

University of Nebraska - Lincoln

DigitalCommons@University of Nebraska - Lincoln

Publications from USDA-ARS / UNL Faculty

U.S. Department of Agriculture: Agricultural
Research Service, Lincoln, Nebraska

2001

INTERLABORATORY COMPARISON OF SOIL PHOSPHORUS EXTRACTED BY VARIOUS SOIL TEST METHODS

Peter J.A. Kleinman
USDA-ARS, pjk9@psu.edu

A. N. Sharpley
USDA-ARS

K. Gartley
University of Delaware

W. M. Jarrell
Oregon Graduate Institute

S. Kuo
Washington State University

See next page for additional authors

Follow this and additional works at: <https://digitalcommons.unl.edu/usdaarsfacpub>

 Part of the [Agricultural Science Commons](#)

Kleinman, Peter J.A.; Sharpley, A. N.; Gartley, K.; Jarrell, W. M.; Kuo, S.; Menon, R. G.; Myers, R.; Reddy, K. R.; and Skogley, E. O., "INTERLABORATORY COMPARISON OF SOIL PHOSPHORUS EXTRACTED BY VARIOUS SOIL TEST METHODS" (2001). *Publications from USDA-ARS / UNL Faculty*. 544.
<https://digitalcommons.unl.edu/usdaarsfacpub/544>

This Article is brought to you for free and open access by the U.S. Department of Agriculture: Agricultural Research Service, Lincoln, Nebraska at DigitalCommons@University of Nebraska - Lincoln. It has been accepted for inclusion in Publications from USDA-ARS / UNL Faculty by an authorized administrator of DigitalCommons@University of Nebraska - Lincoln.

Authors

Peter J.A. Kleinman, A. N. Sharpley, K. Gartley, W. M. Jarrell, S. Kuo, R. G. Menon, R. Myers, K. R. Reddy, and E. O. Skogley

INTERLABORATORY COMPARISON OF SOIL PHOSPHORUS EXTRACTED BY VARIOUS SOIL TEST METHODS

**Peter J. A. Kleinman,^{1,*} A. N. Sharpley,¹ K. Gartley,²
W. M. Jarrell,³ S. Kuo,⁴ R. G. Menon,⁵ R. Myers,⁶
K. R. Reddy,⁷ and E. O. Skogley⁸**

¹USDA-ARS, Pasture Systems and Watershed Management
Research Unit, University Park, PA 16802-3702

²Department of Plant Sciences, University of Delaware,
Newark, DE 19717

³Department of Environmental Science and Engineering,
Oregon Graduate Institute, 19600 N.W. von Neumann Dr.,
Beaverton, OR 97006-1999

⁴Dept. Crop and Soil Sciences, Washington State
University, Puyallup, WA 98371-4998

⁵Agric-Economic Division, IFDC, P.O. Box 2040, Muscle
Shoals, AL 35662

⁶Agronomy Department, 2004 Throckmorton Soil and Plant
Sciences Center, Kansas State University, Manhattan, KS
66506-5501

⁷Department of Soil and Water Sciences, 106 Newell Hall,
University of Florida, Gainesville, FL 32611-0510

⁸UNIBEST, Inc., 3535 Stucky Road, Bozeman, MT 59718

*Corresponding author. E-mail: pjk9@psu.edu

ABSTRACT

The widespread use of soil phosphorus (P) data, either in the context of agronomic or environmental management, requires an explicit understanding of potential errors related to soil P testing. This study compares a variety of soil P extraction methods, each performed by 9 separate laboratories on 24 soils from across the United States. Soil clay content ranged from 0 to 47%, pH from 4.2 to 8.6, and Mehlich-3 P concentration from 2 to 205 mg kg⁻¹. Average interlaboratory coefficients of variation (CVs) ranged from 0.11 to 0.22 for solution extracts (Bray-1 P, Fe-strip P, Mehlich-3 P, and Olsen P) and from 0.11 to 0.70 for saturated paste extracts (resin capsules and resin membranes, incubated for 2, 4, and 7 days). For soil tests based upon solution extracts, Olsen P exhibited the greatest variability among laboratories (CV = 0.22), despite its reputed suitability for a wider range of soils than Bray-1 and Mehlich-3. Soil test data were highly correlated, with the lowest correlations occurring between Olsen and Bray-1 P or Olsen and Mehlich-3 P ($r = 0.77$ and 0.84 , respectively) and the highest correlations occurring between Olsen P and Fe-strip P or Mehlich-3 and Bray-1 P ($r = 0.94$ for both correlations). Results indicate that some common soil test P protocols, when carefully conducted, yield data that may be reliably compared, such as in the compilation of regional and national soil databases.

INTRODUCTION

Concern over the management of phosphorus (P) in agricultural soils has focused attention on soil P analyses and their interpretations. Increasingly, studies of P transport potential conducted at watershed, regional, and national scales have employed soil test P data from several laboratories and have had to compare data from different laboratory extraction methods (1–3). These studies point to the need for better understanding of sources of error in soil P analyses, either as a result of interlaboratory variance, soil specific variability, extract constraints, or a combination of these variables.

Interlaboratory variability in analytical results is routinely monitored by state and regional soil testing consortia as part of quality control efforts (4). Published studies have often reported little interlaboratory error for a variety of soil P analyses. For instance, Wolf and Baker (5) reported strong interlaboratory correlation for Olsen, Bray-1, and Mehlich-1 soil tests conducted on 27

non-calcareous soils. Similarly, Sharpley et al. (6) found a close agreement between iron-oxide strip (Fe-strip) P measured by three different laboratories on a variety of soils (pH 5.5 to 8.0). Elsewhere, Nair et al. (7) examined variability related to P adsorption procedures, reporting strong agreement in isotherm parameters as estimated by four laboratories for a wide range of soils (pH 5.6–7.9).

The suitability of specific soil P tests for soils with various pedogenic properties is well documented. For instance, Bray-1, Mehlich-1, and, to a lesser extent, Mehlich-3, are not considered suitable for calcareous soils because soluble P may be precipitated by CaF_2 , a product of the reaction between NH_4F and CaCO_3 (8). Generally, acid extracts provide inconsistent measures of soil P in calcareous soils (9). Some extraction methods, however, such as Olsen, are considered suitable over a wide range of soils, from acidic to calcareous (10).

Given the limitations of certain extraction methods with different soils, relating data from different soil tests can be problematic, since the relationship between soil tests can be soil specific (9). For instance, Michaelson et al. (11) described variable regression equations relating Bray-1 P and Mehlich-3 P for Alaskan soils derived from two distinct parent materials, with the slope of the regression equations ranging from 1.01 to 1.88 and intercepts ranging from -2.9 to 4.6 mg kg^{-1} (r^2 ranged from 0.85 to 0.96). As a result, comparisons of soil P data across large geographic areas often rely upon interpretations (e.g., relative agronomic P status) to reduce variability and normalize results [e.g., Fixen (1)].

An alternative approach to traditional chemical extraction of P is the use of ion sinks, such as Fe-strips, anion exchange resins, and ion-exchange membranes. Ion sinks have given promising results when applied to soils with diverse chemical and physical properties (12,13). Ion sinks adsorb P onto the sink surface and interact minimally with the soil, whereas chemical extraction results in soil reaction with the extractant.

Another application of ion sink extraction of soil P involves the use of saturated soil pastes rather than high solution:soil ratios. When used in saturated soil pastes, ion sinks are believed to closely mimic rhizosphere conditions and are often better correlated with crop response than traditional solution extracts (14). As the sinks operate with limited chemical extraction, they may be more suited to a wide range of soils, irrespective of management history, than are most solution extractions (13,15–17).

The most common saturated soil paste method for assessing available soil P uses anion-exchange resins to extract soil P. Skogley et al. (18) encapsulated a mixture of anion- and cation-exchange resins in a mesh sphere, or a resin capsule, for use as a universal nutrient testing methodology. Greenhouse studies indicated that the correlation between P uptake by sorghum–sudan grass and resin capsule P were as good or better than those with the Olsen P soil test (19). A similar approach using ion-exchange resin impregnated membranes (resin membranes)

has been investigated by several researchers (15,20). In pot studies, the resin membranes have provided a better index of P availability than conventional solution extraction methods for canola and ryegrass (14). Iron oxide strips have also been successfully used to estimate available inorganic P in a wide range of soils and management systems (12,13,21).

This study examines sources of error for a variety of soil P extraction methods through an interlaboratory comparison of four solution extracts and two saturated soil paste extracts by nine laboratories on 24 different soils. Both the magnitude and potential sources of error are investigated. In addition, associations between soil P tests and soil properties and forms of P are assessed.

MATERIALS AND METHODS

Laboratory Collaboration and Soil Collection

Nine collaborating laboratories were involved in soil selection and sample analysis. These laboratories were IFDC, Muscle Shoals, AL; Kansas State University, Manhattan, KS; Oregon Graduate Institute, Beaverton, OR; University of Delaware, Newark, DE; University of Florida, Gainesville, FL; University of Montana, Bozeman, MT; USDA-ARS, Pasture Systems and Watershed Management Research Unit, University Park, PA; National Agricultural Water Quality Laboratory, Durant, OK; and Washington State University, Puyallup, WA.

Two to three agriculturally and geographically important soils were selected by each of the nine collaborating laboratories (see Table 1 for series and classification). Five kg samples of the surface (A) horizon were collected, air-dried, and sieved (2 mm) and sent to a central laboratory (USDA-ARS, Durant, OK). The soils were thoroughly mixed, sub-sampled, and 250 g of all soils sent to the nine collaborating laboratories. Samples were also sent to the USDA-NRCS, National Soil Survey Laboratory, Lincoln, NE, for standard analysis (Table 1).

Soil Phosphorus Analysis

Each laboratory conducted soil P analyses on the 24 soils by Bray-1, Mehlich-3, Olsen, Fe-strip, resin capsule, and resin membrane methods. In addition, water, CaCl_2 , and a modified Hedley sequential extraction was conducted on all samples by the Pasture Systems and Watershed Management Research Unit, University Park, PA. The protocols used by all laboratories are listed in Table 2 for each method. In all cases, P was determined in filtered

(0.45 μm) and neutralized soil extracts, by the colorimetric method of Murphy and Riley (28).

Iron oxide strips were prepared according to Sharpley (25), resin capsules by Skogley et al. (18), and resin membranes by Abrams and Jarrell (20) (Table 2). Briefly, Fe-strips were prepared by immersing filter-paper circles (15-cm diam., Whatman No. 50) in a solution containing 10 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 100 mL distilled water (10% solution). The paper circles were air-dried and immersed in 2.7 *M* NH_4OH solution for 1 minute to convert FeCl_3 to Fe oxide. The paper circles were again air dried, cut into 10 \times 2 cm strips, and stored for subsequent use. Phosphorus was extracted from soil by shaking a 1 g soil sample and one paper strip in 40 mL of 0.01 *M* CaCl_2 end-over-end for 16 h at 25°C. The strip was removed from the solution, rinsed free of adhering soil particles, and air-dried. Phosphorus retained on the strip was extracted from the strip by shaking it end-over-end with 40 mL of 0.1 *M* H_2SO_4 for 1 h.

Resin capsules were prepared by Dr. Earl Skogley. Sixty g of soil were placed in a container and made into a saturated paste. Equal portions of the paste were transferred into three 50 mL plastic beakers. Each beaker contained 20 g of soil to ensure that the resin capsule was completely surrounded with paste when inserted. A resin capsule was placed in the center of the paste, making sure the soil thoroughly contacted it on all sides. The beakers were capped and maintained at 20°C. Resin capsules were removed from the beakers after 2, 4, and 7 d. Upon removal, resin capsules were thoroughly washed with deionized-distilled water to remove all soil particles. They were then placed in a small plastic bag or container and stored in a refrigerator for subsequent P removal. The resin accumulators were eluted slowly with 50 mL of 2 *M* HCl, at a rate of about 1 mL per min, using a leaching apparatus described by Skogley et al. (18). Phosphorus concentrations in the neutralized leachate were then determined.

Anion exchange resin sheets were cut into 2 \times 2 cm squares and stored in propylene glycol. Prior to use, the resin squares were rinsed in distilled water to remove all the propylene glycol, immersed in 0.1 *M* HCl for 16 h, and finally rinsed with distilled water. Sixty g of soil were placed in a container and made into a saturated paste. Twenty g of soil were then placed in three plastic beakers, a resin-square inserted in the center, the beaker capped, and maintained at 20°C. Resin membranes were removed after 2, 4, and 7 d. The membranes were thoroughly washed with deionized-distilled water until all soil particles were removed. To remove P, the membrane was shaken end-over-end with 40 mL 1 *M* HCl for 4 h, removed, and rinsed with deionized-distilled water. The HCl extraction of membranes was repeated with the P concentration of the first and second extractions measured separately and values added to give the final amount.

A sequential fractionation of soil P was conducted using a modified approach proposed by Hedley et al. (27). Specific modifications included a

Table 1. Properties of 24 Soils Included in Interlaboratory Comparison Study

Series	State	Classification	Particle Size Composition			C _{org} (mg kg ⁻¹)	pH (H ₂ O, 1:1)	CEC (cmol kg ⁻¹)	Bray-1 P (mg kg ⁻¹)
			Clay (%)	Silt (%)	Sand (%)				
Rutlege	DE	Typic Umbraquult	0	6	94	115	5.2	27	104
Matapeake	DE	Typic Hapludult	12	59	29	10	6.2	8	114
Evesboro	DE	Typic Quartzipsamment	2	9	89	10	6.6	4	82
Thurlow	MT	Ustollic Haplargid	29	28	43	24	8.0	20	9
Amsterdam	MT	Typic Haplustoll	20	49	32	4	8.6	14	0
Huntley	MT	Lithic Hapludoll	39	41	20	12	7.9	27	37
Tully	KS	Pachic Argiustoll	30	60	11	28	7.0	24	31
Cove	WA	Vertic Epiaquoll	38	41	22	36	6.2	37	52
Tolke	WA	Alic Hapludand	14	70	17	44	6.0	28	69
Laurelwood	WA	Ultic Haploxeralf	18	67	15	17	5.7	14	91
Crosby	OH	Aeric Epiaqualf	26	51	23	9	7.7	15	64
Paulding	OH	Typic Epiaquept	47	45	8	34	7.5	27	104

Hartsells	TN	Typic Hapludult	19	50	30	22	4.7	11	3
Crete	KS	Pachic Argiustoll	27	65	8	14	5.9	20	43
Eudora	KS	Fluventic Hapludoll	12	63	26	8	6.0	12	104
Captina	AR	Typic Fragiudult	12	67	21	9	4.6	7	86
Sultan	WA	Aquandic Dystroxerept	19	75	7	19	5.7	17	82
Pahokee	FL	Lithic Medisaprist	7	37	56	446	5.2	177	37
Myakka	FL	Aeric Alaquod	0	5	95	24	4.5	6	10
Wauchula	FL	Ultic Haplaquod	0	4	95	7	4.2	2	3
Gallion	OK	Typic Hapludalf	3	31	65	12	5.5	5	114
Cahaba	OK	Typic Hapludult	8	32	60	15	6.5	7	13
Kirkland	OK	Udertic Paleustoll	20	62	18	23	6.1	17	10
Houston Black	TX	Udic Haplustert	46	40	13	8	8.0	39	2
		Mean	19	44	37	40	6.2	24	53
		Maximum	47	75	95	446	8.6	177	114
		Minimum	0	4	7	4	4.2	2	0

Data obtained from NRCS, National Soil Characterization Laboratory.

Table 2. Soil Phosphorus Extractions Conducted on 24 Soils for Interlaboratory Comparison

Extract	Soil:Solution	Extraction Time	Replications	Reference
Bray-1 (0.03 M NH ₄ F, 0.025 M HCl)	1:10	5 min	2	Bray and Kurtz, 1945
Mehlich-3 (0.2 M CH ₃ COOH, 0.25 M NH ₄ NO ₃ , 0.015 M NH ₄ F, 0.013 M HNO ₃ , 0.001 M EDTA)	1:10	5 min	2	Mehlich, 1984
Olsen (0.5 M NaHCO ₃ , pH 8.5)	1:20	30 min	2	Olsen et al., 1954
Fe-strip (10 × 2 cm strips, 0.01 M CaCl ₂)	1:40	16 hrs	2	Sharpley, 1993
Resin capsule	Saturated paste	2, 4, 7 days	3	Skogley et al., 1990
Resin membrane (2 × 2 cm)	Saturated paste	2, 4, 7 days	3	Abrams and Jarrel, 1992
Water soluble P	1:10	1 hr	2	Kuo, 1996
CaCl ₂ P (0.01 M CaCl ₂)	1:10	1 hr	2	Kuo, 1996
Sequential extraction (modified Hedley)	1:40	12 hrs	2	Hedley et al., 1982
Extract 1: Fe-oxide strip (2 × 10 cm)				
Extract 2: 0.5 M NaHCO ₃ (pH 8.5)				
Extract 3: 0.1 M NaOH				
Extract 4: 1.0 M HCl				
Extract 5: perchloric acid digestion				

soil:solution ratio of 1:40, substitution of Fe-strips for resin strips, and substitution of perchloric acid digestion for sulfuric acid/peroxide digestion.

Statistical Analysis

Descriptive statistics were used to assess interlaboratory error for each of the P extraction methods. Data were assessed for normality by the Anderson–Darling test. Non-normal data were logarithmically transformed. Associations between data were evaluated by Pearson's correlation analysis, and regression equations were estimated by least squares regression. Differences in extract data were evaluated by paired, Student's *t* test (29). Data were analyzed with Minitab's statistical software, Release 11 (30).

RESULTS AND DISCUSSION

Average soil P concentrations for tests conducted by the nine laboratories are presented in Table 3 for each of the 24 soils included in the study. The soils included a wide range of soil P levels (1 to 217 mg kg⁻¹ Bray-1 P; 2 to 140 mg kg⁻¹ Olsen P; 2 to 126 mg kg⁻¹ Fe-strip P; 2 to 205 mg kg⁻¹ Mehlich-3 P). The interlaboratory coefficient of variation (CV) for each of the extraction methods is presented, by soil, in Table 3. These values were determined from the sub-samples of each soil provided to the various laboratories.

Interlaboratory Variability—Solution Extracts

Of the four solution extractions, the largest average CV was associated with Olsen extraction (CV = 0.22), followed by Bray-1 (CV = 0.13), Fe-strip (CV = 0.12), and Mehlich-3 (CV = 0.10) (Table 3). Average interlaboratory CVs for these soil tests were higher than those reported in the literature [e.g., Wolf and Baker (5)]. However, our study included a much wider range of soil types than the previous studies of interlaboratory variability. When the five soils with the largest interlaboratory CVs were excluded from the analysis, the average CV improved considerably for Bray-1 (CV = 0.08), Fe-strip (CV = 0.08), Mehlich-3 (CV = 0.06), and Olsen (CV = 0.14). Soils with the largest CVs had the lowest extractable P concentrations and were either the most alkaline (Amsterdam and Houston Black), or most acidic (Hartsells, Myakka and Wauchula) soils in the study.

Olsen P, which should be best suited to the broad range of soils included in this study (10), exhibited the greatest interlaboratory error of any of the standard

Table 3. Interlaboratory Coefficient of Variation for Different Phosphorus Extraction Methods

Soil Series	Soil Test Extracts				Saturated Paste Extracts					
	Mehlich III P	Fe-Oxide Strip P	Bray 1 P	Olsen P	Resin Capsule P (4 day)	Resin Capsule P (7 day)	Resin Capsule P (2 day)	Resin Membrane P (4 day)	Resin Membrane P (7 day)	Resin Membrane P (2 day)
Rutlege	0.10	0.04	0.04	0.03	0.10	0.11	0.07	0.57	0.44	0.45
Matapeake	0.03	0.04	0.13	0.08	0.04	0.07	0.10	0.80	0.59	1.26
Evesboro	0.05	0.06	0.08	0.14	0.11	0.07	0.06	0.62	0.51	0.75
Thurlow	0.04	0.11	0.29	0.10	0.08	0.04	0.11	0.19	0.36	0.30
Amsterdam	0.22	0.23	0.61	0.60	0.29	0.11	0.19	0.65	0.66	1.38
Huntley	0.08	0.10	0.06	0.09	0.03	0.06	0.10	0.14	0.48	0.28
Tully	0.09	0.12	0.05	0.30	0.11	0.09	0.15	0.55	1.02	0.71
Cove	0.03	0.05	0.06	0.03	0.05	0.09	1.48	0.43	0.55	0.41
Tolke	0.06	0.09	0.04	0.23	0.14	0.18	0.13	0.33	0.64	1.04
Laurelwood	0.05	0.06	0.04	0.08	0.17	0.10	0.12	0.29	0.31	0.74
Crosby	0.05	0.09	0.03	0.08	0.06	0.08	0.10	0.37	0.42	0.35

Paulding	0.03	0.03	0.04	0.05	0.05	0.08	0.07	0.27	0.34	0.21
Hartsells	0.60	0.56	0.28	0.52	0.35	0.39	0.32	0.60	0.34	0.28
Crete	0.07	0.13	0.10	0.19	0.04	0.08	0.09	0.44	0.98	1.02
Eudora	0.04	0.04	0.04	0.05	0.06	0.05	0.06	0.38	0.78	0.26
Captina	0.05	0.07	0.04	0.11	0.08	0.08	0.16	0.51	1.18	1.52
Sultan	0.03	0.04	0.04	0.27	0.05	0.10	0.08	0.75	0.75	0.80
Pahokee	0.11	0.08	0.09	0.27	0.02	0.12	0.09	0.17	0.45	0.61
Myakka	0.19	0.13	0.17	0.34	0.06	0.07	0.07	0.18	0.19	0.34
Wauchula	0.21	0.15	0.40	0.51	0.14	0.12	0.22	0.38	0.38	0.34
Gallion	0.02	0.05	0.02	0.14	0.04	0.06	0.10	0.36	0.40	0.39
Cahaba	0.13	0.19	0.13	0.17	0.20	0.22	0.15	0.61	1.73	1.03
Kirkland	0.08	0.22	0.17	0.19	0.24	0.16	0.32	0.70	1.63	1.13
Houston Black	0.17	0.21	0.29	0.60	0.17	0.34	0.23	0.46	0.17	1.18
Mean CV	0.10	0.12	0.13	0.22	0.11	0.12	0.19	0.45	0.64	0.70

Table 4. Mean Phosphorus Values for Analyses Conducted on 24 Soils as Part of the Interlaboratory Comparison

Series	Fe-Strip P	Mehlich-3 P	Olsen	Bray-1	Resin	Resin	Resin	Resin	Resin	Resin Membrane P (7 day)
			P	P	Capsule P (2 day)	Capsule P (4 day)	Capsule P (7 day)	Membrane P (2 day)	Membrane P (4 day)	
(mg kg ⁻¹)										
Rutlege	92	157	89	164	225	311	445	0.60	0.85	0.89
Matapeake	97	193	82	187	229	280	298	0.15	0.15	0.14
Evesboro	43	92	18	88	196	146	173	0.31	0.27	0.21
Thurlow	51	99	43	13	129	160	181	0.85	0.84	0.96
Amsterdam	11	9	4	1	33	42	39	0.13	0.07	0.09
Huntley	29	46	20	36	243	269	361	0.65	0.69	0.83
Tully	20	29	19	29	62	95	124	0.22	0.21	0.43
Cove	126	110	140	86	229	263	427	0.48	0.91	1.83
Tolke	56	79	45	102	78	86	88	0.18	0.11	0.17
Laurelwood	57	97	53	123	53	64	83	0.18	0.15	0.13
Crosby	44	79	49	65	83	115	133	0.53	0.56	0.69

COMPARISON OF SOIL P

Paulding	101	205	114	150	266	387	513	1.73	2.05	2.35
Hartsells	2	5	4	4	8	6	7	0.05	0.06	0.07
Crete	28	44	24	45	110	197	282	0.25	0.19	0.48
Eudora	111	197	81	151	553	805	1304	1.32	1.81	2.89
Captina	53	103	47	90	80	132	172	0.18	0.10	0.53
Sultan	92	122	50	95	215	286	350	0.24	0.25	0.65
Pahokee	43	37	37	25	130	125	354	0.51	0.43	0.63
Myakka	16	12	13	12	233	299	344	6.19	7.77	7.91
Wauchula	4	3	3	3	51	58	71	2.05	2.51	3.06
Gallion	112	203	81	217	423	688	1382	3.21	6.81	10.89
Cahaba	8	13	7	12	16	18	36	0.15	0.11	0.29
Kirkland	9	9	7	10	30	35	67	0.17	0.11	0.31
Houston Black	2	2	2	1	7	12	11	0.12	0.05	0.06
Mean	50	81	43	71	153	203	302	1	1	2
Maximum	126	205	140	217	553	805	1382	6	8	11
Minimum	2	2	2	1	7	6	7	0	0	0

Table 5. Correlation of P in Various Soil Extracts and Corresponding Regression Equations

Regression Equation	r	P (ANOVA)
Solution extracts		
Fe-strip P = $7.25 + 0.53$ Mehlich-3 P	0.93	<0.001
7.67 + 0.99 Olsen P	0.94	<0.001
13.1 + 0.52 Bray-1 P	0.86	<0.001
Mehlich-3 P = $0.10 + 1.61$ Fe-strip P	0.93	<0.001
14.8 + 1.54 Olsen P	0.84	<0.001
10.8 + 0.99 Bray-1 P	0.94	<0.001
Olsen P = $-2.14 + 0.90$ Fe-strip P	0.94	<0.001
5.69 + 0.46 Mehlich-3 P	0.84	<0.001
11.4 + 0.44 Bray-1 P	0.77	<0.001
Bray-1 P = $-0.6 + 1.43$ Fe-strip P	0.86	<0.001
$-0.84 + 0.89$ Mehlich-3 P	0.94	<0.001
13.5 + 1.34 Olsen P	0.77	<0.001
Saturated paste extracts		
Two day resin capsule P = $19.5 + 0.66$ Four day resin capsule P	0.98	<0.001
46.1 + 0.36 Seven day resin capsule P	0.93	<0.001
117 + 42 Two day resin membrane P	0.42	0.037
115 + 34 Four day resin membrane P	0.51	0.011
108 + 29.9 Seven day resin membrane P	0.58	<0.001
Four day resin capsule P = $-19.3 + 1.45$ Two day resin capsule P	0.98	<0.001
37.3 + 0.55 Seven day resin capsule P	0.97	<0.001
148 + 65 Two day resin membrane P	0.44	0.030
142 + 55 Four day resin membrane P	0.55	0.006
129 + 49 Seven day resin membrane P	0.63	<0.001

Seven day resin capsule P = $-72.1 + 2.44$	Two day resin capsule P	0.93	<0.001
$-46.1 + 1.71$	Four day resin capsule P	0.97	<0.001
210 + 108	Two day resin membrane P	0.42	0.043
191 + 99	Four day resin membrane P	0.56	0.004
161 + 93	Seven day resin membrane P	0.68	<0.001
Two day resin membrane P = $0.19 + 0.004$	Two day resin capsule P	0.42	0.037
$0.24 + 0.003$	Four day resin capsule P	0.44	0.030
$0.37 + 0.002$	Seven day resin capsule P	0.42	0.043
$0.11 + 0.65$	Four day resin membrane P	0.96	<0.001
$0.16 + 0.45$	Seven day resin membrane P	0.87	<0.001
Four day resin membrane P = $-0.04 + 0.01$	Two day resin capsule P	0.51	0.011
$0.01 + 0.01$	Four day resin capsule P	0.55	0.006
$0.16 + .003$	Seven day resin capsule P	0.56	0.004
$-0.08 + 1.42$	Two day resin membrane P	0.96	<0.001
$-0.01 + 0.75$	Seven day resin membrane P	0.97	<0.001
Seven day resin membrane P = $-0.19 + 0.01$	Two day resin capsule P	0.58	0.003
$-0.16 + 0.01$	Four day resin capsule P	0.63	<0.001
$-0.004 + 0.01$	Seven day resin capsule P	0.68	<0.001
$0.11 + 1.66$	Two day resin membrane P	0.87	<0.001
$0.11 + 1.25$	Four day resin membrane P	0.97	<0.001

soil tests. Even when the five soils with highest CVs were excluded from the Olsen P data, the average interlaboratory CV was 0.14. Wolf and Baker (5) also found Olsen P to exhibit the highest CV relative to Bray-1, Mehlich-1, and Mehlich-3. Their study of non-calcareous soils found CVs of 0.11 and 0.10 for Olsen P estimates from two state laboratories, more than twice the average CV of the other tests. However, for all soil extractions, laboratory error did not appear to be systematic, as none of the nine laboratories included in the comparison consistently over- or under-estimated soil P concentrations.

Interlaboratory Variability—Saturated Soil Paste Extracts

The average interlaboratory CVs of saturated soil paste extracts were quite variable, depending both on sink type and incubation period. Variability between resin capsule replicates was significantly lower than that of corresponding resin membrane replicates for a given incubation period, as evidenced by the lower CVs ($P < 0.001$, $N = 24$). Average CVs for the capsules were 0.19 (2 day incubation), 0.11 (4 day incubation), and 0.12 (7 day incubation) (Table 3). Increasing the period of incubation from 2 to 4 days reduced average CVs for the resin capsules, although there was little difference in the average 4 and 7 day CVs. Average CVs for the resin membranes were 0.70 (2 day incubation), 0.45 (4 day incubation), and 0.64 (7 day incubation). The inconsistency in resin membrane P results may be due to their low surface area (8 cm^2), which limits the volume of soil desorbing P to the strips, and hence the small quantity of P extracted (i.e., low resolution).

Correlation of Soil Test Data

Table 4 presents mean values, and Table 5 presents correlation coefficients and regression equations for all soil tests included in the interlaboratory comparison. The solution extracts were all significantly correlated ($r = 0.77$ to 0.94 , $P < 0.05$). The weakest correlations between soil tests were found between Olsen P and Bray-1 P ($r = 0.77$), and Olsen P and Mehlich-3 P ($r = 0.84$). The strongest correlations between soil tests occurred between Mehlich-3 and Bray-1 P, as well as Olsen and Fe-strip P ($r = 0.94$). Comparison of linear regressions reveals that Mehlich-3 and Bray-1 P were similar (approximately 1:1), which compares well with the regression equation reported by Tran et al. (31). Average Olsen P and Fe-strip P values were also approximately 1:1 in average concentration. Mehlich-3 and Bray-1 P values were roughly 1.5 times greater than corresponding Olsen P and Fe-strip P values, in general accordance with

Table 6. Additional Phosphorus Extractions Conducted on 24 Soils Used in the Interlaboratory Comparison

Series	Water Soluble P ^a (mg kg ⁻¹)	CaCl ₂ P ^a (mg kg ⁻¹)	NaOH P (mg kg ⁻¹)	Sequential Extraction		
				Fe-strip P (mg kg ⁻¹)	NaHCO ₃ P ¹ (mg kg ⁻¹)	NaOH P ¹ (mg kg ⁻¹)
Rutlege	15.83	5.95	244	70	159	218
Matapeake	6.85	3.51	377	68	190	375
Evesboro	2.38	0.56	71	31	64	106
Thurlow	8.1	2.11	47	42	104	119
Amsterdam	0.52	0.23	0	4	9	10
Huntley	6.79	1.22	43	27	50	78
Tully	2.77	0.58	62	18	44	87
Cove	10.02	1.79	1543	19	373	1497
Tolke	1.65	0.21	968	28	167	833
Laurelwood	2.37	0.03	768	30	142	670
Crosby	4.95	0.89	206	33	105	257
Paulding	17.34	4.92	510	74	275	583
Hartsells	0.51	0.11	46	1	9	37
Crete	3.65	0.83	84	24	55	91
Eudora	21.6	8.86	166	87	149	197
Captina	5.53	1.96	213	37	104	212
Sultan	3.43	0.79	364	52	175	383
Pahoee	4.58	0.59	163	44	134	193
Myakka	8.1	2.65	16	15	22	28
Wauchula	3.13	1.87	20	3	9	15
Gallion	20.38	7.34	255	70	198	275
Cahaba	2.96	0.56	32	9	17	33
Kirkland	2.43	0.34	25	7	21	36
Houston Black	0.27	0.13	1	1	7	12

^a Inorganic P only.

Buondonno et al. (32), who found Mehlich-3 P to be approximately twice Olsen P values.

Saturated paste extract data revealed strong correlation within methods by incubation period ($r = 0.87$ to 0.98), but generally poor correlation between resin capsule and strip data ($r = 0.42$ to 0.68). Resin capsule P increased roughly 1.3 fold between 2- and 4-day incubations and again 1.5 fold between 4- and 7-day incubations. Similarly, resin membrane P increased an average of 1.3 fold between the 2- and 4-day incubations and 1.4 fold between the 4- and 7-day incubations.

Correlation of Soil Test Data with Other Phosphorus Extract Data

Table 6 presents additional data for the soils included in our study. Sequential extraction data and several other soil P extracts were correlated with the interlaboratory soil test data. Average Fe-strip P estimated by the nine laboratories and Fe-strip P estimated as the first step in a sequential extraction was well correlated ($r = 0.83$; Fe-strip $P_{\text{average}} = 7.3 + 1.3 \text{ Fe-strip } P_{\text{seq. extract}}$). Average Fe-strip P data were also compared with two measures of P Intensity, water-soluble P and CaCl_2 P, both of which were correlated with Fe-strip P, but were significantly smaller pools (water soluble P: $r = 0.75$, Fe-strip P = $19 + 4.8$ water soluble P; CaCl_2 P: $r = 0.68$, Fe-strip P = $28 + 11.2 \text{ CaCl}_2$ P).

Average Olsen P estimated by the nine laboratories and NaHCO_3 P measured by sequential extraction was strongly correlated ($r = 0.96$; Olsen-P = $1.14 + 0.39 \text{ NaHCO}_3 P_{\text{seq. extract}}$), although NaHCO_3 P from the sequential fractionation was roughly 2.5 times greater than the Olsen P, probably owing to the longer extraction time (12 hours as opposed to 30 minutes). Mehlich-3 and Bray-1 P were weakly correlated with sequential fractionation NaHCO_3 P ($r = 0.77$ for Mehlich-3 P; $r = 0.71$ for Bray-1 P) and poorly correlated with the sequential fractionation NaOH P ($r = 0.42$ for Mehlich-3 P; $r = 0.44$ for Bray-1 P).

CONCLUSIONS

This study highlights the potential importance of interlaboratory error in the assessment of soil P data. When a wide range of soils was assessed, interlaboratory CVs for all analyses exceeded 0.10. Olsen P, which should be the most reliable measure of available soil P for such a variety of soils, exhibited the largest interlaboratory CV (0.22). Even when five low P, strongly acid, and calcareous soils responsible for greatest interlaboratory CVs were excluded from the analyses, the Olsen P CV was 0.14. The CVs for the other extraction methods were below 0.10. Extraction of P from saturated soil pastes using resin

membranes could not be reliably replicated between laboratories, probably owing to the low resolution of this method. Use of resin capsules, however, resulted in CVs comparable to the standard soil test analyses, and increasing the incubation time for the capsules reduced average interlaboratory variability. When consistent soil extraction and analytical procedures are used, common soil test P methods can provide reproducible values.

ACKNOWLEDGMENTS

Gerald Elwinger, USDA-ARS, University Park, PA, provided valuable advice in conducting statistical analyses for the study.

REFERENCES

1. Fixen, P.E. Soil Test Levels in North America. *Better Crops* **1998**, *82*, 16–18.
2. Lander, C.H.; Moffitt, D. *Nutrients Available from Livestock Manure Relative to Crop Growth Requirements*; Re. Assess. Strat. Plan. Pap. 98-1 USDA-NRCS: Washington, DC, 1998.
3. Sims, J.T.; Edwards, A.C.; Schoumans, O.F.; Simard, R.R. Integrating Soil Phosphorus Testing into Environmentally Based Agricultural Management Practices. *J. Environ. Qual.* **2000**, *29*, 60–71.
4. Sims, J.T. Phosphorus Soil Testing: Innovations for Water Quality Protection. *Commun. Soil Sci. Plant Anal.* **1998**, *29*, 1471–1489.
5. Wolf, A.M.; Baker, D.E. Comparisons of Soil Test Phosphorus by Olsen, Bray P1, Mehlich-I, and Mehlich-3 Soil Test. *Commun. Soil Sci. Plant Anal.* **1985**, *16*, 467–484.
6. Sharpley, A.N.; Indiati, R.; Ciavatta, C.; Rossi, N.; Sequi, P. Interlaboratory Comparison of Iron Oxide-Impregnated Paper to Estimate Bioavailable Phosphorus. *J. Environ. Qual.* **1994**, *23*, 14–18.
7. Nair, P.S.; Logan, T.J.; Sharpley, A.N.; Sommers, L.E.; Tabatabai, M.A.; Yuan, T.L. Interlaboratory Comparison of a Standardized Phosphorus Adsorption Procedure. *J. Environ. Qual.* **1984**, *4*, 591–595.
8. Smillie, G.W.; Syers, J.K. Calcium Fluoride Formation During Extraction of Calcareous Soil with Fluoride II. Implications to Bray-1 Test. *Soil Sci. Soc. Am. Proc.* **1972**, *36*, 25–30.
9. Fixen, P.E.; Grove, J.H. Testing Soils for Phosphorus. In *Soil Testing and Plant Analysis*; 3rd Ed; Westerman, R.L. Ed.; Soil Science Society of America: Madison, WI, 1990, 141–180.

10. Kamprath, E.J.; Watson, M.E. Conventional Soil and Tissue Tests for Assessing the Phosphorus Status of Soil. In *The Role of Phosphorus in Agriculture*; Khasawneh, F.E., Sample, E.C., Kamprath, E.J. Eds.; American Society of Agronomy/Crop Science Society of America/Soil Science Society of America: Madison, WI, 1980, 433–469.
11. Michaelson, G.J.; Ping, C.L.; Mitchell, G.A. Correlation of Mehlich-3, Bray-1, and Ammonium Acetate Extractable P, K, Ca, and Mg for Alaska Agricultural Soils. *Commun. Soil Sci. Plant Anal.* **1989**, *18*, 1003–1015.
12. Menon, R.G.; Hammond, L.L.; Sissingh, H.A. Determination of Plant-Available Phosphorus by the Iron Hydroxide-Impregnated Filter Paper (P_i) Soil Test. *Soil Sci. Soc. Am. J.* **1989**, *52*, 110–115.
13. Sharpley, A.N. Soil Phosphorus Extracted by Iron-Aluminum-Oxide-Impregnated Filter Paper. *Soil Sci. Soc. Am. J.* **1991**, *55*, 1038–1041.
14. Sharpley, A.N.; Sims, J.T.; Pierzynski, G.M. Innovative Soil Phosphorus Indices: Assessing Inorganic Phosphorus. In *New Directions in Soil Testing for Nitrogen, Phosphorus, and Potassium*; Havlin, J., Jacobsen, J., Fixen, P., Hergert, G. Eds.; American Society of Agronomy: Madison, WI, 1994, 115–142.
15. Qian, P.; Schoenau, J.J.; Huang, W.Z. Use of Ion Exchange Membranes in Routine Soil Testing. *Commun. Soil Sci. Plant Anal.* **1992**, *23*, 1791–1804.
16. Somasiri, L.L.W.; Edwards, A.C. An Ion Exchange Resin Method for Nutrient Extraction of Agricultural Advisory Soil Samples. *Commun. Soil Sci. Plant Anal.* **1992**, *23*, 645–657.
17. Yang, J.E.; Skogley, E.O.; Georgitis, S.J.; Schaff, B.F.; Ferguson, A.H. Phytoavailability Soil Test: Development and Verification of Theory. *Soil Sci. Soc. Am. J.* **1991**, *55*, 1358–1365.
18. Skogley, E.O.; Georgitis, S.J.; Yang, J.E.; Schaff, B.F. The Phytoavailability Soil Test (PST). *Commun. Soil Sci. Plant Anal.* **1990**, *21*, 1229–1243.
19. Schaff, B.E.; Skogley, E.O.; Bauder, J.W.; Sieler, D.J. Resin Capsule Adsorption of P for Predicting Plant Response and P Uptake. *Agron. Abstr.* **1992**, *84*, 290.
20. Abrams, M.M.; Jarrell, W.M. Bioavailability Index for Phosphorus Using Ion Exchange Resin Impregnated Membranes. *Soil Sci. Soc. Am. J.* **1992**, *56*, 1532–1537.
21. Menon, R.G.; Chien, S.H.; Hammond, L.L.; Arora, B.R. Sorption of Phosphorus by the Iron Oxide-Impregnated Filter Paper (P_i Soil Test) Embedded in Soils. *Plant Soil* **1990**, *126*, 287–294.
22. Bray, R.H.; Kurtz, L.T. Determination of Total Organic, and Available Forms of Phosphorus in Soils. *Soil Sci.* **1945**, *119*, 190–197.
23. Mehlich, A. Mehlich-3 Soil Test Extractant: A Modification of Mehlich-2 Extractant. *Commun. Soil Sci. Plant Anal.* **1984**, *15*, 1409–1416.

24. Olsen, S.R.; Cole, C.V.; Watanabe, F.S.; Dean, L.A. *Estimation of Available Phosphorus in Soils by Extraction with Sodium Bicarbonate*; USDA Circ. 939 U.S. Government Printing Office: Washington, DC, 1954.
25. Sharpley, A.N. An Innovative Approach to Estimate Bioavailable Orthophosphate in Agricultural Runoff Using Iron Oxide-Impregnated Paper. *J. Environ. Qual.* **1993**, *22*, 597–601.
26. Kuo, S. Phosphorus. In *Methods of Soil Analysis: Chemical Methods, Part 3*; Sparks, D.L. Ed.; SSSA No. 5 Soil Science Society of America: Madison, WI, 1996, 869–919.
27. Hedley, M.J.; Stewart, W.B.; Chauhan, B.S. Changes in Inorganic and Organic Soil Phosphorus Fractions Induced by Cultivation Practices and Laboratory Incubations. *Soil Sci. Soc. Am. J.* **1982**, *46*, 970–976.
28. Murphy, J.; Riley, J.P. A Modified Single Solution Method for Determination of Phosphate in Natural Waters. *Anal. Chim. Acta* **1962**, *27*, 31–36.
29. Snedecor, G.W.; Cochran, W.G. *Statistical Methods*; 8th Ed; Iowa State University Press: Ames, IA, 1991.
30. Minitab, *Minitab User's Guide Release 11*; Minitab, Inc. State College, PA, 1996.
31. Tran, S.T.; Giroux, M.; Guilbeault, J.; Audesse, P. Evaluation of Mehlich-III Extractant to Estimate the Available P in Quebec Soils. *Commun. Soil Sci. Plant Anal.* **1990**, *21*, 1–28.
32. Buondonno, A.; Coppola, E.; Felleca, D.; Violante, P. Comparing Tests for Soil Fertility. 1. Conversion Equations Between Olsen and Mehlich-3 as Phosphorus Extractants for 120 Soils of South Italy. *Commun. Soil Sci. Plant Anal.* **1992**, *23*, 699–716.