

## Experiment 25: The Fertilizer Project- Analysis of Phosphorus in Fertilizer

### Spectrophotometric

#### Construct a Calibration Curve

1. Obtain 20 mL of a standard stock solution of  $5.00 \times 10^{-3}$  into a dry 50-mL beaker
2. Create a working solution by using a Mohr pipet to obtain 3.00 mL of the standard stock solution into a 250-mL volumetric flask and adding enough water to dilute the solution to the 250-mL mark
3. Use a volumetric pipet to place 5.00 mL into a 10 mL volumetric flask, add ~4 mL of heteropoly blue solution, and dilute to the 10 mL mark
4. Pour all of the solution into a medium test tube and label correctly
5. Repeat steps 3 and 4 with 1.0, 2.0, 3.0, and 4.0 working stock solutions
6. Develop a reagent blank by adding 4 mL of the heteropoly blue solution to the 10 mL volumetric flask, add distilled water to the mark, pour into a medium test tube, and label it
  - a. A reagent blank must be made as a reference solution to ensure no  $\text{PO}_4^{3-}$  exists in the distilled water, in addition to make sure no other constituents of the solution contribute to the absorbance
7. Develop the blue color by placing the test tubes in boiling water for 10 minutes
8. Allow the test tubes to cool to room temperature
9. Tilt each test tube to mix the solution with the water that has condensed in the test tube
10. Use a spectrometer to calculate the absorbance spectra for a blank the light path and the reference (the reagent blank solution)
11. Obtain an absorbance spectra for each colored solution ensuring the arrow is pointing toward the light source, the cuvet does not contain air bubbles, the cuvet is filled, and using the same cuvet for each of your colored solutions and rinsing the cuvet with a small amount of the next solution to be measured before filling it up to be measured
12. Print or record your results
13. Using the software "Absorbance Series" to plot your results from step 12 in one graph
14. If any point deviate far from the line of best fit you must remeasure that point
15. Determine your wavelength and print your results
16. Select 'Beer's Law Plot' for the "Analysis" menu and enter absorbance values and concentrations for each colored solution
  - a. This plot is your calibration curve for A vs.  $[\text{PO}_4^{3-}]$

#### Analyzing the Sample

1. Determine the amount of fertilizer needed to make 250 mL of a solution
2. Use the  $\text{KH}_2\text{PO}_4$  (fertilizer) solid to prepare a sample solution
  - a. Place the calculated amount in our case .36g of the fertilizer into a 250 mL volumetric flask

- b. Add 3.00mL of this solution into another 250mL volumetric flask, creating your working solution
3. Prepare three colored solutions using three different volumes of the working solution, 4mL of the heteropoly blue solution, and water into 3 medium sized test tubes
4. Create another reagent blank solution by placing 3mL of the diluted fertilizer solution and water into a 10mL volumetric flask and filling the rest with water.
5. Record and save the absorption spectrum for each solution and generate a Beer's Law plot for the sample data
6. Use the equation from the calibration curve to determine the amount of Phosphate in the each colored sample of the diluted fertilizer by assuming there is one PO<sub>4</sub><sup>3-</sup> in each heteropoly blue formula unit (see calculation 3)
7. Convert the amount of phosphate found in the solution to %P<sub>2</sub>O<sub>5</sub> and compare it to the expected value. (see calculation 7 & 8)

### 1. Amount of fertilizer sample needed.

$$.250L(5 \times 10^{-3}) \times 30.97 \times \frac{100gfertilizer}{11gm} = .35gfertilizer$$

### 2. Calculation of P in colored sample for calibration curve

$$P = KH_2PO_4 = \frac{5.00 \times 10^{-3} mol}{1L} \times \frac{3mL}{250mL} \times \frac{(1)or(3)or(6)mL aliquot}{10mL sol}$$

### 3. Mass of P in 250 mL fertilizer (stock) solution

$$\frac{xxx \times 10^{-6} mol P}{1L} (calibration curve) \times \frac{10mL (colored)}{xxx mL (aliquot)} = (P) in colored solution$$

4.

$$mole P \text{ in colored solution} \times \left[ \frac{250 mL \text{ working solution}}{3 mL} \right] = P \text{ in working solution}$$

$$mole P \text{ in working solution} \times .250L = mol P \text{ in fertilizer sample solution}$$

5.

6.

$$\text{mole P in fertilizer solution} \times \left[ \frac{30.97 \text{ g P}}{1 \text{ mol P}} \right] = \text{g P in fertilizer sample solution}$$

### 7. Calculation of % $\text{P}_2\text{O}_5$ in fertilizer

$$.xxx \text{ g P} \times \left[ \frac{141.9 \text{ g P}_2\text{O}_5}{61.94 \text{ g P}} \right] = .xxx \text{ g P}_2\text{O}_5$$

8.

$$\% \text{ P}_2\text{O}_5 \times \left[ \frac{\text{g P}_2\text{O}_5}{\text{g fertilizer}} \right] \times 100\%$$

### Gravimetric Method

Procedure:

- 1) Weigh out  $0.1 \pm 0.01$  g of  $\text{KH}_2\text{PO}_4$  on an analytical balance.
- 2) Dissolve the sample in about 10 mL of deionized water in a 50 mL beaker.
- 3) Add 10 mL of the 0.4 M  $\text{MgSO}_4$  solution.
- 4) Using a disposable plastic pipet, *slowly* add 1 mL 6M  $\text{NH}_3$  dropwise while stirring; after adding all of the  $\text{NH}_3$ , touch the stirring rod to a piece of red litmus paper.
- 5) Repeat this, adding 1 mL of  $\text{NH}_3$  at a time until the litmus turns blue when the solution contacts it.
- 6) Then add 0.5 mL more of  $\text{NH}_3$ . This will precipitate insoluble  $\text{Mg}(\text{NH}_4)\text{PO}_4 \cdot 6\text{H}_2\text{O}$ . Let the solution stand about 15 min to complete the precipitation.
- 7) Filter off the precipitate using suction filtration. Wash the precipitate with two 5-mL portions of *iso*-propyl alcohol
- 8) Suck air through the filter for about twenty minutes to help dry the precipitate and then carefully scrape it onto a pre-weighed watch glass. Be sure that you get all of the precipitate.
- 9) Let the precipitate dry in air for half an hour and weigh the watch glass with precipitate. If your precipitate is not dry, the weight will continue to change with time.
- 10) Repeat steps 1-9 for a second trial
- 11) Measure mass of product. You will use this to determine the % of  $\text{P}_2\text{O}_5$  (see calculation 3)