Agrosystems

Comparison of Soil-Test Extractants for Potassium, Calcium, Magnesium, Sulfur, and Micronutrients in Idaho Soils

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Core Ideas

- Multinutrient extractants were correlated to standard tests on alkaline calcareous soils
- For the tested nutrients Mehlich-3 generally extracted greater and H3A less than the common tests used in the Western United States
- Mehlich-3 extracted large amounts of Ca when inorganic-C as $CaCO_3$ was greater than 3.3 g kg^{-1} resulting in poor correlation with the ammonium acetate test

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ABSTRACT

Soil fertility and nutrient management programs across Idaho and the western United States should consider the effective comparison of various extractants for nutrient analyses. Common extractants for K, Ca, Mg, S, and micronutrients (Zn, Cu, Mn, Fe, B, Na) vary (e.g., ammonium acetate [AA], Olsen, etc.). The desire to develop effective correlation equations among common tests in the region and those of multinutrient extractants used or proposed in other geographical regions (i.e., Mehlich-3 [M-3] and Haney, Haney, Hossner, Arnold [H3A]) has increased due to the interest in nutrient management planning and in relation to soil health measurements. To investigate these multinutrient tests, 46 primarily alkaline soils were sampled from the 0- to 30-cm depth in agricultural fields in Idaho. Linear regressions between AA and Olsen K and both M-3 and H3A resulted in a significant positive fit (R^2 = 0.69–0.90). Notable issues with increased Ca concentrations occurred in soils with large amounts of inorganic C. Removal of these samples improved the relationship between standard and multinutrient soil tests. Specific micronutrient regressions were problematic when both alkaline and acidic soils were analyzed but improved when separated. Mehlich-3 and H3A extractants were generally well correlated, with the exceptions noted above, to common extractants used in western soils. Thus, this research provides specific correlation equations that could be used for comparison among tests as well as provides evidence of the potential suitability of multinutrient extractants in the region.

Abbreviations: AA, ammonium acetate; AAS, atomic adsorption spectrophotometer; DTPA, diethylene triamine pentaacetic acid; EDTA, ethylene diamine tetra acetic acid; H3A, Haney, Haney, Hossner, Arnold; ICAP-AES, inductively coupled argon plasma–atomic emission spectroscopy; IC, inorganic carbon; M-3, Mehlich-3; NLIN, proc nonlinear split line model; SE, standard error; SOM; soil organic matter.

Soil testing is the primary means to evaluate soil fertility status, nutrient management strategies, and environmental stewardship. Chemical extractions represent the main method used and vary based on the nutrient of interest as well as the inherent properties of the soil being analyzed (Miller et al., 2013; Martins et al., 2015). A wide range of soil tests with different extraction properties have been developed or proposed for recommendations in soils and crops found in the United States (e.g., Olsen et al., 1954; Doll and Lucas, 1973; Lindsay and Norvell, 1978; Mehlich, 1984; Haney et al., 2006). These soil tests provide an index of nutrient availability, and agronomic recommendations are determined based on field correlation and calibration trials for specific crops (Brown, 1987). Comparisons among tests is often impossible, as only the specific test used in the region at the time of development was investigated, thus making comparison or establishment of new tests difficult due to the amount of research efforts needed to correlate and calibrate the tests again.

In the western United States, a large number of soil-extractant procedures are used for the determination of soil nutrient status for agronomic recommendations. These include Olsen and ammonium acetate (AA) for K; AA for Ca, Mg, and Na; turbidimetry for S; diethylene triamine pentaacetic acid (DTPA) for Cu, Fe, Mn, and Zn; and hot-water extraction for B (Miller et al., 2013). The neutral AA extraction (Knudsen et al., 1982) has historically been one of the most widely used extractants for K, Ca, Mg, and Na. In the western United States, many soils are alkaline and have appreciable amounts of inorganic C as $CaCO_3$; therefore, an adjustment of the buffer to pH 8.5 was established to avoid the dissolution of $CaCO_3$ into the solution (Miller et al., 2013; Dari et al., 2019). The Olsen extractant (Olsen et al.,1954) was developed for the measurement of P on high-pH soils where

Table 1. Site description of soil samples used to evaluate soil P test indices as collected at a depth of 0 to 30 cm from agricultural fields in Idaho (Dari et al., 2019).

Identification	Series†	Taxonomic classes of soil	Previous crop‡
		Southern Idaho	
1	Portneuf SiL	coarse-silty, mixed, superactive, mesic Durinodic Haplocaldids	Sugarbeet
2	Sluka SiL	coarse-silty, mixed, superactive, mesic Xeric Haplodurids	Sugarbeet
3	Portneuf SiL	coarse-silty, mixed, superactive, mesic Durinodic Haplocaldids	Sugarbeet
4	Power McCain complex	Power: fine-silty, mixed, superactive, mesic Xeric Calciargids McCain: fine-silty, mixed, superactive, mesic petronodic xeric calcargid	Sugarbeet
5	Sluka SiL	coarse-silty, mixed, superactive, mesic Xeric Haplodurids	Potato
6	Power SiL	fine-silty, mixed, superactive, mesic Xeric Calciargids	Potato
7	Portneuf SiL	coarse-silty, mixed, superactive, mesic Durinodic Haplocaldids	Potato
8	Bahem SiL	coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Potato
9	Declo L	coarse-loamy, superactive, mesic Xeric Haplocalcids	Sugarbeet
10	Fulmer SiL	fine-loamy, mixed, superactive, calcareous frigid Typic Endoaquaolls	Barley
11	Portino SiL	coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Wheat
12	Declo L	coarse-loamy, superactive, mesic Xeric Haplocalcids	Alfalfa
13	Declo L	coarse-loamy, superactive, mesic Xeric Haplocalcids	Wheat
14	Declo L	coarse-loamy, superactive, mesic Xeric Haplocalcids	Oat
15	Declo L	coarse-loamy, superactive, mesic Xeric Haplocalcids	Oat
16	Declo L	coarse-loamy, superactive, mesic Xeric Haplocalcids	Oat
17	Bahem SiL	coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Alfalfa
18	Bahem SiL	coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Potato
19	Rad SiL	coarse-silty, mixed, superactive, mesic Durinodic Xeric Haplocambids	Dry bean
20	Rad SiL	coarse-silty, mixed, superactive, mesic Durinodic Xeric Haplocambids	Corn
21	Portneuf SiL	coarse-silty, mixed, superactive, mesic Durinodic Haplocaldids	Sugarbeet
22	Picabo SiL	coarse-silty, caronatic, frigid, oxyaquic Calcixerolls	Alfalfa
23	Hapur–Picabo SiL	Hapur: fine-loamy, frigid Typic Calciaquolls Picabo: coarse-silty, caronatic, frigid, oxyaquic Calcixerolls	Barley
24	Molyneux L	fine-loamy, mixed, superactive, frigid Ultic Argixerolls	Barley
25	Nyssaton SiL	coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Corn
26	Greenleaf–Owyhee complex	Greenleaf: fine-silty, mixed, superactive, mesic Xeric Calciargids Owyhee: coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Potato
27	Greenleaf–Owyhee complex	Greenleaf: fine-silty, mixed, superactive, mesic Xeric Calciargids Owyhee: coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Wheat
28	Nyssaton SiL	coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	Dry bean
29	Grassy Butte S	sandy, mixed, frigid Typic Haplocalcids	Barley
30	Diston loamy S	sandy, mixed, frigid, Xeric Haplodurids	Potato
31	Ririe SiL	coarse-silty, mixed, superactive, frigid Calcic Haploxerolls	Potato
32	Potell SiL	coarse-silty, mixed, superactive, calcareous, frigid Xeric Torriorthents	Potato
33	Pancheri SiL	coarse-silty, mixed, superactive, frigid Xeric Haplocalcids	Potato
34	Pancheri SiL	coarse-silty, mixed, superactive, frigid Xeric Haplocalcids	Potato
35	Harston FSL	coarse-loamy, mixed, superactive, calcareous, frigid Xeric Torrifluvents	Barley
36	Kucera–Ririe complex	Kucera: coarse-silty, mixed, superactive, frigid Calcic Pachic Haploxerolls Ririe: coarse-silty, mixed, superactive, frigid Calcic Haploxerolls	Fallow
37	Kucera–Ririe complex	Kucera: coarse-silty, mixed, superactive, frigid Calcic Pachic Haploxerolls Ririe: coarse-silty, mixed, superactive, frigid Calcic Haploxerolls	Fallow
38	Iphil–Lostine–Ririe complex	Iphil: coarse-silty, mixed, superactive, frigid Typic Calcixerolls Lostine: coarse-silty, mixed, superactive, frigid Pachic Haploxerolls Ririe: coarse-silty, mixed, superactive, frigid Calcic Haploxerolls	Fallow
39	Ririe–Lostine complex	Ririe: coarse-silty, mixed, superactive, frigid Calcic Haploxerolls Lostine: coarse-silty, mixed, superactive, frigid Pachic Haploxerolls	Barley
40	Arbone–Lostine complex	Arbone: coarse-loamy, mixed superactive, frigid, Calcic Haploxerolls Lostine: coarse-silty, mixed, superactive, frigid Pachic Haploxerolls	Fallow
41	Lostine–Foundem complex	Lostine: coarse-silty, mixed, superactive, frigid Pachic Haploxerolls Foundem: coarse-silty, mixed, superactive, Pachic Haploxerolls	Wheat

(continued)

Table 1. Continued.

Identification	Series†	Taxonomic classes of soil	Previous crop‡
		Northern Idaho	
42	Schnoorson SiL	fine-silty, mixed, active, calcareous, frigid Aeric Fluvaquents	Wheat
43	Nez Perce SiL	fine, smectitic, mesic Xeric Argialbolls	Wheat
44	Palouse–Latahco complex	Palouse: fine-silty, mixed, superactive, mesic Pachic Ultic Haploxerolls Latahco: fine-silt, mixed, superactive, frigid, Argiaquic Xeric Argialbolls	Wheat
45	Latahco–Thatuna complex	Latahco: fine-silt, mixed, superactive, frigid, Argiaquic Xeric Argialbolls Thatuna: fine-silty, mixed, superactive, mesic Oxyaquic Argixerolls	Wheat
46	Uhlorn–Nez Perce complex	Uhlorn: fine-silty, mixed, superactive, mexic Typic Argixerolls Nez Perce: fine, smectitic, mesic Xeric Arigalbolls	Barley

† SiL, silt loam; L, loam; FSL, fine sandy loam; S, sand.

‡ Sugarbeet, *Beta vulgaris* L.; potato, *Solanum tuberosum* L.; wheat, Triticum aestivum L.; alfalfa, *Medicago sativa* L.; oat, *Avena sativa* L.; dry bean, *Phaseolus vulgaris* L.; corn, *Zea mays* L.; barley, *Hordeum vulgare* L.

other tests (Bray and Kurtz, 1945) have noted difficulty in extracting P in alkaline calcareous soils (Ebeling et al., 2008; Dari et al., 2019). In the western United States, Olsen K extracts similar amounts of K as AA, and therefore many labs advantageously use this along with P evaluation. The calcium phosphate turbidimetric method for S is widely used; however, the procedure is difficult for inexperienced technicians and requires substantial repetition to be proficient (Beaton and Platou, 1968; Miller et al., 2013). The DTPA extractant was developed for neutral and calcareous soils. The DTPA extractant is buffered and contains $CaCl_2$ to minimize the dissolution of $CaCO_3$ and occluded nutrients into solution (Lindsay and Norvell, 1978). Hot-water extraction determines the amount of B based on extraction with $CaCl_2$ via the addition of azomethine-H for spectrophotometric analysis (Bingham, 1982; Miller et al., 2013). The large number of extractants, subsequent analyses, and needed instrumentation result in costly and on occasion untimely testing results for producers and a complex array of procedures for laboratories. Despite these issues and the continued efforts to determine a multinutrient extractant that is effective across a wide range of soils, a universal multinutrient extractant has not been established (Holford, 1980; Haney et al., 2006).

The M-3, Mehlich-3 (M-3) extractant was developed on neutral to acidic soils in the southeastern United States to alleviate many of the issues associated with multinutrient extractants and has been widely adopted in the southeastern region (Mehlich, 1984). However, in the western United States, where high pH alkaline and often calcareous soils are common, relatively little work has been conducted to determine the efficacy of the M-3 soil test for multinutrient extraction (Dari et al., 2019). The Haney, Haney, Hossner, Arnold (H3A) extractant, which is a component of the Soil Health Tool (SHT), was recently developed as an attempt to more accurately determine soil nutrient status based on the concept of extracting soil samples near the actual soil pH using organic acids that are naturally occurring from plant root exudates (Rengel, 2002; Baudoin et al., 2003, Haney et al., 2006; Haney et al., 2018). The initial extractant was comprised of lithium citrate, citric acid, malic acid, oxalic acid, ethylene diamine tetra acetic acid (EDTA), and DTPA (Haney et al., 2006). Subsequent revisions of the extractant resulted in the removal of the two synthetic chelators (EDTA and DTPA) and the removal of lithium citrate (Haney et al., 2010, 2017, 2018). The test was developed primarily for the extraction of inorganic N and soil P (Haney et al., 2006) and was revised to improve extraction of Ca, Fe, Al, and K (Haney et al., 2017) where the test is also being used by commercial laboratories to extract other nutrients.

Efforts to establish conversion equations for varying soil-test extractants to standardize soil testing methods and recommendations have previously been conducted in other regions (Wang et al., 2004). To date, no known published research has focused on K, Ca, Mg, S, and micronutrient (Na, Zn, Cu, Mn, Fe, and B) evaluations in predominately alkaline calcareous soils common to the western United States using the multinutrient M-3 and H3A extractants. Therefore, the objectives of our study were to: (i) evaluate the relationship and suitability of conversion equations for K, Ca, Mg, S, and micronutrients by AA, DTPA, Olsen, calcium phosphate turbidimetry, hot-water, M-3, and H3A extraction for a range of primarily alkaline soils collected from the 0- to 30-cm depth from agricultural fields throughout northern and southern Idaho; and (ii) determine whether M-3 and H3A correlation equations can be established and if the extractants can be considered as potential multinutrient extracts for soils in the region.

MATERIALS AND METHODS

Soil Characterization and Site Description

Soil series descriptions are presented in Table 1. Detailed physical and chemical characterization of the individual soils are presented in Dari et al. (2019) and mean values are presented in Table 2. Soil samples for the present study ($n = 46$) were collected in early spring 2015 from agricultural fields throughout northern and southern Idaho. An approximately 0.1-ha area was chosen at each location to collect four subsamples from a depth of 0- to 30-cm using a 7.6-cm bucket auger and composited. The 0- to 30-cm depth is common for the majority of fertilizer nutrient recommendations for various crops in Idaho; however, many of the tested nutrients have no defined recommendations at this time (Brown, 1996; Robertson and Stark, 2003; Stark and Westermann, 2003; Moore et al., 2009). The collected soil samples were dried at 40°C in a forced-convection oven, ground, and homogenized to pass through a 2-mm sieve.

The study region primarily includes the agricultural areas in the Snake River Plains and the Palouse and Nez Perce Prairies in southern and northern Idaho, respectively (USDA-NRCS, 2006). The predominant soil order in the study region in the Snake River plains are characterized by Aridisols where the average rainfall and annual temperature range from 180 to 305 mm, and 5 to 13°C, respectively. The average rainfall and annual temperature of the Palouse and Nez Perce Prairies are 330 to 710 mm, and 8 to 12°C, respectively, and the most common soil order in the region is Mollisols.

Table 2. Soil physicochemical (Dari et al., 2019) and soil tests values (mg kg^{-1}) for K, Ca, Mg, S, and micronutrients (Cu, Zn, Fe, Mn, Na, and B) for the soils collected at 0- to 30-cm depths (n = 46) from agricultural fields in Idaho.†

Parameters	Mean	SE	Min.	Max.
Soil, pH‡	7.9	0.1	5.6	8.6
EC, cmol$_c$ kg^{-1}	252	17	68	600
SOM§, g kg^{-1}	20	2	BDL	56
Inorganic C, g kg^{-1}¶	4.8	1.0	BDL	39
Sand, g kg^{-1}	273	27	56	894
Silt, g kg^{-1}	548	24	17	749
Clay, g kg^{-1}	178	9	20	400
	———————— mg kg^{-1} ————————			
Ammonium acetate extraction				
K	328	22	59	692
Ca	4434	172	2148	6364
Mg	415	21	114	697
Na	96	11	15	321
DTPA extraction				
Cu	1.3	0.1	0.3	2.7
Fe	24	5	4	137
Mn	23	3	4	101
Zn	2.1	0.2	0.2	4.8
Mehlich-3 extraction				
K	350	20	60	664
Ca	5678	459	2088	16040
Mg	569	33	184	1183
S	23	2	6	56
Cu	2.5	0.1	0.4	4.8
Fe	81	9	16	277
Mn	145	10	14	292
Zn	6.0	0.4	1.3	13.2
Na	74	10	7	276
B	1.8	0.1	0.5	4.4
H3A extraction				
K	123	11	20	664
Ca	2140	145	567	4183
Mg	261	16	112	631
S	18	2	3	56
Cu#	0.1	<0.1	BDL	0.5
Fe	30	4	2	105
Mn	11.6	1.5	0.4	38.4
Zn	0.5	0.1	BDL	1.8
Na	82	11	12	318
Olsen extraction				
K	251	19	50	635
Turbidimetric extraction				
S	15	1.6	4.2	47.0
Hot-water extraction				
B	0.5	0.1	0.3	1.4

† SE, standard error; SOM, soil organic matter; AA, ammonium acetate; DTPA, diethylene triamine penta acetic acid; M-3, Mehlich 3; H3A, Haney, Haney, Hossner, Arnold.

‡ The basic soil parameters (soil pH, EC, SOM, inorganic C, soil texture) were analyzed as per the methods described in Dari et al. (2019).

§ The sample detection limit for soil organic matter determination was 10 g kg^{-1}, with soils less than this value noted as below detection limit (BDL).

¶ The sample detection limit for inorganic C determination is 0.3 g kg^{-1}, with soils less than this value noted as BDL.

The sample detection limit for H3A Cu and Zn test was 0.1 mg kg^{-1}, with soils less than this value noted as BDL.

Table 3. Pearson correlations for various soil tests methods (AA, Olsen, DTPA, M-3, H3A) for K, Ca, Mg, S, and micronutrients (Cu, Zn, Fe, Mn, Na, and B) for the soils collected at 0- to 30-cm depths (n = 46) from agricultural fields in Idaho.†

Nutrients	Soil test	Pearson coefficient (r value)		
K		Olsen	H3A	M-3
	AA	0.96***	0.91***	0.94***
	Olsen		0.95***	0.90***
	M-3		0.89***	–
Ca		M-3	H3A	–
	AA	0.77**	0.86***	–
	M-3		0.88***	
Mg		M-3	H3A	–
	AA	0.91***	0.85***	–
	M-3		0.92***	
S		M-3	H3A	–
	Turbidimetry	0.84***	0.94***	
	M-3		0.84***	
Cu		M-3	H3A	–
	DTPA	0.80***	0.50**	
	M-3		0.52**	
Fe		M-3	H3A	–
	DTPA	0.86***	0.81***	
	M-3		0.94***	
Mn		M-3	H3A	–
	DTPA	−0.12	0.56**	
	M-3		0.53**	
Zn		M-3	H3A	–
	DTPA	0.89***	0.38*	
	M-3		0.28	
Na		M-3	H3A	–
	AA	0.98***	0.98***	
	M-3		0.99***	
B		M-3	–	–
	Hot-water	0.89***	–	–

* Significant correlations at P < 0.05.

** Significant correlations at P < 0.01.

*** Significant correlations at P < 0.001.

† AA, ammonium acetate; DTPA, diethylene triamine pentaacetic acid; M-3, Mehlich-3; H3A, Haney, Haney, Hossner, Arnold.

Soil Tests for K, Ca, Mg, S, and Micronutrients (Na, Zn, Cu, Mn, Fe, Al, and B)

For each sample and test combination duplicate soil samples were prepared, extracted, and analyzed per the procedures listed below where the mean of the duplicate values was used for all analyses.

Ammonium Acetate Test for K, Ca, Mg, and Na

The AA extraction was performed by shaking 2.5 g of soil with 25 mL of 1 mol L^{-1} ammonium acetate; NH$_4$OAc extraction solution (buffered to pH 8.5) in an orbital mechanical shaker for 30 min and filtered through Whatman filter paper no. 42. The concentrations of K, Mg, Ca, and Na in the soil extract were determined using an atomic adsorption spectrophotometer (AAS) (Miller et al., 2013).

DTPA Test for Zn, Fe, Cu, and Mn

The DTPA test was performed by shaking 10.0 g soil with 20 mL of 0.005 mol L^{-1} DTPA extraction reagent for 2 h at 25°C and 180 oscillations min^{-1} in a reciprocating mechanical shaker and
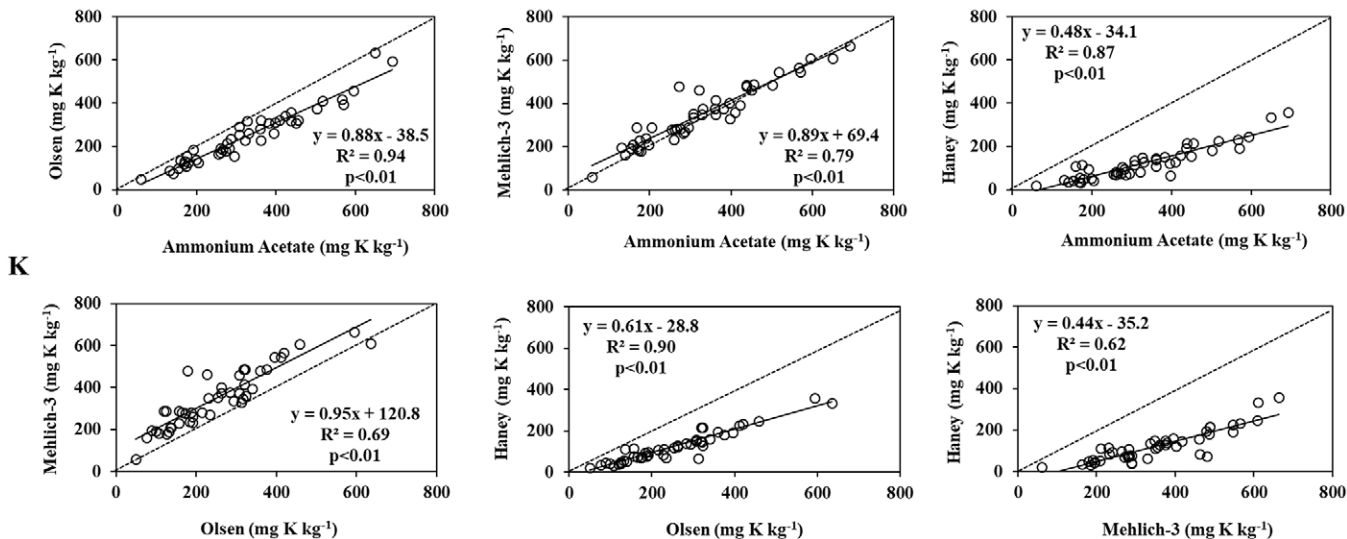
Fig. 1. Comparison of extractability of various soil test methods for K from soil samples collected at a sample depth of 0 to 30 cm from agricultural fields in Idaho. The solid line is the linear regression fit and the dotted line represents a 1:1 relationship between the tests being compared.

filtered through Whatman filter paper no. 42. The concentrations of Zn, Cu, Mn, and Fe were determined by analyzing the extracted solution using an AAS (Miller et al., 2013).

Mehlich-3 Test for K, Ca, Mg, S, and Micronutrients

Soil samples were extracted with M-3 extracting solution (0.2 mol L^{-1} acetic acid; CH_3COOH + 0.25 mol L^{-1} ammonium nitrate; NH_4NO3 + 0.015 mol L^{-1} ammonium fluoride; NH_4F + 0.013 mol L^{-1} nitric acid; HNO_3 + 0.001 mol L^{-1} EDTA buffered to pH 2.50) by shaking 2 g of soil with 20 mL of extractant for 5 min and concentrations of K, Ca, Mg, S, Fe, Mn, Cu, Zn, B, and Na in extracting solution were determined using an inductively coupled argon plasma atomic emission spectroscopy (ICAP-AES) (Mehlich, 1984).

Haney Test for K, Ca, Mg, S, and Micronutrients

Soil samples were analyzed for H3A extracted nutrients by weighing 4 g of sample and extracting with 40 mL of Haney extractant (three organic acids, i.e., 0.0024 mol L^{-1} citric acid, 0.004 mol L^{-1} oxalic acid, and 0.004 mol L^{-1} malic acid at pH 3.75 in plastic centrifuge tubes (Haney et al., 2017). Samples were then shaken for 10 min, centrifuged for 5 min, and subsequently filtered through Whatman 2V filter paper (GE Healthcare UK Ltd). The concentration of K, Ca, Mg, S, Fe, Mn, Cu, Zn, B, and Na were determined by ICAP-AES.

Olsen Test for K

Olsen K was determined by shaking 2.0 g soil with 40 mL Olsen extractant (0.5 mol L^{-1} sodium bicarbonate; $NaHCO_3$) for 30 min in a reciprocating mechanical shaker. After shaking, all samples were filtered through Whatman filter paper no. 42. Potassium in the extracts obtained by each method was determined spectrophotometrically on an automated flow injection analyzer (Olsen et al., 1954).

Calcium Phosphate Turbidimetric Test for S

Soil-extractable S was determined by shaking 10.0 g soil with 25 mL 0.08 mol L^{-1} calcium phosphate $(Ca(H_2PO_4))$ extraction solution for 30 min in a reciprocating mechanical shaker, 0.15 g of activated charcoal (Darco G-6 activated C) was added, shaken for

an additional 3 min, and filtered through Whatman filter paper no. 42. A 10.0-mL aliquot of soil extract was placed in a 25 mL flask, and 1.0 mL of "seed solution" (20 mg L^{-1} S in 5.8 mol L^{-1} HCl) was added and swirled. The container flask was placed on a magnetic stirrer and 0.3 g of $BaCl_2 \cdot 2H_2O$ crystals was added and stirred for 5 min. The concentration of SO_4–S in the soil extracts was determined by ICAP-AES (Miller et al., 2013).

Hot-Water Extraction Method for B

Hot-water–extractable B was determined by boiling 4.0 g soil with 20 mL 0.02 mol L^{-1} calcium chloride $(CaCl_2)$ extracting solution in an incubator at 80°C for 1 h. After boiling, the samples were allowed to filter for 10 min with the concentration of B in the soil extracts determined by ICAP-AES (Hill, personal communication, 2013; Miller et al., 2013).

Statistical Analyses

As noted above, duplicate analyses were conducted for soil tests for each location where the mean value was used for all statistical analyses. Pearson correlations and regression models were conducted to determine the relationship among various soil test methods using SigmaPlot 13.0 (SYSTAT). A proc nonlinear split line (NLIN) model was fit using SAS 9.3 (SAS Institute) to determine the change point in inorganic carbon (IC) of M-3 Ca (McDowell and Sharpley, 2001; Casson et al., 2006; Chakraborty et al., 2011). The change point in the fitted segmented-nonlinear model was directly estimated and the 95% confidence interval reported. The slope obtained from the left-hand line was estimated as a function of the change point and other model parameters to ensure that the two-line segments joined at that particular change point.

RESULTS AND DISCUSSION

The soils were typical of those found in the major agricultural production area across Idaho and were predominately silt loam soils (Table 1). The range of values of K, Ca, Mg, S, and micronutrients (Fe, Al, Cu, Mn, Zn, B, and Na) for the various analyses are reported in Table 2. In general, M-3 extracted the greatest quantity of nutrient, and H3A extracted the least amount of nutrient compared with the commonly used methods in the western United States. This is
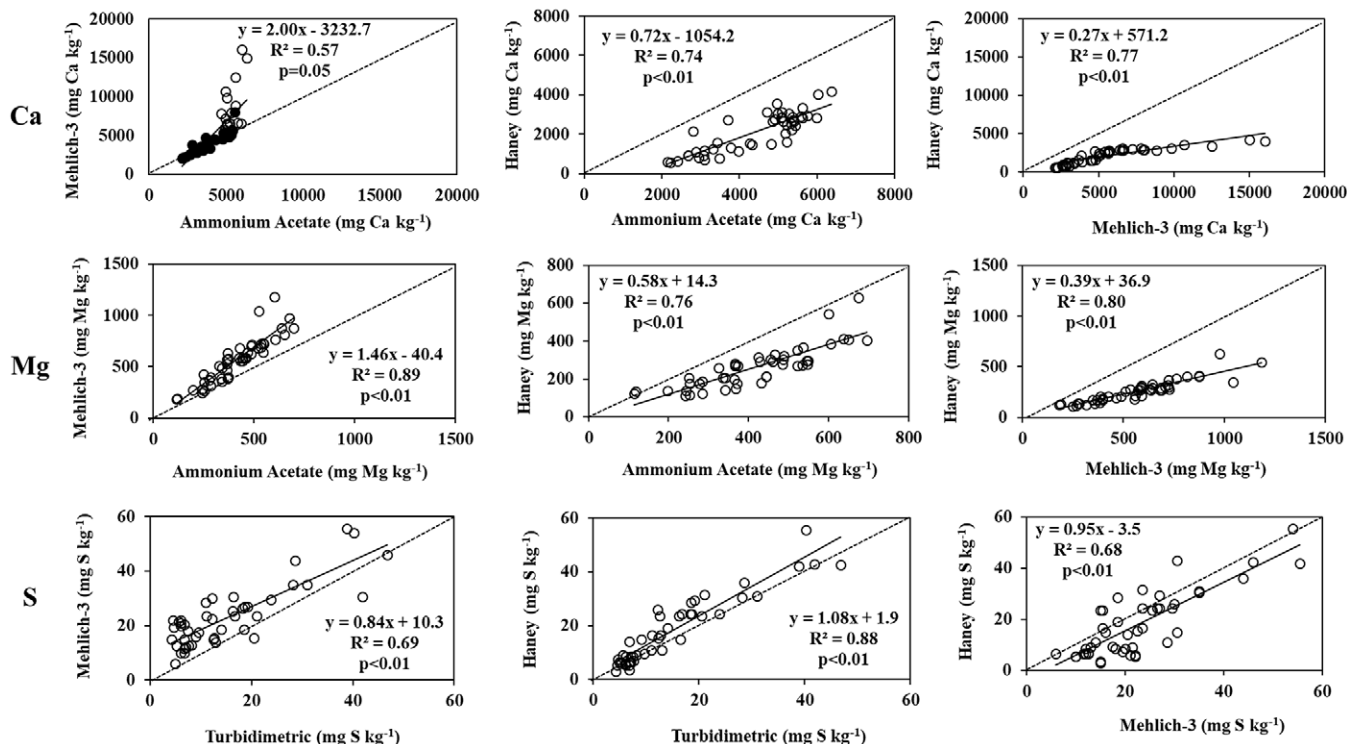
Fig. 2. Comparison of extractability of various soil test methods for Ca, Mg, and S for soil samples collected at a sample depth of 0 to 30 cm from agricultural fields in Idaho. The solid line is the linear regression fit and the dotted line represents a 1:1 relationship between the tests being compared.

largely due to the highly acidic M-3 extractant liberating greater amounts of nutrients and the weakly acidic H3A extractant liberating lesser amounts of nutrients compared with the standard soil-testing procedures in the region.

Comparison among Various Tests for K

Potassium extractants (Olsen, M-3, H3A) were all strongly correlated ($r = 0.89$–0.96), indicating the potential for the establishment of conversion equations between the tests (Table 3). Linear regression analysis indicated that the line fit of the two commonly used tests (Olsen and AA) in the samples analyzed from Idaho was $R^2 = 0.94$ where Olsen extracted less than AA (Fig. 1). Mehlich-3 and AA resulted in an $R^2 = 0.79$ where M-3 extracted at a nearly 1:1 ratio. These results were similar but with a slightly lower R^2 compared with previous work on a wide range of Florida soils by Alva (1993), Louisiana soils by Wang et al. (2004), and Arkansas soil by Martins et al. (2015), where $R^2 > 0.95$ for the previous studies. The relationship between the H3A and the AA test resulted in an $R^2 = 0.87$, where H3A extracted less K than AA; we are unaware of published comparative data from other regions for H3A and the multitude of tests in the current study. Similar results were observed between M-3 and Olsen as well as H3A and Olsen, where $R^2 = 0.69$ and 0.90, respectively. The H3A extractant consistently extracted less K compared with all other extractants. These results and those by Alva (1993), Wang et al. (2004), and Martins et al. (2015) indicate that effective equations could be developed for multinutrient tests (M-3 and H3A) for soil K in varying regions and soils of the United States.

Comparison among Various Tests for Ca, Mg, and S

Correlation for Ca was significant for the AA extractant compared with both M-3 and H3A ($r = 0.77$ and 0.86, respectively) (Table 2). For Mg correlation was 0.91 and 0.85 for M3 and H3A, respectively, and for S correlation was 0.84 and 0.94 for M-3 and

H3A, respectively. The regression fit for M-3 and AA extracted Ca was $R^2 = 0.57$ ($P = 0.05$), where noticeable deviation occurred at the higher end of the curve (Fig. 2). This contrasts with noncalcareous soils where a better relationship between M-3 and AA was observed where $R^2 = 0.91$, 0.95, and 0.97, respectively, as reported by Alva (1993), Wang et al. (2004), and Martins et al. (2015). Pre-
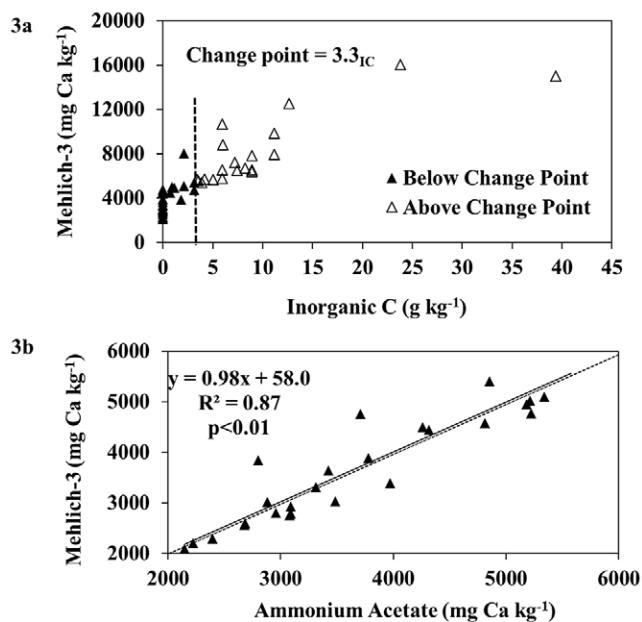

Fig. 3. (a) Mehlich-3 soil Ca test values in response to soil inorganic carbon (IC) content at a sample depth of 0 to 30 cm for soils ($n = 46$) collected from agricultural fields in Idaho. The change point in IC, as indicated by the dotted line, is 3.3 g kg^{-1} with a 95% confidence interval of 2.9 to 3.6 g kg^{-1}. (b) Comparison of extractability of ammonium acetate and M-3 tests for Ca for soil samples ($n = 26$) with IC content below change point (i.e., 3.3 g kg^{-1} IC). The solid line is the linear regression fit and the dotted line represents a 1:1 relationship between the tests being compared.

vious research in alkaline calcareous soils has noted that the highly acidic M-3 test extracts greater quantities of P compared with Olsen, Bray-1, or H3A, likely due to the dissolution of Ca compounds (Dari et al., 2019). Thus, follow-up analyses were conducted based on Dari et al. (2019). A change point analysis was conducted to determine if a threshold IC content could be determined, at which point increased M-3 Ca was measured. The analysis resulted in a change point of 3.3 g kg⁻¹ IC (95% confidence interval of 2.9–3.6 g kg⁻¹), where the

average M-3 Ca was 3799 mg kg⁻¹ (SE = 262) for samples below the threshold ($n = 26$) and 8120 mg kg⁻¹ (SE = 707) ($n = 20$) for samples above the threshold (Fig. 3). Thus, high IC levels were strongly related to high M-3-Ca (Fig. 3). Removal of these high IC sites increased the R^2 to 0.87. These results are in alignment with previous work (Alva, 1993; Wang et al., 2004; Martins et al., 2015) and provide evidence that M-3 extracts more plant available Ca on alkaline calcareous soils as the interaction of IC and the M-3 extracting solution causes greater
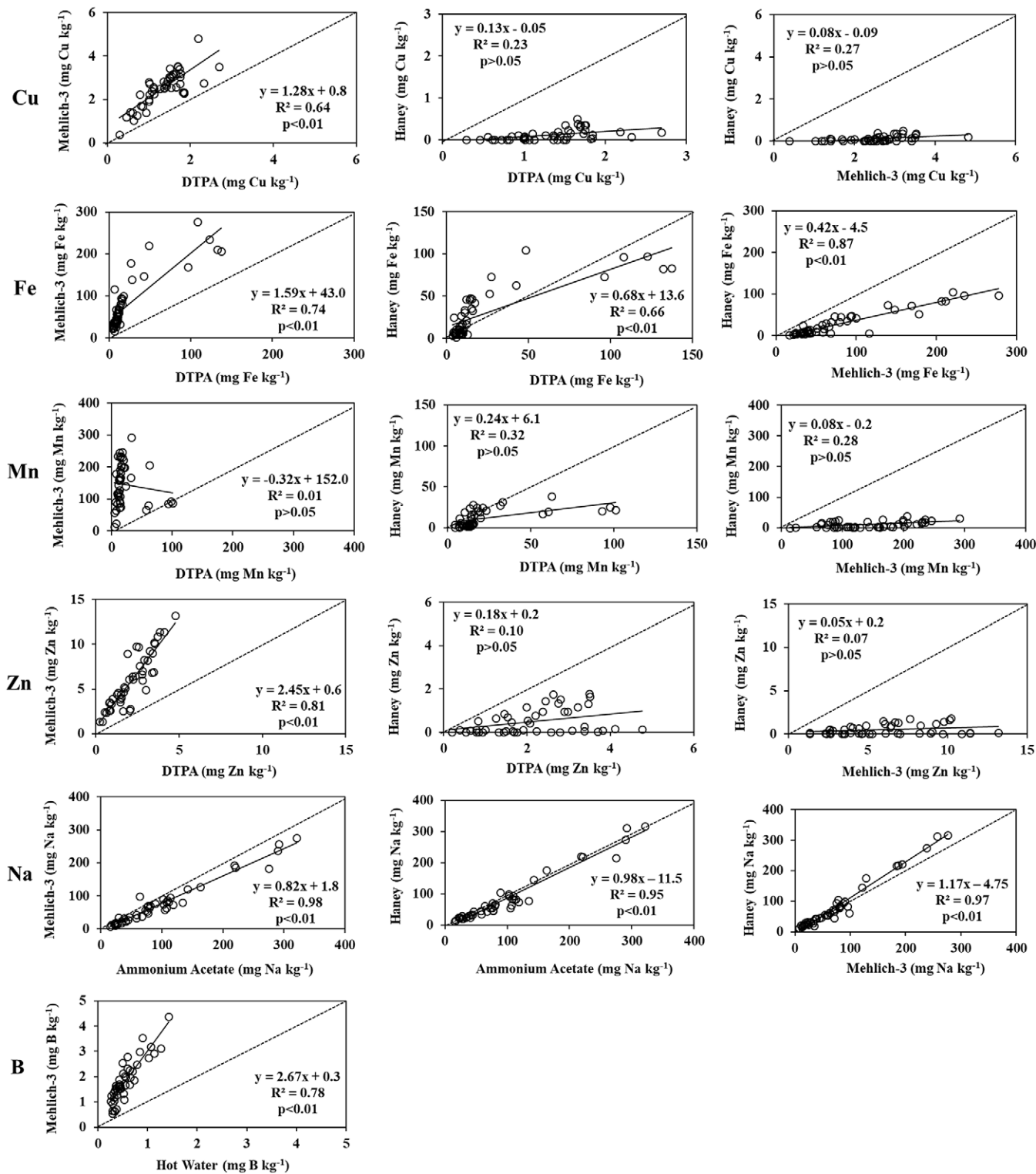

Fig. 4. Comparison of extractability of various soil test methods for micronutrients for soil samples collected at a sample depth of 0 to 30 cm from agricultural fields in Idaho. The solid line is the linear regression fit and the dotted line represents a 1:1 relationship between the tests being compared.

release of Ca as compared to the AA test. The H3A test resulted in less extracted Ca and did not exhibit issues at high IC content as a result of its weakly acidic nature.

Multinutrient Mg extractants (M-3, H3A) regression fit with AA extractant were $R^2 = 0.89$ and 0.76, respectively (Fig. 2). The M-3 extractant compared to R^2 of 0.89 was slightly less or greater than those reported by Alva (1993), Wang et al. (2004), and Martins et al. (2015), where $R^2 = 0.62$, 0.97, and 0.95, respectively. The current results indicated that M-3 Mg was greater than AA extracted Mg, which was similar to Martins et al. (2015) where Wang et al. (2004) reported a nearly 1:1 relationship. For S extractants, previous work (Rao and Sharma, 1997) reported similar correlations of M-3 in acidic Indian soils using multiple S extractants where $r = 0.87$ to 0.96 as compared with the current study where $r = 0.84$ (Table 3). The fit between the turbidimetric test and M-3 and H3A was $R^2 = 0.69$ and 0.88, respectively (Fig. 2). In contrast with the majority of other comparisons, the H3A extractant resulted in greater S extraction than the standard test for the region.

Comparison among Various Tests for Micronutrients

Crop recommendations for micronutrients are generally less well researched compared with macronutrients. Correlations in the current study between multinutrient tests (M-3 and H3A) compared with the DTPA test indicated more variability compared with the macronutrient tests ($r = -0.12$ to 0.98) (Table 3). Similar to previous work, DTPA and M-3 were significantly correlated for Cu, Fe, Zn, and Na; however, in the current study no relationship was measured for Mn (Fig. 4; Walworth et al., 1992; Cancela et al., 2002; Wang et al., 2004). Walworth et al. (1992) reported improvement in specific micronutrient regressions when pH was considered. As notable extremes in values were observed in acidic samples (pH < 7.0; $n = 6$), we removed these acidic samples and reanalyzed the data ($n = 40$, Fig. 5). The regression for Mn was still nonsignificant ($P > 0.05$). Despite the high R^2 values for the initial Fe analysis, the fit about the curve exhibited a large amount of skewness at greater concentrations appearing to represent discrete regression patterns. The reanalyzed fit for Fe ($R^2 = 0.81$) was greater than that reported by Wang et al. (2004) of $R^2 = 0.38$ but similar to $R^2 = 0.75$ as reported by Cancela et al. (2002). The greatest amount of M-3 extractable Fe in the current study was nearly double that reported by Wang et al. (2004) but was similar to Cancela et al. (2002) and may indicate differences in efficiencies in the two methods that occur with increased Fe concentrations. The H3A test resulted in small R^2 values with DTPA Cu, Mn, or Zn, where $R^2 = 0.66$ for H3A compared with DTPA Fe; however, as with M-3, the fit was skewed at higher Fe concentrations (Fig. 4). Removal of acidic soil samples resulted in improvement for Cu, Fe, and Mn for the H3A test, indicating that either separate curves are needed for alkaline and acidic soils or that the specific test may not be suitable at higher pH ranges when appreciable IC is present (Fig. 5). For Cu, Fe, Mn, and Zn, the M-3 test generally extracted greater amounts, whereas H3A extracted lesser amounts compared with DTPA. These differences are due to the acidic nature of the M-3 extractant releasing more of the tested micronutrient into solution at low pH or potentially due to differences in the extracting chelate retaining the micronutrient in solution.
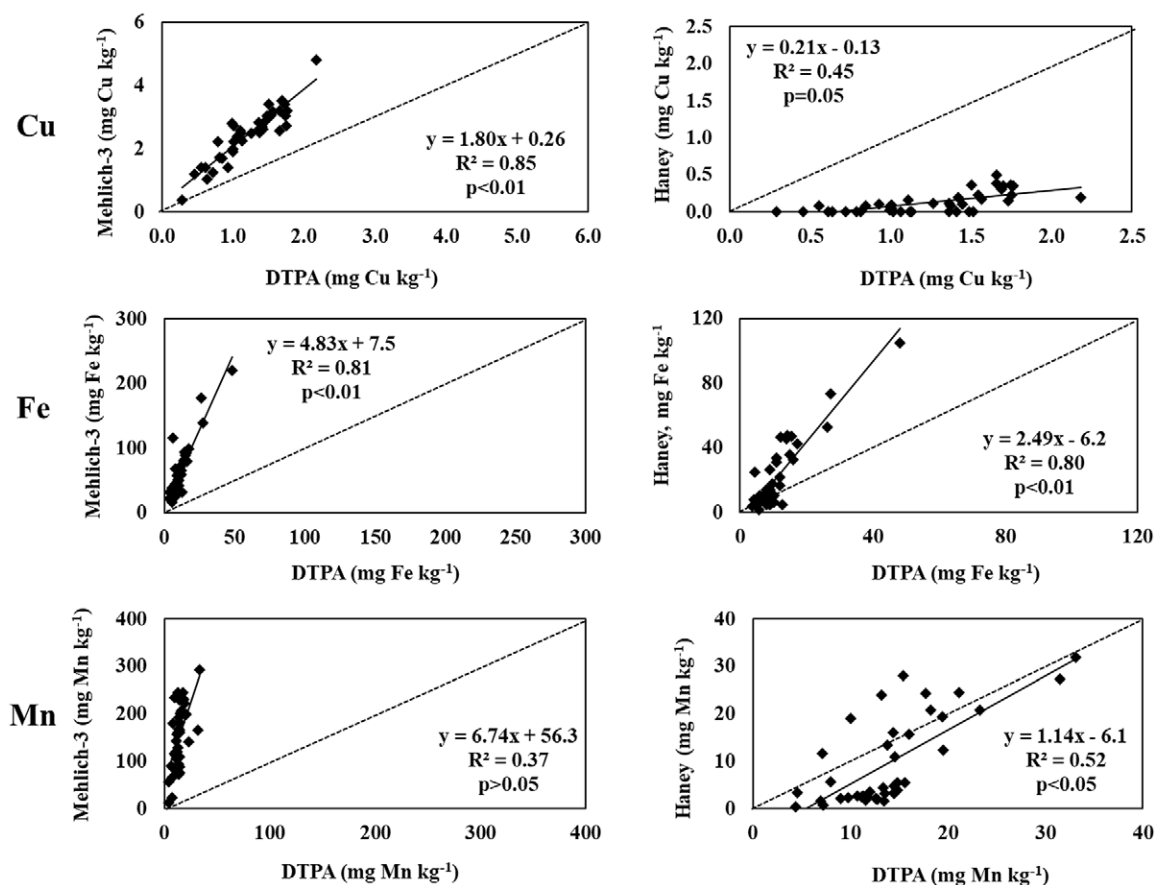

Fig. 5. Comparison of extractability of diethylene triamine penta acetic acid (DTPA), Mehlich-3, and Haney, Haney, Hossner, Arnold (H3A) for soil samples having a soil pH below 7.0 collected at a sample depth of 0 to 30 cm from agricultural fields in Idaho. The solid line is the linear regression fit and the dotted line represents a 1:1 relationship between the tests being compared.

The M-3 test resulted in greater values compared with the hot-water B extraction (Fig. 4). The relationship between the hot-water extraction and M-3 for B resulted in an $R^2 = 0.78$ where previous literature has reported $R^2 = 0.57$ in Alaskan soils (Walworth et al., 1992).

CONCLUSIONS

Both the M-3 and H3A extractants were promising in terms of the possibility of development of effective equations between these multinutrient tests and common standard tests in alkaline soils; however, notable exceptions occurred. Overall, the greater acidity associated with the M-3 was a primary factor that resulted in greater measured concentrations of nearly all nutrients as measured via the standard tests (AA, Olsen, calcium phosphate turbidimetric, and DTPA) and would over-extract Ca in alkaline calcareous soils. Results from the H3A extractant were less consistent where values were less (Olsen), greater (calcium phosphate turbidimetric), and relatively similar (AA). These results clearly indicate the lack of usefulness in attempting to compare multinutrient tests without proper correlation equations or updated correlation calibration studies from crop response trials. Care must be taken concerning scatter about the fitted line, and goodness of fit must also be considered (e.g., Fe), but despite this concern, our study presents conversion equations for the majority of the tested nutrients. The possible use of M-3 as a universal soil extractant and the interest in using the SHT and associated H3A make comparison data to standard soil tests critical for appropriately interpreting data. Although conversion equations have been developed that can be useful for comparison between methods, crop correlation and calibration studies would be needed to validate agronomic response as related to the soil test and specific crop response parameters.

CONFLICT OF INTEREST DISCLOSURE

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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