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Ion Chromatography as an Alternative to Standard Methods for Analysis of Macro-nutrients in Mehlich 1 Extracts of Unfertilized Forest Soils

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ABSTRACT

This study evaluates ion chromatography (IC) as an alternative to atomic absorption (AA) and inductively-coupled plasma spectrometry (ICP) for analysis of potassium (K), magnesium (Mg), and calcium (Ca), and as an alternative to antimonylmolybdate colorimetry and ICP for analysis of phosphorus (P) macro-nutrients

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in Mehlich 1 extracts. Soils typical of pine forests in the southeastern United States were tested. IC correlates well with AA and ICP for K and Ca. hut not for Mg, unless conditions are chosen that resolve Mg from manganese (Mn). IC does not correlate very well with colorimetry for P at extract levels below 2mg kg-' or in extracts with high levels of dissolved organic matter complexes of aluminum (Al) and iron (Fe). ICP results for P exceed both IC and colorimetry by 3 -5 mg kg-' for all soils tested. The merits of IC relative to AA, ICP, and colorimetry for forest soil testing are discussed.

Key Words: Ion chromatography; Soil; Forest; Analysis; Mehlich 1; K; Ca; Mg; P; Colorimetry; ICP; Atomic absorption; Macro-nutrients.

INTRODUCTION

Since 1983, this laboratory has conducted a region-wide study of the growth-response of loblolly pine (*Pinus taeda* L.) to control of competing vegetation at 13 sites throughout the southeastern United States.“’ These sites were selected relative to commonly occurring soil series for the major physiographic provinces in the pine forests of the region. Initially in 1984, composite soil samples were collected from each site and analyzed for available P, K, Mg, and Ca after extraction with Mchlich 1 solution.[2] P was analyzed by antimonylmolybdate colorimetry[3] and K, Mg, and Ca by AA spectrometry, following standard practice at most soil testing laboratories in the southeastern U.S. at the time.[4] At the 15-yr mark of the study all sites were re-sampled to assess long-term effects of forest management treatments and vegetation growth on soil quality. By this time IC had become an established method in research laboratories for analysis of both anions and cations in soil solutions.[5–14] IC is an economical alternative to ICP for labs with limited resources and sample loads, but it has not so far been applied to Mehlich 1 soil extracts. Mehlich 1 has remained the accepted multiple macro-nutrient extractant for soil-testing laboratories in most southeastern states except Mississippi and Louisiana[15,16] despite the introduction of Mehlich 3.[17–20] As we attempted to, apply IC analyses to re-sampled soils the question of comparability arose. This work reports conditions for IC analysis of Mchlich 1 extracts for P (as orthophosphate) and for K, Mg, and Ca, and discusses the suitability of IC as an alternative method to AA, ICP, and colorimetry for analysis of macro-nutrients in unfertilized forest soils.

MATERIALS AND METHODS

Soil Sample Collection and Processing

Each location in the region-wide study contained a gridwork of sixteen 0.10 ha treatment plots comprised of four replications each of four postplanting treatments: no control, complete woody control, complete herbaceous control, and complete control of associated vegetation for the first three to five years. For the yr-1.5 soil sampling, four 1.91 cm diameter soil cores were collected from each treatment plot at depths of 0–15, 15–30, and 30 60 cm around five evenly-spaced tree positions (20 cores plot-‘). The cores were carefully composited by depth in plastic buckets at the site, and transferred to freezer bags for shipment to the Auburn laboratory. Each of the 48 composites from each location was stored at 4°C and then air-dried at room temperature, ground to pass through a 2 mm sieve, and again stored at 4°C in a freezer bag. For the purpose of analytical method comparison, 140 composites from the yr- 15 sampling were extracted with Mehlich 1 and the extracts were each analyzed for K, Mg, and Ca by IC, ICP, and AA and for P by IC, ICP, and colorimetry. Seven out of the thirteen sampling locations were chosen to give geographic diversity and to avoid redundancy of soil types (Fig. 1; Table 1). Based on prior analytical data, 20 composites from each of these locations were selected to give the broadest range of nutrient levels and also to include all soil depths from each location.

Soil Sample Extraction and Cleanup

Standard soil test extraction protocols were scaled up fourfold to provide sufficient extract volume for comparative analyses. Approximately 20 ± 0.02 g of each composite soil sample was weighed into a plastic weighing boat and transferred to a 250-mL high density polyethylene wide-mouth bottle. Approximately 80 ± 0.1 mL of Mehlich 1 extracting solution (0.05 M HCl+ 0.0125 M H_2SO_4) was added to each flask using an automatic repeating dispenser. The bottles were sealed with polypropylene screw caps, shaken 1.5 min at 180 oscillations min^{-1}, and allowed to settle. (Soil test labs in Georgia, Florida, Tennessee, South Carolina, North Carolina, and Virginia use a 5 min shaking time; Louisiana and Mississippi use 15 min, and Alabama uses 30 min.)“” The extracts were carefully decanted through cones of 9.0 cm Whatman No. 1 filter paper into 150 mL beakers. (The filter paper was prewashed with 0.05 M HCl for 75 min, then with 18.2 MΩ water for 15 min, and then

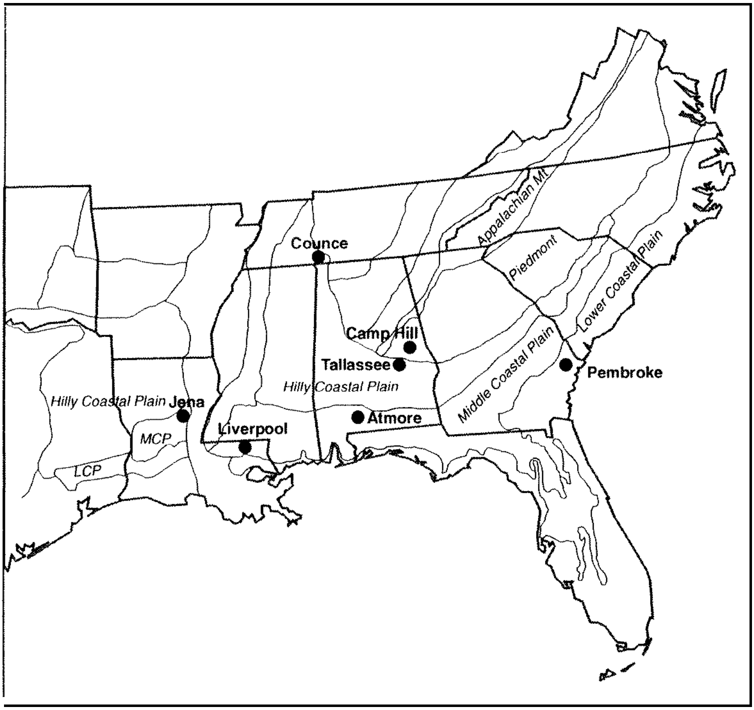

Figure 1. Map of site locations for soil sample collection.

air-dried for 2 d.) For K, Mg, and Ca analysis, about 5 mL of the filtered extract was decanted into a Dionex HDPE sample vial with 20 µM frit filter cap. For P analysis, 5 mL was passed through a Dionex OnGuard®-H cartridge to remove polyvalent cations such as Fe and Al that would contaminate the analytical column. Each cartridge was prewashed with 2mL of Mehlich 1 extracting solution at a rate of 2 mL min^{-1}, followed by the sample extract. The first 3 mL ofextract was discarded and the last 2mL was collected in a Dionex sample vial for analysis. ICP analysis of the eluents showed that each OnGuard®-H cartridge could be reused up to 10 times without breakthrough of Fe or Al for any of the sites studied.

AA, ICP, and Colorimetric Reference Analyses

A 15 20 mL portion of each extract was transferred to a 30 mL HDPE sample bottle for submission to the cooperating laboratory

Table 1. Study locations selected for soil sampling.

Site location *coordinates*	Soil series	Soil classification
Pembroke, GA N32°7′48″ W81°35′26″	Mascotte	Sandy, siliceous, Thermic Ultic Alaquods
Atmore, AL N31°15′27″ W87°17′17″	Orangeburg	Fine-loamy, kaolinitic, Thermic Typic Kandiudults
Jena, LA N31°40′27″ W92° 10′39″	Ruston	Fine-loamy, siliceous, Thermic Typic Paleudults
Liverpool, LA N30°55′7″ W90°43′27″	Tangi	Fine-silty, siliceous, Thermic Typic Fragiudults
Tallassee, AL N32°31′38″ W85°48′42″	Cowarts	Fine-loamy, kaolinitic, Thermic Typic Kanhapludults
Counce, TN N35°9′52″ W87°58′17″	Silerton	Fine-silty, siliceous, Thermic Typic Paleudults
Camp Hill, AL N32°49′42″ W85°35′48″	Cecil	Clayey, kaolinitic, Thermic Typic Kanhapludults

(Auburn University Soil Testing Lab). Phosphorus was analyzed using the standard antimonylmolybdate-ascorbic acid colorimetric method of Watanabe and Olsen,[3] measuring absorbance at 740 nm. Determination of K was by AA at 766.5 nm on undiluted extract. Determinations of Mg and Ca were by AA at 285.2 and 422.7 nm, respectively, after 1:4 dilution with 0.5% lanthanum in Mehlich 1 solution. Confirmatory tests for K, Mg, Ca, P, Mn, Fe, and Al were performed by ICP using a Jarrell-Ash 9000.

Ion Chromatographic Analyses

The ion chromatograph used was a Dionex DX300 system (Dionex Corp., Sunnyvale, CA) comprising a gradient pump, CDM-3 conductivity detector, eluent degas module, AS40 automatic sampler, and Rheodyne 9126 automatic sample injection valve. Instrument control and data handling was by computer interface to a Pentium II PC running Dionex Peaknet 5.1 software under Windows 95.

Analysis for P (as orthophosphate anion) was performed using a Dionex AS4A-SC (4 x 250mm) anion exchange column with an AG4A-SC (4 x 50mm) guard column and a Dionex ASRS-ULTRA 4mm self-regenerating membrane suppressor. The eluent was 1.8 mM Na_2CO_3 + 1.7 mM $NaHCO_3$ pumped at 2.0 mL min^{-1}. The suppressor was operated in the recycle mode with the eluent serving as the regenerant after passing through the detector cell. A current of 100 mA across the suppressor membranes kept the background conductivity below 1000 nS. External standards of 0, 0.1, 0.5, 2.0, and 10.0 mg L^{-1} orthophosphate in Mehlich 1 extraction solution were injected before every 30 sample injections along with two extracts of a control soil. A 50-µL sample loop was used. The following instrument detection limits (IDL) were observed, based on 3 times the standard deviation of the lowest calibration standard:

Orthophosphate	0.15 mg kg^{-1} in extract	0.60 mg kg^{-1} in soil
Phosphorus	0.05 mg kg^{-1} in extract	0.20 mg kg^{-1} in soil

Direct injection of Mehlich 1 extracts severely overloaded the column, causing the chloride peak to tail significantly and the sulfate peak front to encroach upon the small phosphate peak. Degradation of the column set due to adsorption of dissolved organic matter was carefully monitored to ensure adequate resolution. The retention time of sulfate was measured prior to every sample set by injection of a dilute sulfate standard (in water) and the guard column was replaced when the sulfate retention time fell to 80% of new column performance. Guard columns could be partially regenerated for reuse by cleaning with 1 N HCl for 1 h.

Analyses for K, Mg, and Ca (as cations) were performed using two different column-eluent systems: (1) a Dionex CS-12 (4 x 250mm) cation exchange column plus a CG-12 (4 x 50 mm) guard column with 22 mN H_2SO_4 eluent pumped at 1.0 mL min^{-1}; (2) a Dionex CS-12A (4 x 250 mm) analytical column plus a CG-12A (4 x 50mm) guard column with 20mN methanesulfonic acid eluent pumped at 1.0 mL min^{-1}. In both cases a Dionex CSRS-ULTRA 4mm self-regenerating suppressor was operated in the chemical suppression mode with an AutoRegen® module pumping recycled 100 mM tetrabutylammonium hydroxide at 10 mL min^{-1}, maintaining a baseline conductivity of 1.5 2.0 µS. The cation regenerant cartridge in the AutoRegen® module was replaced whenever the baseline conductivity rose above 3 µS.

External standards including a blank were prepared in Mehlich 1 extraction solution and injected before every 30 sample extract injections (including two control soil extracts). Calibration standard levels were 0, 2.5, 10, 25, and 100 mg L^{-1} for K and Ca, and 0, 1.25, 5, 12.5, and 50mg L^{-1} for Mg. All cations showed linear conductivity detector response over this calibration range, but column overloading occurred at higher Ca levels as revealed by peak "fronting" and reduced retention times. Detection limits were never an issue with these soils, so a 2-μL sample loop was used to minimize overloading and retention time migration. No retention time loss was observed, even after 2000 soil extract injections.

Extracts of soil from one site (Pembroke, Georgia; Mascotte Series) were heavily contaminated with dark brown dissolved organic matter. After only a few injections of Mascotte extracts the CSRS suppressor membrane (*downstream* from the analytical column) became too contaminated to function. To protect the IC system these extracts were pretreated by passing them through OnGuard®-P cartridges which removed this brown organic matter. The cartridges, mounted in a vacuum manifold, were pre-washed with 10 mL Mehlich 1 extraction solution, and then 5 mL of extract was passed through, discarding the first 3 mL and collecting the final 2 mL. These cleanups were time-consuming and were not required for samples from the other sites.

Linear correlations were performed on the comparative nutrient data using the REG procedure in SAS (Version 8.0). In all cases the probability level of no linear correlation between methods was <0.0001.

RESULTS AND DISCUSSION

Ion chromatography results for K (Fig. 2) and Ca (Fig. 3) showed excellent linear correlations with both AA and ICP ($R^2 = 0.98$ for K and 0.99 for Ca; $n = 140$). IC results for Mg did not correlate quite as well with either AA ($R^2 = 0.80$; $n = 140$) or with ICP ($R^2 = 0.82$; $n = 140$), particularly at levels below 70 mg kg^{-1} in soil. The *difference* between IC-Mg levels and AA-Mg levels did correlate significantly with ICP-measured levels of Mn ($R^2 = 0.91$; $n = 140$, data not shown). Comparison of Mg results on a site-by-site basis revealed that IC-Mg levels significantly higher than AA-Mg levels occurred mainly on three sites: Camp Hill, AL (Cecil), Atmore, AL (Orangeburg), and Counce, TN (Silerton). Examination of the ICP results for these sites confirmed high levels of Mn in all three. Tests with mixed standard solutions of Mg and

Mn revealed that the CS-12 cation exchange column could not resolve the Mg peak from the Mn peak.

It was subsequently found that a CS-12A column, an improved version of the CS-12, was able to resolve Mg from Mn if eluted with 20mM methanesulfonic acid instead of sulfuric acid. Reanalysis of the extracts using a CS-12A column gave IC results for Mg, which correlated very well with both AA and FCP results ($R^2 = 0.996$; Fig. 4). Results for K and Ca using the CS-12A column were equivalent to those using the CS-12 column and so are not reported. For all three cationic nutrients, the slope of the correlation line was lower when IC data was compared to ICP data than when IC data was compared to AA data. This is consistent with the ability of the 10,000°K ICP plasma source to ionize colloidal-size clay mineral particles that pass through the coarse Whatman No. 1 cellulose fiber filter paper (11 µm) used in standard soil test procedures and remain suspended in the extract filtrate.

Ion chromatography and AA techniques are more selective than FCP. They measure only free ions in solution- -not those occluded in

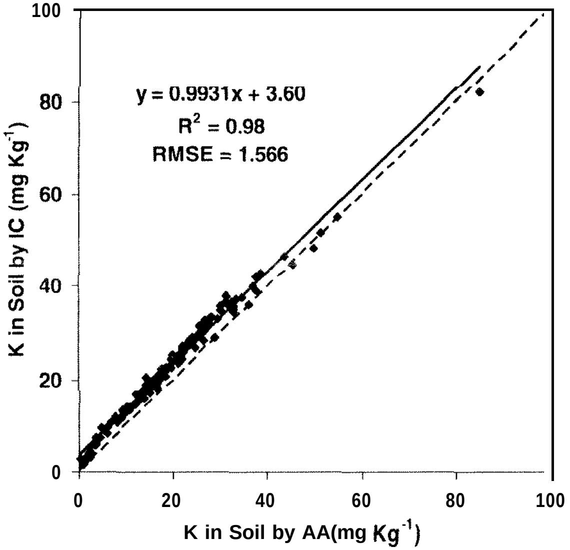

Figure 2a. **Method** comparison of IC results for **K** in Mehlich I soli extracts with AA results for the same extracts. In Figs. **2 5** the dotted line indicates the 1:1 line where data points would be located if there was 100% conformity between methods. The linear regression equation, correlation coefficient, and root mean square error are shown ($n = 140$).

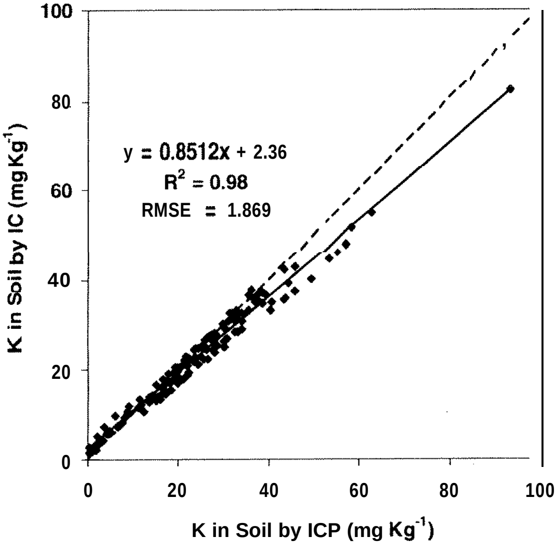

Figure 26. Method comparison of IC results for K in Mehlich 1 soil extracts with ICP results for the same extracts (n = 140).

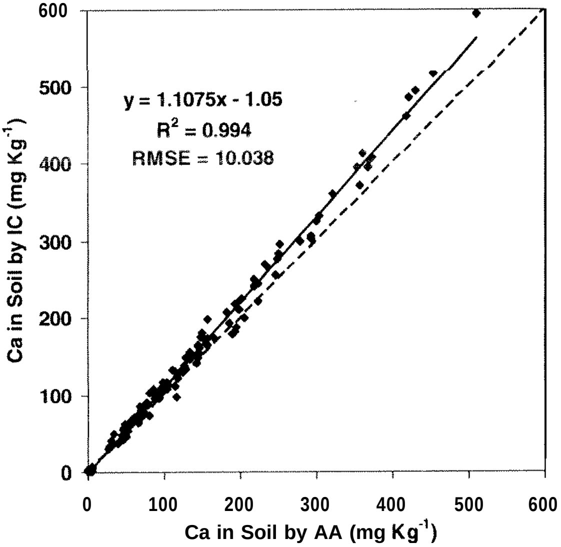

Figure 3a. Comparison of IC results for Ca in Mehlich 1 soil extracts with AA results for the same extracts (n = 140).

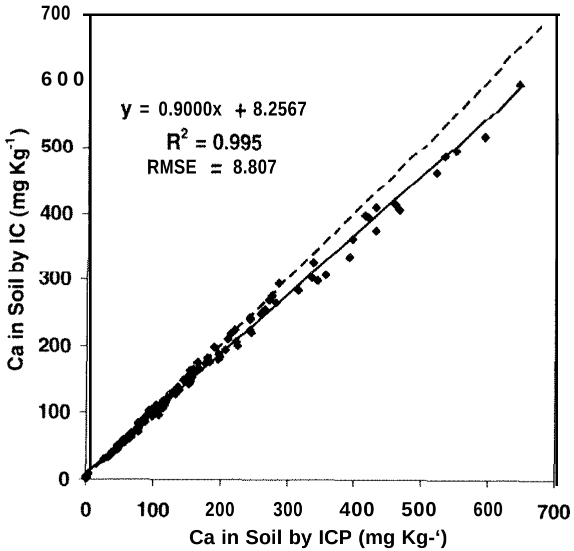

Figure 36. Comparison of IC results for Ca in Mehlich I soil extracts with ICP results for the same extracts (n = 140).

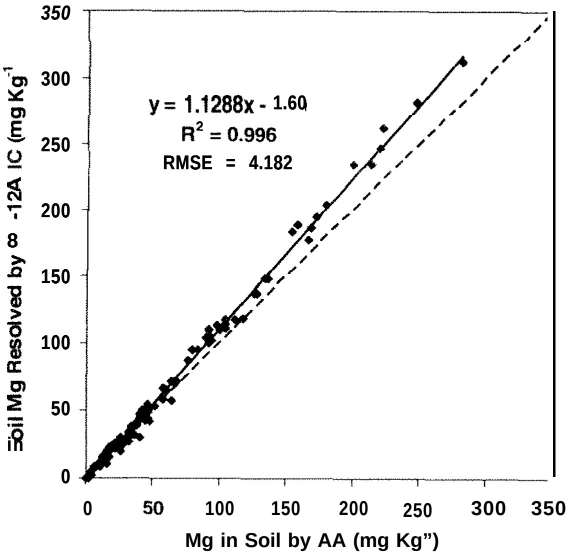

Figure 4a. Comparison of IC results for Mg in Mechlich I soil extracts (using the CS-12A column) with AA results for Mg in the same extracts (n = 140).

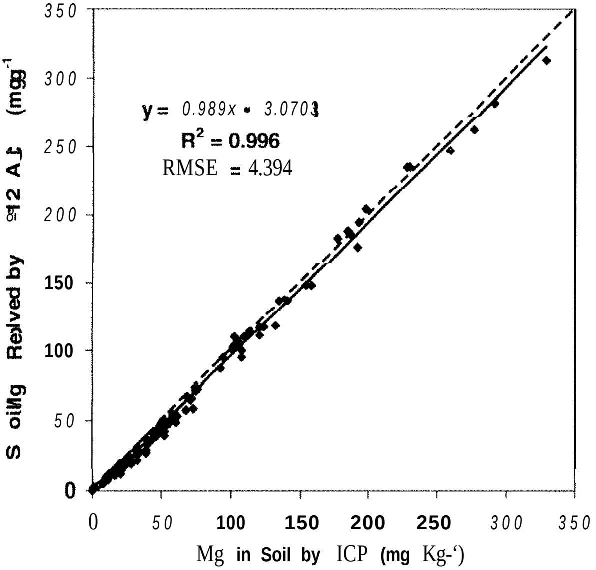

Figure 4b. Comparison of IC results for Mg in Mechlich 1 soil extracts (using the CS-12A column) with ICP results for Mg in the same extracts (*n* = 140).

suspended clay particles. The excess response of IC over AA for Mg (1.13:1) and Ca (1.11:1) is consistent across the analytical range, and could be a systematic error caused by disagreement between the calibration standards used for the different methods. Direct AA analysis of our IC cation standards by the Auburn University Soil Testing Laboratory gave 12.5 mg/L for Mg (12.5 nominal) and 25.3 mg/L for Ca (25 nominal). A possible cause of reduced AA response for Mg and Ca might also be the practice of diluting the soil extracts 1:4 with 0.5% lanthanum solution prior to AA analysis of Mg and Ca to reduce matrix interferences.

Phosphorus results by IC did not correlate especially well with colorimetric P results ($R^2 = 0.90$) and still less well ($R^2 = 0.71$) with ICP results for P (Fig. 5). IC and colorimetric P results were not well correlated for low P soils near the detection limit (<2 mg kg^{-1} P). The high P soils, Cowarts and Mascotte, showed higher P by colorimetry than by IC, and ICP results for P were 3–5 mg kg^{-1} higher than either IC or colorimetric P results for all soils tested (Fig. 6). The excess ICP results for P correlated significantly ($R^2 = 0.67$; n = 140, data not shown) with ICP-measured levels of Al.

Phosphorus exists in the soil-groundwater system in many forms, including inorganic ions (orthophosphate, polyphosphates), soluble organic compounds (phytates, phospholipids), colloidal size suspended minerals (clays), and dissolved organic complexes (humates, fulvates).[21–25] During the 5–30 min contact period with the Mehlich 1 solution each of these P sources begins to approach an equilibrium state in the soil solution. It has been shown that significant reservoirs of P can be within and on the surface of colloidal size clay mineral particles that pass through membrane filters as fine as 0.22 μm,[26] let alone the coarse 11 μm Whatman No.1 paper used in routine soil tests. Even more significant P reservoirs can occur in dissolved organic matter bound to humate- Al--Fe-complexes.[27,28] This is especially true for sandy lower coastal plain soils like Mascotte[29] (Pembroke, Georgia). Each analytical method interacts differently with these P reservoirs in the aqueous Mehlich 1 extract.

Ion chromatography is the most selective; it detects only dissolved orthophosphate (as HPO_4^{-2}). Colorimetry can respond not only to dissolved orthophosphate, but to additional P leached out by the strongly acid (pH 1-2) antimonylmolybdate color reagent from the suspended

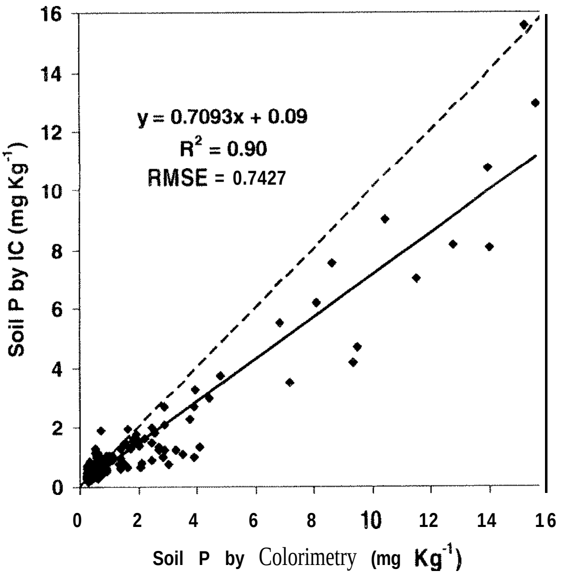

Figure 5a. Comparison of IC results for P in Mehlich 1 soil extracts with colorimetric results for P in the extracts ($n = 140$).

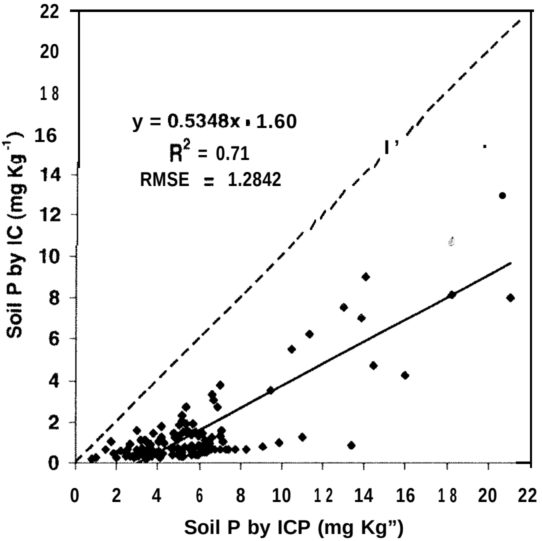

Figure 56. Comparison of IC results for P in Mechlich I soil extracts with ICP results for P in the same extracts ($n = 140$).

clay particles that pass through the Whatman No. l filter paper.[21,25,27,28] ICP is the most indiscriminate method of the three, converting all P species, organic, dissolved, adsorbed, and occluded, to ions in the plasma source---in effect measuring *total dissolved P*, analogous to Kjeldahl or persulfate digestion.[16,25] Because this extra ICY-liberated P is not measured by the traditional colorimetric method.” including it in routine

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“Ralph J. DiCosty (Research Soil Scientist, USDA Forest Service, Athens, Georgia; private communication) has noted that the original 1953 Mehlich procedure[2] specified adding 250 mg activated charcoal powder to the soil sample to decolorize the extract prior Lo extraction and filtration. This would remove most of the P bound to dissolved organic matter, a major source of excess P detected by ICP. A checkout of this procedure revealed extremely high levels of phosphorous contamination in charcoal obtained from hone meal (>2400 μg g^{-1}) and even in acid-washed Norit-A (350 μg g^{-1}). Only charcoal derived from combustion of acetylene was free of P contamination. Soil testing laboratories using ICP to measure P in soil extracts might well consider adopting this step, so long as they avoid using contaminated charcoal.

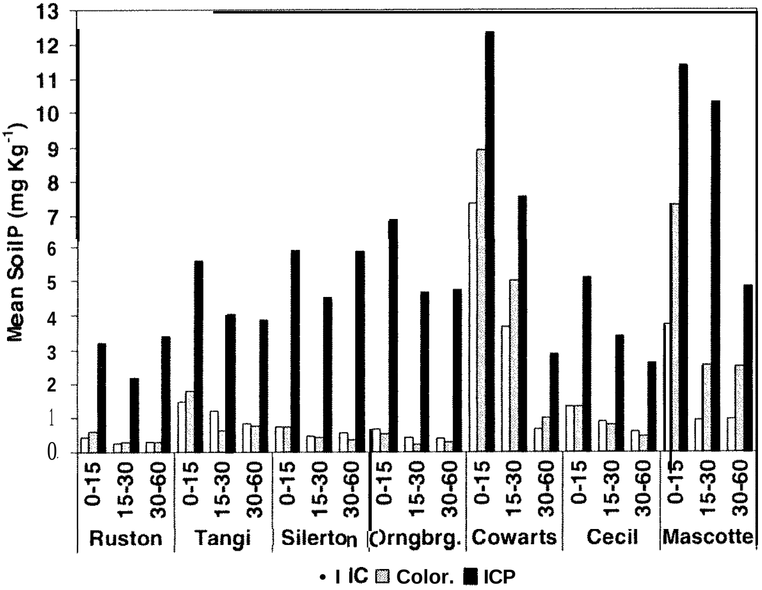

Figure 6. Mean soil P results by analysis method, soil series, and core depth (cm).

soil test reports can cause underestimation of P fertilization requirements, especially for the P-deficient soils characteristic of forests in the southeastern U.S.

CONCLUSIONS

It was found that IC is a valid and convenient alternative method for the analysis ofcationic soil nutrients K, Mg, and Ca in Mehlich 1 extracts from a wide variety of forest soil types common to the southeastern U.S. The presence of Mn at high levels in some of these soils can positively bias the IC results for magnesium unless a column-eluent system capable of resolving these ions is used. The CS- 12A column with methanesulfonic acid eluent is the only IC system we have found that is presently capable of resolving Mg from Mn without resorting to eluent programming. The 12 min IC analysis time is offset by the ability to determine all three cation specks in a single run and by the ability to run up to 60 samples without manual intervention. IC could be cost-effective as a supplementary or alternative technique to ICP or AA for research laboratories with limited personnel, low equipment budgets, and small or infrequent sample loads.

Phosphorus is more problematic due to the multiplicity of forms in which P occurs in the filtered extract. The acidic colorimetric reagent partially hydrolyzes each of these P sources to orthophosphate.[29] IC does not subject the extracts to this additional acidification prior to analysis. Extra sample cleanup and constant performance monitoring arc necessary to manage the continuous degradation of the anion-exchange column from adsorption of humate-Fe-Al complexes. The critical separation between the small orthophosphatc peak and the large trailing sulfate peak of the Mehlich 1 solution can degrade after only a few injections, necessitating reanalysis and frequent costly column replacements. Large-volume injection to offset the marginal sensitivity of the IC conductivity detector for orthophosphatc further aggravates sulfate peak overload and the resulting phosphate-sulfate peak resolution problem. For all of these reasons IC is probably not a valid or cost-effective substitute for colorimetric analysis of P in Mehlich 1 soil extracts at the present time. Nevertheless, it gives a much closer approximation of traditional colorimetric P results than does ICP for all the soils tested in this study. The unique selectivity of IC for orthophosphate allows direct determination of this most available form of P without the need for fractionation, holding the potential for more accurate fertilization recommendations for forest management and more sensitive monitoring of site productivity changes than either ICP or traditional colorimetric methods.

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