UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

A Comparison of Methods for Estimating Extractable Amounts of 17 Constituents in Samples of Mine Soil from Coal Strip Mines in the Western Energy Region

By

R. C. Severson, M. L. Tuttle, J. L. Peard, and J. G. Crock

Open File Report 81-180

1981

CONTENTS

Page

ILLUSTRATIONS

ILLUSTRATIONS (Continued)

TABLES

A Comparison of Methods for Estimating Extractable Amounts of 17 Constituents in Samples of Mine Soil from Coal Strip Mines in the Western Energy Region

by

R. C. Severson, M. L. Tuttle, J. L. Peard, and J. G. Crock

INTRODUCTION

Most research in soil testing has been applied to agricultural soils and agronomic crops. Because of increasing concern for monitoring metal levels in natural soils and native plants, and in rehabilitated mine spoil and reclamation plants, many of the "availability" measures common in agronomy are being applied directly to problems in reclamation of mined land. Melsted and Peck (1973) discuss the problem of transferring soil-test technology among regions of underdeveloped countries where no adequate research background has yet been developed; such transfers can result in invalid interpretations and judgments. A similar problem exists in transferring soil-test technology from fertilized agricultural soils and crops to rehabilitated mine spoil and reclamation plants. Only minimal research has been conducted to determine whether or not the agronomic soil tests provide reliable "availability indexes" for the plant-soil systems of mined land. Consequently, the application of common soil tests to rehabilitated mine land is currently limited (Berg, 1978). For example, the "critical" or "toxic" levels specified in mined-land rehabilitation regulations probably are not supported by adequate research to permit valid interpretations and sound judgments. As

Cope and Rouse (1973, p. 36) point out, values obtained for soil extracts do not, in themselves, directly reflect amounts available to plant roots; the values become meaningful only when they are related to differences in plant growth or nutrient uptake.

DTPA (diethylenetriaminepentaacetic acid) is a soil extractant that has been used on both agronomic soils and rehabilitated mine spoils. Lindsay and Norvell (1969, 1978) have shown that DTPA is both theoretically and functionally a suitable extractant for assessing "available" levels of Cu, Fe, Mn, and Zn in agricultural soils. Soltanpour and Schwab (1977) have modified this DTPA extractant so that it will be useful for assessing "available" levels of NO_3, P, and K in addition to Cu, Fe, Mn, and Zn. They have shown (Soltanpour and Schwab, 1977) that their ammonium bicarbonate-DTPA extractant test is highly correlated with the Lindsay DTPA extractant test (Lindsay and Norvell, 1969, 1978) for Cu, Fe, Mn, and Zn; with the Olsen P test (Olsen and Dean, 1965); and with the ammonium acetate K test (Pratt, 1965).

We suspect that DTPA will continue to be used to estimate "available" levels of metals in soils and that the necessary research background for this extractant for rehabilitated mine land and reclamation plants will be forthcoming. Persons conducting this basic research will be aided by information on the working range of metals in rehabilitated land from 12 coal strip mines in the western energy regions (fig. 1). They should also find useful the comparisons of the DTPA extracts (Lindsay and Norvell, 1969, 1978) and exchangeable and water-soluble cations with the NH_4HCO3-DTPA extracts (Soltanpour and Schwab, 1977) of the same samples of topsoil and spoil material from these rehabilitated coal strip mines.

Acknowledgments

The following persons, all with the U.S. Geological Survey in Denver, either prepared and analyzed the samples or were responsible for development of instrumental capabilities for analyzing the soil extracts: D. B. Hatfield, T. T. Law, F. E. Lichte, R. E. McGregor, C. S. E. Papp, and George Riddle.

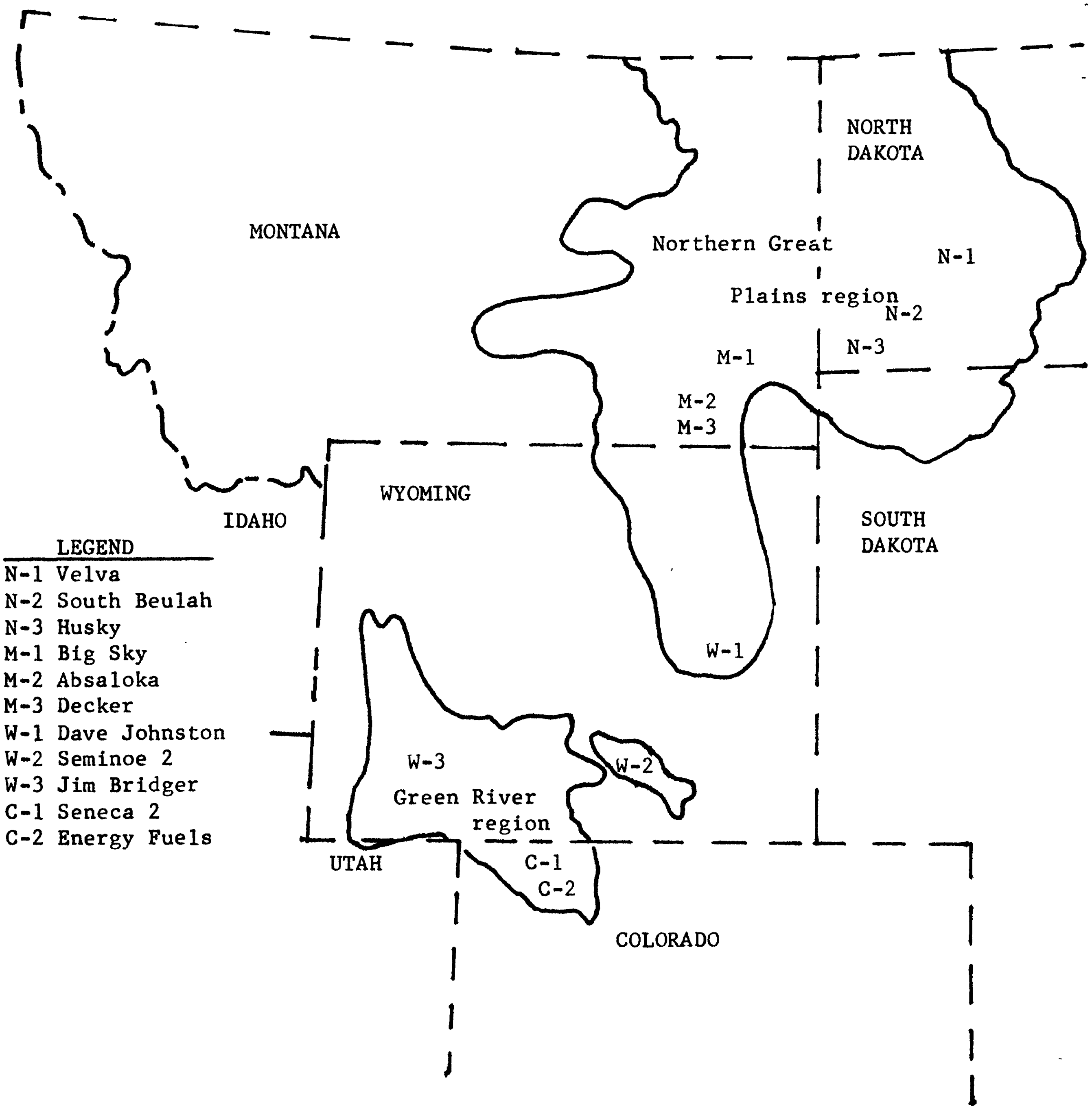

Figure 1.--General locations of the coal strip mines sampled in the western energy regions.

METHODS

Sample Collection and Preparation

Soils were collected in the following way at each of the 12 coal strip mines shown in figure 1. A random traverse across the site was made and at ten locations topsoil and spoil samples were collected. The exact soil sampling locations were dictated by the presence of desirable plant material. Where grasses were sampled, the topsoil sample was obtained by digging around the plant clump to a depth of about 10 cm, extracting the plant clump with soil attached to the roots, and collecting the soil particles (topsoil, spoil, or some combination) adhering to the roots of the plant. Where a legume or plant other than a grass was sampled, the topsoil material to a depth of about 10 cm was collected within a radius of about 20 cm from the plant. At each location where topsoil was collected, a second sample of material (called spoil material) was collected below the 10-cm level after the topsoil had been removed. True spoil material was sampled only at some mines where topsoil was shallow, because the "spoil" sample was collected only to a depth of 30-40 cm below the surface. The spoil material was sifted through a stainless steel screen with 1-cm openings, and the material larger than 1 cm was discarded.

All samples were air dried at ambient temperature. The dry samples were disaggregated in a motor-driven mortar and pestle, and the fraction passing a 2-mm (10-mesh) sieve was saved. More than about 90 percent of the material in all topsoil samples passed the 2-mm sieve after disaggregating, except for samples collected at the Jim Bridger Mine, of which about 80 percent of the topsoil material passed the sieve after disaggregating. For spoil material samples, the fraction passing the 2-mm sieve was quite consistent from site to site. All samples were processed and analyzed in a random sequence so that any systematic biases would be converted to random errors.

Lindsay's DTPA Extract

All samples were extracted with a solution of 0.005 M DTPA (diethylene triaminepentaacetic acid), 0.1 M triethanolamine, and 0.01 M calcium chloride at a pH of 7.3 (Lindsay and Norvell, 1969, 1978). The extraction procedure is described in detail by Crock and Severson (1980). The extraction was performed in the following general way. For each sample, 15.00 g of the air-dried soil and 30.00 mL of the DTPA solution were added to a 125-mL polyethylene Erlenmeyer flask. The flask was stoppered tightly with a polyethylene thimble and shaken on a reciprocating shaker at 240 cycles per minute for two hours. The solution was decanted into a 50 mL polyethylene centrifuge tube and centrifuged for 5 to 6 minutes at 2000 rpm. The solution then was filtered through an 11-cm Whatman* 41 filter paper into a 2-oz polyethylene bottle, and the solutions were acidified with four drops of concentrated nitric acid. Eight metals were determined using atomic absorption spectrometry (A.A.S.) standard procedures: Cd, Co, Cu, Fe, Mn, Ni, Pb, and Zn.

---

*Use of a brand name in this report is for descritpion purposes only and does not imply endorsement by the U.S. Geological Survey.

Soltanpour's DTPA Extract

Soils were extracted using the method of Soltanpour and Schwab (1977). An extracting solution of 0.005 M DTPA and 1.0 M ammonium bicarbonate was prepared and adjusted to pH 7.6 with nitric acid. For each sample, 10.0 g of soil and 20.0 mL of extracting solution were placed into a 125-mL polyethylene Erlenmeyer flask. The unstoppered flask was shaken for 15 minutes on a reciprocating shaker at 180 cycles per minute. The extract was filtered through Whatman 41 filter paper. A 10.0-mL aliquot of the filtrate was placed in a polyethylene bottle and acidified with 5.0 mL of 6 N nitric acid. The acidified extracts were analyzed by ICP-OES (inductively coupled argon plasma-optical emission spectroscopy) utilizing interelemental spectral corrections (F. E. Lichte, oral communication, June 1980).

Exchangeable and Water-Soluble Cation Determinations

The exchangable cations were determined from an extraction of the soil using a 1.0 N ammonium acetate solution at pH 7.0 (Chapman, 1965). Crock and Severson (1980) outline in detail this method and some minor modifications. The extraction procedure is summarized as follows. For each sample, 33 mL of the 1.0 N ammonium acetate solution and 4.00 g of the air-dried soil were added to a 65-mL culture tube (20 mm by 225 mm), and the tube was shaken horizontally on a reciprocating shaker at 240 cycles per minute for 5 minutes. The solution was centrifuged at 2000 rpm for 5 minutes and then decanted into a 100-mL volumetric flask through a funnel containing Whatman 41 filter paper. The ammonium acetate wash procedure was repeated two additional times. Four drops of 0.1-percent sodium hexametaphosphate were added, and the solution was brought to volume (100 mL) using the 1.0 N ammonium acetate solution. This solution was analyzed for exchangable Ca, Mg, K, and Na by A.A.S. standard methods.

Water-soluble cations were determined from a saturation paste of the soil. This extract has been described by Bower and Wilcox (1965) and was performed in the following general way. For each sample, 200.0 to 500.0 g of air-dried soil was added to an 800-mL plastic, disposable beaker of known weight; demineralized water was added to a known weight of soil until the saturation criteria were met. These criteria are that the paste glistens, flows freely off of the mixing spatula, and has no free-standing water. The paste was allowed to set covered for 4 hours, weighed to obtain the amount of water used, and transferred to a Buchner funnel containing Whatman 41 filter paper; the paste was filtered under low vacuum (20 to 30 torr) until the paste cracked and drew air. The filtrate was centrifuged at 2000 rpm for 5 minutes, filtered through a Whatman 41 filter paper into a 2-oz polyethylene bottle, and acidified with nitric acid. Details of this method and minor modifications are given by Crock and Severson (1980). The resulting solution was analyzed for water-soluble Ca, Mg, K, and Na using A.A.S. standard methods.

Available B in soil was estimated by a hot-water extraction. The extraction procedure is outlined by Crock and Severson (1980) and is summarized as follows. For each sample, 20.0 g of air-dried soil, 20.0 mL of demineralized water, and 0.5 mL of 10-percent barium chloride were added to a 4-oz polyethylene bottle. The bottle was immersed in a boiling water bath for 30 minutes, removed and cooled to room temperature, filtered through a Whatman 41 filter paper, and acidified with nitric acid. Boron was determined with a DC argon plasma at 249.7 nm.

Statistical Analysis

The relation between the amount of an element extracted by Soltanpour's method and the amount of the same element extracted by other standard methods was evaluated using linear regression analysis. Prediction equations were calculated using the following form:

$$\log Y = \underline{a} + \underline{b} \log X$$

where Y is the estimate of the element amount extracted by Soltanpour's method; X is the estimate of the element amount extracted by other standard methods; $\underline{b}$ is the slope; and $\underline{a}$ is the intercept.

The relative precision for constituents measured by each method was determined by computing an $\underline{F}$-ratio, using the variance of replicate determinations. Because there was no reason to believe one method should be consistently more precise than another method, the largest variance was divided by the smallest variance, and a two-tailed test of hypothesis was used to determine the critical value of the $\underline{F}$ statistic. Such a test is realistic only if the samples analyzed by the different methods have homogeneous variance between them. A test for homogeneity of variance was not performed, but such homogeneity generally can be confirmed or rejected by visually comparing geometric deviations for each constituent for the samples analyzed by the two different methods.

All constituents were transformed by taking their common logarithm prior to statistical analysis. This transformation was made because histograms of the data for all samples were positively skewed; many values occurred near the lower end of the detection range, and few values occurred near the upper end of the range. For most constituents, histograms of the log-transformed data approximate a normal distribution more closely than do untransformed data. The effect of this transformation can be seen by comparing figure 2 (untransformed DTPA extractable Fe) and figure 3 (log-transformed DTPA extractable Fe). Figure 3 shows that the data in figure 2, when transformed by the common logarithm, are more evenly dispersed throughout the observed range. If untransformed data are used to calculate a prediction equation, an extremely high value will influence the slope of the equation to a greater extent than will several small values. The transformation of the data results in having a more equal weighting for samples with high and low concentrations in determining the slope of the equation.

RESULTS AND DISCUSSION

Prediction Equations

Regression equations for relations between Soltanpour's method and the other standard methods are given in table 1. The observed range, geometric mean, and geometric deviation for each constituent and for each method are given in table 2. Table 2 also shows means and ranges for constituents determined in the samples used by Soltanpour and Schwab (1977) to develop prediction equations for relations between their method and other standard methods. Table 2 shows that the observed ranges for the 205 mine-spoil samples of this study are greater than those for the 481 samples of agricultural soils reported by Soltanpour and Schwab. For Cu and Fe, the range is an order of magnitude greater, while for Mn, Pb, and K the range is about 1.5 to 2.0 times greater. Therefore, any prediction equations deemed valid for relating these methods will expand the working range for Soltanpour's DTPA extract when used for mine soils.

The slopes of the equations in table 1 indicate that similar amounts of DTPA-extractable Cd, Fe, Ni, and Zn, of exchangeable Na, and of hot-water-extractable B are measured in Soltanpour's extract and the other methods. For all of these prediction equations, except B, the coefficient of determination is sufficiently large that the equations presented in table 1 should be valid and useful. Coefficients of determination show that more than 60 percent of the total variation between analyses by the two methods is explained for all the aforementioned metals and for DTPA-extractable Cu as well.

Table 1.--Prediction equations for relations between Soltanpour's method and other standard methods for determining soil constituents.

[Prediction equations are of the form: $\log Y = \underline{a} + \underline{b} \log X$ where Y is an estimate of the variable from Soltanpour's method and X is the variable measured by other standard methods; me, millieqivalent (1 me = 1/1,000 of the equivalent weight).]

Variable (X,Y)	Intercept (a)	Slope (b)	Coefficient of Determination (r^2)
	Extractable, in ppm		
Cd	-0.16	0.86	0.68
Co	-1.46	.62	.26
Cu	.34	.76	.71
Fe	.02	.97	.78
Mn	.11	.76	.41
Ni	.08	1.01	.74
Pb	.17	.52	.39
Zn	.13	1.00	.87
	Exchangeable, in me /100 g		
Ca	.35	-.09	.05
Mg	.08	.46	.30
K	-.05	.75	.53
Na	-.17	1.00	.79
	Water Soluble, in me/L		
Ca	1.24	-.02	.01
Mg	1.34	.10	.07
K	.78	.25	.23
Na	.21	.53	.59
	Hot-Water Soluble, in ppm		
B	-.87	1.13	.28

Table 2.--Summary statistics for variables determined on mine-soil samples by Soltanpour's method and other standard methods, and data reported in the literature.

[In the present study, 205 samples were analyzed by each method; The data reported in the literature represent analyses of 481 samples; ---, not determined; me, milliequivalent (1 me = 1/1,000 of the equivalent weight).]

Variable, Unit of measure	Present Study						Reported[1]	
	Geometric mean	Geometric deviation	Observed range	Geometric mean	Geometric deviation	Observed range	Arithmetic mean	Observed range
	Soltanpour's DTPA Extract			Lindsay's DTPA Extract				
Cd, ppm	0.06	2.37	0.04 - .46	0.06	2.30	0.02 - .22	---	---
Co, ppm	.08	1.79	.06 - .8	.2	1.62	.1 - .7	---	---
Cu, ppm	2.4	1.93	.4 - 9.8	1.1	2.08	.2 - 6.7	[2]0.7	0.4 - 1.4
Fe, ppm	26	2.86	1.8 - 710	28	2.61	6.6 - 490	8.4	.1 - 40
Mn, ppm	6.5	1.65	.8 - 20	8.6	1.53	1.4 - 27	[2]4.3	1.3 - 11
Ni, ppm	1.0	2.05	.2 - 5.6	.9	1.84	.2 - 3.3	---	---
Pb, ppm	1.1	1.64	.3 - 4.4	.6	1.81	.1 - 2.9	---	---
Zn, ppm	1.4	2.78	.06 - 15	1.0	2.59	.1 - 9.5	1.4	.1 - 10
					Exchangeable			
Ca, me/100g	1.7	1.29	.3 - 3.7	21	1.82	4.2 - 88	---	---
Mg, me/100g	2.5	1.57	.8 - 6.8	4.8	1.71	.1 - 13	---	---
K, me/1000g	.5	1.61	.1 - 1.9	.5	1.59	.1 - 1.7	.7	.05 - 1.3
Na, me/100g	.2	2.86	.01 - 3.5	.2	2.54	.1 - 3.5	---	---
					Water-Soluble			
Ca, me/L	17	1.29	2.8 - 37	5.3	3.22	.1 - 47	---	---
Mg, me/L	25	1.57	7.6 - 68	3.5	3.32	.1 - 67	---	---
K, me/L	4.9	1.61	1.3 - 19	.5	2.47	.1 - 4.0	---	---
Na, me/L	1.6	2.86	.06 - 35	1.0	4.58	.1 - 43	---	---
					Hot-Water Soluble			
B, ppm	.14	4.43	.05 - 9.2	1.1	2.01	.5 - 26	---	---

[1] Soltanpour and Schwab, 1977

[2] 50 samples, rather than 481 samples were analyzed.

About 25 percent more Cu is extracted by Soltanpour's method than by Lindsay's method. The strong correlations established between Soltanpour's extract and the other methods for these metals will result in greater laboratory efficiency, because each sample will need fewer extractions for a given number of determinations and all constituents can be determined simultaneously by ICP-OES. Coefficients of determination show that between 50 and 60 percent of the total variation is explained for exchangeable K and water-soluble Na when measured by Soltanpour's extract, indicating that these equations are of marginal value for predictive purposes. For the remaining elements--DTPA-extractable Co, Mn, and Pb; exchangeable Ca and Mg; water-soluble Ca, Mg, and K; and hot-water-soluble B--the predition equations are not of much value, as shown by the low coefficients of determination. Even though many of the equations are of limited or no value, the data on the range for each constituent in western energy regions mine soil should be of use. Further research is needed to determine if the Soltanpour method will be a useful soil test for predicting plant growth and metal uptake on reclaimed mine land. The data provided here will provide an indication of the ranges in concentration to be expected in mine soils and may aid laboratory researchers in designing their experiments to encompass these ranges.

The following table shows the slopes of the prediction equations for untransformed and log-transformed data for the present study and the slopes for the untransformed data reported by Soltanpour and Schwab (1977).

Variable	Present Study		Soltanpour
	log-transformed	untransformed	untransformed
Cu	0.76	1.43	1.69
Fe	.97	1.17	.86
Mn	.76	.71	.81
Zn	1.00	1.36	1.29
K	.75	.94	.70

The slopes reported by Soltanpour are similar to those for the untransformed data from the present study, except for Fe and K. However, the slopes based on the log-transformed data differ greatly from the untransformed data of the present study and from those reported by Soltanpour, except for Mn and K. The reasons for these differences have been explained previously in the "Statistical Analysis" section. For the constituents in the table above, and for those in table 1, the prediction equations based on transformed data are probably more realistic than those based on untransformed data, because each sample used in the calculation had a more equal weighting following transformation than it would have had if untransformed data were used. The similarity in slope for transformed and untransformed Mn data can be explained by the fact that these data fit normal and lognormal distributions about equally well. The appropriate distribution for Soltanpour's data used to compute the slopes in the above table cannot be determined from the information provided. Furthermore, it cannot be assumed that the distributions are similar to those of the present study, because the present study used samples of unfertilized reclaimed mine spoil while Soltanpour used samples of fertilized agricultural soils.

Relative Precision

Fourteen of the samples were randomly selected and split for duplicate determinations by each of the methods. All sample preparation and laboratory procedures necessary to obtain a value for an element's concentration in a sample are included in this estimate of analytical error variance. The *F* ratios for precision between Soltanpour's method and the other standard methods are as follows:

Variable	*F* ratio	Variable	*F* ratio	Variable	*F* ratio
Cd (DTPA)	*3.6*	Pb (DTPA)	9.5	Ca (w.s.)	45
Co (DTPA)	3.2	Zn (DTPA)	*1.2*	Mg (w.s.)	45
Cu (DTPA)	*1.6*	Ca (ex.)	4.6	K (w.s.)	11
Fe (DTPA)	*1.1*	Mg (ex.)	128	Na (w.s.)	16
Mn (DTPA)	*3.9*	K (ex.)	2.6	B (h.w.)	*17*
Ni (DTPA)	5.2	Na (ex.)	3.0		

(DTPA), DTPA extractable; (ex.), exchangeable; (w.s.), water-soluble; (h.w.), hot-water extractable; underscored values indicate that Soltanpour's method is less precise than the standard method, and vice versa for those without underscoring; the critical *F* value is 6.4 at a 0.05 probability level

Soltanpour's method is significantly less precise than the standard methods for B only, whereas the standard methods for DTPA-extractable Pb, exchangeable Mg, and all water-soluble constituents are significantly less precise than the Soltanpour method. As mentioned previously, such ratios are only valid if homogeneous variance can be shown to occur between the samples analyzed by each method. Homogeneity is indicated if the geometric deviations (table 2) are similar for the two methods being compared. Deviations for water-soluble Ca, Mg, K, and Na, and hot-water-soluble B are not similar, and, therefore, these constituents do not meet the requirement of homogeneous variance. For constituents with heterogeneous variance, both heterogeneous variance and relative precision are included in the _F_-ratios; thus, little inference can be made about the relative precision of each method for water-soluble cations and B. Deviations are very close for the other constituents (table 2). Consequently, the _F_-ratios presented in the above table should reflect the relative precision of the various methods for each constituent. Soltanpour's DTPA method of extraction provides results with precision similar to Lindsay's DTPA method, with the exception of Pb, and similar to the method for exchangeable cations, with the exception of Mg.

Scatter Diagrams

Scatter diagrams are useful for determining visually whether a low coefficient of determination might be due to a nonlinear relation between constituents, a few random data "outliers" reflecting errors in sample preparation and analysis, a group of data "outliers" reflecting a peculiarity of a group of samples, or excessive scatter reflecting the inadequacy of the method for the constituent. Figures 3 through 19 (following the bibliography) show scatter diagrams for each of the constituents considered in this study. Scatter diagrams for those constituents having a large enough coefficient of determination to indicate that prediction equations may be useful (Fe, fig. 3; Cd, fig. 4; Cu, fig. 6; Ni, fig. 8; Zn, fig. 10; and Na, fig. 14) show a relatively close grouping of points throughout the range of the data. The diagrams also indicate that a linear model is appropriate to describe the relation. The few data points with large deviations from the imaginary regression line in each of these figures have been determined to most probably be due to some laboratory or analytical error, rather than to differences in amounts extracted from the same sample by the different methods. This determination was made by comparing the data for the deviating sample with the rest of the samples collected at the same strip mine. Samples collected at each mine all tend to show similar concentrations, and therefore, an anomalous value for a single sample determined by only one method suggests can error in some calculation or transcription of the data.

Samples showing large deviations in the scatter diagram for exchangeable K (fig. 13) are the result of errors in analysis, as described above. However, the general scatter of data points about the imaginary regression line is probably due to the differences in samples and the different extractants. Soltanpour's method, on the average, extracted an amount of K similar to that determined to be exchangeable (table 2). However, at the lower end of the range Soltanpour's method extracts more K than is measured as exchangeable, and the data show more scatter about the imaginary regression line than at the upper end of the range. Perhaps Soltanpour's method extracts exchangeable K plus K from some other soil constituent. The amount from the "other" constituent markedly affects the sum total K concentration, (exchangeable plus "other"), at the low end of the range but does not have as great an influence at the high end of the range. The "other" constituent may also be more consistent from sample to sample and may account for some of the large scatter of data points observed in fig. 13, because the precision of the two methods is similar. (See table in the "Relative Precision" section.)

Of the remaining constituents with homogeneous variance between samples (Co, fig. 5; Mn, fig. 7; Pb, fig. 9; Ca, fig. 11; and Mg, fig. 12), only Pb and Mg exhibit significant differences in analytical precision. From scatter diagrams, it is not possible to observe this difference. Again, any samples showing large deviations from the imaginary regression lines in these figures are probably due to the analytical errors discussed previously. The large scatter observed throughout the range in each of these figures probably indicates that each method acted differently on each sample--that is, each extractant reacted with several different soil constituents and probably with differing effectiveness on each.

Soltanpour's method extracted similar amounts of Ca (figs. 11 and 15) from all samples, regardless of the amount measured as exchangeable or water-soluble. The constant amount of Ca extracted from sample to sample by Soltanpour's method is probably due to the limited solubility of Ca in a solution of carbonates as Ca^{2+} and $CaCO_3^{o}$. The solution will rapidly become saturated with $CaCO_3(s)$ and the solubility of the carbonate will be the controlling factor in the amount of Ca measured in the solution (Garrels and Christ, 1965, p. 74-92).

SUMMARY

Prediction equations for the relations between Soltanpour's and Lindsay's DTPA methods for Cd, Cu, Fe, Ni, and Zn; and between Soltanpour's method and the exchangeable Na determination procedure indicate in each case that either method may be used for these constituents in soil with some confidence that the chosen method will correlate with the other. The prediction equations presented in this report show slopes and coefficients of determination similar to those presented by Soltanpour and Schwab (1977) for untransformed Cu, Mn, and Zn data. When the data are transformed by the common logarithm, the slopes of the prediction equations differ greatly. A log transformation was deemed necessary for the constituents measured on topsoil and spoil samples in the present study, because histograms of the data for each constituent were positively skewed. For the remaining constituents (DTPA-extractable Co, Mn, and Pb; exchangeable Ca, Mg, and K; water-soluble Ca, Mg, K, and Na; and hot-water-soluble B), a poor relation to Soltanpour's DTPA extraction was found.

Even though there are similarities for some constituents and differences for other constituents when determined by the different methods, the value of each method will depend on how well it predicts a constituent's availability--that is, how well the amount in soil correlates with plant uptake or the amount measured in plant tissue. Many of the proven agronomic soil tests have been applied to the evaluation of native soils and plants or to rehabilitated mine soil and reclamation plants without adequate basic research to determine their validity. Because these soil tests have not been adequatly tested as measures of plant uptake, the prediction equations reported here may be only of secondary importance. However, the observed range measured for each constituent should be of primary importance for those persons conducting basic research. The data on observed ranges for the constituents in topsoil and spoil from the western energy regions will give the researcher information necessary for designing experiments that will encompass this range.

The methods of Soltanpour and Lindsay provide similar relative precision of duplicate analyses for all DTPA-extractable metals except Pb. For Pb, Soltanpour's method is more precise than Lindsay's method for these samples of topsoil and spoil. Similarly, Soltanpour's DTPA extract method was found to be about as precise as the other procedure for exchangeable Ca, K, and Na, but not for Mg. Soltanpour's method was more precise for Mg. Inhomogeneous variance between samples for water-soluble Ca, Mg, K, Na, and B invalidates any estimate of precision between methods.

Scatter diagrams were useful for determining which relations would be better represented by nonlinear equations, which relations were poor because of a peculiarity of a group of samples, and which relations were poor because of some laboratory or analytical errors. Linear equations appear adequate to describe relations between the two methods for all constituents. The excessive scatter about the regression line for many constituents appears to result both from variance between methods and variance between samples, indicating that the effectiveness of each extractant differed with different soil constituents. In general, data for a few samples for each constituent showed errors in analysis, probably in transcription of data. It is generally difficult to distinguish between these errors and differences in amounts extracted from the same sample by different methods when only a single analysis of the sample is available. Because of this difficulty, the values suspected of being in error were not removed from the analysis or corrected before statistical analysis. We are, however, reasonably certain that laboratory or analytical errors can be identified here because all samples collected from a single coal strip mine had similar concentrations of each constituent.

REFERENCES CITED

Berg, W. A., 1978, Limitations in the use of soil tests on drastically disturbed land, _in_ Schaller, F. W., and Sutton, Paul, eds., Reclamation of drastically disturbed land: Madison, Wisc., American Society of Agronomy, p. 653-664.

Bower, C. A., and Wilcox, L. V., 1965, Soluble salts, _in_ Methods of soil analysis, pt. 2, Chemical and microbiological properties: Madison, Wisc., American Society of Agronomy p. 933-951.

Chapman, H. D., 1965, Cation-exchange capacity, _in_ Methods of soil analysis, pt. 2, Chemical and microbial properties: Madison, Wisc., American Society of Agronomy p. 891-901.

Cope, J. T., Jr., and Rouse, R. D., 1973, Interpretation of soil test results, _in_ Walsh, L. M, and Beaton, J. D., eds., Soil testing and plant analysis: Madison, Wisc., Soil Science Society of America, p. 35-54.

Crock, J. G., and Severson, R. C., 1980, Four reference soil and rock samples for measuring element availability in the western energy regions: U.S. Geological Survey Circular 841, 16 p.

Garrels, R. M., and Christ, C. L., 1965, Solutions, minerals, and equilibria: New York, Harper and Row, 450 p.

Lindsay, W. L., and Norvell, W. A., 1969, Development of a DTPA micronutrient soil test: Madison, Wisc., American Society of Agronomy, Agronomy Abstracts, p.84.

________1978, Development of a DTPA soil test for Zn, Fe, Mn, and Cu: Soil Science Society of America Journal, v. 42, p. 421-428.

Melsted, S. W., and Peck, T. R., 1973, The principles of soil testing, in Walsh, L. M., and Beaton, J. D., eds., Soil testing and plant analysis: Madison, Wisc., Soil Science Society of America, p. 13-22.

Olsen, S. R., and Dean, L. A., 1965, Phosphorus, in Methods of soil analysis, pt. 2, Chemical and microbiological properties: Madison, Wisc., American Society of Agronomy p. 1035-1049.

Pratt, P. F., 1965, Potassium, in Methods of soil analysis, pt. 2, Chemical and microbiological properties: Madison, Wisc., American Society of Agronomy, p. 1022-1030.

Soltanpour, P. N., and Schwab, A. P., 1977, A new test for simultaneous extraction of macro- and micro-nutrients in alkaline soils: Communications in Soil Science and Plant Analysis, v. 8, no. 3, p. 195-207.

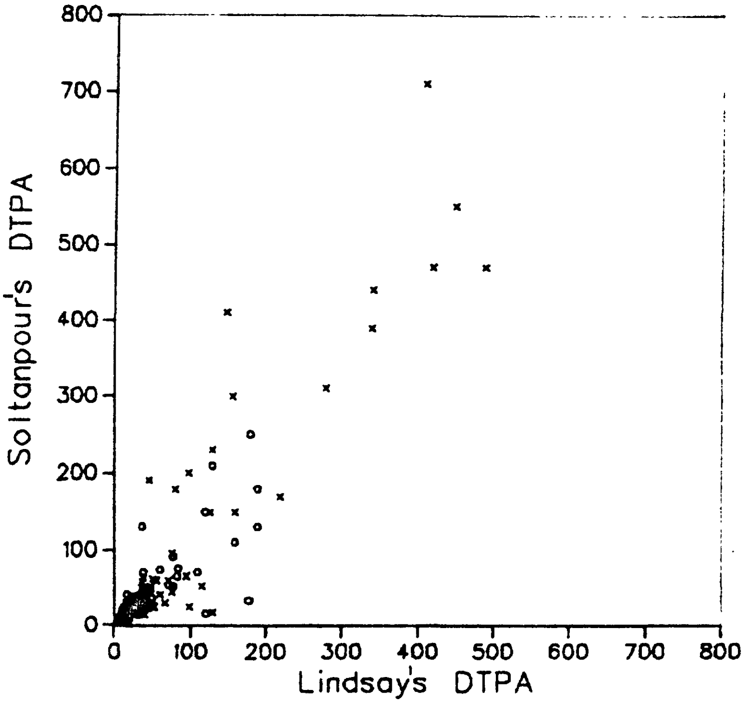

Figure 2.--Scatter diagram showing the relation between two methods for extractable Fe, in parts per million, on an arithmetic scale.

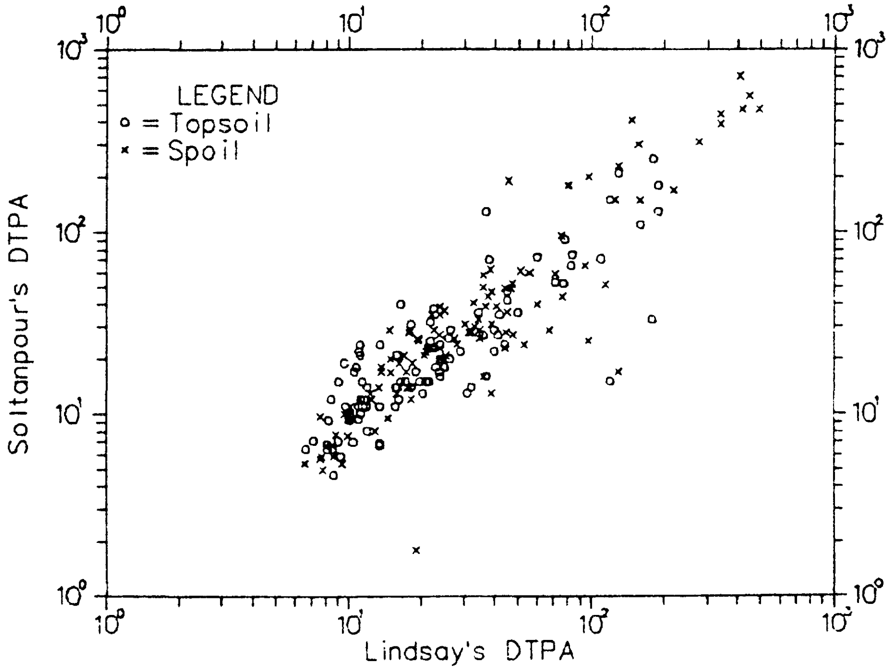

Figure 3.--Scatter diagram showing the relation between two methods for extractable Fe, in parts per million, on a logarithmic scale.

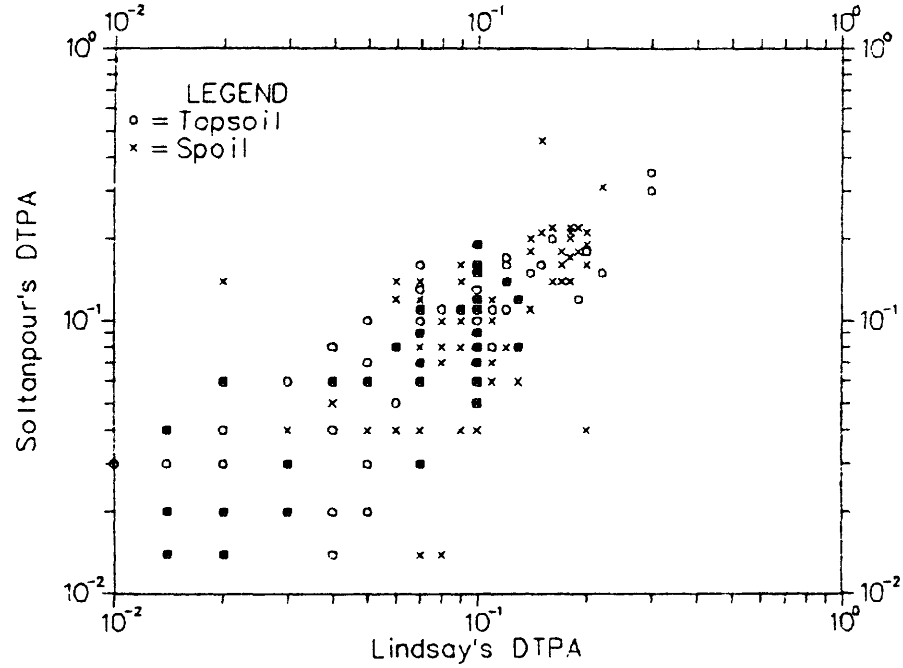

Figure 4.--Scatter diagram showing the relation between two methods for extractable Cd, in parts per million, on a logarithmic scale.

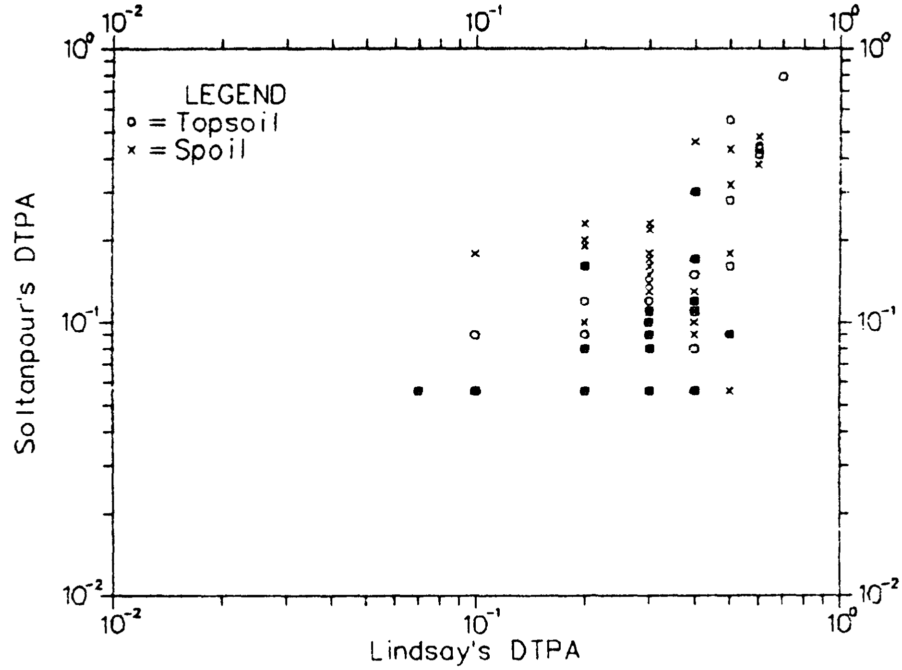

Figure 5.--Scatter diagram showing the relation between two methods for extractable Co, in parts per million, on a logarithmic scale.

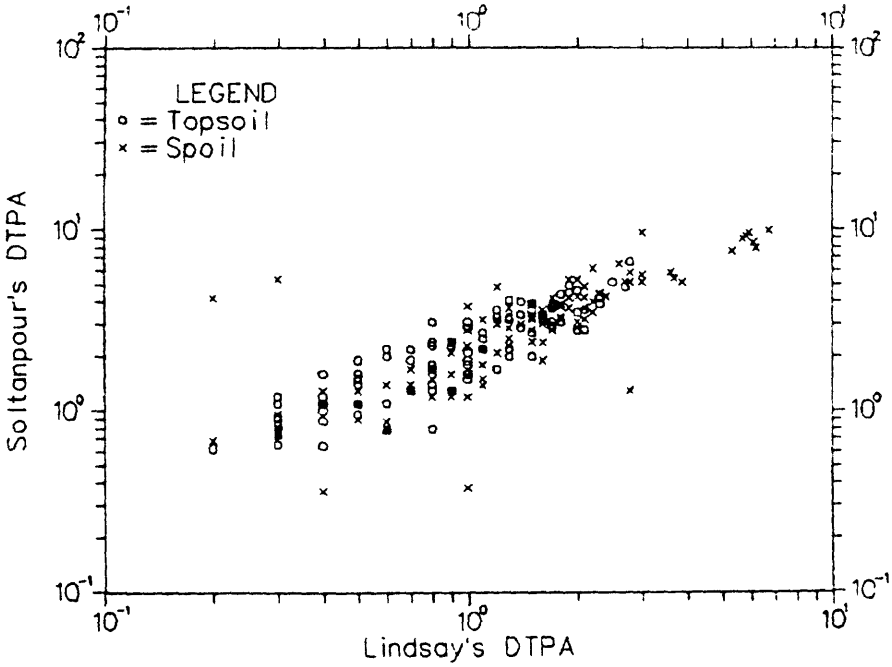

Figure 6.--Scatter diagram showing the relation between two methods for extractable Cu, in parts per million, on a logarithmic scale.

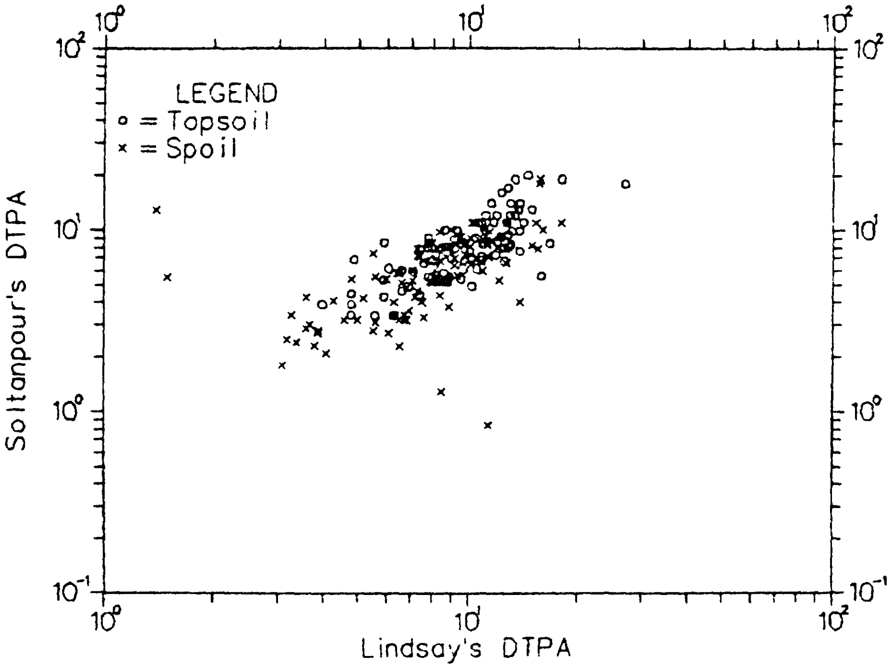

Figure 7.--Scatter diagram showing the relation between two methods for extractable Mn, in parts per million, on a logarithmic scale.

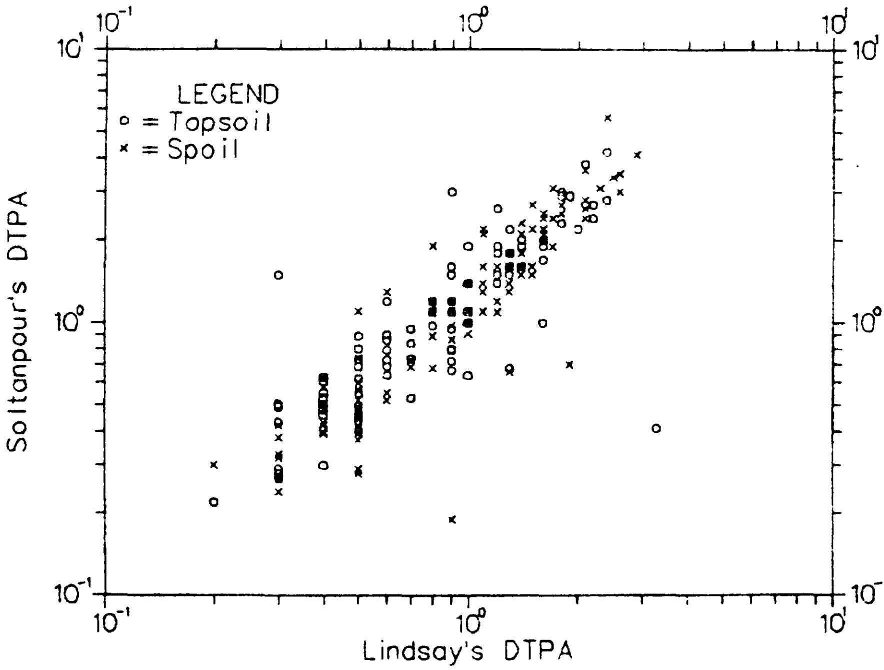

Figure 8.--Scatter diagram showing the relation between two methods for extractable Ni, in parts per million, on a logarithmic scale.

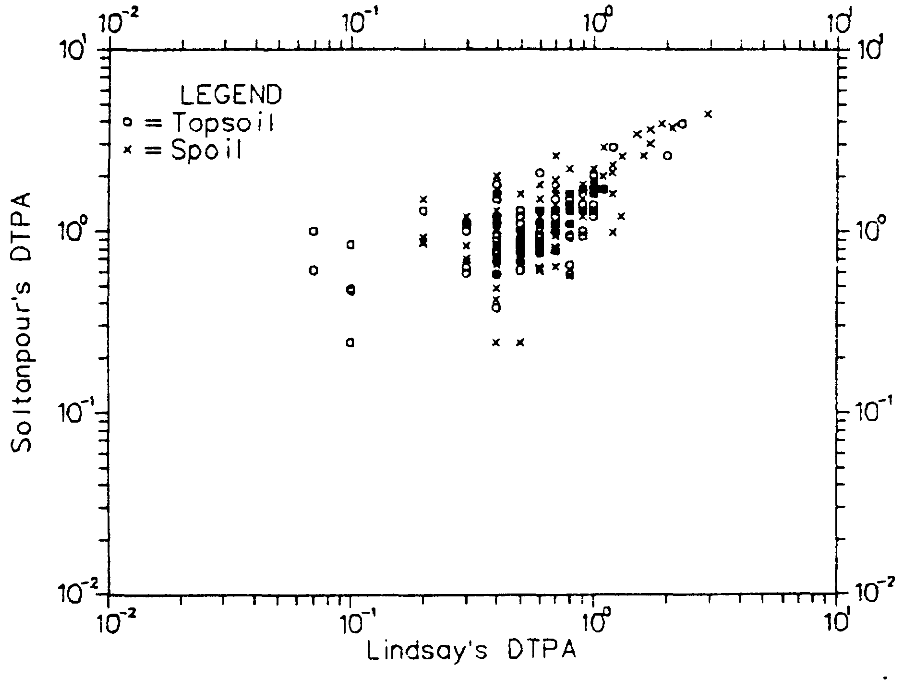

Figure 9.--Scatter diagram showing the relation between two methods for extractable Pb, in parts per million, on a logarithmic scale.

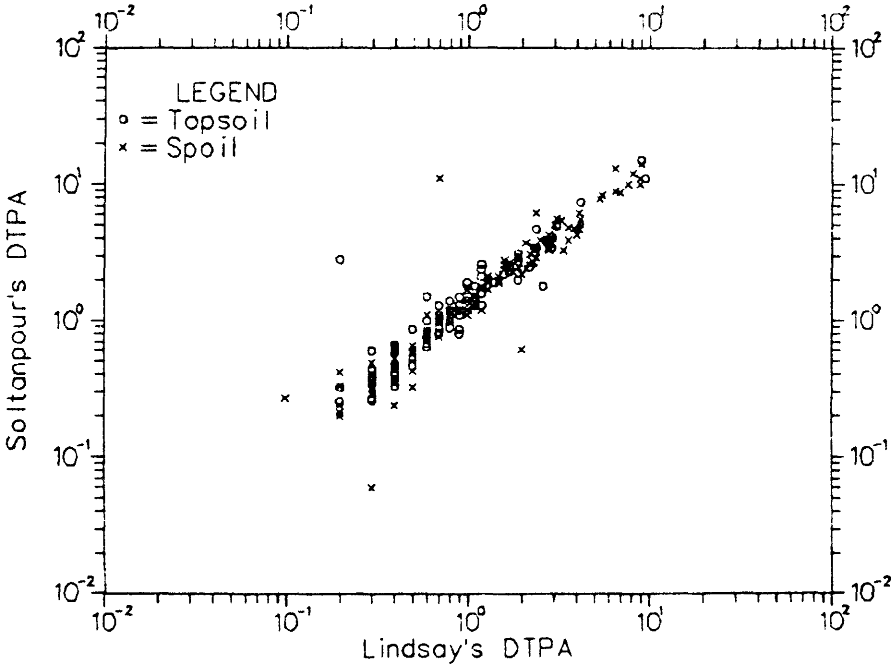

Figure 10.--Scatter diagram showing the relation between two methods for extractable Zn, in parts per million, on a logarithmic scale.

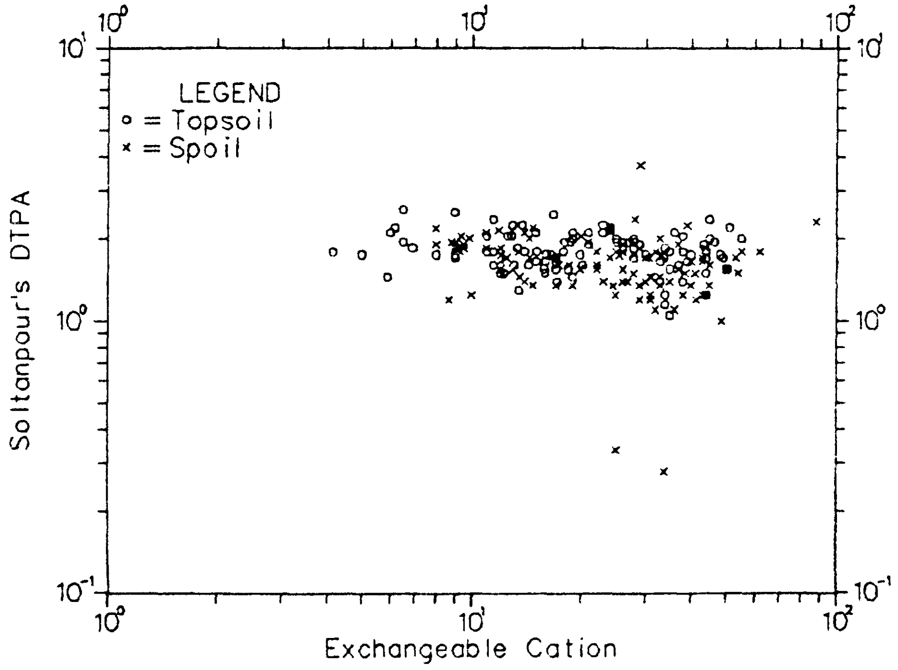

Figure 11.--Scatter diagram showing the relation between methods for exchangeable and extractable Ca, in me/100g, on a logarithmic scale.

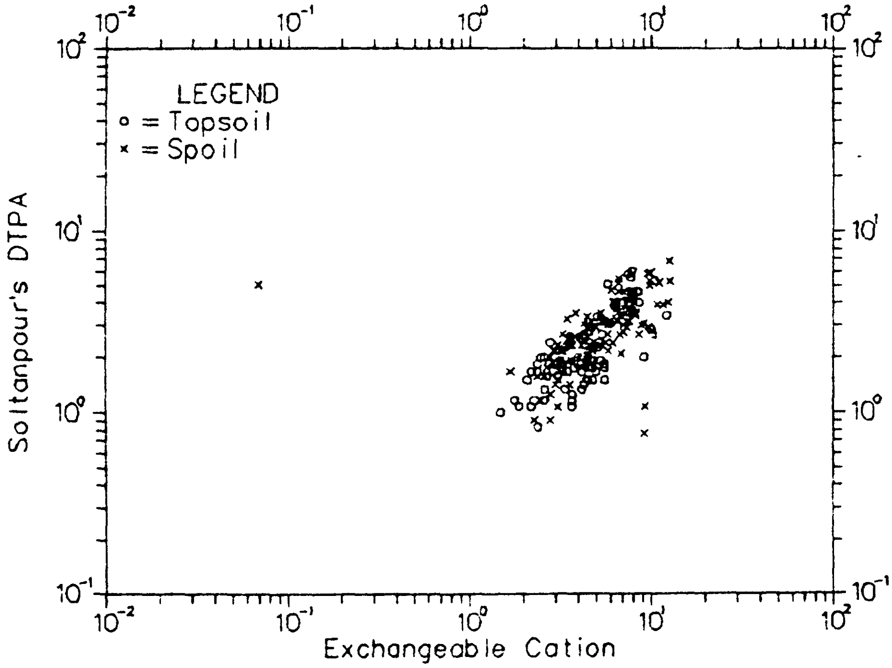

Figure 12.--Scatter diagram showing the relation between methods for exchangeable and extractable Mg, in me/100g, on a logarithmic scale.

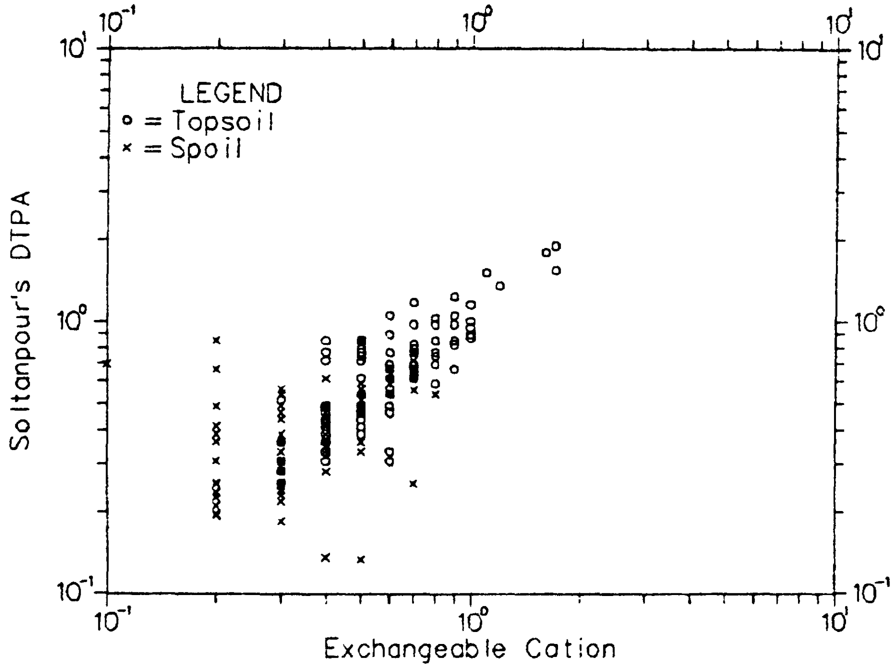

Figure 13.--Scatter diagram showing the relation between methods for exchangeable and extractable K, in me/100g, on a logarithmic scale.

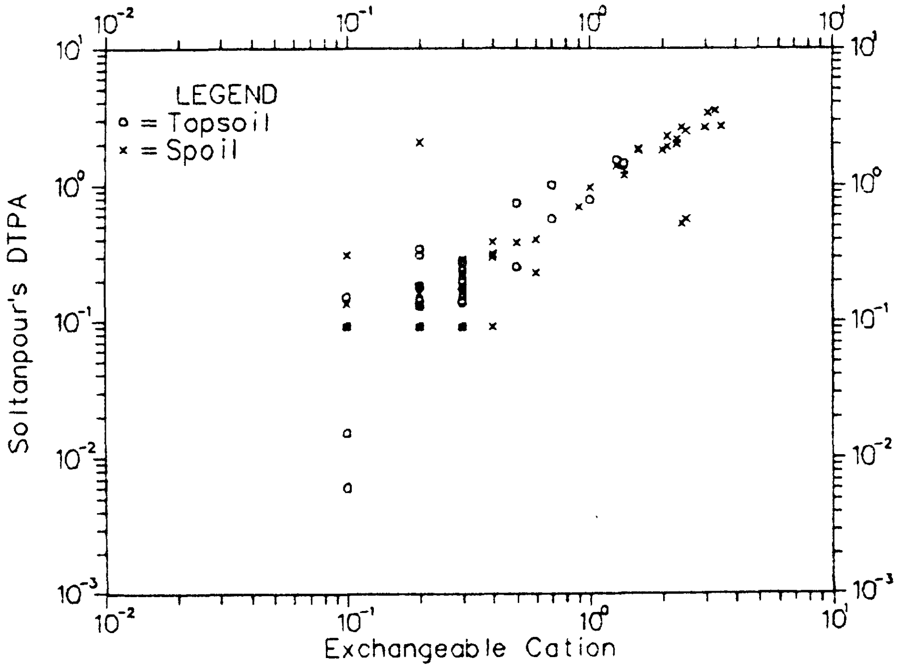

Figure 14.--Scatter diagram showing the relation between methods for exchangeable and extractable Na, in me/100g, on a logarithmic scale.

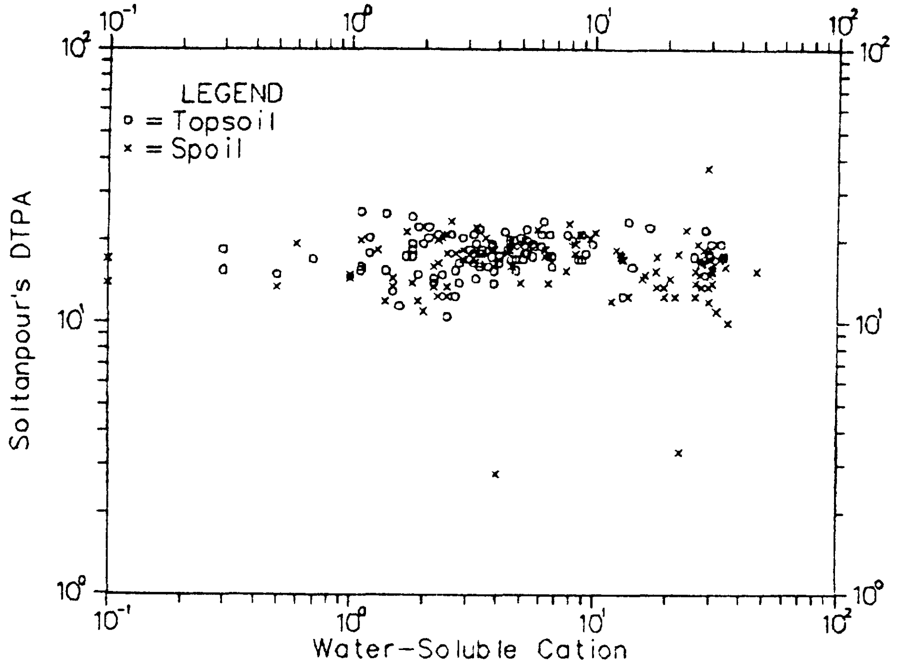

Figure 15.--Scatter diagram showing the relation between methods for water-soluble and extractable Ca, in me/L, on a logarithmic scale.

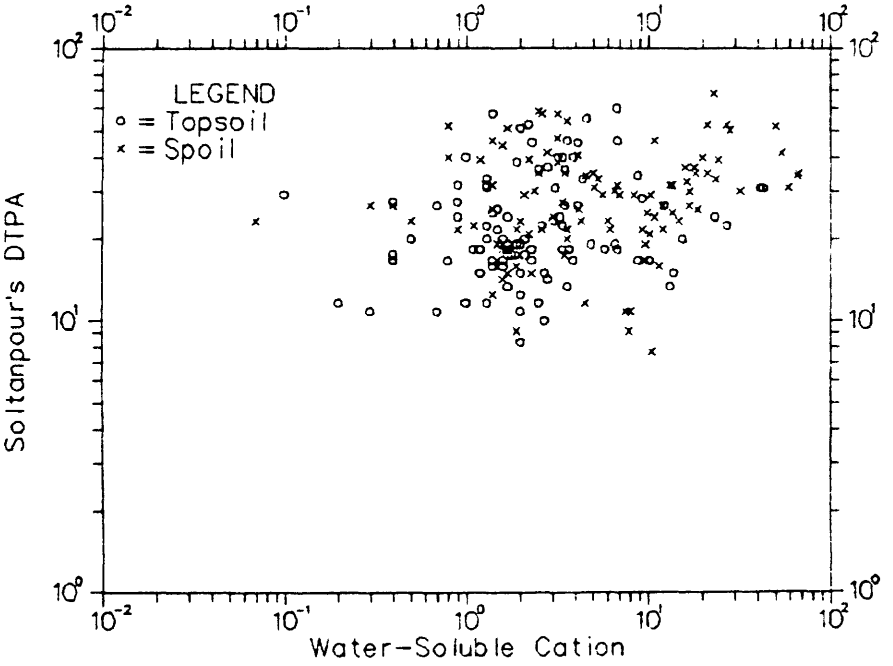

Figure 16.--Scatter diagram showing the relation between methods for water-soluble and extractable Mg, in me/L, on a logarithmic scale.

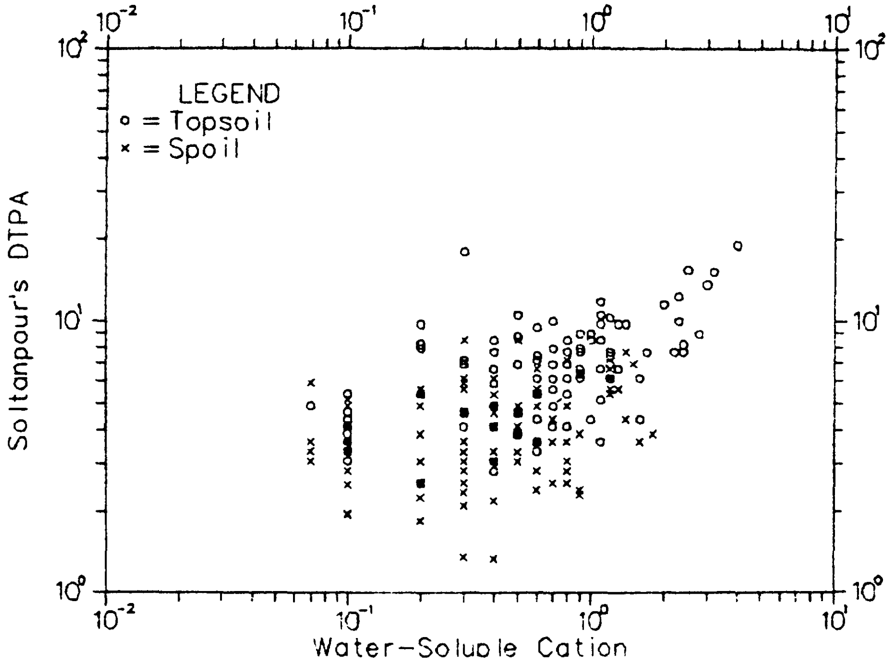

Figure 17.--Scatter diagram showing the relation between methods for water-soluble and extractable K, in me/L, on a logarithmic scale.

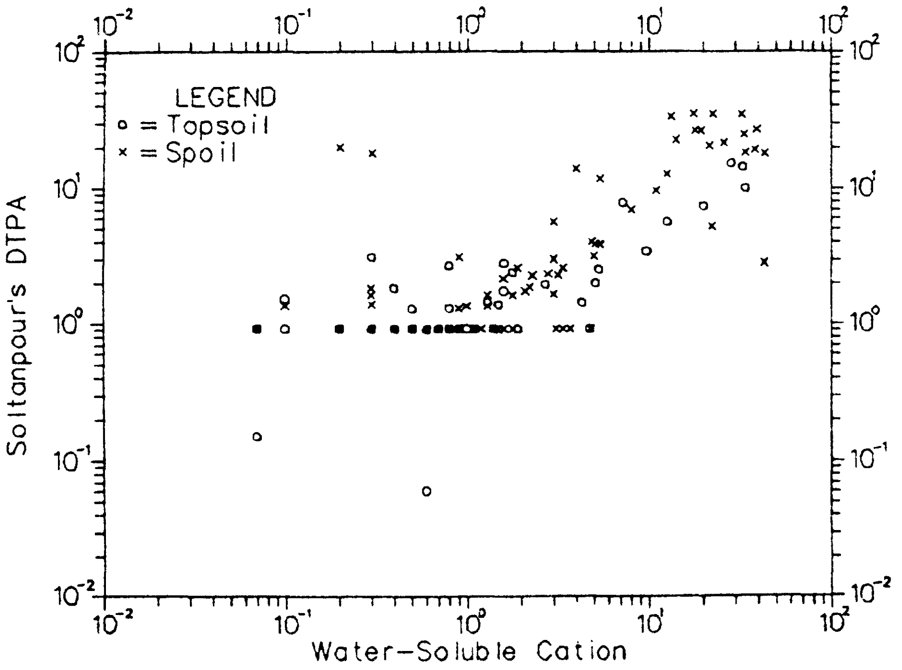

Figure 18.--Scatter diagram showing the relation between methods for water-soluble and extractable Na, in me/L, on a logarithmic scale.

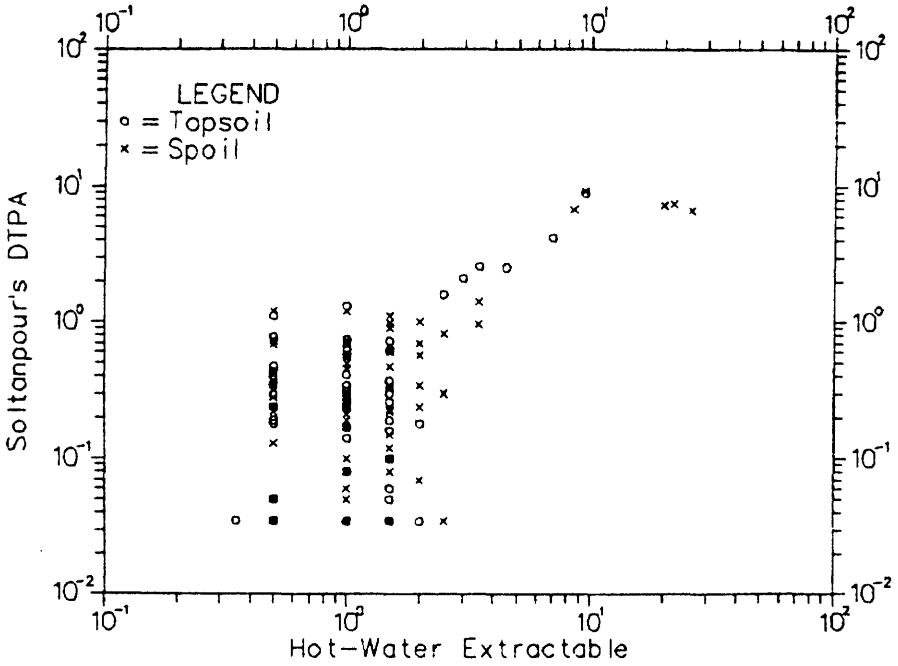

Figure 19.--Scatter diagram showing the relation between methods for hot-water-soluble and extractable B, in parts per million, on a logarithmic scale.