To close the yellow note, click once to select it and then click the box in the upper left corner.
To open the note, double click (Mac OS) or right click (Windows) on the note icon.

Determination of Phosphorus Content in River Water

Phil McBride, Eastern Arizona College, Thatcher, AZ

INTRODUCTION

Description

In this experiment, students discover that a pure sample can become contaminated during preparation for analysis. The students colorimetrically analyze water samples from a river for phosphorus. During the sample preparation, one group of students (Gila Laboratory) decolorizes the "murky" water with activated carbon, while the other group (Graham Laboratory) decolorizes the water with activated carbon that has been acid washed. Activated carbon contains small amounts of phosphorus. Acid washing removes the phosphorus. The data is compared, and the students then try to determine why the results differed.

Student Audience

This lab is intended for college students taking General Chemistry or Organic Chemistry.

Goals for the Activity

The goals of this activity are to

- help students discover the importance of sample preparation when running an analysis,
- help students see the importance of researching all of the steps in the method of analysis to ensure that contamination of the sample will not occur, and
- enable students to apply knowledge of the spectrophotometer and calibration curves to a real-world scenario.

Recommended Placement in the Curriculum

This lab should be conducted after the students have learned how to use the spectrophotometer. It would fit nicely within chapters that deal with concentration.

STUDENT HANDOUT
Gila Laboratory

Purpose

The purpose of the lab is to determine the amount of phosphate in water samples as well as to identify preparation techniques that must be considered in analyzing a sample.

Scenario

A lake community called Calais is situated 60 miles downriver from the resort community of Lille. Calais Lake is formed from a large dam on the Bailleul River. Several new 18-hole golf courses are being built at Lille.

The people of Calais are complaining that their lake water contains high levels of phosphorus, which they claim are killing the fish. They believe that the builders of the new golf courses are trying to jump-start their grass by applying large amounts of phosphorus and that much of this phosphorus is running off the golf courses into the river, eventually ending up in Calais Lake. The community of Calais has filed suit against the community of Lille asking that they clean up the lake.

You work for Gila Laboratory and have been hired by the community of Lille to determine whether phosphorus is entering the river at Lille. You must have the results back in three days for the court hearing. GOOD LUCK!

Materials

Apparatus

- spectrophotometer, for use at 400 to 490 nm
  The wavelength of choice depends on the desired sensitivity. Lower wavelengths are more sensitive. However, be aware that iron(III) causes interference at low wavelengths, especially around 400 nm. The recommended wavelength for this experiment is 400 nm.

Reagents

- phenolphthalein indicator (aqueous solution)
- 6 M HCl, hydrochloric acid
- activated carbon
- vanadate-molybdate reagent
- standard phosphate solution: Dissolve in distilled water 0.2195 g anhydrous KH_2PO_4 and dilute to 500 mL; this gives a 100 ppm of P as PO_4^{3-} solution

Safety, Handling, and Disposal

It is your responsibility to specifically follow your institution's standard operating procedures (SOPs) and all local, state, and national guidelines on safe handling and storage of all chemicals and equipment you may use in this activity. This includes determining and using the appropriate personal protective equipment (e.g., goggles, gloves, apron). If you are at any time unsure about an SOP or other regulation, check with your instructor.

The vanadate-molybdate reagent and analyzed samples should be collected in a properly labeled waste container and then disposed of according to established local ordinances. Any untreated water samples can usually be disposed of down the drain, but make sure to abide by established local ordinances.

Procedure

1. Devise a plan of action with a very basic outline showing how you will determine if the Bailleul river is being contaminated with phosphorus from the community of Lille.

2. Test the pH of your water samples. If the pH is greater than 10, add 1 drop of phenolphthalein indicator to a 50.0-mL sample. Add 6 M HCl dropwise until the pink color disappears. Dilute to 100 mL in a 100-mL volumetric flask.

3. As you have noticed, the water samples are murky. Describe what you can do to clear up the sample.

4. Gravity-filter your samples. After filtering, remove the excessive color in the sample by shaking about 50 mL of sample with 0.20 g activated carbon in an Erlenmeyer flask for 5 minutes. Filter again to remove the carbon.

5. Look at the apparatus and reagents that are available for your use. Devise a method to determine the amount of phosphorus in the water samples. (Hint: Refer to the "Color Development Procedure," which follows.)

6. Pipet 10.0 mL prepared sample into a 25-mL volumetric flask. Add 4 mL vanadate-molybdate reagent and dilute to 25 mL with distilled water. Prepare a blank in which 10 mL distilled water is substituted for the sample. After 10 minutes or more, measure the absorbance of the sample versus a blank at a wavelength of 400 nm.

Color Development Procedure: Preparing Phosphate Standards and Determining Concentration

1. Prepare 100 mL of a 1-ppm standard by pipetting 1.00 mL of the 100-ppm P standard into a 100-mL flask. Dilute to 100 mL with distilled water. Repeat this procedure to prepare 3-, 5-, and 7-ppm standards.

2. To establish the calibration curve, treat each standard as follows:

   a. Pipet 10 mL of the 1-ppm standard into a 25.0-mL volumetric flask.

   b. Pipet 4.0 mL of the vanadate-molybdate reagent into this flask and dilute to 25 mL with distilled water. Mix thoroughly.

   c. Repeat with 3-, 5-, and 7-ppm standards.

   d. Prepare a blank by pipetting 4.0 mL of the vanadate-molybdate reagent into a 25.0-mL volumetric flask and bring to volume with distilled water.

   e. After 10 minutes but no more than 30 minutes, measure the percent transmittance at 400 nm.

3. Prepare a calibration curve by plotting absorbance as a function of ppm phosphate-phosphorus.

$$absorbance = -log\left(\frac{\%T}{100}\right)$$

4. Using the calibration curve, calculate the ppm of phosphorus in the river water samples. Submit your results to the Court (your instructor).

Questions

1. The Court (your instructor) will provide the results of both "laboratories." Record these results below:

Graham Laboratory	Gila Laboratory

2. Find a laboratory group that works for the Graham Laboratory. Discuss any discrepancies found between the two groups. Review the procedure that you followed and arrive at a conclusion to explain any discrepancies.

3. List at least two sample preparation considerations that must be taken into account to obtain accurate results in this lab.

References

American Public Health Association. *Standard Methods for the Examination of Water and Wastewater/Prepared and Published Jointly by American Public Health Association, American Water Works Association, Water Pollution Control Federation*, 16th ed.; Washington, DC, 1985; pp 445–446.

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Materials

Apparatus

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  The wavelength of choice depends on the desired sensitivity. Lower wavelengths are more sensitive. However, be aware that iron(III) causes interference at low wavelengths, especially around 400 nm. The recommended wavelength for this experiment is 400 nm.

Reagents

- phenolphthalein indicator (aqueous solution)
- 6 M HCl, hydrochloric acid
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- standard phosphate solution: Dissolve in distilled water 0.2195 g anhydrous KH_2PO_4 and dilute to 500 mL; this gives a 100 ppm of P as PO_4^{3-} solution

Safety, Handling, and Disposal

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The vanadate-molybdate reagent and analyzed samples should be collected in a properly labeled waste container and then disposed of according to established local ordinances. Any untreated water samples can usually be disposed of down the drain, but make sure to abide by established local ordinances.

Procedure

1. Devise a plan of action with a very basic outline showing how you will determine if the Bailleul river is being contaminated with phosphorus from the community of Lille.

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3. As you have noticed, the water samples are murky. Describe what you can do to clear up the sample.

4. Gravity-filter your samples. Weigh out 1.000 g of activated carbon. Acid wash the carbon by washing with 50 mL 6M HCl. Rinse the carbon several times with water to remove all traces of the acid. Remove the excessive color in the sample by shaking about 50 mL of sample with 0.200 g acid-washed activated carbon in an Erlenmeyer flask for 5 minutes. Filter again to remove the carbon.

5. Look at the apparatus and reagents that are available for your use. Devise a method to determine the amount of phosphorus in the water samples. (Hint: Refer to the "Color Development Procedure," which follows.)

6. Pipet 10.0 mL prepared sample into a 25-mL volumetric flask. Add 4 mL vanadate-molybdate reagent and dilute to 25 mL with distilled water. Prepare a blank in which 10 mL distilled water is substituted for the sample. After 10 minutes or more, measure the absorbance of the sample versus a blank at a wavelength of 400 nm.

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    c. Repeat with 3-, 5-, and 7-ppm standards.

    d. Prepare a blank by pipetting 4.0 mL of the vanadate-molybdate reagent into a 25.0-mL volumetric flask and diluting to 25 mL with distilled water.

    e. After 10 minutes but no more than 30 minutes, measure the percent transmittance at 400 nm.

3. Prepare a calibration curve by plotting absorbance as a function of ppm phosphate-phosphorus.

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INSTRUCTOR NOTES
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Time Required
This activity requires approximately 3 hours.

Group Size
This activity may be conducted individually or in pairs.

Materials
Apparatus

- spectrophotometer, for use at 400 to 490 nm
  The wavelength of choice depends on the desired sensitivity. Lower wavelengths are more sensitive. However, be aware that iron(III) can cause interference at low wavelengths, especially around 400 nm. The recommended wavelength for this experiment is 400 nm. This may have to be adjusted if there is a significant amount of iron in your "river" water.

Reagents

- phenolphthalein indicator (aqueous solution)
- 6 M HCl, hydrochloric acid
- river water samples (if not collected)
  Prepare samples between 1 ppm and 7 ppm as described in the "Color Development Procedure."
- activated carbon (Choose a type that has a high phosphorus content.)
- vanadate-molybdate reagent:
  Solution A: Dissolve 25 g ammonium molybdate, $(NH_4)_6Mo_7O_{24} \cdot H_2O$, in 300 ml distilled water.
  Solution B: Dissolve 1.25 g ammonium metavanadate, NH_4VO_3, by heating to boiling in 300 mL distilled water. Cool and slowly add 330 mL concentrated HCl. Cool Solution B to room temperature.
  Pour Solution A into Solution B, mix, and dilute to 1 L. This solution will have a yellow color.
- Standard phosphate solution: Dissolve in distilled water 0.2195 g anhydrous KH_2PO_4 and dilute to 500 mL; this gives a 100 ppm of P as PO_4^{3-} solution.

Safety, Handling, and Disposal
As the instructor, you are expected to provide students with access to SOPs, MSDSs, and other resources they need to safely work in the laboratory while meeting all regulatory requirements. Before doing this activity or activities from other sources, you should regularly review special handling issues with students, allow time for questions, and then assess student understanding of these issues.

The vanadate-molybdate reagent and analyzed samples should be collected in a properly labeled waste container and then disposed of according to established local ordinances. Any untreated water samples can usually be disposed of down the drain, but make sure to abide by established local ordinances.

Points to Cover in the Pre-Lab Discussion

- Have one or two students from each "laboratory" collect water samples from an area river. The amount of sample depends on the number of students that are in your lab. Be sure to have the students collect about twice as much sample as will be needed by the entire lab.
- Tell the students where to dispose of their chemical waste.
- Review the use of the spectrophotometer.
- Review how to make and use a calibration curve.

Plausible Answers to Questions

Procedure Step 1. Plan of action:

- *Test the pH of the water samples.*
- *Collect water samples from the river several miles above the community of Lille.*
- *Collect water samples from the river several miles below the community of Lille.*
- *Analyze the different samples for phosphorus.*

Procedure Step 3. To clear up the sample:

- *Filter the water samples.*
- *Add decolorizing carbon to the water samples.*

Procedure Step 5. Procedure for the analysis:

The actual procedure is given in the student handout. The students have a list of the apparatus and reagents on their handout. They should be able to come up with a basic procedure that includes the use of the spectrophotometer to determine the phosphorus content.

Color Development Procedure Step 4. Determination of phosphorus in water samples:

Make up several phosphorus standards and run those standards on the spectrophotometer to prepare a calibration curve. Run the water samples in the same manner and then use the calibration curve to determine the amount of phosphorus in the samples.

Question 2. Comparison of laboratory results:

The two different laboratories should show different amounts of phosphorus for representative samples. After talking between laboratories the students should determine that one laboratory acid washed their activated carbon and the other did not. This is the only procedural difference between the two laboratories.

Question 3. At least two sample preparation considerations:

a. Make sure that the water samples are homogeneous.
b. Use phosphorus-free activated carbon or acid wash the carbon before use.
c. Make sure that all glassware is clean.

Extensions and Variations

- The students could determine the amount of phosphate in detergents (Kennedy, pp 107–108). They could then wash some glassware with the same detergent, rinse it, and determine whether any phosphate remains. This will help them see that contaminants can be introduced into the sample by "cleaning the glassware and apparatus." (Many detergents are free of phosphorus.)
- The students could determine the amount of phosphorus in cola drinks.

References

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