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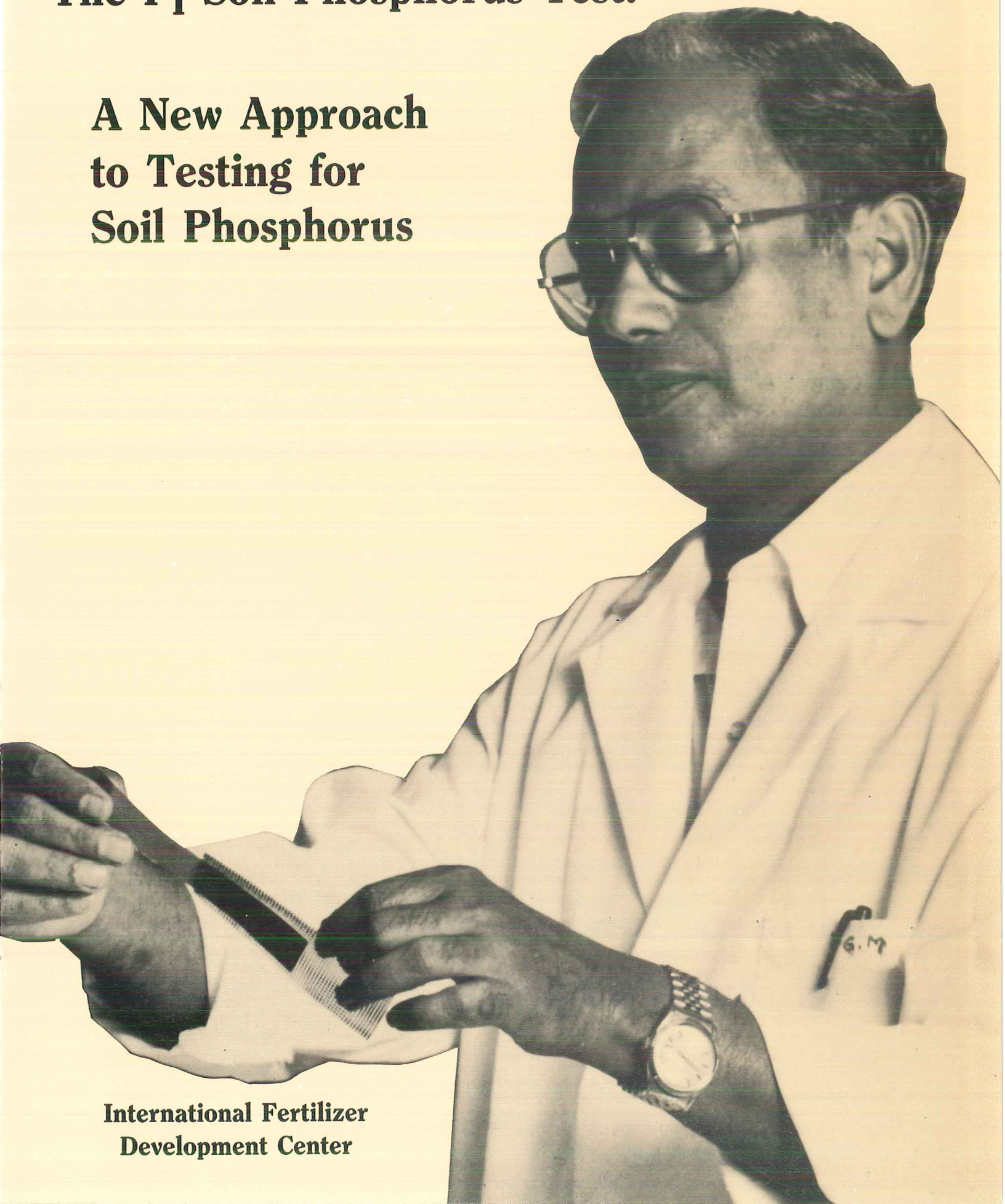
The P_i Soil Phosphorus Test:

A New Approach to Testing for Soil Phosphorus



International Fertilizer Development Center

The P_i Soil Phosphorus Test:

A New Approach to Testing for Soil Phosphorus

R. G. Menon
S. H. Chien
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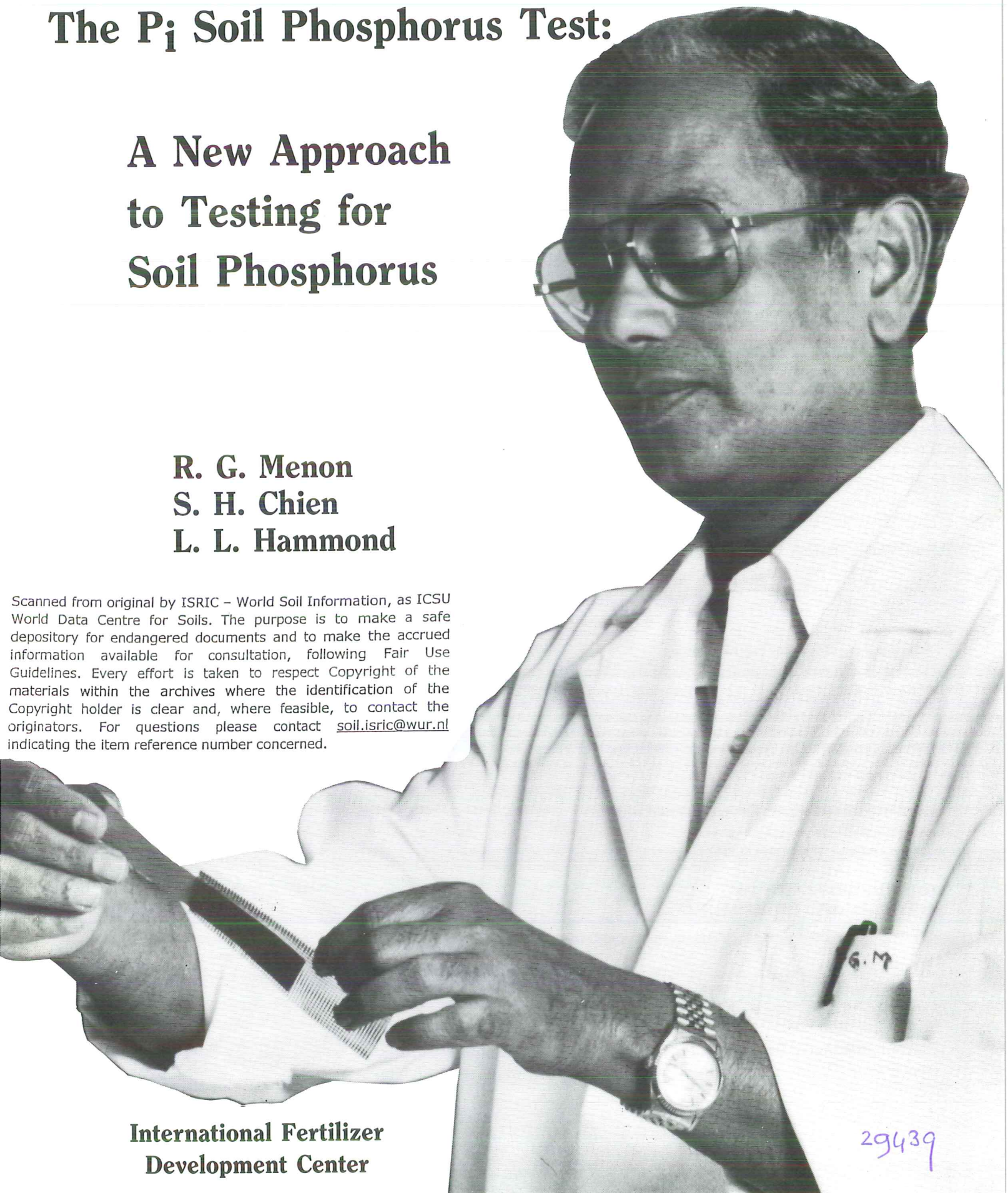

International Fertilizer Development Center

Abstract

There are many soil tests for determining plant-available phosphorus (P) in soils. Some of these work well in certain types of soil and not in others. Some have wider applicability but are not used in large-scale routine soil testing due to analytical difficulties or inconvenience.

A new approach to soil testing for plant-available P is being evaluated at the International Fertilizer Development Center (IFDC), using iron oxide-coated filter paper strips (P_i strips) as collectors for the P in the soil suspension. The new soil test, the P_i -P test, has been found to work well in acid as well as alkaline and calcareous soils. Laboratory and greenhouse studies have shown that the P_i test has a good potential as a reliable soil test for P with wide applicability. Moreover, the technology is appropriate to soil testing institutions in developing countries of the world. However, before the test can be used for fertilizer recommendations, the P_i test needs to be calibrated with field data. Work on this has already been initiated.

For reliability and precision, standardization of methodology is very important. This handbook describes the P_i methodology. It explains in simple terms the various steps involved in the preparation and use of the P_i strips. No attempt has been made in evolving guidelines for the interpretation of the P_i-P data for fertilizer recommendations. The handbook is intended for those who are interested in including the new test in their soil test calibration studies and for those who are cooperating with IFDC in P_i research.

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The P_i Soil Phosphorus Test: A New Approach to Testing for Soil Phosphorus

What is the P_i Test?

The P_i test is a new approach to testing for soil phosphorus. This test, instead of using extracting solutions to dissolve phosphorus, uses strips of paper coated with iron oxide (P_i strips) as collectors or sinks for the phosphorus in the soil. The P_i strips do not react with the soil but only adsorb the phosphorus that enters the soil suspension.

When the P_i strip is placed in a soil-water suspension, the iron oxide on the strip adsorbs the phosphorus in the suspension and retains it. The strip is then taken out, and the phosphorus retained is dissolved in dilute acid and measured.

Why Another Test?

An ideal soil test should predict the nutrient response of a wide variety of crops but, at the same time, be independent of the chemical and mineralogical properties of the soil. In all of the soil tests in use, except for the anion exchange resin and water extraction methods, phosphorus is removed by using extracting solutions. These solutions not only dissolve the phosphorus available for plant use but may also dissolve some phosphorus that is not available. Besides, these methods were developed to measure phosphorus from soils having specific characteristics and may not work equally well in other soils. Acid extractants like Bray 1 and Bray 2, Mehlich reagent, etc., work very well in acid soils, but they are not suitable for calcareous soils. Olsen extractant works well in calcareous and acid soils but underestimates phosphorus in soils treated with phosphate rocks.

The water extraction and anion exchange resin methods do not have the limitations of extracting solutions. However, these two methods are not suitable for large-scale analysis. The resin method is tedious and time consuming. Water extraction works well in soils with measurable amounts of phosphorus, but in tropical soils, soluble phosphorus is so low that there is difficulty in measuring it accurately.

Increasing interest has been shown in using nonconventional phosphate fertilizers such as phosphate rock or partially acidulated phosphate rock. All of the commonly used soil testing methods, however, are designed for making fertilizer recommendations for water-soluble phosphate fertilizers, e.g., single superphosphate (SSP) and triple superphosphate (TSP), and can either overestimate or underestimate available P from the nonconventional phosphate fertilizers. Thus, a new soil testing method that is independent of fertilizer source and soil properties is needed to determine plant-available phosphorus.

How Does the P_i Test Work? (Theory)

Because the iron oxide (Fe-gel) impregnated on the P_i paper is in the amorphous (noncrystalline) form, it has a very large specific surface area. The amorphous Fe-gel reacts immediately with phosphate ions in solution and forms water-insoluble iron phosphate on the P_i paper. Thus, the P_i paper serves as a sink for continuous depletion of phosphate ions in solution. The rate and amount of phosphate adsorption by the P_i paper depend on the forms and quantities of the phosphate present in the soil sample. Because the desorption of phosphate from the soil particles to the P_i paper is a diffusion-controlled process, the P_i extraction simulates the natural process in which a plant's roots absorb the phosphate ions from the soil solution under field conditions.

How is it Done?

The methodology originally proposed (*1*) was later refined and simplified (*2, 3*). The modified procedure is described here.

Preparation of the P_i Strip

Equipment Needed (Figure 1)

1. Two trays, one approximately 25 cm long, 17 cm wide, 5 cm deep (glass, enamel, porcelain, or plastic), and the other 20 cm long, 10 cm wide, and 5 cm deep (metal, glass, or enamel).
2. Crystallizing dish, 15 cm in diameter and 6 cm deep.
3. Clothespins for hanging paper (wood or plastic).

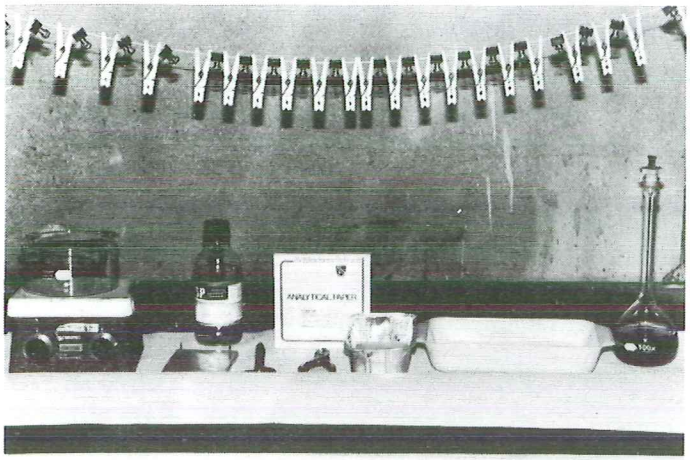

Figure 1. Equipment to Prepare P_i Strips

4. Paper cutter or pair of scissors.
5. Paper punch for making holes.
6. Magnetic stirrer and stirring rods (optional).
7. Waxing assembly. This can be made as follows:
   a. Prepare 6 aluminum plates 10 cm long, 12 cm wide, and 2 mm thick.
   b. Drill 2 holes, 2 mm in diameter, 1 cm down from the top and 1 cm from the side as shown in Figure 2.
   c. On one of the plates, fix two rods 2 cm long and 2 mm in diameter. These could be screws with flat ends or even nails. This will serve as the bottom plate (Figure 3).
   d. Prepare a template 12.5 cm long and 12 cm wide with the corners rounded off to fit the curvature of a 15-cm-diameter filter paper (Figure 4). The templates can be made from an aluminum plate or from stiff cardboard or plastic.

Chemicals

1. Iron chloride, 10% solution in water. Dissolve 100 g pure phosphorus-free iron chloride ($FeCl_3 \cdot 6H_2O$) in distilled water. Dilute to 1 L with distilled water and mix well. (Instead of iron chloride, pure iron nitrate could also be used. In this case, prepare 20% solution.)
2. Ammonia solution. Mix 500 mL ammonium hydroxide (30% NH_3) with 500 mL distilled water. Mix well.
3. Paraffin wax, solid.
4. Filter paper. Whatman No. 541 or Schleicher and Schuell (S and S) No. 589-WH. hardened paper, 15 cm in diameter.

Procedure

1. Pour the iron chloride solution into the tray to a depth of about 1-2 cm.

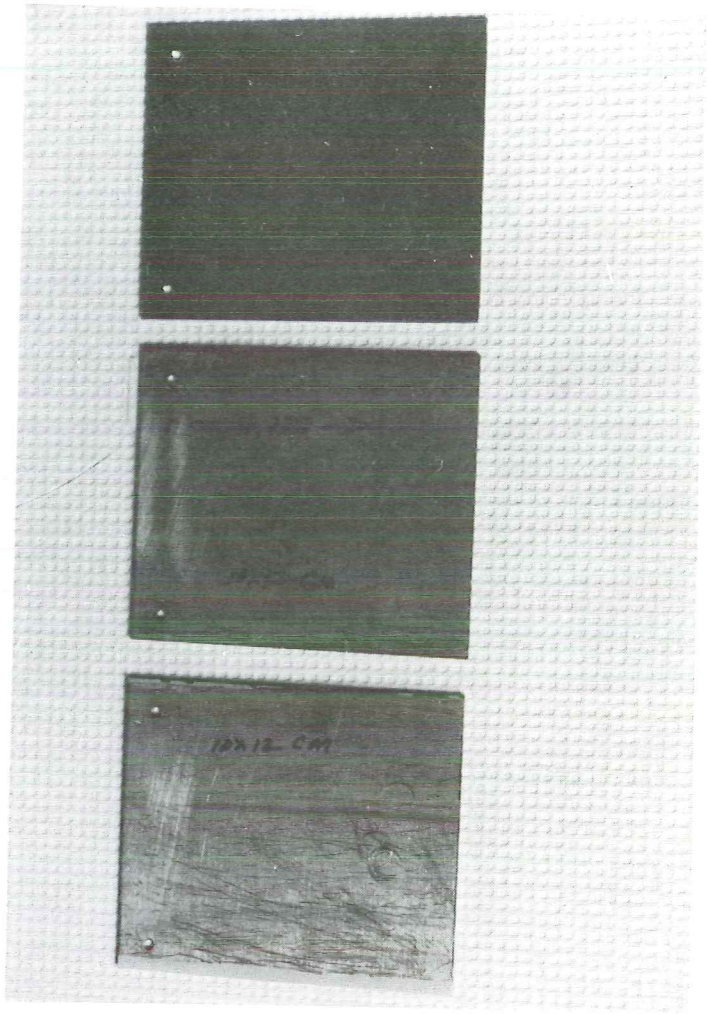

Figure 2. Aluminum Plates for Waxing Assembly.

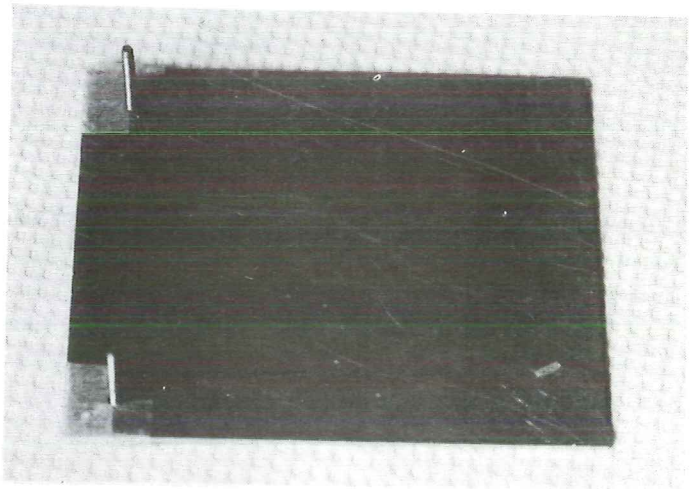

Figure 3. Bottom Plate of the Waxing Assembly.

Figure 4. Template.

2. Pull the filter paper circles swiftly through the solution, making sure that the paper is completely immersed in it momentarily (Figure 5). Hang the paper to dry for about 1 hour at room temperature, using the clothespins. This could be done conveniently by tying a cord to the nails fixed on the walls of a fume hood and attaching the clothespins to it (Figure 6).
3. When the filter paper circles are dry, they will be yellow in color. Remove them from the clothespins.
4. Place the crystallizing dish on the magnetic stirrer in the fume hood. Turn the exhaust system on. Pour the ammonia solution into the dish to a depth of 2 cm or so.
5. Place the iron chloride-saturated filter paper circles on the crystallizing dish as a cover. Up to four papers can be kept at one time. Turn the stirrer on and stir the ammonia solution gently. Although ammoniation will occur more quickly with stirring, the magnetic stirrer and stirring are not essential (Figure 7).

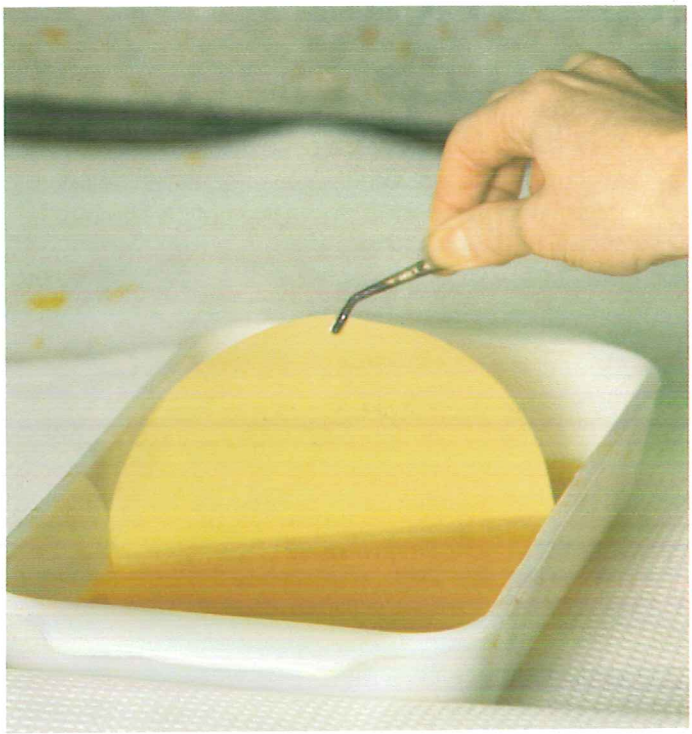

Figure 5. Filter Paper is Drawn Through Iron Chloride Solution.

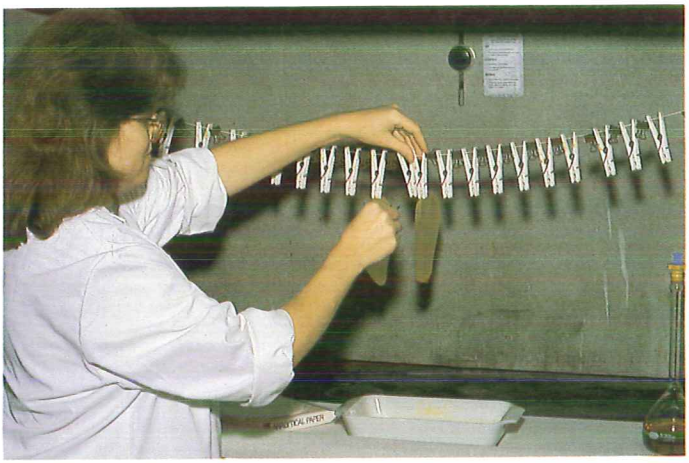

Figure 6. Clothesline Used for Drip Drying Filter Paper.

Figure 7. Iron Chloride-Saturated Paper is Ammoniated.

6. As the ammonia vapor reacts with the iron chloride on the paper, the iron chloride is converted into iron oxides and the color of the paper changes from yellow to brown. When all the iron chloride has been converted into iron oxides, as indicated by the intensity and uniformity of the brown color, turn the stack of paper over to expose the other side to ammonia.
7. When both sides of the paper are uniformly brown, remove them (Figure 8).

Figure 8. Paper Before and After Ammoniation.

8. Using the paper cutter or pair of scissors for cutting and the template for a guide, cut the paper circles to rectangles 12.5 cm long and 12 cm wide (Figure 9).

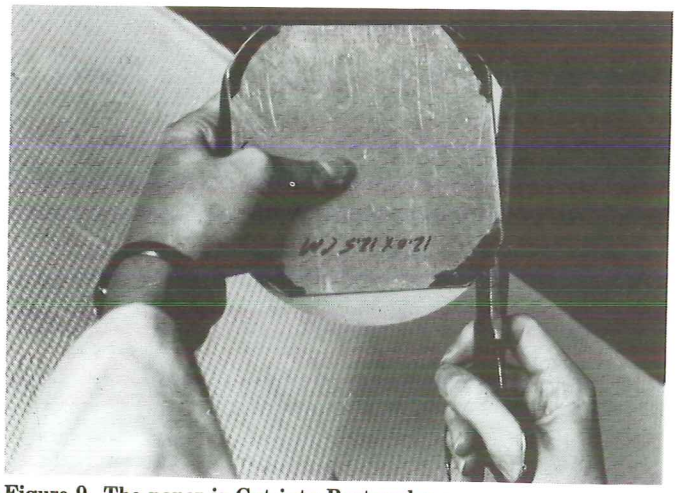

Figure 9. The paper is Cut into Rectangles.

9. With the paper punch, punch two holes in the filter paper to correspond to the holes on the metal plates of the waxing assembly (Figure 10).
10. Place the paraffin wax in a metal tray and melt at a low heat.

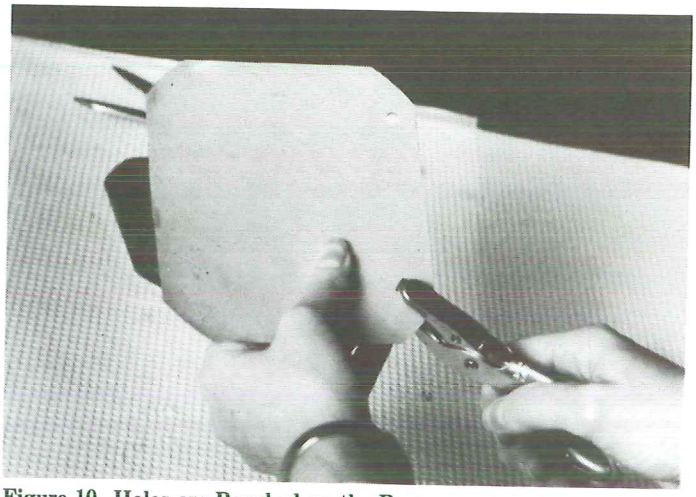

Figure 10. Holes are Punched on the Paper.

11. Mount one of the filter paper sheets on the bottom plate of the waxing assembly. Mount an aluminum plate over it, then another filter and so on. Mount up to five filter sheets alternating with the metal plates. The paper thus mounted will have 1.25-~~mm~~ cm-wide edges protruding outside (Figure 11).

Figure 11. Paper is Mounted on the Waxing Assembly.

12. Holding the waxing assembly tight, dip the protruding ends of the paper in the molten wax, one side at a time. The metal plates prevent the wax from creeping inside beyond the protruding ends of the paper and also prevent the edges from sticking together (Figure 12).
13. Separate the plates and remove the filter paper sheets from the waxing assembly. Use a spatula to scrape off excess wax from the waxed area (Figure 13).

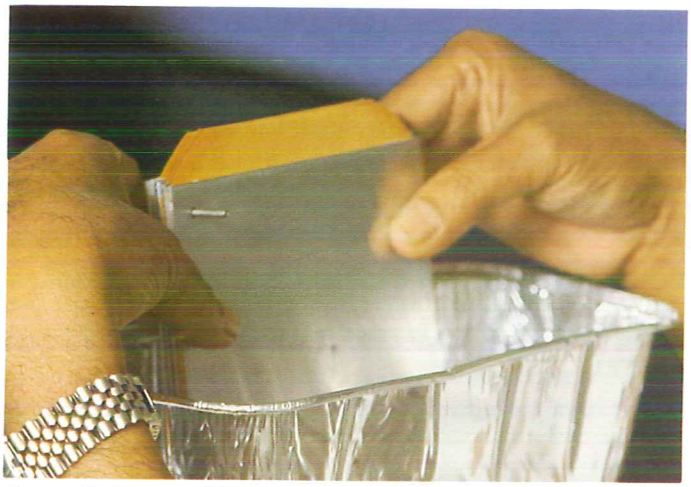

Figure 12. Paper is Dipped in Molten Paraffin Wax.

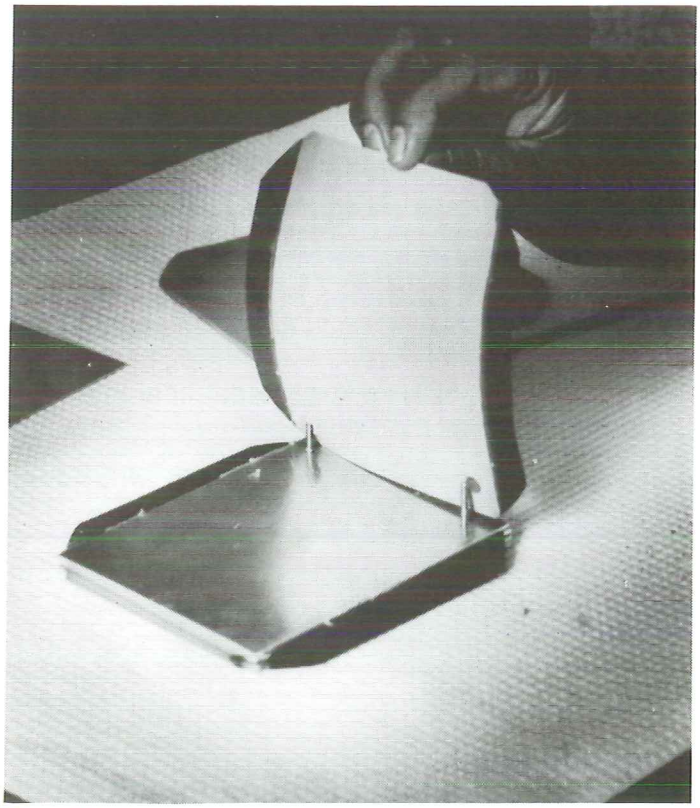

Figure 13. Paper is Removed From the Waxing Assembly.

14. Using the paper cutter or pair of scissors, cut the paper into strips 12.5 cm long and 2 cm wide with a waxed area 1.25 cm wide on either end. Each strip will have a reactive surface of 40 cm^2. Because the part of the sheet with holes will be discarded, four good strips can be obtained from each sheet (Figure 14).

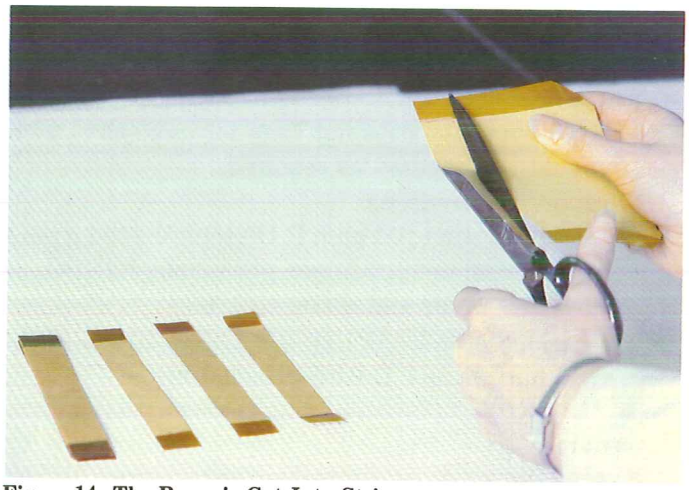

Figure 14. The Paper is Cut Into Strips.

Extraction of Phosphorus, Using P_i Strips, and Measurement (Laboratory Procedure)

Equipment Needed

1. Shaker, reciprocal.
2. Spectrophotometer.
3. Shaking bottle, wide mouthed, with screw cap, 100-mL capacity.
4. Funnels.
5. Volumetric flasks, 25 mL.
6. Pipettes, beakers, cylinders, etc.
7. Nylon mesh bags, 12.5 cm long and 2.5 cm wide. These can be made from nylon mesh cloth, mosquito nettings, etc., either by stitching or using a sealing machine.

Chemicals

1. Calcium chloride solution, 0.01 M. Dissolve 14.702 g $CaCl_2 \cdot 2H_2O$ in distilled water and dilute to 10 L.
2. Sulfuric acid, 0.2 N. Place 1 L distilled water in a 2-L Pyrex beaker. Carefully measure 56 mL concentrated sulfuric acid in a graduated cylinder. Pour the acid slowly down the sides of the beaker into the water. Mix by stirring with a glass rod. Let the solution become cold. Dilute it to 10 L with distilled water. Mix well.

3. Ammonium molybdate reagent.
   a. Dissolve in 250 mL of warm distilled water 12 g of ammonium molybdate $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$.
   b. Dissolve 0.2908 g antimony potassium tartrate $[K(SbO)C_4H_4O_6 \cdot \frac{1}{2}H_2O]$ in 100 mL distilled water.
   c. Add 148 mL concentrated sulfuric acid to 800 mL distilled water. Allow the solution to cool.
   d. Add solution a and b to c. Mix thoroughly. Dilute to 2 L with distilled water.
4. Phosphorus reagent. Dissolve 1.056 g ascorbic acid in 200 mL of the ammonium molybdate reagent. Mix well. This reagent does not keep for more than 24 h. It should be prepared as required.
5. Phosphorus standards.
   a. Stock solution 100 ppm P. Dissolve 0.4393 g pure, dry potassium dihydrogen phosphate, KH_2PO_4, in distilled water and dilute to 1 L.
   b. Working standards 1 ppm P. Dilute 10 mL of the 100 ppm P to 1 L with distilled water.

Procedure

1. Extraction
   a. Weigh 1 g soil (2 mm fraction) and transfer to a 100-mL shaking bottle with screw cap (Figure 15).
   b. Add 40 mL $CaCl_2$ solution to the bottle (Figure 16).
   c. Place one P_i strip inside the nylon bag and place it in the bottle. Close the bottle tight with the screw cap (Figures 17, 18).
   d. Place the bottle in the shaker. Shake for 16 h (overnight) at low speed (180 oscillations per minute) (Figure 19).
   e. At the end of the shaking period, take the strip out of the nylon bag. Rinse the strip with distilled water to remove any soil particles sticking to it (Figure 20). Place the strip on a paper towel for a few minutes to dry. It is not necessary that the strip be completely dry.
   f. Transfer the strip to another shaking bottle. Add 40 mL 0.2 N sulfuric acid (Figure 21). Close the bottle tight and shake for 1 h in the shaker.
   g. Filter and save the filtrate for P measurement (Figure 22).
2. Measurement
   a. Pipette 2.5, 5.0, 7.5, 10, and 20 mL of the 1 ppm standard to 25 mL volumetric flasks. For the control, transfer 20 mL 0.2 N H_2SO_4 to another flask.

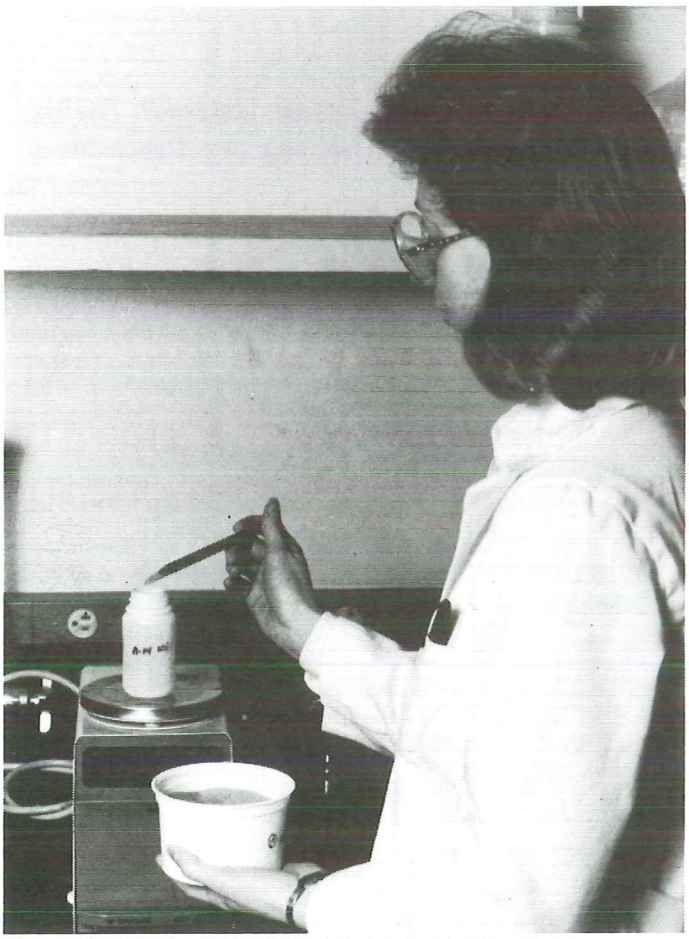

Figure 15. One Gram Soil is Weighed Into Shaking Bottle.

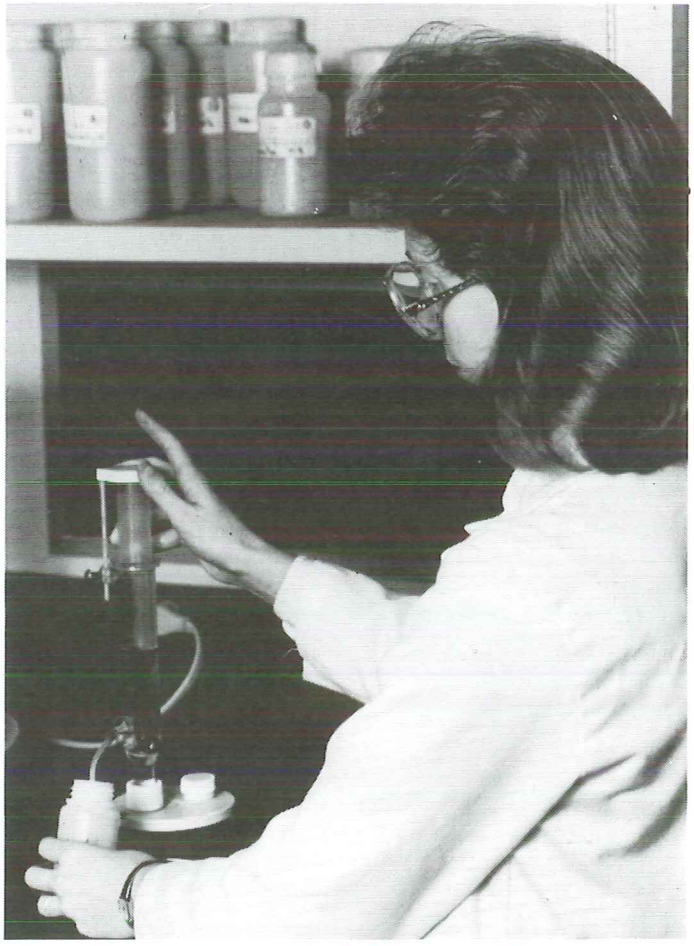

Figure 16. 40 mL Calcium Chloride Solution is Added.

Figure 17. P_i Strip is Placed in Nylon Sheath.

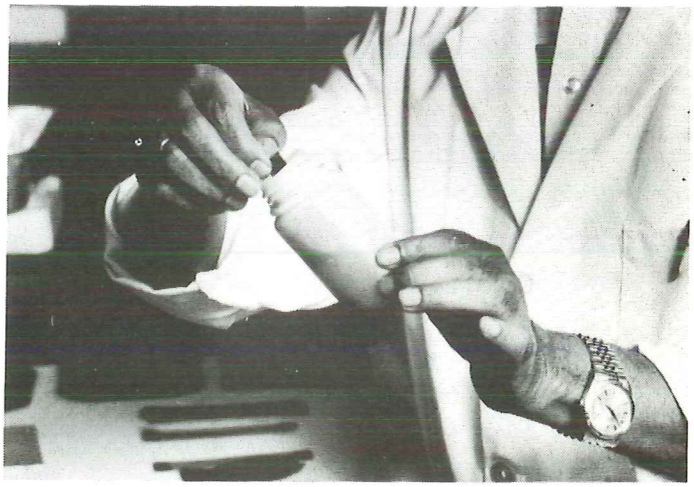

Figure 18. The Strip Enclosed by the Sheath is Placed in the Shaking Bottle.

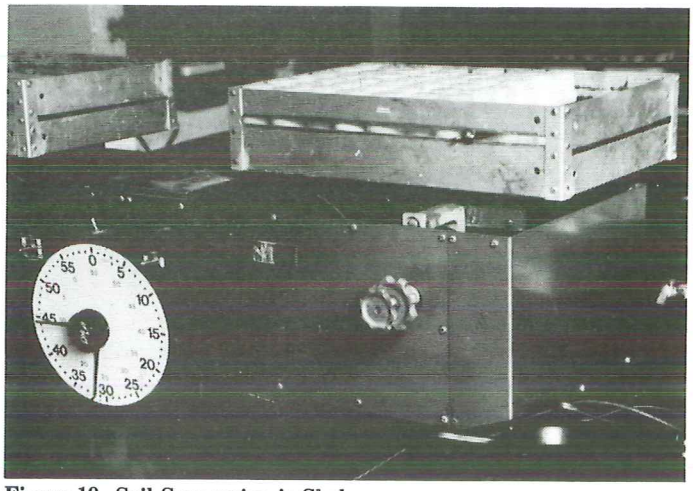

Figure 19. Soil Suspension is Shaken.

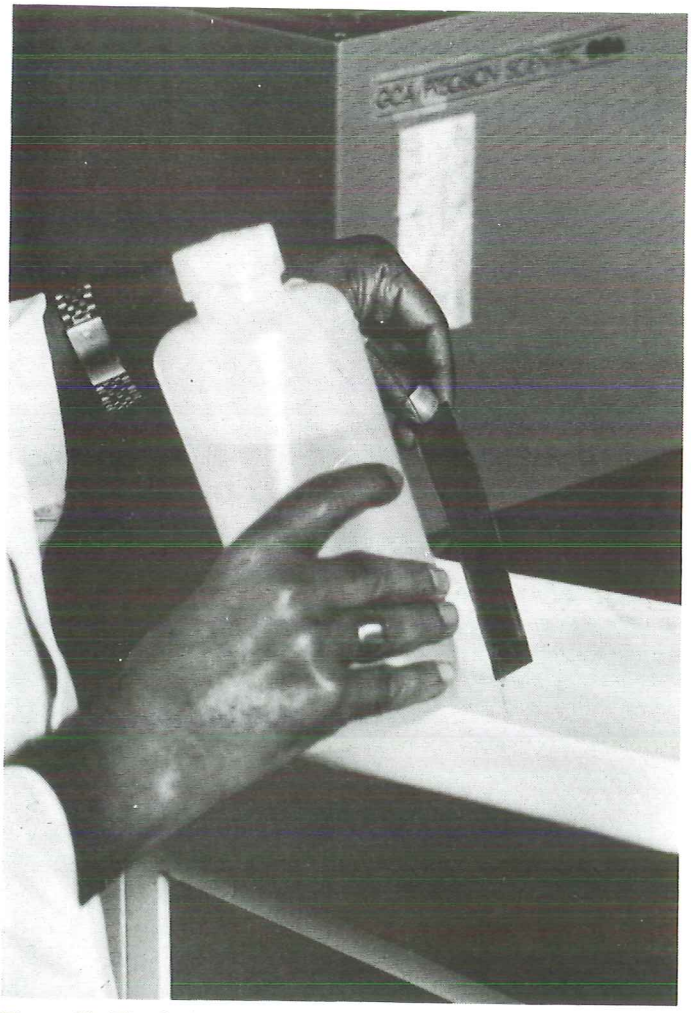

Figure 20. The Strip is Removed and Rinsed With Water.

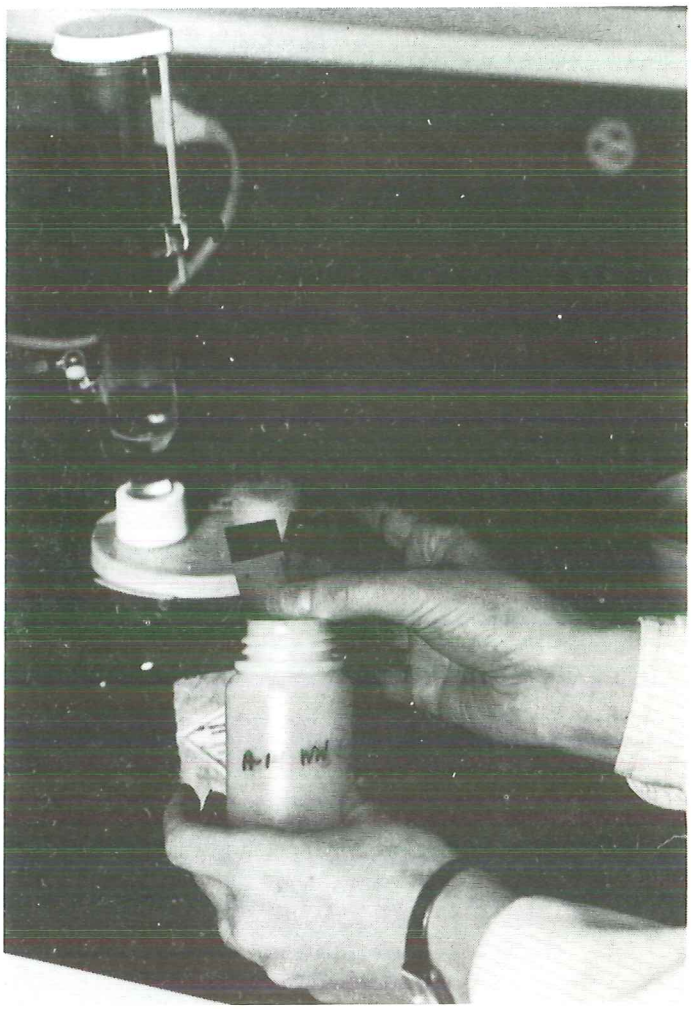

Figure 21. The Strip is Placed in the Bottle and 40 mL Sulfuric Acid is Added.

Figure 23. 20 mL of the Extract is Pipetted Into Volumetric Flask.

Figure 22. After Shaking, the Solution is Filtered.

b. Add 4 mL phosphate reagent to each flask. Dilute to 25 mL with distilled water and mix well. Let stand for 30 min. The concentrations of phosphorus in the colored solutions will be 0, 0.1, 0.2, 0.3, 0.4, and 0.8 ppm P.

c. Pipette 20 mL of the soil extract into a 25-mL flask (Figure 23). Add 4 mL phosphate reagent (Figure 24), dilute to 25 mL with distilled water, and let stand for 30 min. (Figure 25). If the color is too deep, pipette a lower volume of extract. Bring it up to 20 mL with water.

d. Adjust the wavelength of the spectrophotometer to 880 nm. Read the concentration of the standards against the blank (Figure 26).

   If the instrument reads the concentration of the P in solution directly, calibrate the instrument using the top standard. If the instrument reads percent transmission, read %T for all the standards.

e. Read the concentration or %T for the soil extracts.

Figure 24. Coloring Reagents are Added.

Figure 25. Solutions After Color Development.

Figure 26. Concentration of Phosphorus is Measured Using Spectrophotometer.

f. Prepare a graph by plotting %T for the standards against concentration. Read the concentration of P in the extracts from the graph.

Calculation

If c is the concentration of P in the colored solution, read from the instrument or from the graph

$$\text{ppm P in the soil} = c \times \frac{25}{V} \times \frac{40}{W}$$

Where: 25 is the volume of the colored solution, V is the mL extract taken to develop the color, 40 is the total volume of the extract, W is the weight of soil taken.

Thus, $\text{ppm P in the soil} = c \times \frac{1000}{V \times W}$;
if W is 1 and V is 20, ppm P = c x 50.

How Good is the P_i Test?

Research carried out thus far indicates that the P_i test can be a good "general soil test" for phosphorus. This new test offers several important advantages.

1. Unlike other P tests now being used, the P_i test works well in all types of soil—acidic, alkaline, or calcareous—and is especially suited for the phosphorus-deficient, acid soils of the tropics.
2. Limited pot trials carried out at IFDC have shown that the test can be used in soils fertilized with conventional fertilizers like superphosphates as well as nonconventional materials such as phosphate rocks, partially acidulated phosphate rocks, or phosphate

rocks compacted with soluble fertilizers like superphosphates. The other tests can overestimate or underestimate plant-available phosphorus from soils treated with phosphate rocks or modified rocks.

3. The P_i test is inexpensive, and the technology is appropriate to any laboratory in the less developed countries. The P_i paper strips can be prepared in any laboratory, and the test itself is easy to perform. Overnight shaking may pose some difficulty in countries faced with power shortages. However, tests are currently being carried out to extract phosphorus by surface contact between the P_i strips and moist soil, and the results look promising. This would eliminate the need to shake the soil suspension overnight.

Further research is needed to test the suitability of the P_i test under field conditions. Research is also being carried out to develop a field test in which P_i strips would be embedded in the soil, and the blue color would be directly developed on the paper in proportion to the concentration of phosphorus retained on the paper. The color would then be compared with standard color charts. This procedure would enable the extension agent or the farmer to extract and measure phosphorus in the soil at the site instead of sending the soil sample to the laboratory.

Acknowledgments

The idea of using iron oxide-coated paper to extract phosphorus was originally suggested by Dr. H. A. Sissingh (Retired), Institute of Soil Fertility, Haren, the Netherlands.

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