

DEVELOPMENT OF INTERNAL FOREST SOIL REFERENCE SAMPLES AND TESTING OF DIGESTION METHODS

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ABSTRACT

Our research requires determinations of total elemental concentrations of forest soils. The lack of certified forest soil reference materials led us to develop internal reference samples. Samples were collected from three soil horizons (Oa, B, and C) at three locations having forested, acidic soils similar to those we commonly analyze. A shatterbox was used to homogenize the nine samples. The Radiation Laboratory of the University of Massachusetts, Lowell completed neutron activation analyses (NA) on our samples. Our laboratory performed five digestion procedures: microwave [nitric acid (HNO_3), hydrochloric acid (HCl), fluoboric acid (HBF_4), hydrogen peroxide (H_2O_2)], block [sulfuric acid (H_2SO_4), selenious acid (H_2SeO_3), and H_2O_2], hot plate [hydrofluoric acid (HF) and perchloric acid (HClO_4)], and flux [lithium borate (LiBO_2) and lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$)]. Concentrations of phosphorus (P), iron (Fe), copper (Cu), manganese (Mn), magnesium (Mg), sodium (Na), aluminum (Al), calcium (Ca), and potassium (K) were measured by direct current plasma spectrometry (DCP). The results of the digestion procedures were compared with results from the neutron activation analyses (NA) allowing us to choose the most effective digest procedure. Each digestion procedure obtained varied elemental recovery rates (RR) ranging from Al with <10% RR for a C horizon sample digested in a block digester to Ca with >125% RR for a B horizon sample digested on a hot plate when compared to the neutron activation analyses values. Overall, the maximum recovery rates were achieved by the flux, microwave, and hot plate procedures.

INTRODUCTION

Due to the lack of certified chemical analyses for forest soil samples (especially for organic horizons), we needed to establish internal reference samples. Nine samples were collected from three forested sites in New England with acidic soils, including Oa, B, and C horizons from each site. After homogenizing the samples with a shatterbox, our laboratory performed five different digestion procedures (Figure 1). Though only Al and Ca are presented in this poster, we also measured elemental concentrations of P, Fe, Cu, Mn, Mg, Na, and K by DCP. Neutron activation analyses on the nine samples were completed by the Radiation Laboratory at the University of Massachusetts, Lowell. The NA results represented the expected

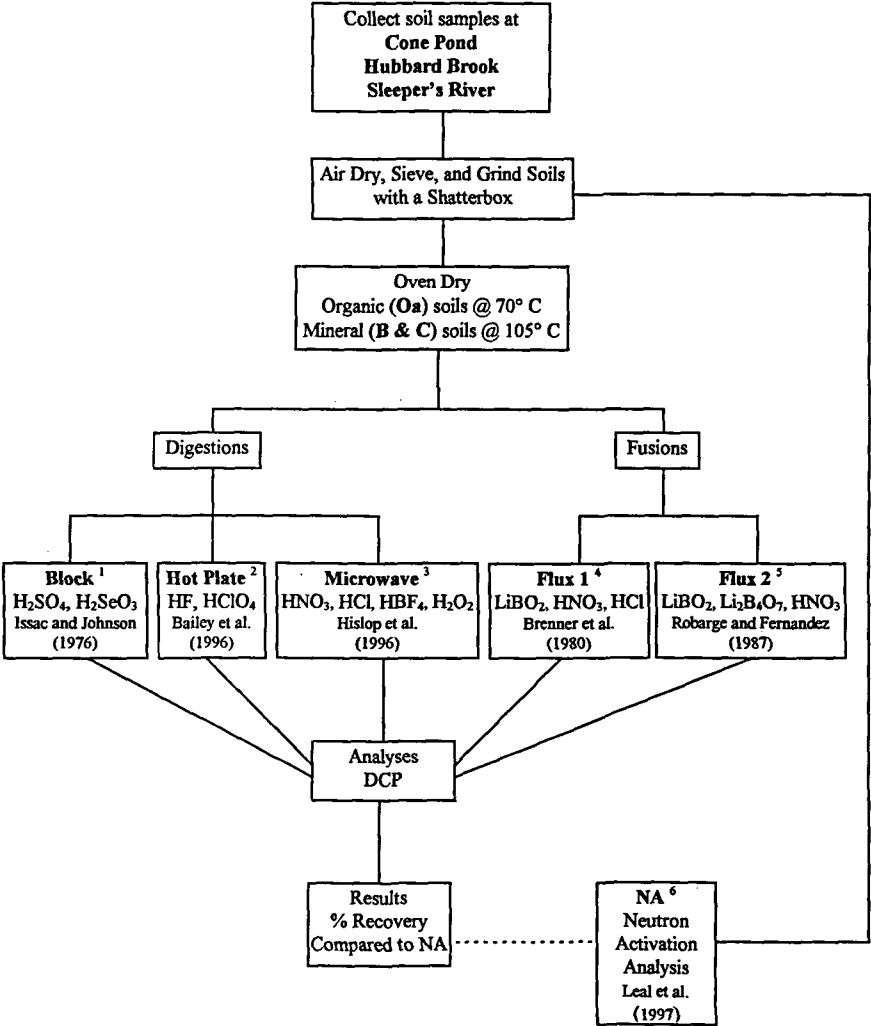


FIGURE 1. Flow chart for sampling and laboratory procedure.

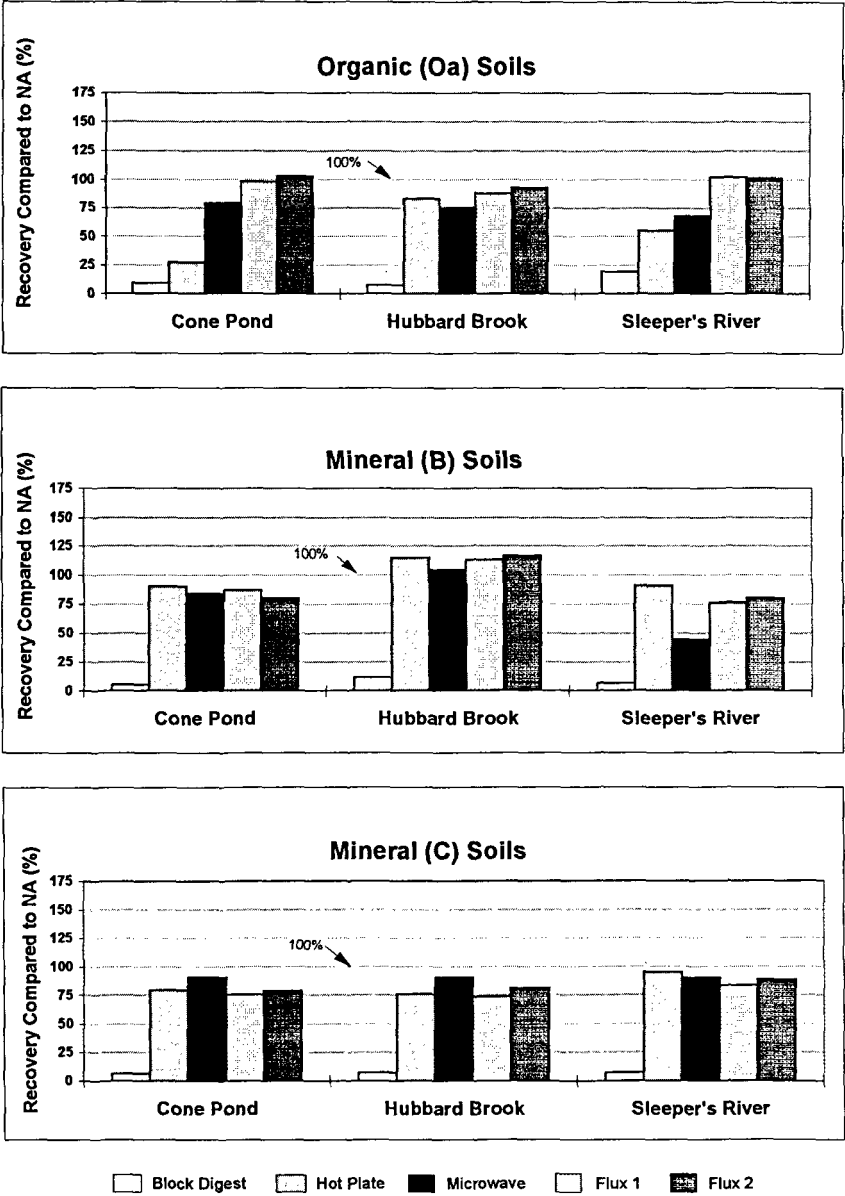


FIGURE 2. Aluminum recovery rates for three soil horizons at three New England sites.

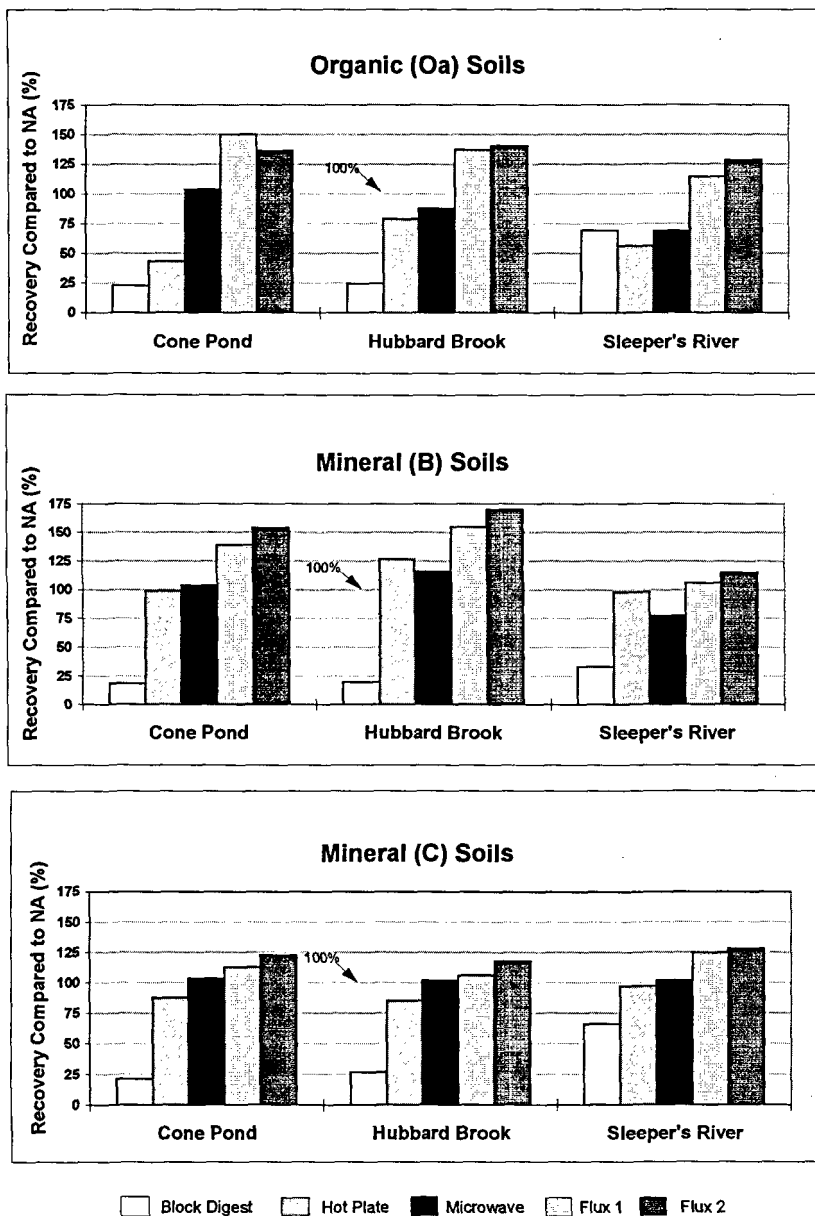


FIGURE 3. Calcium recovery rates for three soil horizons at three New England forested sites.

values from which we could determine recovery rates of the five different digest procedures (Figure 1).

DISCUSSION

Total Aluminum

The block digest method had as low as a 7% recovery rate for Al (Figure 2). Compared to NA, this method does not obtain all the Al in any of the samples either by site or by horizon. The greatest recovery was for the Oa horizons by the flux 2 method. This method obtained the range of 93 to 103% recovery for the three samples. The flux 2 method achieved between 80 to 117% on all three B horizon samples. The microwave procedure produced >90% recovery for all C horizon soils (Figure 2).

Total Calcium

The block digestion method had better recovery rates for Ca on the organic (Oa) soils (Figure 3). The block digestion recovery rates drop off to <35% with the B horizon samples and <67% with the C horizon soils. Both flux procedures achieved between 114 to 171% recovery on all organic samples. The microwave technique produced recoveries between 68 and 104% for organic soils with two samples above 80%. The B horizon soils had a recovery range from 98 to 127% for the hot plate technique. Both flux methods were above the 105% recovery rate for the B horizon soils. The B soils recovery rates were between 77 to 117% for the microwave procedure. For both flux procedures, Ca recovery rates were >106% for the C horizon samples. The recovery rate for the hot plate method on the C soils was <98%. The microwave technique obtained Ca recovery rates between 101 to 103% for the C horizon samples (Figure 3).

CONCLUSION

In general, each horizon and each site responded differently with each method and element. It is apparent that organic content plays a definite role in the dissolution of some elements. One will have to choose the method that achieves the best elemental recovery for their needs.

RECOMMENDATIONS

1. Prior to the digestion of unknown samples, check the digest or flux method recovery rates with matched reference materials.
2. If a matched reference material is not available, recognize the need to develop an internal reference sample similar to the unknowns.
3. Realize that the recovery rates might differ from element to element. You might have to choose a procedure based on a single element recovery rate and be willing to accept lower accuracy for one element than another.

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