

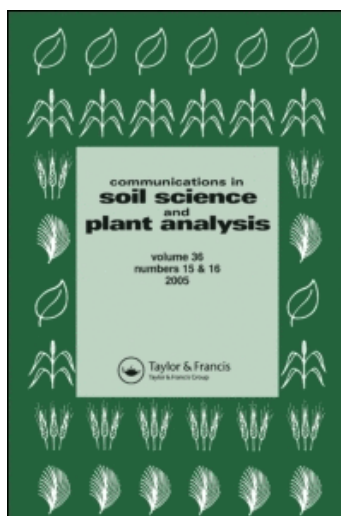
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Publisher Taylor & Francis

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Communications in Soil Science and Plant Analysis

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597241>

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To cite this Article Wilson, Michael A., Burt, Rebecca and Lee, Chris W. 'Improved Elemental Recoveries in Soils with Heating Boric Acid Following Microwave Total Digestion', Communications in Soil Science and Plant Analysis, 37: 3, 513 – 524

To link to this Article: DOI: 10.1080/00103620500449377

URL: <http://dx.doi.org/10.1080/00103620500449377>

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Improved Elemental Recoveries in Soils with Heating Boric Acid Following Microwave Total Digestion

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Abstract: Microwave digestion of soils for elemental analysis commonly uses hydrofluoric acid (HF) because of insolubility of aluminosilicate minerals in other acids. Boric acid is added following digestion to complex F in solution. Low recoveries of calcium (Ca), aluminum (Al), and magnesium (Mg) of soil reference materials led to this investigation of a secondary heating of the boric acid with digested soil. The objectives were to evaluate boric acid (H_3BO_3) concentrations needed to complex F from 4 mL HF and to evaluate soil characteristics that may contribute to the formation of metal-fluoride complexes that decrease recovery following digestion. Four standard soil reference materials and a variety of soil samples ($n = 75$) were evaluated. Heating 20 mL 2.5% H_3BO_3 with a digested standard reference soil produced recoveries of 94, 98, and 99% for Al, Ca, and Mg, respectively, compared to 46% for Al and Mg and 37% recovery for Ca in extracts where H_3BO_3 was added but not heated. Two other concentrations of H_3BO_3 were tested with slightly improved recoveries, and results suggest that 20 mL of a 4.5% H_3BO_3 solution was sufficient to maximize recoveries. Digestion of soil samples by both the nonheated and heated H_3BO_3 methods showed that recovery difference between the two methods ranged from 0 to 100% for Al and Ca. Assuming that this difference in recovery was related to the formation of metal fluorides, correlation with clay and C in soils may reflect the positive or negative influence of these constituents on the formation of these complexes, respectively.

Keywords: Hydrofluoric acid, metal-fluoride complexes, geochemistry, total analysis

Received 6 August 2004, Accepted 2 September 2005

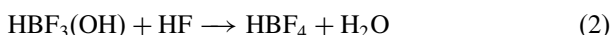
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INTRODUCTION

Microwave digestion in decomposition vessels with sealed covers is regarded as a rapid, efficient technique for elemental analysis of soils. The method has advantages such as containment of volatile elements and decreased risk of contamination over open plate digestion or fusion methods (Hossner 1996). A widely used microwave digestion procedure is EPA Method 3052 (Environmental Protection Agency 1996) that uses a combination of acids (HF, HNO₃, and HCl). This method is regarded as efficient, easy to use, safe, and robust. It avoids the problem of incomplete digestion associated with methods that do not incorporate HF in the digestion (such as EPA Method 3051).

Hydrofluoric acid is used for digestion of silica or aluminosilicate minerals (e.g., quartz, feldspars) and is especially efficient at temperatures greater than 100°C and at elevated pressures. The efficiency of HF is related to the complexing ability of the fluoride anion. Initially, SiF₆²⁻ ions are formed in acidic solution, and these are converted to volatile SiF₄ (Dulski 1996). The utility of the F⁻ ion is apparent in its use in other soil extracts, such as Bray P-1, where F decreases Al activity via formation of Al-F complexes, thereby increasing P desorption in acid soils (Kuo 1996).

A standard procedure when using HF for soil digestion is to add boric acid (H₃BO₃) to the digested sample. Boric acid is used to “neutralize” excess HF and complex fluoride (forming tetrafluoroboric acid) in solution:



The removal of HF prevents volatilization of SiF₄ (improving Si recovery) and prevents formation of insoluble metal fluoride complexes. It also improves safety for sample handling and allows use of silica torches during ICP analysis. The rate-determining step is creation of fluoroboric acid. It has been suggested that the solution could be treated with 10 times excess of H₃BO₃ to overcome the slow kinetics (Kingston et al. 1997). However, high H₃BO₃ concentrations would result in increased dissolved solids in the extract, increasing detection limits during ICP analysis.

The routine procedure in the USDA-Natural Resources Conservation Service Soil Survey Laboratory (Lincoln, NE) is to add a H₃BO₃ solution following HF digestion of soils for major elemental analysis in our laboratory. Recoveries of elements from soil reference materials have been generally good (85–100%) for elements such as phosphorus (P), iron (Fe), and silicon (Si), but lower (50–65%) for Al, Ca, and Mg. In addition, the coefficient of variation (CV) for replicate samples is less than 5% for most elements, whereas the three suspect elements have CV 10% or greater. Early testing of the method to improve the Al, Ca, and Mg recoveries and reproducibility was unsuccessful. Formation of insoluble metal fluorides was suspected, but no visible evidence (small particulates or cloudy digestion solution) existed.

It was suggested that the H_3BO_3 (added following the acid digestion) may need to be heated in the microwave to further the reaction (D. Barclay, personal communication, March 2003). Review of the literature confirms others have used microwave heating to facilitate the reaction of the H_3BO_3 with the HF (Coedo et al. 1998; Feng et al. 1999). These two studies used small amounts of H_3BO_3 due to the limited amount of HF (≥ 0.2 mL) in the sample. The digestion procedure in the USDA laboratory requires 4 mL HF, resulting in a much larger volume of H_3BO_3 required. This study was initiated to examine the influence of a second heating of a digested sample with H_3BO_3 on recovery of Al, Ca, and Mg. The specific objectives were to 1) quantify the improvement in elemental recoveries of standard reference materials and other soils from heating H_3BO_3 in the microwave and 2) examine characteristics of soils that may influence the formation of metal fluorides during the initial digestion procedure.

EXPERIMENTAL

The study used a CEM Mars 5 microwave oven¹ (CEM Corp., Matthews, NC) with a 14 position-HP500 Plus vessel and rotor (vessels composed of PFA, sleeves composed of advanced composite). Four standard reference materials were tested: 1) CRM-LO-C (loam soil from High Purity Standards Corp., Charleston, SC), 2) NIST (National Institute of Standards and Technology, Gaithersburg, MD) SRM (standard reference material) 2709 (San Joaquin Soil), 3) NIST SRM 2710 (highly contaminated Montana soil), and 4) NIST SRM 2711 (moderately contaminated Montana soil). The initial method (based on EPA 3052 and designated here as the "original method") digested 250 mg soil in a microwave oven (closed vessel) by using 9 mL concentrated HNO_3 , 4 mL 48% HF, and 3 mL concentrated HCl. The samples were heated in the microwave to 180°C and then digested for 9.5 min. The vessels were allowed to cool, a 2.5% w/v H_3BO_3 solution was added to the sample, and the entire contents quantitatively transferred to a 100-mL volumetric flask with additional H_3BO_3 solution. Extracts were analyzed for Al, Ca, Fe, potassium (K), Mg, manganese (Mn), Na, P, and Si by using a Perkin Elmer 3300DV ICP-OES instrument (Perkin Elmer Corporation, Wellesley, MA) operating in radial mode, with a cross-flow injector and alumina torch. Data from this original method are reported in this study for the CRM-LO-C reference soil and soil samples used in the third experiment.

The initial experiment tested the elemental recovery between the original method and a modified method for the CRM-LO-C reference soil. This modification tested the addition of 20 mL 2.5% H_3BO_3 to the vessel following initial digestion, with resealing and heating in the microwave at 160°C for

¹Mention of tradenames does not constitute an endorsement by USDA-NRCS.

10 min. Following heating, samples were brought to a 100-mL final volume with additional 2.5% H_3BO_3 , resulting in a final H_3BO_3 concentration in samples of 2.1%. All reference materials in this and the second experiment were run in triplicate for all treatments. Results were evaluated by calculating percent recovery defined as the difference between certified value and experimental value, divided by certified value, times 100.

A second experiment evaluated two other H_3BO_3 concentrations to evaluate if recoveries of the standard reference soils could be improved: (a) 20 mL 4.5% and (b) 30 mL 4.5%, with reheating in the microwave at 160°C for 10 min. Following heating, samples were brought to a final volume with 1.9% H_3BO_3 for a final H_3BO_3 concentration of 2.1%. Data were evaluated by calculation of arithmetic mean, CV, and percent recovery. Fisher's least-significant difference (LSD) procedure ($p < 0.05$) was performed to determine if means were significantly different (Manugistics 2002).

A third experiment used soil samples ($n = 75$) with a wide range in Al and Ca concentrations from Missouri, Virginia, Alabama, and other states. Samples include horizons from over 20 pedons, representing the upper 2 m of soil. These samples were digested by the original method (no H_3BO_3 heating) and by the method of heating 20 mL 4.5% H_3BO_3 to evaluate if specific soil properties could be related to the difference in elemental recovery between methods. Bulk samples were air dried and sieved to less than 2 mm to remove rock fragments. Elemental analysis was performed on less than 2 mm soil material ground to less than 150 μm . The following laboratory analyses were also performed on the less than 2 mm soil, and these methods are described in Soil Survey Investigations Report No. 42 (Burt, 2004): particle-size, total C, acid ammonium oxalate-extractable Fe (Feo), dithionite-citrate extractable Fe (Fed), carbonates, and pH in water. Recovery difference for soil samples is defined as the difference in elemental concentration from the two H_3BO_3 treatments. Independent variables are determined for simple regression models at the 90, 95, and 99% confidence levels ($p < 0.1$, 0.05, and 0.01, respectively). Coefficient of determination and correlation coefficient are herein designated as r^2 and r , respectively.

RESULTS AND DISCUSSION

Results from digestion of the reference soil CRM-LO-C for the first experiment (Table 1) indicates that heating the digested sample with 20 mL 2.5% H_3BO_3 in the microwave resulted in improved recovery for all elements compared with the nonheated H_3BO_3 treatment (original method). This initial experiment of heating H_3BO_3 solution with the sample resulted in recoveries of 94, 98, and 99% for Al, Ca, and Mg, respectively. This finding compared with recoveries of 46% for Al and Mg and 37% for Ca obtained when the H_3BO_3 added to samples was not heated. These improved recoveries

Table 1. Recovery for the soil reference material CRM-LO-C based on treatment following microwave digestion (n = 3)

Parameter	Al	Ca	Mg	Fe	K	Mn	Na	P	Si
No heating of boric acid									
Mean (mg kg ⁻¹)	22980a ^c	1219a	1670a	22891a	12648a	1121a	3652a	955a	317775a
CV ^a	10.4	9.7	27.0	1.0	2.9	1.2	0.9	2.4	2.8
Recovery (%) ^b	46	37	46	90	85	84	90	96	—
20 mL 2.5% boric acid; heated									
Mean (mg kg ⁻¹)	46752b	3218bc	3552b	24011c	14356b	1363b	3854b	1013b	334059b
CV	0.7	0.9	0.8	0.6	0.0	1.9	0.6	5.3	1.8
Recovery (%)	94	98	99	95	96	102	95	101	—
20 mL 4.5%; heated									
Mean (mg kg ⁻¹)	47615b	3209b	3561b	24432bc	15127b	1367b	4008b	1007ab	331530ab
CV	1.4	0.9	1.1	0.8	0.5	1.0	0.9	2.4	1.8
Recovery (%)	96	98	99	96	102	102	98	101	—
30 mL 4.5% boric acid; heated									
Mean (mg kg ⁻¹)	45539b	3366c	3552b	23249ab	13992b	1340b	3749b	976ab	322338ab
CV	1.4	1.1	1.4	1.5	1.4	1.3	1.8	3.5	1.6
Recovery (%)	92	103	99	92	94	100	92	98	—

^aCV, coefficient of variation.
^bRecovery based on certified values reported for the soil material.
^cMeans for elements within a column followed by the same letter are not statistically different by Fisher's least-significant difference procedure at the 95% confidence level (*p* < 0.05).

are likely the result of greater complexation of the fluoride by the H_3BO_3 , preventing precipitation (solubilizing) metal fluorides of Ca, Al, and Mg in the digestate. The CVs for these elements ranged from 9.7 to 27% on the original method but were less than 1% on samples with heated H_3BO_3 . Recovery of other elements (Fe, K, Mn, Na, P, and Si) ranged from 95 to 102% in samples with heated H_3BO_3 . The relative increase in recoveries for these elements was not as large, because their recoveries in nonheated H_3BO_3 extracts ranged from 84 to 96%. Differences were statistically significant ($p < 0.05$) between methods for all elements (Table 1).

Kingston et al. (1997) indicates that a wide range of H_3BO_3 concentrations are regarded as suitable for complexing HF in samples. Based on the stoichiometry of the reaction in Eqs. (1) and (2), it would require 1.49 g H_3BO_3 to neutralize the 1.92 g HF (4 mL 48% HF) added to our sample. The final H_3BO_3 concentration of 2.1% equates to 2.1 g total H_3BO_3 in solution, more than stoichiometrically sufficient to complex all F. Although the initial test added only 0.5 g H_3BO_3 (20 mL 2.5%) to the sample for heating, recovery greatly improved. This was evidence that heating this non-stoichiometrically equivalent amount of H_3BO_3 decomposed most of the fluoride complexes present in the digestion solution.

Following our initial improvement in elemental recovery, we further tested the amount of H_3BO_3 heated, comparing the elemental recovery that results from the addition of 20 mL 2.5% H_3BO_3 with 20 mL and 30 mL 4.5% H_3BO_3 solutions on the four reference materials (Tables 1–4). Results suggest a small improvement in recovery from increasing the H_3BO_3 added in the initial heating test (0.5 g) to 0.9 and 1.35 g for the 20 and 30 mL 4.5% H_3BO_3 solutions, respectively. Statistical analysis found little difference in elemental means for the CRM-LO-C reference soil, but most elements had improved recoveries and elemental means were significantly different for the 4.5% H_3BO_3 additions. The 20 mL 4.5% H_3BO_3 treatment proved best overall based on both percent recovery and statistical analysis.

A variety of soils ($n = 75$) were used to compare the elemental recovery between the original method and the addition of 20 mL 4.5% H_3BO_3 . It is assumed that the decreased recovery in the original method was associated with metal fluoride complexation and that nearly 100% recovery was achieved via heating H_3BO_3 with sample extracts. The recovery difference between methods ranged from 0 (no difference in recovery for an element) to over 100% increase in recovery from heating the H_3BO_3 with the sample. Recovery differences averaged 31, 75, and 60% for Al, Ca, and Mg, respectively. There were also slightly improved recoveries for K, Na, and Mn. Coefficients of determination and correlation coefficients (Table 5) were determined for Al and Ca for several properties. Overall, correlations as well as the range of recovery (Figure 1) were generally higher for Ca than Al. The relationship with these elements was greatest for clay, silt, total C, and pH, suggesting that these properties may be most strongly related to the recovery between these methods. The correlations were low

Table 2. Recovery for the soil reference material NIST SRM-2709 based on treatment following microwave digestion (n = 3)

Parameter	Al	Ca	Mg	Fe	K	Mn	Na	P	Si
20 mL 2.5% boric acid; heated									
Mean (mg kg ⁻¹)	65883a ^c	18235a	12679a	33854a	18644a	515a	11187a	608a	278919a
CV ^a	2.1	1.3	3.8	0.7	0.8	0.6	0.8	4.8	0.5
Recovery (%) ^b	88	96	84	97	92	96	96	98	94
20 mL 4.5%; heated									
Mean (mg kg ⁻¹)	72140b	18566a	14186b	34585b	19986b	546b	11728b	642a	273777b
CV	1.4	0.9	1.8	1.0	1.1	0.8	0.8	8.2	1.0
Recovery (%)	96	98	94	99	98	102	101	104	92
30 mL 4.5% boric acid; heated									
Mean (mg kg ⁻¹)	70521b	18490a	14332b	33435a	18921a	558c	11142a	667a	279664a
CV	1.1	1.0	1.0	1.0	1.1	1.0	1.1	1.5	0.9
Recovery (%)	94	98	95	96	93	104	96	108	94

^aCV, coefficient of variation.
^bRecovery based on certified values reported for the soil material.
^cMeans for elements within a column followed by the same letter are not statistically different by Fisher's least-significant difference procedure at the 95% confidence level ($p < 0.05$).

Table 3. Recovery for the soil reference material NIST SRM-2710 based on treatment following microwave digestion (n = 3)

Parameter	Al	Ca	Mg	Fe	K	Mn	Na	P	Si
20 mL 2.5% boric acid; heated									
Mean (mg kg ⁻¹)	59666a ^c	11711a	7778a	31980a	19593a	9757a	10539a	909a	269275ab
CV ^a	3.3	2.6	4.5	1.9	2.4	2.0	1.9	1.6	1.6
Recovery (%) ^b	93	94	92	95	93	97	92	86	93
20 mL 4.5%; heated									
Mean (mg kg ⁻¹)	62653b	12207b	8144ab	33077c	21136c	9962a	11289c	1059c	270220a
CV	0.9	0.7	0.6	0.6	0.7	0.7	0.8	0.6	0.3
Recovery (%)	97	98	96	98	100	99	99	99	93
30 mL 4.5% boric acid; heated									
Mean (mg kg ⁻¹)	61909ab	12432b	8265b	32357bc	20159b	9904a	10832b	1002b	276195b
CV	0.6	0.2	0.2	0.3	0.5	0.3	0.4	1.6	0.3
Recovery (%)	96	99	97	96	96	98	95	95	95

^aCV = coefficient of variation.
^bRecovery based on certified values reported for the soil material.
^cMeans for elements within a column followed by the same letter are not statistically different by Fisher's least-significant difference procedure at the 95% confidence level (*p* < 0.05).

Table 4. Recovery for the soil reference material NIST SRM-2711 based on treatment following microwave digestion (n = 3)

Parameter	Al	Ca	Mg	Fe	K	Mn	Na	P	Si
20 mL 2.5% boric acid; heated									
Mean (mg kg ⁻¹)	59368a ^c	26666a	9307a	27113a	22426a	593a	10792a	795a	280763a
CV ^a	2.4	2.3	2.8	2.2	2.2	2.3	2.3	4.2	2.7
Recovery (%) ^b	91	93	89	94	92	86	95	92	92
20 mL 4.5%; heated									
Mean (mg kg ⁻¹)	63223c	27565c	9901b	28310b	24597b	636b	11775b	847a	279991a
CV	0.5	0.3	0.6	0.7	0.5	0.5	0.6	0.9	3.9
Recovery (%)	97	96	94	98	100	93	103	99	92
30 mL 4.5% boric acid; heated									
Mean (mg kg ⁻¹)	61333b	27213ab	9893b	27291a	22902a	646b	11082a	795a	286635a
CV	0.6	0.6	0.6	0.9	0.7	0.8	0.8	5.4	0.9
Recovery (%)	94	94	94	94	93	94	97	92	94

^aCV, coefficient of variation.
^bRecovery based on certified values reported for the soil material.
^cMeans for elements within a column followed by the same letter are not statistically different by Fisher's least-significant difference procedure at the 95% confidence level (*p* < 0.05).

Table 5. Coefficients of determination (r^2) and correlation coefficients (r) for the difference in Ca and Al recoveries between unheated and heated boric acid treatments with several soil properties (n = 75)

Dependent variable	Soil property	r^2	r
Ca difference	Total Ca	13.6	-0.37***
	Total Ca + Al	8.5	0.29**
	Clay	42.6	0.65***
	Silt	40.8	-0.64***
	Fed	20.9	0.46***
	Feo	3.3	-0.18 ^{NS}
	Carbonate	5.8	-0.24**
	Total C	29.4	-0.54***
	pH(H ₂ O)	34.1	-0.58***
Al difference	Total Al	4.9	0.22*
	Total Ca + Al	1.4	-0.19 ^{NS}
	Clay	3.7	0.19*
	Silt	7.1	-0.27**
	Fed	2.5	0.16 ^{NS}
	Feo	0.1	-0.04 ^{NS}
	Carbonate	10.3	-0.32***
	Total C	21.2	-0.46***
	pH(H ₂ O)	32.8	-0.57***

NS, not statistically significant.
*, significant at the 0.1 probability level.
**, significant at the 0.05 probability level.
***, significant at the 0.01 probability level.

for such properties as total Ca, total Al, and sum of Ca plus Al in the samples, as well as Fed, Feo, and carbonate content.

The correlations between recovery difference and soil properties suggest an inverse relationship (Table 5) between both Ca and Al recovery difference and pH ($r = -0.58$ and -0.57 , respectively). Figure 1(a, b) shows a wide range of recovery differences at a lower pH, whereas recovery differences decreased at the more alkaline soil pH. This finding suggests that alkaline soils had decreased F complexation of Al and Ca in the nonheated samples. The inverse relationship (Figure 1c, d) between total C and recovery difference for Ca and Al ($r = -0.54$ and -0.46 , respectively) suggest that increased C in soils may play a role in preventing formation of metal fluorides. Clay and silt (Figure 1e, f, g, h) were significantly related to Ca recovery differences ($r^2 = 42.6$ and 40.8), whereas there was a much weaker relationship with Al ($r^2 = 3.7$ and 7.1% , respectively). Recovery differences increased with clay percentage, suggesting that during the digestion process, clay (with the relatively large surface area per unit weight compared to sand and silt) may present reaction surfaces for Ca-F complexation in nonheated digests.

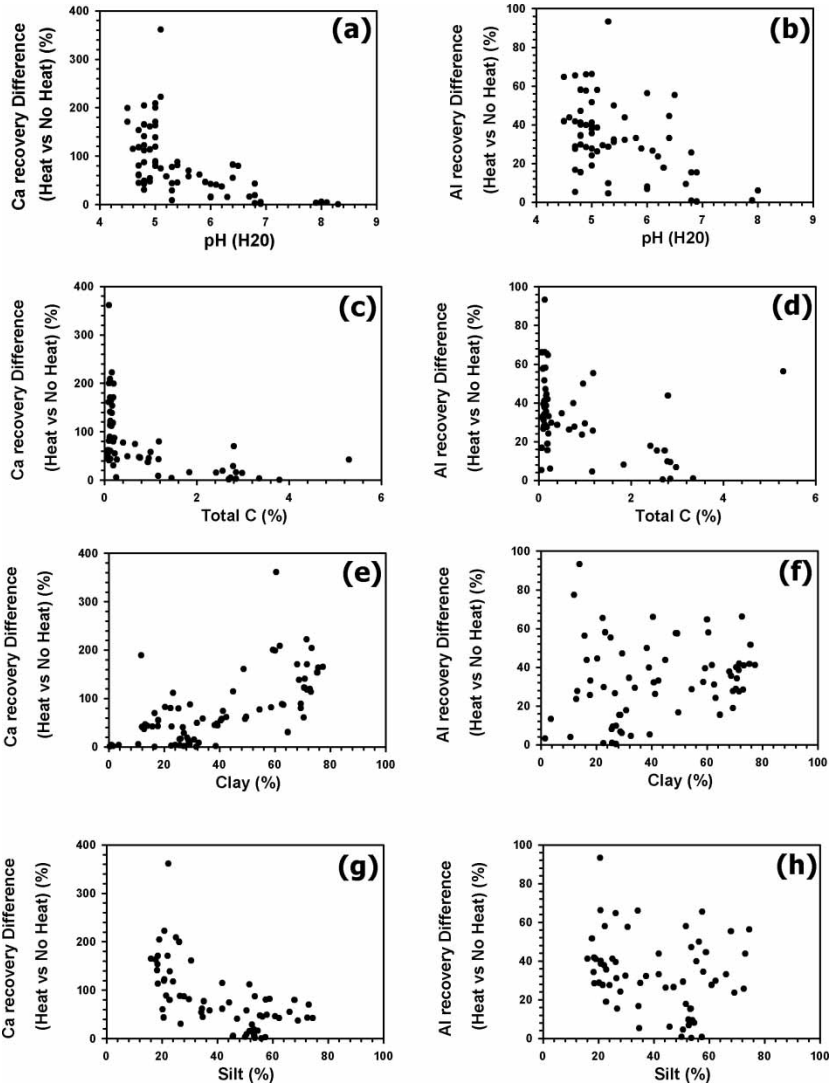


Figure 1. Difference in recovery of Ca and Al compared with pH, total C, clay, and silt in soil samples. Recovery difference is the measured Ca and Al between soil samples with digests that received the heated 20 mL 4.5% boric acid treatment vs. samples that had only boric acid added with no heat treatment.

CONCLUSIONS

The addition of H_3BO_3 following HF digestion increases the recovery of elements during analysis by decreasing volatilization losses (Si) and solubilizing metal fluoride complexes (Ca, Al, and Mg). Heating of H_3BO_3 with

the digested soil sample has been found to be important to initiate the complexation reaction forming HBF_4 . Results of this study indicate that a 20 mL 4.5% H_3BO_3 solution added following initial HF (4 mL 48% HF) digestion was sufficient to increase recoveries of Al, Ca, and Mg in a soil reference material to 95–100%. The CV of these elements for replicate samples decreased, and small increases in recovery were also demonstrated for other elements (Fe, K, Mn, Na, P, and Si). Small amounts of H_3BO_3 (less than the stoichiometric equivalent of F added) heated with the digested soil were sufficient to decompose metal fluoride complexes with the balance of the H_3BO_3 added following heating. This study used the Ca and Al recovery differences between heating and not heating of the H_3BO_3 in extracts as an indicator of the amount or degree of metal fluoride formation in the initial digestion. Correlations of these recovery differences with clay and total C suggest that surfaces of soil particles may serve as loci for formation of these complexes, whereas the inverse correlation with C suggest that the organic matter in these soils may decrease this complex formation.

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