

Measuring Soil Phosphates Using Ion-Exchange Resins

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A Final Project for Freshman Chemistry

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A final project in freshman chemistry gives students the opportunity to apply previously learned concepts to a practical problem. The final project described here uses ion-exchange resins to isolate phosphate ions from a soil matrix and the phosphate is measured as yellow vanadomolybdophosphoric acid using a spectrophotometer. The students are required to use many laboratory skills and theoretical concepts learned throughout freshman chemistry: metric conversions, pipetting, spectrophotometry, molarity, solution and standard preparation, titration, weak acid equilibria, solubility product, and ion chromatography. The laboratory experience is designed to be completed in 3–4 laboratory sessions near the end of a full year of freshman chemistry.

Reports of projects such as the one described here being used in undergraduate chemical education have been limited (1, 2). The final project allows students to begin gaining the skills needed for an undergraduate research project or capstone experience. The importance of a capstone experience was emphasized in the Boyer Commission's Report (3), "All the skills of research developed in earlier work should be marshaled in a project that demands the framing of a significant question or set of questions, the research or creative exploration to find answers, and the communication skills to convey the results to audiences both expert and uninitiated in the subject matter." The laboratory experience reported here allows students to begin developing those skills early in their undergraduate education, rather than waiting until the end of their senior year.

The overall model of the laboratory experience is as follows:

1. *A problem to be solved.* The problem ideally requires the student to use as much freshman year chemistry as possible and at least some library research. A team of two to four students is formed to work on the project.
2. *Background laboratory experiences.* If the students need special lab skills to solve a particular problem, they need to be provided with the laboratory experiences and skills to approach the problem intelligently. Examples include use of a spectrophotometer, preparation of calibration curves, or uses of ion-exchange materials.
3. *Laboratory work that will require multiple laboratory sessions to complete.* This allows the student to develop a feeling of proficiency in the lab and better models a workplace situation.
4. *Record keeping.* The students are required to keep a laboratory notebook of all work performed. Spreadsheets are used to develop calibration curves, absorption spectra, and statistical analysis of the data.
5. *Final Report.* A formal written report is required from each student. The format of a research journal article is used, as shown in the student guidelines.^W

The chemistry of soils and fertilizers provides an opportunity to teach many fundamental concepts at the freshman level. A plant needs a variety of nutrients in the soil in order to grow properly, and one of the more important nutrients is phosphate. Phosphate becomes part of ATP during photosynthesis. The ionic forms of phosphorus in soil that are available to plants are HPO_4^{2-} and H_2PO_4^- (4).

For a plant to absorb the appropriate nutrients through the root system, the pH must be within certain limits. A pH below 4.0 can contain free acids and a pH of 6.5 to 7.0 is desirable for many farm crops (5). The wrong pH can increase the availability of some nutrients to toxic levels while others form insoluble precipitates and become unavailable to plants. Since the most common pH problem is too low a pH (below 6.0), farmers in the U.S. control pH by adding limestone, CaCO_3 , to the soil to increase the pH. Both the nutrients and the limestone are spread on the soil by large spreader trucks. To save time and money, it is tempting to spread both limestone and fertilizer from the same truck. One problem with this practice is that limestone can react with the phosphates in the fertilizer to form insoluble or slightly soluble calcium phosphate compounds such as CaHPO_4 and $\text{Ca}(\text{H}_2\text{PO}_4)_2$.

Soil testing is used to determine the nutrient levels in the soils so that the appropriate amount of fertilizer can be applied for a particular crop. Traditionally, the testing has been accomplished by extracting the various nutrients with an extracting solution and analyzing for the nutrients in the extract (6). These numbers are then related back to crop performance through field trials. A more recent technique is the use of mixed-bed, cation–anion resins to emulate a plant root (7). The resin beads are held within a mesh capsule and placed in the wet soil. The anion and cation nutrients are absorbed into the resin much as they are absorbed into a plant root. The resin is then rinsed with acid to displace the ions and the resulting acid solution is analyzed for the various plant nutrients.

In this experiment, the students are given a scenario in which two neighbors apply phosphate fertilizer to their lawns, but one of them applies limestone simultaneously with the fertilizer. In the scenario, the neighbor who applies the lime discovers that his lawn is showing a phosphorus deficiency. The students are asked to design an experiment to demonstrate why applying lime and phosphate fertilizer simultaneously would cause a deficiency in phosphorus. The effect of solubility of compounds in this practical application is illustrated by allowing the student to discover that calcium carbonate will react with phosphates in soils to produce insoluble compounds. It should be noted at this point that, keeping in mind that

Table 1. Experiments Providing Background Experience for the Final Project

Topic	Experiment	Skills or Concepts Reinforced; Student Activities
Intermolecular forces, solids and liquids	Determining the exchange capacity of an ion-exchange resin	Molarity, normality, solution preparation Titrations Introduction to ion exchange Intermolecular forces
Solutions	The absorption spectrum of vanadomolybdophosphoric acid: an introduction to spectrophotometry	Preparation of solutions Molarity, mg/L Pipetting Beer-Lambert law Absorption spectra Spreadsheets
Solutions, continued	Chromatographic separation of phosphoric acid from Cola beverages (7)	Practical application of above principles
Equilibrium	An experiment on chemical equilibria that involves preparing a precipitate of calcium phosphate	Chemical equilibrium Solubility product Le Châtelier's principle

these are freshman students, they are given leading questions to guide their thinking. They are also reminded that the *process* of solving the problem (doing a literature search, keeping an accurate notebook, using good laboratory practices, being able to communicate their findings effectively) is the most important part of this laboratory experience.

The students are provided with appropriate background experiences to prepare them for the project by modifying the laboratory experiments used during the second and third quarters. The topics covered and associated experiments are listed in Table 1.

To make this experiment more reproducible in other geographical locations, sand is substituted for soil. One portion of sand (about 750 mL) is treated with powdered CaCO_3 followed by KH_2PO_4 and a second portion of sand is treated with KH_2PO_4 only. To measure the amount of phosphate that would be found in the "soil", a commercially prepared mixed-bed, anion-cation exchange resin capsule is used to absorb the phosphate ions (PST-1 resin capsules; they are available from Unibest, Inc., 3535 Stucky Rd., Bozeman, MT 59718, and at the time of this writing, they cost \$5.00 each). The capsule is buried approximately 3–5 cm below the surface of the wet sand (after a "rain" of about 100 mL) and is allowed to sit for 24–48 hours. This setup is illustrated in Figure 1. The phosphate ions are removed from the resin with 1 M H_2SO_4 and analyzed colorimetrically.

One would expect to find lower phosphate concentrations in the sand to which the calcium carbonate had been applied. The first test of this experiment with students got very mixed results. The students reasoned that the method of application

of the phosphate was allowing the phosphate to get past the calcium carbonate before it had a chance to react. For example, if a solution of phosphate is simply poured onto the layer of powdered CaCO_3 , the solution could splash the CaCO_3 outward and go directly down into the sand before any insoluble calcium phosphates could form. The *students* suggested that the phosphate be sprayed on as a solution to allow for an even application. In the second year of testing the experiment, a variety of methods of applying the phosphate were tried, but only those using the spray technique achieved the expected results.

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Supplemental Material

Supplemental material for this article is available in this issue of *JCE Online*.

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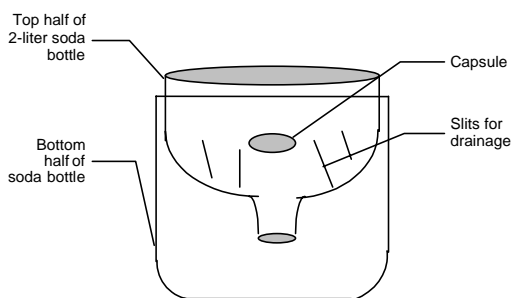


Figure 1. Diagram of the experimental setup.