**

Materials:

Scale
5 grams phosphate rock
2 - 250 mL beakers
DI (nano) water
50 mL graduated cylinder
Whatman filter paper
Funnel
2 - test tube racks
10 - 15ml test tubes
4 - sterile pipettes
Colored pencils

Procedures:

Phosphoric Acid Production

1. Weigh a 5 gram sample of phosphate rock and place in a pyrex container (graduated cylinder, beaker).
2. Moisten with DI (nano) water.
3. Carefully add 20 ml of the 20% sulfuric acid solution. Allow to sit until obvious chemical reaction subsides (5-10 minutes).
4. Filter through Whatman filter paper.
5. Allow material (gypsum) to dry. Save liquid (phosphoric acid).

Colorimetric Procedure

1. Place ten (10) 15 ml centrifuge tubes in two rows of 5 each. Add 5 ml DI water to each tube.
   a. To one row of five, **Series A**, add the following drops of dilute phosphoric acid using a transfer pipette.

Tube 1	Tube 2	Tube 3	Tube 4	Tube 5
0 drops	2 drops	4 drops	6 drops	8 drops

   b. To the second row of tubes, **Series B**, add the following drops of phosphoric acid using a transfer pipette.

Tube 1	Tube 2	Tube 3	Tube 4	Tube 5
0 drops	2 drops	4 drops	6 drops	8 drops

2. Add 1.5 ml (or 15 drops) of molybdovanadate reagent with a 3 ml syringe to each tube in Series A and Series B.
3. Mix each tube thoroughly.
4. Allow to stand for 5 to 10 minutes to allow the reaction to progress. Note color difference from one tube to the next.
5. After color has reached its maximum, add 5 drops of ascorbic acid solution to each tube in Series B using a clean transfer pipette.
6. Mix well and allow to stand for 5 to 10 minutes. Note color difference from one tube to the next.

Data

Sketch and color your final product.

Analysis/Conclusion

1. What is the role of sulfuric acid in chemical processing?
2. Identify the solutes and solvents used in this demonstration.
3. What is solubility? Identify the factors that affected the rate of solubility.
4. How do acids and bases impact industry and the environment?
5. Currently phosphogypsum has to be "stacked" due to EPA regulations. What are costs and benefits to this current method of disposal? What could be some alternative strategies for utilizing or disposing of the gypsum?
6. Why is phosphogypsum sometimes referred to as a *co-product* rather than a *by-product*?