# CORRELATION OF FIELD AND GREENHOUSE RESPONSES

## TO PHOSPHORUS FERTILIZER WITH SOIL TESTS

FOR AVAILABLE PHOSPHORUS

By

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## I INTRODUCTION

One of the objectives of the field of soil chemistry and fertility has been to determine the fertilizer needs of a soil by means of chemical methods. Bray  $(4)^{\perp}$  reviewed the historical development of this aspect of the science and noted that, in general, three methods have been followed in establishing the fertilizer needs of a given soil; the experiment field method, the pot culture method and the chemical studies of the soil and the plants growing on the soil. Early studies involved total analysis of the soil, but no practical recommendations for the needs of the soil were secured. Later on more promising procedures were developed and rather broad correlations between chemical tests and field responses were obtained. At this stage, the basic concepts involved not only the idea of measuring the availability of the element in question, but also the procedure to be followed. For example, it was thought that the chemical method of extraction should imitate closely the absorption by plant roots. Advances in this branch of knowledge make these concepts appear inadequate. The latest concept of an extracting solution is one that can extract all of that form or forms of the element being tested for and which have, therefore, "a purely chemical basis". The chemical methods must be correlated with fertility studies using the crops and soils on which the tests are to be applied.

Williams (41) stated that in determining the so-called available phosphorus, many methods have been developed in attempts to assess the I Figures in parentheses refer to Literature Cited. fertilizer requirement of the soil or its ability to supply phosphates to growing plants. Most of these methods are empirical and must be correlated with field experiments before they can be used as a source of information. The chemical methods usually extract the soil with a solution which, it is hoped, will dissolve mainly that fraction of the soil phosphate which would be utilized by the crop.

Olsen et al. (27) discussed the characteristics of a useful chemical extractant for available soil phosphorus. It will (a) measure all or a definite proportion of the various forms of phosphorus in the same relative amounts as they are absorbed by plants during the growing season; (b) correlate to a high degree with plant uptake of phosphorus and yield response to added phosphorus over a wide range of soil types; (c) minimize the secondary precipitation and adsorption reactions that may occur during extraction; and (d) be adaptable to routine-test procedures.

Oklahoma soils have phosphorus fertility as a specific problem. In the present work, an attempt is made to find a chemical extractant for available soil phosphorus capable of correlating in an acceptable degree with field and greenhouse results.

#### II LITERATURE REVIEW

#### Soil Phosphorus Compounds

Because the relatively large number of forms of phosphate in the soil have different solubility behaviors, the procedures to identify them have been developed very slowly. In some cases, there is not a sharp delineation among certain fractions.

Russell (29) grouped the soil phosphorus compounds in three categories: (a) inorganic phosphorus in neutral soils, probably a calcium phosphate or hydroxyapatite; (b) inorganic phosphorus in acid soils, presumably combinations with iron and aluminum oxides; and (c) organic phosphorus compounds, presumably of value after mineralization by microorganisms. Dean (11) proposed a fractionation of total soil phosphorus by means of an alkali followed by an acid extraction. He found four groups: (a) inorganic alkali soluble-iron and aluminum phosphates and small amounts of mono and dicalcium phosphate if they are present in the soil; (b) acid soluble, but alkali insoluble-apatites and tricalcium phosphate; (c) organic phosphates, definite organic combinations of phosphorus; and (d) insoluble phosphorus of unknown chemical composition.

From the point of view of the phosphate-carrying particle of natural soils, Burd and Murphy (6) suggested a division into two defined classes: those which dissolve in acids (both ions enter in solution) and those which do not dissolve in acid, but which either

hydrolyze in alkaline solutions or release phosphate only as a result of anion exchange. Chang and Jackson (7) recently related the distribution of inorganic phosphorus in soils with the degree of chemical weathering. The chemical weathering sequence was calcium phosphate, aluminum phosphate, iron phosphate and occluded phosphate. The latter category refers to iron phosphate and aluminum-iron phosphate coated with iron oxide.

In discussing soil phosphorus in relation to its availability to plants, Truog (37) used the designations of "readily available" and "difficulty available" rather than available and unavailable. He noted that "readily available" exists largely as calcium phosphate and the "difficulty available" as basic or ferric phosphate. Chang and Jackson (7) stated that the various forms of phosphates with their different solubilities give an indication of the availability of phosphorus in the soil, although the solubility is to a great extent determined by the amount of surface of the various forms.

Investigators have found that some soils which have very low amounts of available phosphorus do not respond to phosphate applications or the response obtained is not accord with the phosphorus deficiency. The explanation given has been "phosphorus fixation". Cooke (8) defined phosphorus fixation as the precipitation of phosphate from solution to forms which are useless to plants or are insoluble in the conventional extractant. Gardner and Kelly (17) suggested that the addition of monocalcium phosphate to calcareous soils may result in carbonato-apatite, a compound which is relatively insoluble.

In acid soils, the iron and aluminum hydroxides are responsible for phosphate fixation. In a potentiometric titration of iron and

aluminum chlorides in the presence of phosphate, Swenson and his coworkers (35) detected inflection points in the titration curves which indicate that for each metal ion, two hydroxyls and one phosphate ion reacted to form a basic phosphate. They proposed the following mechanism of phosphate fixation by iron or aluminum.

AL 
$$(H_2O)_3 \stackrel{OH}{=} H_2PO_1 \stackrel{AL}{=} (H_2O)_3 \stackrel{OH}{=} H_2PO_1 + OH$$

In chemical and greenhouse experiments Murphy (26) noted that soils with a kaolinitic type of clay have a high capacity to fix soluble phosphates. Stout (34) proposed a mechanism for the reaction between kaolinite and phosphate.

$$R \stackrel{OH}{=} 0H + H_2 PO_{\downarrow} + H^+ \xrightarrow{PO_{\downarrow}} R \stackrel{O}{=} P + 0 + 3H_2 O$$

Dean (11) found in Hawaiian soils that: (a) phosphorus added to acid soils tends to accumulate in the alkali soluble form; (b) phosphorus added to neutral or calcareous soils tends to accumulate in acid soluble, but alkali insoluble forms; (c) upon dispersion of the soil in the presence of a dilute acid extracting solution there may be a reaction between soil constituents and the readily soluble phosphorus; and (d) the insoluble forms of soil phosphorus are probably of primary origin. Wild (40) grouped the retained soil phosphates in the following order: iron and aluminum compounds, calcium **and** magnesium compounds, clay minerals, compounds with other inorganic soil constituents (quartz, manganese, and titanium), and compounds with organic matter.

#### Extractants of Soil Phosphorus

#### Acid Extractions

For a long time, the available phosphorus of soils has been

extracted by means of dilute solutions of organic or inorganic acids. Good correlations between the phosphorus extracted and plant responses have been obtained for some soils, but poorer correlations have been noted when a wide variety of soils is employed.

Cooke (8) pointed out two main causes of poor correlation between the amount of phosphate extracted by acids and the amount of phosphate taken up by plants. The first cause was that dilute acid extractions were based on the premise that plants dissolve plant nutrients through the activity of acids exuded by their roots. New evidences in plant nutrition have shown that plants may acquire nutrients by direct exchange between root and soil particles. Secondly, acid extracting solutions are more acid than most field soils. Thus, the acid extractants dissolve phosphate compounds that are not normally available to plants. Elimination or diminution of phosphate re-fixation during dilute acid extraction is of a great importance. Cooke also cited three types of mechanisms of phosphate fixation that may occur when a soil is extracted with dilute acid; (a) chemical precipitation by soluble salts of iron, aluminum, titanium and manganese; (b) adsorption by the hydrous oxides of iron, aluminum, titanium and manganese and (c) adsorption by alumino-silicates.

Fraps (14) employed N/5 HNO<sub>3</sub> and concluded that this acid extractant dissolves calcium phosphates completely, but only dissolves mineral aluminum phosphates or basic ferric phosphates to a slight extent. He recommended this extractant method for calcareous soils. Truog (37) used 0.002 N  $H_2SO_4$  successfully in Wisconsin soils. He stated that this extractant dissolves the readily available phosphate (calcium phosphate) and that this weak extractant does not bring into solution iron oxide or

organic matter that may interfere in the colorimetric reading. Harper (18) worked with 0.2 N  $H_2SO_{l_1}$  as extractant in a study of soil samples from Oklahoma and other states. By using this method, he was able to establish response limits of phosphorus for alfalfa, cotton, oats, sweet clover, wheat, corn, soybeans, cowpeas and grain sorghums. Beater (2) used N/21  $H_2SO_{l_1}$  as a phosphorus extractant in a rapid method for obtaining readily soluble phosphate.

Harper (19) also used 0.1 N acetic acid as a phosphate extractant with very good results. He stated that this solution will dissolve considerable amounts of freshly precipitated iron, aluminum and manganese phosphate. He also emphasized the fact that when there is some calcium phosphate in the soil, the strength of the solution will keep the dissolved phosphate from being adsorbed by the soil particles. This method is not recommended for calcareous soils because considerable amounts of occluded phosphate is dissolved. Morgan (25) extracted soil phosphates with 0.5 N acetic acid buffered at approximately 4.8 with sodium acetate. Cooke (8) also employed 0.5 N acetic acid alone and with different reagents in his study of the phosphate fixation during acid extractions. He concluded that in soils where fixation is caused largely by hydrous oxides, selenious-acid mixtures will give more satisfactory values than acetic acid alone. It is possible that selenite ions replace hydroxyl groups on the surface of the hydroxide. This same author pointed out that acids used to extract soils should not dissolve compounds which are useless to crops. Citric acid is thought to dissolve iron phosphate from some soils and give false high values for available phosphorus.

In most Arizona soils, McGeorge (23) found that phosphorus availability is related to the CO<sub>2</sub> production of the soil. Based on this

evidence, he proposed a phosphorus extraction method employing CO<sub>2</sub> for calcareous soils. The extraction is mainly a function of hydrogen ions which must be sufficient to overcome the effect of calcium in precipi-tating the phosphate extracted.

# Fluoride Extraction

Bray and Kurtz (5) proposed two methods in which they included  $NH_{l_{4}}F$  besides the dilute acid. Method No. 1 for adsorbed forms (0.025 N HCl + 0.03 N  $NH_{l_{4}}F$ ) and method No. 2 for combined forms (0.1 N HCl + 0.03 N  $NH_{l_{4}}F$ ). These authors noted that these extractants will remove proportional parts of each soil phosphate form and that the amount of phosphate extracted is largely influenced by the more readily soluble portion of each form.

The fluoride ion has been employed in several other studies. Turner and Rice (38) worked with neutral  $\mathrm{NH}_{4}\mathrm{F}$  in studying the phosphate adsorption characteristics of Al(OH)<sub>3</sub> and Fe(OH)<sub>3</sub> gels. They found that the  $\mathrm{NH}_{4}\mathrm{F}$  reacted with Al(OH)<sub>3</sub> gel to form  $(\mathrm{NH}_{4})_{3}\mathrm{AlF}_{6}$  and the phosphate adsorbed by these gels was more or less completely released by the fluoride action. The Fe(OH)<sub>3</sub> gel was apparently not attacked by the fluoride and the phosphate adsorbed was not released. Seatz (31) had a similar evidence in phosphate desorption studies. His results showed that the fluoride ion is more effective in desorbing P<sup>32</sup> from aluminum phosphate than from iron phosphate.

Kurtz et al. (22) performed a study to remove adsorbed phosphates with different salt solutions as extracting agents. They found that the replacing ability of the different anions was in the following order: fluoride, oxalate, citrate, bicarbonate, borate, acetate, thiocyanate,

sulfate, and chloride. They also explained that the fluoride ion, which has a small radius, forms stable complexes with iron, aluminum, and silicon ions and is isomorphous with the phosphate ion in numerous compounds. Dickman and Bray (12) chose the fluoride ion as a more selective extractant of the adsorbed forms of phosphate. They stated that the fluoride ion is extremely reactive, is stable in neutral solutions, and offers no complications in the subsequent colorimetric determination of phosphate. The fluoride ion might be expected to replace OH groups from the surface of kaolinite. Jackson (21) found that the inclusion of acid in the Bray and Kurtz extractants causes the dissolution of the more active calcium phosphate and prevents precipitation.

#### Alkaline Extractions

 $NaHCO_3$  and  $K_2CO_3$  are among the alkaline extracting solutions that have been used in available phosphorus determinations. Olsen et al. (27) developed the 0.5 M NaHCO<sub>3</sub> method in an attempt to find a chemical extractant which would measure not only a definite proportion of the various forms of phosphorus available to plants, but also would minimize the secondary precipitation reactions which occurred during the extraction process. They proposed two major mechanisms that may take place during the NaHCO<sub>3</sub> extraction: (a) calcium phosphate increases in solubility in NaHCO<sub>3</sub> at pH 8.5 as a result of the repression of the Ca ion activity (common ion effect of carbonate ions in the presence of solid phase CaCO<sub>3</sub>); and (b) the bicarbonate, carbonate, and hydroxyl ions replace phosphate ions on the surface of the soil particles. In calcareous soils, they found a very good correlation between phosphorus extracted by NaHCO<sub>3</sub> and "A" values. The "A" value is defined as amount of soil phosphorus that

is as available to the plants as phosphorus in a standard phosphate fertilizer which has been mixed with the soil.

Gardner and Kelly (17) obtained very acceptable results using 1% potassium carbonate in determining the available phosphorus in Colorado soils. This method has value for soils of similar composition because there is a correlation between the alkaline and acid-extractable phosphorus. The  $K_2CO_3$  method is evidently not adaptable where the soils vary widely in chemical composition, especially if non-calcareous soils are included with calcareous soils. Das (9) reported that the action of 1%  $K_2CO_3$  in calcareous soils is two-fold. A reaction takes place with any dicalcic or other phosphates present in these soils with the production of insoluble tricalcic phosphate and soluble potassium phosphate plus a release of phosphate from some organic compounds.

#### Water Extraction

Water-soluble phosphorus determinations have been performed with considerable success as an indicator of crop response. Olsen (27) cited Bingham (1949) and Martin (1950) from California, who obtained a high correlation between water-soluble phosphate and "A" values. Their data indicate that the water-soluble phosphate tends to give lower correlations with "A" values as the number of soil types involved increases. Fried and Shapiro (16) reported that if different soils are arranged in the order of their amounts of plant-available phosphorus, there is little relationship between the plant-available phosphorus and the phosphorus in the initial water or soil solution extracts. However, if only soils showing a similar

pattern of phosphate release are compared, the initial water extract is a relatively better measure of plant-available phosphorus. Kurtz and his co-workers (22) suggested that the phosphate released by water is due to hydrolysis. Thus, replacement of the phosphate by the hydroxyl ion of water is proportionally great, especially when large amounts of adsorbed phosphate are present.

## III MATERIALS AND METHODS

Six Oklahoma soils were employed in this study: Norge, sample from the Agronomy farm at Perkins; Carey, sample from wheat fertility plots near Custer City; Hollister, soil sampled from wheat fertility plots on the Altus irrigation station; Tabler, sample from Renfrow; Minco, sample taken from wheat fertility plots near Hydro; and Pond Creek, sampled from wheat fertility plots near Carrier. A bulk soil sample (0 to 6 inches deep) of about 200 pounds and a smaller one of about 2 pounds from below the arable layer were taken at each location. The 200 pound soil samples were ground and screened through a 4-mesh sieve. A small portion of each soil was put aside for chemical and physical analyses and the rest was employed in the greenhouse experiment.

## Soil Analyses

The mechanical analysis was determined by the hydrometer method (3). The pH was measured in a thick paste (made by moistening the soil with distilled water) with a glass electrode pH meter (30). The percentage of organic matter was obtained by oxidation with sodium dichromate in the presence of sulfuric acid (39). The percent nitrogen was determined by a modification of the method recommended by the Association of Official Agricultural Chemists (1). Selenium was used instead of copper or mercury and sodium sulfate was added to the digestion flask

to raise the boiling point (20). Available potassium was measured by analyzing the ammonium acetate leachate of the soil with the flame photometer (28). The cation exchange capacity was obtained by displacing the cations with ammonium acetate. The excess ammonium ions were washed from the soil with methanol and then the ammonium ions were replaced with magnesium oxide in the distillation phase (1). Exchangeable calcium, magnesium and sodium were analyzed in the leachate with the flame photometer (28). Some of the physical and chemical characteristics of the soils are shown in Table I.

#### Greenhouse Experimental Procedure

In the greenhouse experiment, a randomized complete block design was used with five fertilizer treatments, six soils, and three replications. Ten pounds of surface soil were placed in each pot and treatments equivalent to 0, 20, 40, 80, and 160 pounds per acre of available  $P_2O_{ij}$  were added to the Hollister, Minco, Tabler, Pond Creek, and Carey soils. For the Norge soil, the phosphate treatments employed were slightly different; equivalent to 0, 18, 36, 72 and 144 pounds of available P205 per acre. These rates corresponded with those used in a wheat fertility study at Perkins. In addition the equivalent of 40 pounds of nitrogen and 40 of available K<sub>2</sub>O per acre were applied to all soils to prevent any deficiency of these nutrients. The source of phosphorus was superphosphate (20% available  $P_2O_5$ ); the nitrogen carrier was ammonium nitrate (33.5% N); and pottasium was supplied as muriate of potash (60% available  $K_2^0$ ). The fertilizer treatments were placed 2 inches deep. Fifteen seeds of Concho wheat were planted in each pot at a 1 inch depth. After fertilization and planting, the pots were

TABLE	Ι
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PHYSICAL AND CHEMICAL CHARACTERISTICS OF THE SIX SOILS USED IN THE GREENHOUSE EXPERIMENT

Scil			••••••••••••••••••••••••••••••••••••••			Arroil	Cation Exchange	Perc	ent Satur	ation
Series	Texture	Depth	рH	<b>%0.</b> М.	%N	K K	(m.e.)	Ca	Mg	Na
Carey	Silt loam	surface	6.70	1.33	0.08	344	8.00	33.75	42.37	1.62
tt	Silt loam	subsoil	7.80	1.28	0.06	158	10.28	68.97	37.06	1.26
Hollister	Loam	surface	7.50	1.79	0.10	800	21.68	32.79	16.33	2.17
11	Silty clay loam	subsoil	7.95	1.79	0.06	3.74	25.72	30.52	22.51	3.42
Minco	Silt loam	surface	6.50	1.57	0.08	640	9.06	32.01	15.01	0.99
<b>#1</b>	Sandy loam	subsoil	6.55	1.62	0.06	260	12.16	33.63	15.13	1.15
Norge	Sandy loam	surface	5.60	0.90	0.06	272	5.30	20.00	12.26	1.51
11	Sandy clay loam	subsoil	5.60	0.92	0.08	152	10.73	<b>19.2</b> 0	16.96	0.84
Tabler	Silty Clay loam	surface	5.10	1.86	0.09	320	15.05	18.14	14.02	1.93
<b>FE</b>	Silty clay	subsoil	6.40	1.83	0.08	272	27.43	27.05	20.05	6.67
Pond Creek	Silt loam	surface	5.50	1.81	0.10	568	10.56	18.94	12,21	0.47
#P	Silty clay loam	subsoil	6.20	1.81	0.07	364	15.30	20.39	16.99	0.72

:

watered. When germination was complete, the plant population of each pot was thinned to 10 plants. Adequate soil moisture was maintained throughout the growing period. After two months growth, the forage was harvested by cutting off the plants about 3/4 of an inch above the surface of the soil. The green samples were oven dried at  $60^{\circ}$ C. for 48 hours and then the weights of the oven-dry samples were recorded.

## Soil Phosphorus Extraction Procedures

The available phosphorus determinations were made by the following procedures: 0.1N acetic acid method (19); 0.025N HCl + 0.03N NH<sub>1</sub>F (Bray and Kurtz No. 1) with two soil-solution ratios, 1:7 and 1:50 (5); 0.1N HCl + 0.03N NH<sub>1</sub>F, (Bray and Kurtz No. 2) (5); 0.02N H<sub>2</sub>SO<sub>4</sub>, county agent's method (20); 0.2N H<sub>2</sub>SO<sub>4</sub> method (18); 0.5M NaHCO<sub>3</sub> method (27); and distilled H<sub>2</sub>O method (21).

The soil solution ratios employed by these methods were as follows: O.1N acetic acid, 1:40; Bray No. 1, 1:7 and 1:50; Bray No. 2, 1:7; O.02N  $H_2SO_{14}$ , 1:12; O.2N  $H_2SO_{14}$ , 1:12; and O.5M NaHCO<sub>3</sub>, 1:20. The O.1N acetic acid method was a leaching procedure, while the others were tests in which the extractants were shaken with the soil. In the O.5M NaHCO<sub>3</sub> method, the samples were shaken for 30 minutes on a mechanical shaker. A brief period of hand shaking was used in Bray No.1, Bray No. 2, 0.2N  $H_2SO_{14}$ , 0.02N  $H_2SO_{14}$ , and distilled  $H_2O$  procedures.

In all cases, an ammonium molybdate-HCL solution was the complexing agent. The concentration of the acid was approximately 8 N except in the Bray No. 1 and 0.5M NaHCO<sub>3</sub> methods in which the concentration of the acid used was somewhat lower. Stannous chloride was the reducing agent for all methods except Bray No. 2 and 0.2N H<sub>2</sub>SO<sub>1</sub>. These

procedures employed an organic reducing solution (5 grams pmethyl-aminophenol-sulfate plus 15 grams of NaHSO<sub>2</sub>).

All the samples were read on a Bausch and Lomb electrophotometer with a red filter. The readings were compared with a previously prepared standard curve and the ppm. of phosphorus in the soil were calculated.

## Statistical Analyses

The analysis of variance of the greenhouse yields was made according to the procedure described by Snedecor (33). The multiple range test proposed by Duncan (13) was utilized in testing the significance among treatment means.

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In the correlation and linear regression studies, the procedures discussed by Snedecor (33) were used. The amount of phosphorus extracted by a given extracting solution was the independent X and the percentage yield was the dependent Y. The percentage yield was cited by Fried and Dean (15) and by the soil test work group of The National Soil Research Committee (36).

Percentage yield= <u>yield without phosphate fertilizer</u> X 100 yield with adequate phosphate fertilizer

In the present investigation, the yield with the adequate phosphate fertilizer was assumed to be the 40-80-40 treatment for both the field and the greenhouse.

#### IV RESULTS AND DISCUSSION

#### Phosphorus Extraction Investigations

The amount of phosphorus extracted from the surface soil and subsoil by the different extracting solutions varied widely. These results are reported in Table II. In most cases the amount of phosphorus found in the surface soil was higher than that in the subsoil.

The relatively high values obtained with 0.2 N  $H_2SO_{l_1}$  method as compared with 0.02 N  $H_2SO_{l_1}$  suggest that the higher acid concentration of the extractant may have dissolved phosphorus compounds not readily available to plants. Miller and Axley (24) extracted available phosphorus from soils with several concentrations of  $H_2SO_{l_1}$  in the presence of 0.03 N NH<sub>4</sub>F. They found that 0.05 N  $H_2SO_{l_1}$  dissolved more phosphorus from soils treated with rock phosphate than 0.03 N  $H_2SO_{l_1}$ . However, the latter concentration gave a better relationship between the quantity of phosphorus extracted and the yields of corn, wheat and red clover.

In general, the 0.1N acetic acid procedure extracted the second largest amounts of phosphorus among all the methods used. It is possible that this acid dissolved certain quantities of occluded phosphorus in some soils. Harper (19) pointed out that this extractant may dissolve phosphorus which is useless to plants from soils which do not show secondary precipitation when extracted or from calcareous soils.

In comparing the amounts of phosphorus extracted by the Bray No. 2 (0.1N HCL + 0.03N NH<sub> $\mu$ </sub>F) and by the Bray No. 1 (0.025N HCL + 0.03N NH<sub> $\mu$ </sub>F)

	TABIE	II
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PHOSPHORUS EXTRACTED FROM SURFACE AND SUBSOILS BY DIFFERENT EXTRACTING SOLUTIONS (expressed in ppm.)

		0.2 <b>n</b> H <sub>2</sub> SO <sub>l4</sub>	0.1N Acetic Acid	0.02N H <sub>2</sub> SO <sub>4</sub>	Bray No.2	Bray No.l (1:50)	Bray No.l (1:7)	0.5M NaHCO3	н <sub>2</sub> 0
Carey :	surface	585.0	95.0	31.8	40.3	18.1	4.5	20.0	2.9
11 (	subsoil	840.0	202.5	12.3	4.2	6.6	2.5	12.5	1.6
Hollis	ter surface	352.5	82.5	18.0	20.4	8.1	11.9	42.5	5.3
11	subsoil	236.3	32.5	1.2	4.5	6.3	2.2	5.0	2.9
Minco :	surface	453.8	97.5	40.5	43.1	21.6	11.7	55.0	5.5
u i	subsoil	352.5	83.8	23.7	21.9	6.3	3.2	10.0	2.5
Norge a	surface	23.3	3.8	2.3	4.1	10.3	3.9	10.0	2.5
11	subsoil	13.9	2.0	1.3	3.8	5.0	1.9	4.0	3.3
Tabler	surface	32.3	8.4	5.1	8.9	17.2	8.9	25.0	2.9
Ħ	subsoil	135.0	3.6	2.6	9.5	7.5	2.5	5.0	3.2
Pond Ci	reek surface	217.0	60.0	20.3	22.8	18.4	10.5	40.0	3.3
tt.	" subsoil	93.8	8.0	3.2	7.9	9.1	4.3	15.0	1.9

methods, the concentrations of the acid may account for the values detected. Bray and Kurtz (5) developed the 0.025N HCL + .03N  $\rm NH_{1/2}F$  procedure for "adsorbed phosphorus" and the 0.1N HCL + .03N  $\rm NH_{1/2}F$  method for "acid-soluble" and "adsorbed phosphorus" forms. These investigators also indicated that the results are strongly influenced by the more readily soluble portion of each form. Smith et al. (32) found in both acid and calcareous soils that 0.025N HCL + 0.03N  $\rm NH_{1/2}F$  method with the soil-solution ratio 1:50 extracted more phosphorus and gave better correlation values than the ratio 1:7. In the present work the ratio of 1:50 also extracted more than twice as much phosphorus as the 1:7 ratio, but the correlation values were better for the 1:7 soil-solution ratio.

In the case of 0.5 M NaHCO<sub>3</sub>, the amounts of phosphorus extracted by this method showed a very close relationship with the field and greenhouse responses. Olsen et al. (27) proposed that the 0.5M NaHCO<sub>3</sub> method may be adaptable to a wide variety of soil conditions. However, the requisition of a certain previous skill for its manipulation may restrict this method as a widely used routine test procedure.

The water extraction method removed the smallest amounts of phosphorus of all the methods tested. The relationship between the amounts extracted and the greenhouse responses was very acceptable, but the relationship with the field responses was considerably poorer. It is possible that the low amounts of phosphorus extracted by this method will improve chances of contamination.

#### Greenhouse Experiment

In all the soils tested, there was an increase in forage yield with

the addition of phosphate fertilizer. The yield results are indicated in Table III.

#### TABLE III

	GREENHOUSE EXPERIMENT (expressed in grams per pot)					
		Fer	tilizer Trea	atments		
Soil Series	40-0-40	40-20-40	40-40-40	40-80-40	40-160-40	
Carey	1.32*	2.79	3.64	3.25	3.88	
Hollister	2.16	2.26	3.11	2.95	3.57	
Minco	3.27	3.66	3.79	4.41	4.68	
Tabler	1.31	2.76	3.75	3.69	3.48	
Pond Creek	1.70	2.33	2.89	2.91	2.81	
	Fertilizer Treatments					
	40-0-40	40-18-40	40-36-40	40-80-40	40-160-40	
Norge	1,02	2.87	2.84	3.24	2.94	

# FORAGE YTELDS OBTAINED IN THE OVEN\_DRY WHE AT

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Each value is an average of 3 replications.

The yields obtained from the check pots varied from 1.02 grams for the Norge soil to 3.27 grams for the Minco soil. The efficiency of the phosphate application, measured by the differences in yield between the check pots and the pots producing maximum yields, was in the following order: Carey 2.57 gm, Tabler 2.44 gm, Norge 2.22 gm, Hollister 1.41 gm, Minco 1.41 gm and Pond Creek with 1.21 gm.

On the Carey soil, there was a considerable increase in yield from 0 up to 40 pounds per acre of  $P_2O_5$ , a small decrease from 40 to 80 pounds per acre, and then an increase again from 80 to 160 pounds per acre. For the Hollister soil the increase in forage yield from the first increment of  $P_2O_5$  was very small, but from 20 to 40 pounds the increase was appreciable. A leveling off occurred at the 40 and 80 pounds rates and again there was an increase from 80 to 160 pounds. Small, but steady increases in yield with increasing phosphate applications were found on the Minco soil. The largest increase was observed when the rate was increased from 40 pounds to 80 pounds of  $P_{205}$  per acre. On the Norge soil, there was a relatively large increase in forage yield from 0 to 18 pounds of  $P_205$  treatment and a leveling off at the 18 and 36 pounds applications. The maximum yield was produced with 72 pounds of phosphate, while the 144 pound rate resulted in a small decrease. For the Tabler soil, there were considerable increases in yields from 0 up to  $\mu$ 0 pounds per acre of P<sub>2</sub>0<sub>5</sub>. At the 80 and 160 pound rates, slight decreases in yields were obtained. In the case of the Pond Creek soil, small increases in forage yields were noticed as the amount of  $P_{2}O_{5}$  per acre was increased up to 80 pounds. A slight decrease in yield occurred when 160 pounds of  $P_2O_5$  per acre were added.

For four soils; Carey, Norge, Tabler and Pond Creek, the forage yields obtained with the phosphate fertilizer treatments were significant at the 1% level. On two soils, Minco and Hollister, the calculated F values were very close to the 5% level. The analysis of variance and the multiple range test of significance at the 5% level are shown in Table IV.

# TABLE IV

# ANALYSIS OF VARIANCE AND MULTIPLE RANGE TEST OF WHEAT FORAGE YIELDS OBTAINED FROM A PHOSPHATE FERTILIZER STUDY IN THE GREENHOUSE

Carey Soil

Analysis of Variance						
	Source Treatment Blocks Error	df 1 2 8	<u>M.S.</u> 3.091 0.354 0.378	Calc. F 8.177 <sup>**</sup>		
	S	hortest si	gnificant ra	anges		
	p: Rp:	(2) 1.157	(3) 1.203	(4) 1.232	(5) 1.249	
Treat Means	40-0-40 1.316	40-20-40 _2.790	40-80-40 3.250	40-40-40 3.635	40-160-40 3.883	
Hollister S	oil					
		Analysis	of Variance	9		
	Source Treatments Blocks Error	<u>df</u> 2 8	M.S. 1.053 0.063 0.28l <sub>1</sub>	Calc. F 3.708		
Norge Soil						
		Analysis	of Variance	)		
	Source Treatments Blocks Error	df 4 2 8	M.S. 2.359 0.149 0.0798	Calc. F 29.487**		
	S	hortest sig	gnificant ra	inges		
	p: Rp:	(2) •532	(3) •553	(4) •566	(5) •574	
Treat. Means	40-0-40 1.023	40-36-40 2.839	40-18-40 2.891	40-144-40 3.942	40-72-40 3.242	

\*Any two means not underscored by the same line are significantly different the 5% probability level.

\*\*Indicates significance at 1% level of confidence.

# TABLE IV (CONTINUED)

# ANALYSIS OF VARIANCE AND MULTIPLE RANGE TEST OF WHEAT FORAGE YIELDS OBTAINED FROM A PHOSPHATE FERTILIZER STUDY IN THE GREENHOUSE

# Minco Soil

Analysis of Variance						
	Source Treatments Blocks Error	df 4 2 8	M.S. 0.985 0.128 0.304	<b>Calc.</b> F 3.240		
Tabler Soil						
		Analysis	of Variance	)		
	<u>Source</u> Treatments Blocks Error	df 4 2 8	M.S. 3.143 0.160 0.163	Calc. F 19.382**	*	
	S	hortest sig	gnificant ra	inges		
	p: Rp:	(2) •760	(3) •790	(4) .809	(5) .820	
Treat. Mean <sup>*</sup>	40-0-40 1.307	40-20-40 2.756	40-160-40 3.480	40-80-40 3.691	40-40-40 3.746	
Pond Creek	Soil	******				
· · · · · · · · · · · · · · · · · · ·		Analysis	of Variance			
	Source Treatments Blocks Error	df 4 2 8	M.S. 0.815 0.224 0.041	Calc. F 19.878**	¢	
	S	hortest sig	gnificant ra	nge s		
	p: Rp:	(2) .381	(3) •396	(4) .406	(5) .411	
Treat. Means	40=0-40 1.701	40-20-40 2.327	40-160-40 2.814	40-40-40 2.894	40-80-40 2.911	

\*Any two means not underscored by the same line are significantly different at the 5% probability level.

\*\*Indicates significance at 1% level of confidence.

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Data from field experiments conducted by the Agronomy Department of Oklahoma State University were employed in this investigation. The results of these experiments are shown in Table V.

#### TABLE V

# WHEAT YIELDS FROM SOIL FERTILITY PLOTS (expressed in bushels per acre)

· · ·	Fertilizer Treatments						
Soil Series	40-0-0	lio-20-0	40-140-140	40-80-0	n shift a shi s		
Carey	41.3*	42.6	44.2	45.5			
Hollister	51.5	49.5	53.1	54.3			
Minco	32.9	36.9	35.3	34.4			
Tabler	20.7	23.0	24.3	<b>25.</b> 3			
Pond Creek	34.9	35.7	35.8	34.5			
		Fer	tilizer Trea	atments			
	40-0-40	40-18-40	40-36-40	40-72-40	40-14 <b>4</b> -40		
Norge	15.8	22.4	23.1	22.0	22.7		

Each figure is the average of three replications.

In general, most of the field plots showed a similar pattern of phosphate fertilizer responses as those observed in the greenhouse experiments. However, the Pond Creek soil did not respond to phosphate treatments in the field studies.

# Correlation and Linear Regression Studies

The percentage yields for the greenhouse and field were obtained

by dividing the yield of the check treatment by the yield of 80pounds per acre of  $P_2O_5$  treatment, in the case of 5 experiments and by the 72 pounds per acre treatment for the Norge experiment. These percentage yields are represented in Table VI.

#### TABLE VI

Soil Series	Greenhouse	Field
Carey	$\frac{1.32 \times 100}{3.25} = 40.49\%$	$\frac{41.3 \times 100}{45.5} = 90.77\%$
Hollister	$\frac{2.16}{2.95} \times 100 = 73.45\%$	$\frac{51.5 \times 100}{54.3} = 94.84\%$
Minco	$\frac{3.27}{4.41} \times 100 = 74.23\%$	<u>32.9</u> x 100 = 95.64% 34.4
Norge	$\frac{1.02}{3.24} \times 100 = 31.55\%$	$\frac{15.8}{22.0} \times 100 = 71.82\%$
Tabler	$\frac{1.31}{3.69} \times 100 = 35.41\%$	$\frac{20.7 \times 100}{25.3} = 81.82\%$
Pond Creek	1.70 x 100 = 58.43% 2.91	<u>34.9</u> x 100 = 101.16% 34.5

GREENHOUSE AND FIELD PERCENTAGE YIELDS

It is apparent that the percentage yields in the field were proportionally greater than the percentage yields in the greenhouse with the exception of the Pond Creek soil.

For seven of the extracting solutions employed, the correlation values between the amount of phosphorus extracted and the percentage yields were above 0.5 and in only one case below this figure. Table VII shows the correlation values for the greenhouse experiment and the field studies. These analyses are reported in more detail in Appendix Tables I and II for the greenhouse and field respectively.

Technooting	Correlation Value		
Solution	Greenhouse	Field	
0.2N H2504	0.4997	0.6174	
O.lN Acetic Acid	0.7245	0.7865	
0.02N H2SO4	0.6284	0.7053	
Bray No. 2	0.5337	0.6697	
Bray No. 1 (1:50)	0.0864	0.4081	
Bray No. l (l:7)	0.8560	0.7054	
0.5M NaHCO3	0.9411	0.8060	
н <sub>2</sub> 0	0.9456	0.5900	

TABLE VII

CORRELATION COEFFICIENTS FOR GREENHOUSE AND FIELD

In the greenhouse when the r values were below 0.75, the respective r value for the field tended to be higher. This was observed for  $0.2N H_2SO_4$ , 0.1N Acetic acid,  $0.02N H_2SO_4$ , Bray No. 2 (0.1N HCL + 0.03N NH\_4F) and Bray No. 1 (0.025N HCL + 0.03N NH\_4F) with a 1:50 ratio methods. The reverse trend was noticed in Bray No. 1 (0.025N HCL + 0.03N NH\_4F) at a 1:7 ratio, 0.5M NaHCO<sub>3</sub> and distilled H<sub>2</sub>O extraction procedures when the r values were greater than 0.75.

The  $H_2O$ , Bray No. 1 (1:7), and 0.5M NaHCO<sub>3</sub> extracting solutions gave the highest correlation coefficients for the greenhouse. Olsen et al. (27) reported similar results in comparing different extracting solutions with the 0.5M NaHCO<sub>3</sub>.

The 0.5M NaHCO<sub>3</sub>, 0.1N Acetic acid and Bray No. 1 (1:7) methods had the best correlations in the field. The r value for 0.1N Acetic acid

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in the field was about 0.06 higher than in the greenhouse; for the 0.5M NaHCO<sub>3</sub> the r value was about 0.13 lower in the field compared to the greenhouse; and for Bray No. 1 (1:7) the r value was about 0.15 lower in the field compared to the greenhouse. The lowest correlation values observed were for Bray No. 1 (1:50) method in both the greenhouse and field.

Considering both the field and greenhouse, the highest correlation coefficients were found for the distilled  $H_2O$ , 0.5M NaHCO<sub>3</sub> and Bray (1:7) procedures. The comparison of soil test results with plant responses in the greenhouse should give a better measure of the ability of the soil test method to estimate the phosphorus supplying power of the soil. The comparison with the field results should be even more valuable in predicting crop responses because the field data include effects of climate availability of subsoil phosphorus and other factors.

The linear regression analysis for each method was performed to visualize the way the data fit with the sample regression lines. In each figure are included the greenhouse and field regression lines for a particular method. (Figures 1-8)



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#### V SUMMARY AND CONCLUSIONS

In this investigation a comparison of several phosphorus extracting solutions was made in an attempt to obtain a reliable method of available phosphorus extraction for Oklahoma soils. Correlation values for these methods under greenhouse and field conditions were determined.

From the results of these experiments, it may be concluded:

- 1. The more acid extracting solutions removed the greatest amounts of phosphorus from the soil.
- 2. Among the methods tested,  $0.2N H_2 S_4$  gave the highest value for extracted phosphorus and the distilled  $H_2 0$  the lowest.
- 3. Yield responses to phosphate fertilizer applications showed a similar pattern in both the greenhouse and field with the exception of the Pond Creek soil which responded significantly in the greenhouse, but not in the field.
- 4. The efficiency of the phosphate treatments in the greenhouse based on the size of the response was in the following order: Carey, Tabler, Norge, Hollister, Minco and Pond Creek.
- 5. In the correlation studies, all the r values were above 0.5 in both the greenhouse and field with the exception of the Bray No. 1 (0.025N HCL + 0.03N  $NH_4F$ ) at a ratio of 1:50 procedure.

6. It was apparent that when the r value for the greenhouse was greater than 0.75 the respective value for the field was lower. The reverse trend was observed when the r value was less than 0.75.

7. The best correlation coefficients for the greenhouse were those obtained for the 0.5M NaHCO<sub>3</sub>, Bray No. 1 (0.025N HCl + 0.03N NH<sub>4</sub>F) with a ratio of 1:7 and distilled water procedures, while in the field the best values were for 0.5M NaHCO<sub>3</sub>, Bray No. 1 (0.025N HCL + 0.03N NH<sub>4</sub>F) at a ratio of 1:7 and 0.1N acetic acid methods.

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APPENDIX

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Extractant Used	∕zx <sup>2</sup>	≥y <sup>2</sup>	Exy/(((x <sup>2</sup> ) ((y <sup>2</sup> )	Value
0.2N H2 <sup>SO4</sup>	259,695.93	1821.12	10866.88 \(259,695.93) (1821.12)	0.4997
O.lN Acetic Acid	8931.19	1821.12	<u>2922.06</u> \(8931.19) (1821.12)	0.7245
0.02N H2SO4	1099.89	1821.12	$\frac{889.33}{\sqrt{(1099.89)(1821.12)}}$	0.62.84
Bray No. 2	1261.21	1821.12	<u>808.89</u> \(1261.21) (1821.12)	0.5337
Bray No. 1 (1:50)	136.40	1821.12	<u>43.08</u> \(136.40) (1821.12)	0.0864
Bray No. 1 (1:7)	63.24	1821.12	$\frac{290.49}{(63.24) (1821.12)}$	0.85.60
0.5M NaHCO <sub>3</sub>	1380.12	1821.12	$\frac{11491.97}{(1380.12) (1821.12)}$	0.9411
H <sub>2</sub> 0	8.92	1821.12	$\frac{120.53}{(8.92) (1821.12)}$	0.9456

CALCULATIONS FOR GREENHOUSE CORRELATION COEFFICIENTS

# APPENDIX TABLE II

Extractant Used	≤x <sup>2</sup>	≥y <sup>2</sup>	$\leq xy/(zx^2)$ ( $zy^2$ )	Value
0.2N H2SO14	259,695.93	575.20	7546.23 \(259,695.93) (575.20)	0.6174
O.1N Acetic Acid	8931.19	575.20	$\frac{1782.73}{\sqrt{(8931.19)(575.20)}}$	0.7865
0.02N H2SO4	1099.89	575.20	$\frac{561.01}{(1099.89)(575.20)}$	0.7053
Bray No. 2	1261.21	575.20	<u>570.45</u> \(1261.21) (575.20)	0.6697
Bray No. 1 (1:50)	136.40	575.20	11)4.30 V(163.40) (575.20)	0.4081
Bray No. 1 (1:7)	63.24	575.20	$\frac{134.53}{\sqrt{(63.24)(575.20)}}$	0.7054
0.5M NaHCO 3	1 <b>3</b> 80 <b>.</b> 12	575 <b>.2</b> 0	<u>718.13</u> (1380.12) (575.20)	0.8060
H_0 2	8.92	575.20	<u>)12.26</u> (8.92) (575.20)	0.5900

# CALCULATIONS FOR FIELD CORRELATION COEFFICIENTS

#### VITA

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