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Release of Native and Amended Boron From Arid Zone Soils After Varying Incubation Times

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Abstract: In this study, we evaluated the boron (B) release from soils containing elevated native B and examined the extent to which incubation time affected B release. Five soils varying in initial pH and clay content were selected for the study. The soils were spiked with five varying doses of B as H_3BO_3 (0–100 mg B kg^{-1}) and equilibrated for a total of 5 months at field capacity water content. The soils were incubated in a temperature-controlled room at 25°C. At monthly intervals, the soils were thoroughly mixed and subsamples were removed. The subsamples were extracted with diethylenetriaminepentaacetic acid–sorbitol, boiling water, and 0.1 M NaCl to provide various B extraction protocols. Boron desorption decreased as a function of time for at least some of the extractions and treatments for all five soils. This is consistent with published literature that added B becomes less extractable with increased incubation time. Comparison between the three extractants revealed no statistically significant differences in amounts of adsorbed B for three of the soils. For the two other soils, the order of desorbed B amount was hot water soluble < 0.1 M NaCl extractable < diethylenetriaminepentaacetic acid–sorbitol extractable. The decrease in B release with respect to incubation time approached zero for all five soils for most of the extractant solutions, as evidenced by slopes that were not statistically significantly different from zero at the 95% level of confidence for almost all treatments of all three extractions. This result suggests that a 5-month incubation is adequate to obtain an accurate assessment of the amount of B available for release. Because the decrease in extractable B with time for four of the soils is not large, a very good indication of extractable soil B can be obtained from a 1-month incubation study.

Key words: Adsorption, desorption.

B<small></small>oron (B) is an essential micronutrient required for plant growth. However, the concentration range between plant deficiency and toxicity is smaller for B than for any other nutrient element. Boron deficiency often occurs in humid regions where rainfall exceeds potential transpiration, especially on sandy or acid soils. Boron toxicity is common in arid and poorly drained areas. At very high concentrations, B can also be toxic to growth and reproduction of avian species, such as ducks (Smith and Anders, 1989; Hoffman et al., 1990).

Soil solution B concentrations are controlled by adsorption-desorption reactions, release from tetrahedral sites of clay minerals (Jasmund and Lindner, 1973) and amorphous aluminosilicate phases, or dissolution of B-containing minerals (Su and Suarez, 2004). It is generally assumed that B adsorption and desorption reactions occur virtually instantaneously and reversibly so that B adsorption results can be used to describe B desorption behavior. This was indeed the case for some soils, where the B desorption isotherm closely corresponded to the adsorption isotherm (Hatcher and Bower, 1958; Elrashidi and O'Connor, 1982). For other soils, hysteresis was reported and the B desorption isotherm did not mirror the adsorption isotherm (Elrashidi and O'Connor, 1982).

The rate of B desorption from soils was found to decrease over time after initial B addition and was attributed to B diffusion out of the interior of clay mineral particles (Griffin and Burau, 1974). Boron fixation by the clay minerals kaolinite and smectite increased as incubation time was increased from 1 to 4 weeks (Parks and White, 1952). Boron retention in a Norwegian forest soil doubled on incubation for 8 weeks from that obtained after 17 h (Letho, 1995). Up to 82% of B applied to Finnish soils was fixed to hot-water extraction after 100 days of incubation, as opposed to 49% after 20 h (Saarela, 1985). It has been suggested that native adsorbed B is held with greater tenacity in soils than B that has been artificially added (Rhoades et al., 1970).

In most soils, adsorption-desorption processes control the amount of soluble B in solution. Boron soil tests are designed to measure plant-available B and thus provide a measure of B release from both native and added B sources. Three different extractants were used in this study to measure B desorption. Historically, the most common soil test for B has been the hot water–soluble procedure described by Berger and Truog (1940). A diethylenetriaminepentaacetic acid (DTPA)–sorbitol extract has been recommended by the North American Proficiency Testing Program to estimate soil availability of B and the trace metal nutrients Zn, Cu, Mn, and Fe (Miller et al., 2000). Release of native B from arid zone soil samples has been characterized by extraction into sodium chloride (Goldberg et al., 2005).

The objectives of this study were (i) to determine the release of native and added B from arid zone soils as a function of incubation time and (ii) to evaluate three extractants for their ability to measure extractable adsorbed B in soils.

MATERIALS AND METHODS

The top 10 cm of five arid zone soils from California were collected from surface horizons of the soil series as follows: Tranquillity in 2004, Twisselman in June 2009, and Arlington, Ramona, and Hanford in August 2009. Sampling coordinates, soil types, and land use are provided in Table 1. The soils were air-dried and sieved, and the less than 2-mm fraction was used for analysis. The sieved soils were thoroughly homogenized and stored in 5-gallon plastic buckets. Both the Tranquillity and the Twisselman soils contained native amounts of B, a moderate amount for the Tranquillity and a large amount for the Twisselman. Total B contents of the soils were determined using *aqua regia* digestion according to method 3051A (USEPA, 1997).

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TABLE 1. Soils Sampled for the Study

Soil Series	Coordinates	Soil Type	Land Use
Tranquillity	36°49′57″N, 120°33′8″W	Haplic Durixeralf	Arable land
Twisselman	35°44′4″N, 119°43′14″W	Typic Torriorthent	Arable land
Arlington	33°58′48″N, 117°20′28″W	Sodic Haploxerert	Arable land
Ramona	33°57′52″N, 117°20′35″W	Typic Haploxeralf	Orchard
Hanford	33°57′55″N, 117°20′12″W	Typic Xerorthent	Orchard

The soil physical and chemical characteristics are provided in Table 2. Soil pH was determined at a soil/0.1 M NaCl solution ratio of 1:5. Water content at −1 kPa pressure was determined using a pressure plate apparatus. Particle size distributions were obtained using the basic hydrometer method (Gee and Bauder, 1986). Surface areas were determined using ethylene glycol monoethyl ether adsorption (Cihacek and Bremner, 1979). Carbon contents were determined using a carbon coulometer. Inorganic carbon content (IOC) was measured using an acidification module and heating to 70°C. Organic carbon content (OC) was determined as the difference between total carbon content measured by furnace combustion at 950°C and IOC. Iron and aluminum oxides were extracted using a citrate/citric acid buffer and hydrosulfite as described by Coffin (1963). Iron and Al concentrations in the extracts were measured by inductively coupled plasma optical emission spectrometry (ICP-OES).

Incubation experiments were carried out at the field capacity of each soil. The water contents at field capacity were considered to correspond to −1 kPa pressure and are provided in Table 2. Samples of 500 g of air-dry soil were added to 1-L plastic containers and equilibrated with appropriate amounts of boric acid solution to achieve the same water content as that determined at −1 kPa pressure and added soil B contents of 0, 3, 8, 20, or 50 mg B kg^{-1} soil. The 0 mg B kg^{-1} added soil treatment was achieved by the addition of deionized water. The soil/solution mixtures were homogenized by thorough stirring, the containers were capped, and the systems were incubated in a constant-temperature room for a total of 5 months at 25°C. Once a week, the lids were removed briefly to aerate the soils and to check by smell that reducing conditions had not developed. After 1 month, the containers were weighed and the water loss was recorded. At monthly intervals, the soils were thoroughly mixed and one fifth of the soil/solution mixture was removed. The treatments were replicated three times.

The subsamples were split and analyzed for hot water-soluble B, DTPA-sorbitol–extractable B, and 0.1 M NaCl-extractable B. The extraction analyses were always performed without drying the soils from their field capacity water content. This was done to avoid changes in the proportions of adsorbed to soluble B that occur from wetting and drying regimes in soils.

Hot water–soluble B was determined using the method of Berger and Truog (1940), with the simplification developed by Gupta (1967). Samples consisting of 25.0 g of soil plus the water needed to attain water content at −1 kPa pressure were weighed into 150-mL Pyrex beakers, and 50.0 mL of deionized water were added. The slurries were stirred well with a plastic rod, covered with a watch glass, placed on a hot plate, and boiled for 5 min. After cooling, the beakers were weighed and deionized water was added to make up any weight loss. The slurries were stirred well and filtered immediately using two Whatman No. 42 filter papers. The filtrates were analyzed for B concentration using ICP-OES spectrometry.

The DTPA-sorbitol–extractable B was determined using the method of Miller et al. (2000). The extraction solution contained 0.005 M DTPA, 0.01 M CaCl$_2$, 0.1 M triethanolamine adjusted to pH 7.3, and 0.2 M sorbitol. Samples consisting of 10.0 g of soil plus the water needed to attain water content −1 kPa pressure were added to 50-mL polypropylene centrifuge tubes and equilibrated with 20.0 mL of the DTPA-sorbitol extraction solution on a reciprocating shaker for 2 h. After reaction, the samples were centrifuged and filtered using Whatman No. 42 filter paper. The filtrates were analyzed for B concentrations using ICP-OES spectrometry.

Sodium chloride–extractable B was determined as described by Goldberg et al. (2005). Samples of 2.5 g of soil plus the water needed to attain water content −1 kPa pressure were added to 50-mL polypropylene centrifuge tubes and equilibrated with 25.0 mL of an electrolyte solution of 0.1 M NaCl on a reciprocating shaker for 20 h. After reaction, the samples were centrifuged and the solutions were analyzed for pH and filtered through 0.45-μm membrane filters. The filtrates were analyzed for B concentration using ICP-OES spectrometry.

RESULTS AND DISCUSSION

Boron desorption as a function of reaction time was determined for the soils by incubating for a total of 5 months from

TABLE 2. Soil Physical and Chemical Characteristics

Soil	pH 1:5	$\Theta g^{\dagger}$ %	Clay %	SA m^2 g^{-1}	IOC %	OC %	Fe %	Al %	B mg kg^{-1}
Tranquillity	7.3	40.6	31.1	131	0.022	0.78	0.850	0.0595	86.4
Twisselman	7.7	35.2	17.5	78.1	0.0215	0.60	0.398	0.0408	307.
Arlington	7.2	20.5	5.8	13.8	0.0085	0.52	0.333	0.0222	36.7
Ramona	7.4	10.9	2.1	7.03	0.0048	0.14	0.225	0.0155	27.1
Hanford	7.0	15.2	9.6	17.5	0.00525	0.72	0.489	0.0314	44.9

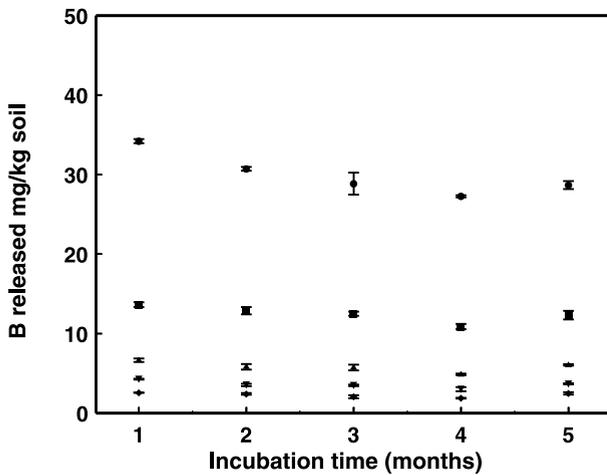†Θ_g: gravimetric water content at −1 kPa pressure.

FIG. 1. Boron desorption as a function of incubation time from Tranquillity soil using the hot water–soluble extraction. Boron additions are represented by diamonds = 0, down triangles = 3, up triangles = 8, squares = 20, and circles = 50 mg B/kg soil.
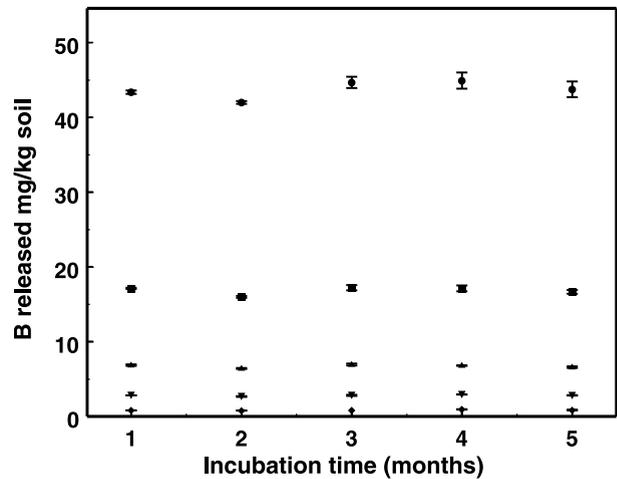

FIG. 3. Boron desorption as a function of incubation time from Arlington soil using the hot water–soluble extraction. Boron additions are represented by diamonds = 0, down triangles = 3, up triangles = 8, squares = 20, and circles = 50 mg B/kg soil.

September 2009 to February 2010. Boron desorption from the soils as a function of time is presented in Figs. 1 to 5 for the hot water–soluble extraction. Tables 3 to 5 present the first and 5-month desorption data for all three extractants. Boron desorption generally decreased as a function of time for each extraction and for all of the treatments for all five soils. Statistically significant decreases with time at the 95% level of confidence are indicated in boldface on Tables 3 to 5. Only a small subset of data showed statistical significance at the 95% level. However, for the Tranquillity soil, all three extractants at all five concentrations showed a decrease with time (Tables 3–5). Similarly, for the Ramona soil, DTPA-sorbitol and NaCl-extractable B decreased during 5 months for all treatments. For the Hanford soil, all treatments for all three extractants, except one, decreased with time (14 of 15 treatment extractions). For the Twisselman soil, all five B levels added showed an increase

with time when DTPA-sorbitol extraction was used (Table 4). This extractant removed the greatest amount of B from this soil, suggesting that DTPA-sorbitol may be more effective than the other two extractants in removing native B. This result is consistent with the literature that added B becomes less extractable with increased incubation time.

For the Twisselman (Fig. 2), Arlington (Fig. 3), Ramona (Fig. 4), and Hanford (Fig. 5) soils, B desorption was not statistically significantly different at the 95% level of confidence as a function of time at most B concentrations determined by the hot water–soluble method. The Tranquillity soil (Fig. 1) desorbed progressively less B as a function of time. The Tranquillity soil has the capacity to adsorb appreciable amounts of added B, despite containing a moderate amount of native B. The Twisselman soil (Fig. 2), which contains a large amount of native B, released appreciable amounts of native B, in addition
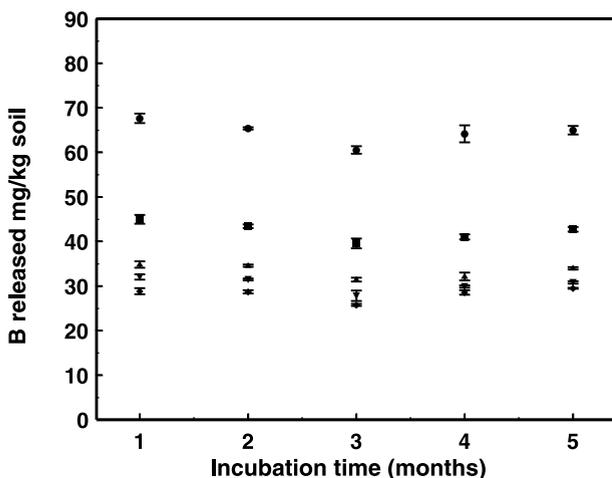

FIG. 2. Boron desorption as a function of incubation time from Twisselman soil using the hot water–soluble extraction. Boron additions are represented by diamonds = 0, down triangles = 3, up triangles = 8, squares = 20, and circles = 50 mg B/kg soil.

FIG. 4. Boron desorption as a function of incubation time from Ramona soil using the hot water–soluble extraction. Boron additions are represented by diamonds = 0, down triangles = 3, up triangles = 8, squares = 20, and circles = 50 mg B/kg soil.
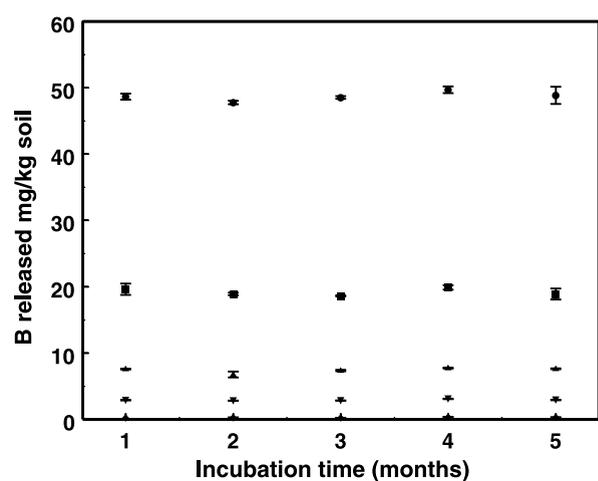
FIG. 5. Boron desorption as a function of incubation time from Hanford soil using the hot water–soluble extraction. Boron additions are represented by diamonds = 0, down triangles = 3, up triangles = 8, squares = 20, and circles = 50 mg B/kg soil.
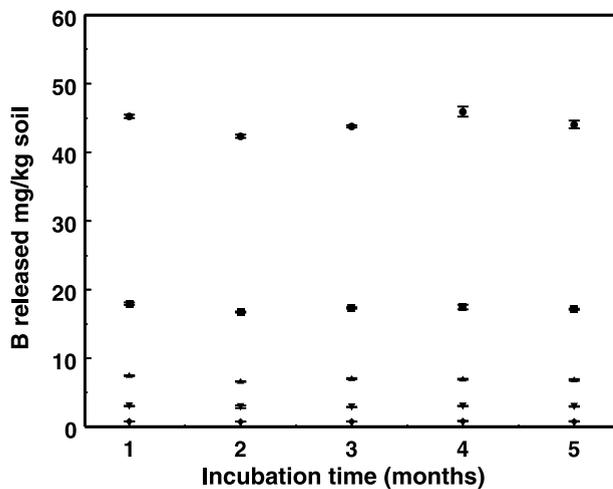
to desorbing added B. Results for hot water–soluble B are presented in the figures but show less B decreases with time than the other two extractants (Tables 3–5).

Comparison between the three extractants revealed no statistically significant differences at the 95% level of confidence in amounts of adsorbed B for the Arlington, Ramona, and Hanford soils. For the Tranquillity (Fig. 1) and Twisselman (Fig. 2) soils, the order of desorbed B amount was hot water soluble < 0.1 M NaCl extractable < DTPA-sorbitol extractable. Increased B release by the DTPA-sorbitol extraction over aqueous extracts including hot water soluble has also been observed previously for the Tranquillity soil (Goldberg et al., 2002). These two soils were the only ones containing significant quantities of native B. This result may also be because DTPA-sorbitol buffers the soil at a pH value below the native pH attained by the aqueous extracts, where B adsorption would be reduced.

Boron release stabilized for all five soils for all hot water-soluble extractions, as evidenced by slopes that were not statistically significantly different from zero at the 95% level of confidence. For the DTPA-sorbitol extraction, all slopes were not statistically significantly different from zero at the 95% level of confidence, except for all treatments for the Tranquillity soil, the 50-mg kg^{-1} treatment for the Ramona soil, and the 8-, 20-, and 50-mg kg^{-1} treatments for the Hanford soil. For the NaCl extraction, all slopes were not statistically significantly different from zero at the 95% level of confidence, except for all treatments for the Tranquillity soil and the 3- and 8-mg kg^{-1} treatments for the Arlington, Ramona, and Hanford soils. These results would indicate that a 5-month incubation period should be adequate to obtain an accurate assessment of the amount of

TABLE 3. Hot Water–Soluble B as a Function of Incubation Time and mg B/kg Soil Added

Soil		0	3	8	20	50
Tranquillity	1 month	2.57 ± 0.02	**4.27 ± 0.07**	6.65 ± 0.22	13.64 ± 0.31	**34.20 ± 0.25**
	5 months	2.48 ± 0.15	**3.68 ± 0.07**	6.06 ± 0.09	12.32 ± 0.54	**28.67 ± 0.50**
Twisselman	1 month	28.84 ± 0.69	32.03 ± 0.58	34.83 ± 0.73	44.98 ± 1.00	67.64 ± 1.08
	5 months	29.49 ± 0.10	30.80 ± 0.27	33.99 ± 0.27	42.73 ± 0.52	64.97 ± 0.95
Arlington	1 month	0.81 ± 0.01	2.82 ± 0.01	**6.86 ± 0.10**	17.13 ± 0.08	43.47 ± 0.22
	5 months	0.85 ± 0.06	2.81 ± 0.03	**6.59 ± 0.11**	16.66 ± 0.26	43.76 ± 1.04
Ramona	1 month	0.24 ± 0.01	2.89 ± 0.03	7.58 ± 0.05	19.61 ± 0.86	48.65 ± 0.46
	5 months	0.30 ± 0.02	2.92 ± 0.04	7.62 ± 0.06	18.90 ± 0.82	48.85 ± 1.30
Hanford	1 month	0.80 ± 0.01	3.02 ± 0.04	7.15 ± 0.06	**17.94 ± 0.18**	45.24 ± 0.26
	5 months	0.78 ± 0.04	2.96 ± 0.05	6.83 ± 0.13	**17.14 ± 0.08**	44.06 ± 0.56

Statistically significant decreases with time at the 95% level of confidence are indicated in boldface.

TABLE 4. The DTPA-Extractable B as a Function of Incubation Time and mg B/kg Soil Added

Soil		0	3	8	20	50
Tranquillity	1 month	**3.60 ± 0.05**	5.51 ± 0.17	**9.12 ± 0.21**	**19.06 ± 0.40**	**45.81 ± 0.86**
	5 months	**3.23 ± 0.09**	4.94 ± 0.15	**8.16 ± 0.16**	**16.92 ± 0.05**	**38.96 ± 0.37**
Twisselman	1 month	36.63 ± 0.10	39.32 ± 0.27	43.47 ± 0.21	54.72 ± 0.24	82.00 ± 0.90
	5 months	37.58 ± 0.27	40.67 ± 0.20	44.98 ± 0.38	55.80 ± 0.45	82.37 ± 0.21
Arlington	1 month	0.65 ± 0.04	2.72 ± 0.04	5.78 ± 0.99	16.80 ± 0.39	44.78 ± 2.16
	5 months	0.76 ± 0.02	2.63 ± 0.03	6.41 ± 0.12	16.42 ± 0.31	42.78 ± 2.16
Ramona	1 month	0.18 ± 0.01	**2.71 ± 0.08**	**7.49 ± 0.20**	**21.08 ± 0.50**	**51.26 ± 0.36**
	5 months	0.16 ± 0.02	**2.39 ± 0.05**	**6.87 ± 0.04**	**18.03 ± 0.10**	**47.55 ± 0.55**
Hanford	1 month	0.67 ± 0.06	**2.70 ± 0.01**	**7.08 ± 0.10**	**18.73 ± 0.50**	**48.35 ± 0.68**
	5 months	0.64 ± 0.02	**2.45 ± 0.07**	**6.25 ± 0.05**	**16.55 ± 0.13**	**44.11 ± 0.46**

Statistically significant decreases with time at the 95% level of confidence are indicated in boldface.

TABLE 5. The NaCl-Extractable B as a Function of Incubation Time and mg B/kg Soil Added

Soil		0	3	8	20	50
Tranquillity	1 month	**2.80 ± 0.02**	**4.76 ± 0.09**	**8.22 ± 0.17**	**17.22 ± 0.11**	**40.67 ± 0.93**
	5 months	**2.37 ± 0.07**	**3.84 ± 0.02**	**6.14 ± 0.07**	**12.61 ± 0.62**	**30.48 ± 0.40**
Twisselman	1 month	31.92 ± 0.39	34.18 ± 0.69	37.66 ± 0.49	**47.14 ± 0.33**	71.47 ± 1.25
	5 months	31.09 ± 0.38	33.31 ± 0.10	36.71 ± 0.83	**45.56 ± 0.40**	69.25 ± 0.10
Arlington	1 month	0.73 ± 0.02	2.84 ± 0.09	**7.20 ± 0.19**	17.04 ± 0.51	**45.15 ± 0.78**
	5 months	0.69 ± 0.02	2.62 ± 0.02	**6.45 ± 0.00**	16.51 ± 0.50	**42.52 ± 0.43**
Ramona	1 month	0.16 ± 0.04	**2.75 ± 0.01**	7.82 ± 0.28	19.44 ± 1.33	49.87 ± 1.86
	5 months	0.15 ± 0.02	**2.47 ± 0.05**	6.83 ± 0.05	18.17 ± 0.16	46.94 ± 1.54
Hanford	1 month	0.57 ± 0.03	**2.76 ± 0.06**	6.85 ± 0.16	**17.69 ± 0.07**	45.49 ± 0.25
	5 months	0.55 ± 0.03	**2.40 ± 0.11**	6.24 ± 0.03	**16.78 ± 0.19**	45.72 ± 0.39

Statistically significant decreases with time at the 95% level of confidence are indicated in boldface.

B available for release. Given that the decrease in extractable B with time is not large (except for the Tranquillity soil), a very good indication of extractable soil B may be obtained from a 1-month incubation study for most practical applications.

REFERENCES

Cihacek, L. J., and J. M. Bremner. 1979. A simplified ethylene glycol monoethyl ether procedure for assessing soil surface area. Soil Sci. Soc. Am. J. 43:821–822.

Coffin, D. E. 1963. A method for the determination of free iron oxide in soils and clays. Can. J. Soil Sci. 43:7–17.

Berger, K. C., and E. Truog. 1940. Boron deficiencies as revealed by plant and soil tests. J. Am. Soc. Agron. 32:297–301.

Elrashidi, M. A., and G. A. O'Connor. 1982. Boron sorption and desorption in soils. Soil Sci. Soc. Am. J. 46:27–31.

Gee, G. W., and J. W. Bauder. 1986. Particle-size analysis. *In* A. Klute, et al. (ed.) Methods of Soil Analysis. Part 1, 2nd Ed. Agron. Monogr. 9. ASA and SSSA, Madison, WI, pp. 383–411.

Goldberg, S., P. J. Shouse, S. M. Lesch, C. M. Grieve, J. A. Poss, H. S. Forster, and D. L. Suarez. 2002. Soil boron extractions as indicators of boron content of field-grown crops. Soil Sci. 167:720–728.

Goldberg, S., D. L. Corwin, P. J. Shouse, and D. L. Suarez. 2005. Prediction of boron adsorption by field samples of diverse textures. Soil Sci. Soc. Am. J. 69:1379–1388.

Griffin, R. A., and R. G. Burau. 1974. Kinetic and equilibrium studies of boron desorption from soil. Soil Sci. Soc. Am. Proc. 38:892–897.

Gupta, U. C. 1967. A simplified method for determining hot water-soluble boron in Podzol soils. Soil Sci. 103:424–428.

Hatcher, J. T., and C. A. Bower. 1958. Equilibria and dynamics of boron adsorption by soils. Soil Sci. 85:319–323.

Hoffman, D. J., M. B. Camardese, L. J. Lecaptain, and G. W. Pendleton. 1990. Effects of boron on growth and physiology in mallard ducks. Environ. Toxicol. Chem. 9:335–346.

Jasmund, K., and B. Lindner. 1973. Experiments on the fixation of boron by clay minerals. Proc. Int. Clay Conf. 1972:399–412.

Letho, T. 1995. Boron retention in limed forest mor. For. Ecol. Manag. 78:11–20.

Miller, R. O., B. Vaughan, and J. Kutoby-Amacher. 2000. Extraction of soil boron with DTPA-sorbitol. Soil Plant Anal. Spring:4–10.

Parks, W. L., and J. L. White. 1952. Boron retention by clay and humus systems saturated with various cations. Soil Sci. Soc. Am. Proc. 16: 298–300.

Rhoades, J. D., R. D. Ingvalson, and J. T. Hatcher. 1970. Laboratory determination of leachable soil boron. Soil Sci. Soc. Am. Proc. 34: 871–875.

Saarela, I. 1985. Plant-available boron in soils and the boron requirement of spring oilseed rapes. Ann. Agric. Fenn. 24:183–265.

Smith, G. J., and V. P. Anders. 1989. Toxic effects of boron on mallard reproduction. Environ. Toxicol. Chem. 8:943–950.

Su, C., and D. L. Suarez. 2004. Boron release from weathering of illites, serpentines, shales, and illitic/palygorskitic soils. Soil Sci. Soc. Am. J. 68:96–105.

U.S. Environmental Protection Agency. 1997. Method 3051a: Microwave-Assisted Acid Dissolution of Sediments, Sludges, Soils, and Oils, 2nd Ed. U.S. Government Printing Office, Washington, DC.