EXPERIMENT 25
THE FERTILIZER PROJECT—ANALYSIS OF PHOSPHORUS IN FERTILIZER

INTRODUCTION

In this project we will investigate the phosphorus content of soluble fertilizers. The idea is to simulate the type of problem that might be solved in a real job situation. The scenario is that an analytical company has been hired to evaluate the phosphorus content of water-soluble plant fertilizers (phosphorus is generally present in the form of phosphate ion in fertilizers). A decision must be made about how to do the analysis. You are part of a team in this company who has been assigned the task of developing the method needed to perform this analysis.

Typically, the steps in developing a method for a specific type of analysis are as follows:

1) The scientific literature is researched to find similar types of analyses—ones that analyze for the phosphorus content of samples that aren't necessarily fertilizers. These are referred to as **standard procedures**. Two procedures that analyze the phosphorus content of the salt KH_2PO_4 are given in this handout.

2) The published procedures are modified to specifically analyze the material of interest—in this case, fertilizer. Modifications include determining how much of the fertilizer sample to use in the analysis, and whether any unique properties of the fertilizer (presence of insoluble impurities, color) will interfere with the published procedure.

3) After a number of analyses have been performed using the different published procedures, the data is analyzed, and a decision is made as to which procedure is most appropriate. Some considerations in the choice will be: the ability of inexperienced lab technicians to execute the procedures reliably in a reasonable amount of time, the reproducibility (precision) of the results obtained, the accuracy of the results obtained, the cost of the reagents and equipment required, and the ease of disposing of any waste generated in the analysis. One way to make such a decision would be to establish a team to make a recommendation, and that is what we shall do here.

PROCEDURE

Each team will consist of four members with assigned roles. The **team leader** does just what the name suggests—organizes the tasks, assigns equivalent work to each team member (including him/herself). The **recorder** keeps a systematic record of the results provided by each team member and the conclusions of discussions reached. The **assistant team leader** assembles the final group report to be submitted. The **information coordinator** is responsible for formulating questions to be asked of the instructor, obtaining the answers and conveying the information obtained to the other team members.

At the start of the project each team will develop two protocols—one for a **gravimetric** analysis of the fertilizer and one for a **spectrophotometric** analysis. A preliminary version of each protocol will be submitted and returned for revision after suggestions from the instructor. The final versions of the

protocols will be submitted one lab period after receiving the preliminary protocol back from the instructor. Each team member will receive a score *only for the protocol they worked on*, which will be twenty percent (20%) of each person's total grade for the project. At the end of the project, each team will submit a series of tables summarizing the results from all team members (25%), and an essay describing the environment impact of phosphates (13.33%). In addition, each team member will write an individual report using his/her own data and the shared data of team members to recommend one of the analytical methods; 31.33% of the grade will be based on this report. Ten percent (10%) of the grade will be based on an evaluation of each team member's contribution to the project, both by the TA and the other team members.

Fertilizers contain nutrients required by plants. Among these are the elements N, P (reported as P_2O_5) and K (reported as K_2O). The composition is reported on the container as three numbers such as 13-13-13, which means that the formulation contains 13% by weight N, 13% by weight P_2O_5 and 13% by weight K_2O. Even though fertilizers do not actually contain P_2O_5 (which would react violently with water) or K_2O, it is customary for historical reasons to report the content in this way. In fact, P is generally present in fertilizers as the phosphate ion PO_4^{3-}. Many products are over formulated; that is, they contain more of some ingredient than is promised on the label. Also, the content of a particular sample may vary as much as ±0.5% because of the problem of achieving complete homogeneity in the mixing of solids. Thus, in evaluating analytical procedures, the agreement among large numbers of results is more important than obtaining the exact percentage on the label (unless the result obtained is ridiculously different).

Two types of analytical methods will be used. In **spectrophotometric analysis** the substance of interest (in our case phosphate, PO_4^{3-}) is converted to a colored compound. The intensity of the color produced can be measured and is proportional to the amount of phosphate present. In **gravimetric analysis** phosphate is precipitated as an insoluble compound which can then be weighed. The standard methods for analysis are described in **APPENDICES 25B and 25C.**

WHAT EACH TEAM AND TEAM MEMBER IS EXPECTED TO DO

The team is responsible for the following:

1. Writing an **essay** of about 500 words detailing the environmental significance of phosphate. **APPENDIX A** gives some suggestions for researching this essay via the Internet.
2. Developing a set of directions (a **protocol**) for analyzing the fertilizer sample. Two team members will develop a protocol for the gravimetric procedure and the other two for the colorimetric procedure; *only one protocol for each method should be submitted.* The intent of the protocol is to provide detailed information for someone to 1) **perform the procedure** and 2) **do the necessary calculations to determine the $\%P_2O_5$ in the fertilizer sample** (typically, a lab technician with limited chemical knowledge is assigned to perform the analysis). The protocol should be typed (the calculations may be written by hand).

   Protocol procedure: You will be using the procedures given in **APPENDICES B and C** as a basis for your fertilizer procedure. Note that the procedures given are for the analysis of a known phosphate sample; much of your protocol procedure will be identical. However, when you get to the **Sample Analysis** portion of the procedure, **you will need to modify each procedure to analyze for fertilizer, rather than the known sample**. Some ideas on how to modify the procedure are given in the **Protocol Considerations** section after each procedure.

Sample calculations to be included in the protocol are given below:

Gravimetric method:

- The amount of fertilizer sample needed for the analysis
- The mass of phosphorus atoms present in the $Mg(NH_4)PO_4$-$6H_2O$ product
- The percent of P_2O_5 in the fertilizer sample

Spectrophotometric method:

- the concentration of PO_4^{3-} in any one of the solutions used to generate the calibration curve;
- how the fertilizer sample is diluted to the desired concentration (the mass of fertilizer to be weighed out);
- the mass of P in the fertilizer sample solution;
- the % of P_2O_5 in the original fertilizer sample.

Note that these calculations will not include actual measured data, but simply show how the calculations are set up.

The preliminary versions of these protocols will be turned in; your TA will make comments and return them to the team for revision.

Each **team member** is expected to:

1. Execute tasks in designing protocols and researching the essay as assigned by the team leader.
2. Give legible copies of all results to the team recorder.
3. Make at least two determinations of the % phosphate in a pure sample of known phosphate content (KH_2PO_4) and in the sample of commercial fertilizer by the method for which he/she helped develop the protocol.
4. Teach this method to a team member who developed the other protocol and help him/her make measurements as in 3.
5. Learn the second analytical method from a team member and make measurements as in 3.
6. Share the results with all other team members. This will give a total of eight measurements for the fertilizer sample and four for the known phosphate sample for each procedure—two from each team member—as well as four for the known phosphate sample for each procedure. Ideally, the measurements in 3 and 5 should all agree with one another within 1.5% of the quantity being measured. If they do not, some decision as to why must be made. Does one person's data seem out of line? If so, what is the problem? Measurements may need to be repeated until each person is satisfied that accurate results are being obtained through improved lab technique as a result of practice. (This is important because one needs to choose procedures that will give the same results no matter which lab tech does the analysis.)
7. Assist in compiling the team data into a series of tables.
8. Write a report which
    a. incorporates a standard **INTRODUCTION;**