

## USING $^{14}\text{C}$ AS A TRACER OF CARBON ACCUMULATION AND TURNOVER IN SOILS

G. M. MILTON and S. J. KRAMER

Environmental Research Branch, AECL Chalk River Laboratories, Chalk River  
Ontario, Canada, K0J 1J0

**ABSTRACT.** Three very different Canadian soils—clay soils of the St. Lawrence Lowlands, sandy forest soils of the Ottawa Valley, and organic-rich sediments from a wetland on the Canadian Shield—have been cored, sliced and separated into different density fractions, and the radiocarbon content of these soil fractions measured. In two of the areas sampled, cores were obtained close to operating nuclear reactors, as well as from beyond their region of influence. As a consequence, it has been possible to ascertain the depths of penetration of both the weapons-testing pulse (peaking in 1963), and a 25–50-yr chronic reactor input of  $^{14}\text{C}$ .

The percentage of carbon stored in different density fractions varied with soil type. Turnover times for bulk soil organic carbon, estimated from soil degassing rates, have been compared with those predicated on the residual “bomb”  $^{14}\text{C}$  in background cores, and/or on the ratio of reactor-emitted  $^{14}\text{C}$  retained in the soils to the total deposited during the lifetime of operation. Residence times for the heavy carbon fraction present at depths below the influence of anthropogenic inputs have also been estimated.

The accumulated data will be incorporated in a revised soil model, adjusted for the parameters deemed to be most important to carbon turnover rates under Canadian conditions.

### INTRODUCTION

Information on the parameters most affecting carbon sequestration in soils are of great importance to modelers of radiocarbon behavior, to agricultural and forestry specialists, and also to all those who are generating, or likely to suffer from the consequences of, increased  $\text{CO}_2$  in the atmosphere. A recent publication of *Forestry Canada* (Kurz *et al.* 1992) has provided improved information on biomass and soil carbon pools in different forest sectors of Canada, and outlined areas requiring further studies, *e.g.*, soil carbon content and dynamics, especially in peatlands. While the chief purpose of the work discussed in this paper was the identification of the extent to which reactor emissions of  $^{14}\text{C}$  are being sequestered in soils near CANDU reactor sites, and for how long, in order to improve existing models, we believe the information gained can be applied to some of the broader questions relating to carbon cycling.

Carbon in soils tends to fall into three main pools: a fast turnover pool (small detrital,  $t_{1/2}$  3–20 yr), a medium turnover pool (large detrital,  $t_{1/2}$  20–100 yr) and a slow turnover pool (humified organic matter,  $t_{1/2}$  >100 yr). The mean life may vary depending on soil characteristics, such as soil density ( $d$  in  $\text{g cm}^{-3}$ ). For example: the  $1 < d < 2$  fraction may contain the small quantity of long-lived carbon present in a forest soil, but only the intermediate age fraction in a clay soil. Similarly, the carbon fraction with  $d < 1$  in an anaerobic soil is found to survive much longer than does a similar fraction in an aerobic soil.

The primary literature concerning the use of the  $^{14}\text{C}$  spike resulting from nuclear weapons testing as a tracer of “new” carbon behavior in soils, and the considerable body of knowledge available on the average residence times of carbon in the various soil carbon pools, was reviewed up to 1994 in a paper reporting on the early findings in this study (Milton *et al.* 1995). More recent measurements elsewhere have delineated the effects of temperature (altitude and latitude) on carbon turnover rates (Trumbore, Chadwick and Amundson 1996, and Alexandrovskiy and Chichagova 1998, respectively), and looked more closely at the most resistant portions of the heavy soil fraction (*e.g.*, Harrison 1996; Leavitt, Follett and Paul 1996; Cherkinsky 1996).

During the past 50 yr, an additional source of anthropogenic  $^{14}\text{C}$  has been available for field measurements, but very little use has been made of it by soil scientists. We refer to the small but very measurable quantities of  $^{14}\text{C}$  emitted from the stacks of nuclear generating stations. While the dispersion of this signal is highly localized, in those areas adjacent to the reactors it provides a chronic pulse in new vegetation, and hence in the soils. Our study was designed to tell us whether the behavior of this chronic pulse mimics that of the earlier shorter stratospheric input during weapons testing, and what are the differences in its behavior in two quite different soils. A third soil, which has received only the bomb pulse as anthropogenic input, has been included in order to follow the movement of this spike under the anaerobic conditions prevailing in a seasonal wetland.

### Sampling Locations

All three sites investigated in this study are located in the province of Ontario, Canada. Sampling locations are indicated in Figure 1, and information on soil type and climatic conditions previously reported in Milton *et al.* (1995) have been enlarged to include the third site (Table 1).

The Pickering Nuclear Generating Station (PNGS) is located on the north shore of Lake Ontario, *ca.* 50 km east of Toronto. The general geological designation of the region is the St. Lawrence Lowlands. Soils in the area are primarily grey-brown podzol of the Darlington Series, resting on the St. Lawrence platform of Paleozoic shales and carbonates. The climate is temperate. The original forests growing at the site were extensively cleared for cultivation following settlement by European and North American immigrants in the late 18th century. The first reactor units at this station were brought into service in the early 1970s.

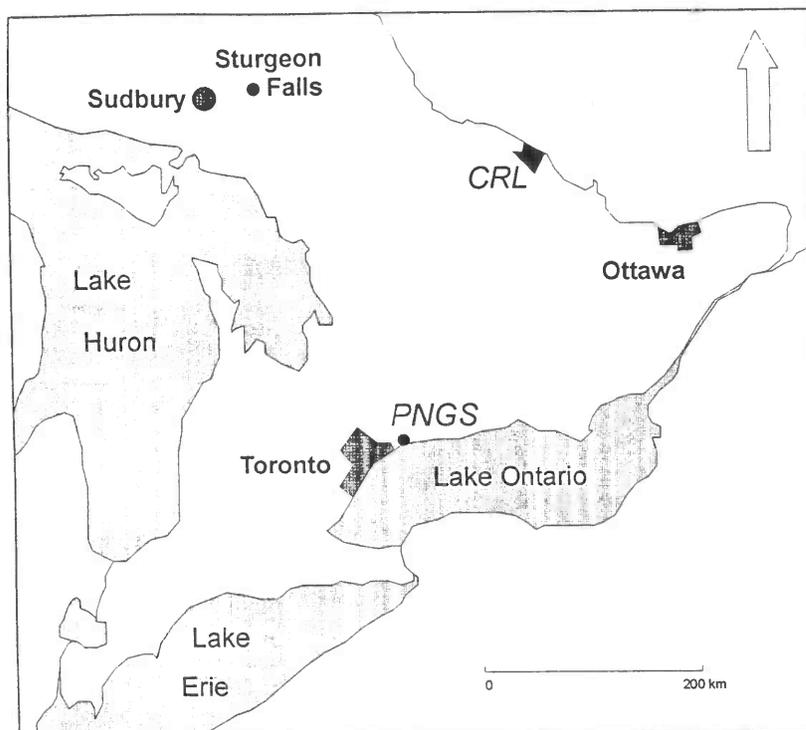


Fig. 1. Map of Ontario, indicating the geographic location of our three study areas

TABLE 1. General Characteristics of the Study Sites

	Pickering	Chalk River	Sturgeon Falls
Latitude/principal land use	40°N suburban housing, agriculture	46°N lumbering	46.3°N lumbering
Climate/temperature, precipitation, climate zone	-20 to 30°C -830 mm 90% rain 10% snow Zone 5(a)*	-30 to 30°C -900 mm 80% rain 20% snow Zone 4(b)*	-30 to 30°C ~900 mm 80% main 20% snow Zone 4(a)*
Soil type†	Darlington Series, grey-brown podzol	Upland Series, orthic humo-ferric podzol	Azilda Series, orthic humic gleysol
Underlying rock	St. Lawrence platform, Paleo- zoic shales, carbon- ates	Pre-Cambrian granites & gneisses	Precambrian granites & gneisses
Vegetation	Deciduous trees/ scrub/grass	Mixed temperate forest/scrub	Mixed temperate- boreal forest, swampy under- growth
Net primary productivity (g × c × m <sup>-2</sup> × a <sup>-1</sup> )‡	240–560	240–560	400–740
Soil pH	7.7	4.9	3.4–4.6

\*Canadian Department of Agriculture

†In Canadian Classification System

‡From Whittaker and Likens (1973)

The Chalk River Laboratories (CRL) are located in the upper Ottawa Valley, *ca.* 200 km west of Ottawa, while our third site lies just outside Sturgeon Falls, north of Lake Huron and *ca.* 50 km from Sudbury, the closest urban center. (A detailed description of this site can be found in Robertson and Cherry (1989).) Both areas lie on the Canadian Shield of Precambrian granites and gneisses, and both have been, and continue to be, extensively logged. The climatic zone is rated north-temperate, with a considerably shorter growing season than that of the St. Lawrence Lowlands. While the vegetation at the CRL and Sturgeon Falls locations is very similar, and both sites are recharge zones and very acid, the former is situated on a bench high above the Ottawa River, while the latter site is a seasonal wetland, with the water table essentially at the surface during spring runoff and the fall rainy season. As a consequence, the orthic humo-ferric podzol at the upland site (CRL) is very thin, while close to 30 cm of organic material (orthic humic gleysol) has built up on the top of the podzol at the wetland site. The NRU reactor at CRL has been in operation since the late 1950s; there are no operating reactors within the sphere of atmospheric influence of Sturgeon Falls.

Cores taken at sites adjacent to, but beyond the influence of, the reactor locations (Petawawa in the Ottawa Valley and Peterborough in the St. Lawrence Lowlands) were used as "background" cores for the two reactor sites. We assume that anthropogenic <sup>14</sup>C inputs at these locations have been limited to those arising from nuclear weapons testing.

## MATERIALS AND METHODS

The methods of sampling, fractionation and  $^{14}\text{C}$  analysis have remained essentially unchanged from those described in Milton *et al.* (1995). In brief, core sections were sieved to remove components  $>2$  mm, oven-dried and digested in dilute HCl to remove carbonates, followed by washing to a neutral pH. Density fractionation of some sections into three components was carried out using sodium metatungstate (density  $2.82\text{ g cm}^{-3}$ ). To avoid potential density gradients in the liquid phase (Plewinsky and Kamps 1984), we have refrained from centrifuging these solutions, allowing sufficient time for gravity separation.

In a number of cases, insufficient material was recovered following fractionation, particularly in the  $d<1$  fractions, to permit  $^{14}\text{C}$  measurement by our usual technique of combustion followed by liquid scintillation counting (LSC). These samples were sent to the Isotracer Laboratory, University of Toronto, for analysis by accelerator mass spectrometry (AMS).

To check for possible land disturbance during the last 40 yr (*i.e.*, since the introduction of anthropogenic tracers to our soil cores following nuclear weapons testing), we have measured the  $^{137}\text{Cs}$  content of the soil profiles by high-resolution gamma spectrometry, prior to any treatment of the samples beyond slicing, drying and determination of the bulk mass density.

## RESULTS AND DISCUSSION

### Bulk Mass Density and Organic Carbon Profiles

Bulk mass density (*i.e.*, oven-dried mass/field volume) and organic carbon are inversely correlated in the Ottawa Valley and St. Lawrence Lowland soils (Figs. 2a,b; the data points represent averages for a large number of cores from both reactor and background sites. Lines drawn in these and all subsequent graphs are intended as guides for the eye only.). The inverse correlation is less apparent in the wetland soil, especially in the top 10 cm.

The organic carbon stored in the top 20 cm of the profile has been summed to compare our measured value for southern Ontario ( $6.4\text{ kg C m}^{-2}$ ) with the estimate of Trumbore (1993) for Sierra Nevada Utsis (0–23 cm,  $5.2\text{ kg C m}^{-2}$ ), a not very dissimilar soil in another temperate zone. The agreement between the two is reasonably good.

The total organic carbon contained in the profile (top 40–50 cm) can be used in conjunction with measured de-gassing rates to estimate bulk turnover times for organic carbon (Table 2). Bulk carbon turnover rates were shown to be low in both the Ottawa Valley and the St. Lawrence Lowlands. However, we had expected, on the basis of total carbon present in the profile, that the Ottawa Valley turnover rate would be even shorter than this. These values are closely tied to the large percentage of organic

TABLE 2. Bulk Carbon Turnover Rates Measured in the Top 40 cm of Cores from Three Sites

Site	Total organic carbon ( $\text{kgC m}^{-2}$ )		Soil respiration ( $\text{gC m}^{-2}\text{ a}^{-1}$ )		Bulk turnover rate (yr)	
	Measured	Estimated*	Measured†	Estimated*	Measured	Estimated*
Ottawa Valley	6.5	6.9–13.4	350	463	19	15–29
St. Lawrence Lowlands	11.5	7.9–13.4	528	500	22	16–27
Seasonal Wetland	16.1	21	--	140	--	92

\*Values taken from Raich and Schlesinger (1992)

†Adjusted to reflect assumption that 30% soil respiration derived from root respiration

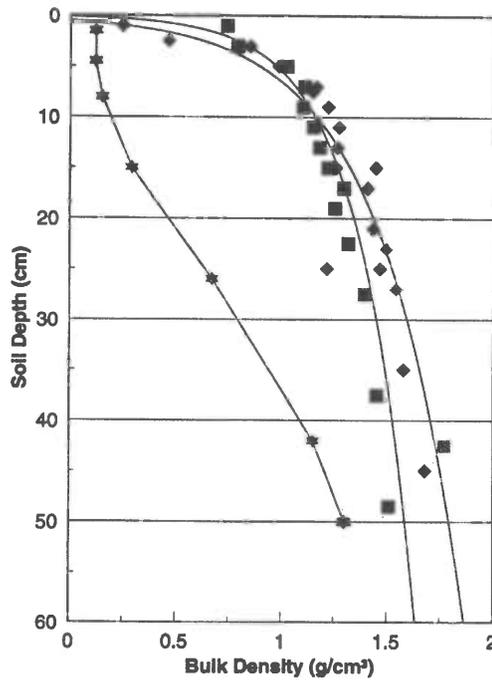


Fig. 2a. Depth profiles of bulk mass density. ■ = St. Lawrence Lowlands, ◆ = Ottawa Valley, \* = wetland near Sturgeon Falls.

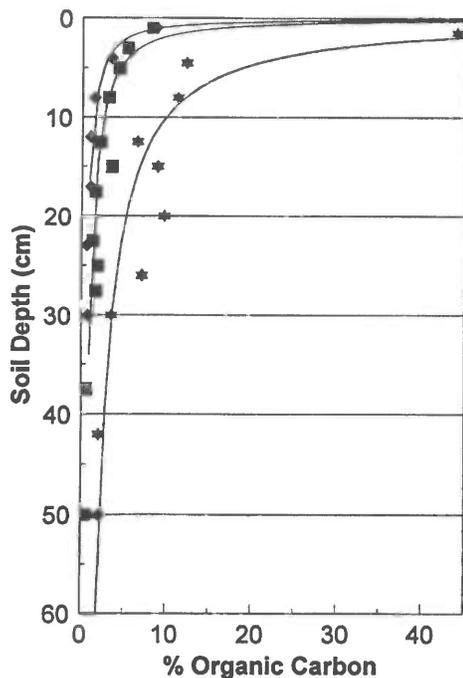


Fig. 2b. Depth profiles of percent organic carbon. ■ = St. Lawrence Lowlands, ◆ = Ottawa Valley, \* = wetland near Sturgeon Falls.

carbon that is present in litter and low density materials in the surface layer, which are broken down most readily by microbial action, which is accelerated at low pH. Degassing rates were measured at this site at the end of the growing season, and consequently may be too low. Bulk turnover rates were estimated to be as long as 100 yr under the anaerobic conditions existing in a seasonal wetland.

### Bulk $^{14}\text{C}$ Profiles

Bulk profiles of  $^{14}\text{C}$  with depth at CRL and PNGS were presented in Milton *et al.* (1995) (Fig. 3a); these are repeated here to allow comparison with three background cores (Fig. 3b). The total  $^{14}\text{C}$  activity in the top 20 cm of the background cores, in which we expect to see the major influence of the weapons testing pulse, ranges from 0.9 kBq m<sup>-2</sup> in the Ottawa Valley soil to ca. 1.4 kBq m<sup>-2</sup> in southern Ontario. The total  $^{137}\text{Cs}$  present in the Ottawa Valley cores is close the expected signal at this time, indicating that there has been very little soil disturbance at these coring sites since the 1950s. Consequently, we believe this difference between the residual “bomb”  $^{14}\text{C}$  remaining in these background cores from Petawawa and Peterborough supports our earlier hypothesis that the bulk turnover time reported for the Ottawa Valley in Table 2 is too long. The residual “bomb” addition is considerably greater in the wetland soil, at  $\approx 3.5$  kBq m<sup>-2</sup>, reflecting the longer bulk turnover time estimated in Table 2. Assuming the accuracy of the 100-yr turnover time at Sturgeon Falls, we estimate bulk turnover times of 14 yr and 20 yr at Petawawa-CRL and Peterborough-PNGS, respectively.

Although it is difficult to verify the absence of “bomb”  $^{14}\text{C}$  at depth in these cores, residence times for the small carbon component present below the depth of anthropogenic inputs (>35–50 cm) are estimated from the  $^{14}\text{C}$  specific activity present to be >1200 yr at Petawawa (Ottawa Valley), >1500 yr in the wetland soil, and >1600 yr at Peterborough (St. Lawrence Lowlands). (These latter values are probably low because, as indicated in a subsequent section, strong acid hydrolysis should have been performed prior to measurement.)

Cores taken near the two reactor sites (Fig. 3a) show the influence of heightened inputs of  $^{14}\text{C}$  down to greater depths than were estimated for “bomb” penetration. This is probably not a true difference in penetration, but rather an indication of our inability to detect a small addition of “new” carbon when carbon concentrations are so low, unless its specific activity is markedly enhanced above that originally present.

These heightened inputs can be estimated from the difference between the total activity stored in the top 40 cm (ca. 2.0 kBq m<sup>-2</sup> at CRL and 8 kBq m<sup>-2</sup> at PNGS), and the activity measured in the background cores (Table 3). The ratios of these inputs to the total estimated deposition at those locations (Table 4) again indicate that the turnover time at Chalk River is indeed shorter than that at Pickering, even if the longer time of operation of the NRU reactor is taken into account. At both sites, it must be assumed that at least 50–75% of the year-end litter is lost to the atmosphere without ever entering the soil column.

### Fractionation Profiles for PNGS and CRL Cores

Three-dimensional profiles of soil density and organic carbon distributions are presented in Figures 4a,b and 5a,b, respectively. As noted earlier, particle mass densities increase very sharply with depth in both soils; in the Ottawa Valley soil, the bulk of the organic carbon is present in the upper 10 cm only. While the light and intermediate fractions make up only a small percentage of the soil at this site, the light fraction is a significant contributor to carbon storage in the top 10 cm and the intermediate fraction plays a major role at all depths studied. It is not surprising, then, that the carbon turnover time for these soils is very short.

Soils of density >2 predominate in the St. Lawrence Lowlands core at all depths. However, in this case most of the carbon present is associated with that fraction, although the intermediate fraction plays an important role down to 30 cm. Carbon turnover times would be expected to be somewhat longer in this soil than in one in which the carbon is predominantly in the light fraction.

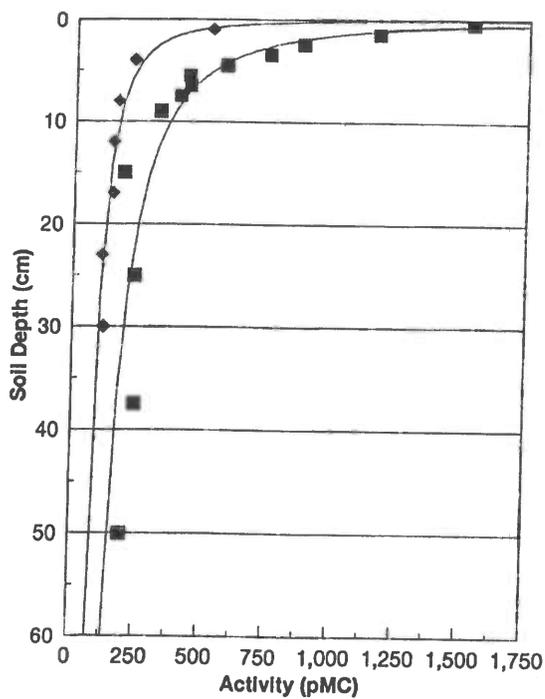


Fig. 3a. Depth profiles of  $^{14}\text{C}$  in bulk soil samples from two reactor sites. ■ = PNGS, ◆ = CRL. Statistical errors  $\leq 5\%$ .

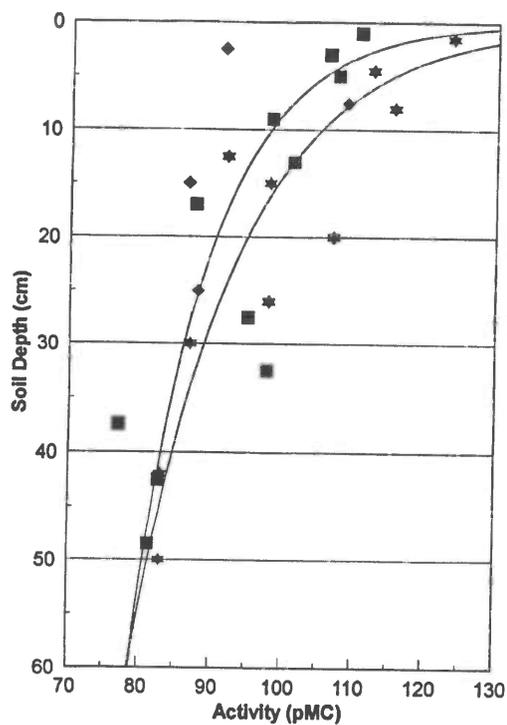


Fig. 3b. Depth profiles of  $^{14}\text{C}$  in bulk "background" soils, far from reactor sites. ■ = St. Lawrence Lowlands, ◆ = Ottawa Valley, \* = wetland near Sturgeon Falls. Statistical errors  $\leq 10\%$ .

TABLE 3.  $^{14}\text{C}$  Storage in the Top 40 cm of Cores from "Background" and Reactor Sites

Site	Depth of anthropogenic $^{14}\text{C}$ penetration (cm)	Estimated total $^{14}\text{C}$ (kBq m $^{-2}$ )
<i>Ottawa Valley</i>	30	
a) CRL (.25 km from reactor)		2.0
b) Background		0.9
		1.1
<i>St. Lawrence Lowland</i>	~40	
a) PNGS (0.9 km from reactor)		7.6
b) Background		1.4
		6.2
<i>Sturgeon Falls</i>	~50	
a) Background		3.5

TABLE 4. Estimates of the Ratio of Deposited to Stored  $^{14}\text{C}$  in Soils Near Reactor Sites

Site	Lifetime of operations	Distance from stack (km)	Estimated $^{14}\text{C}$ deposited (kBq m $^{-2}$ )	Reactor $^{14}\text{C}$ stored in soil (kBq m $^{-2}$ )	Estimated ratio stored/deposited
CRL	42	0.25	16	1.1	0.07
PNGS	25	0.9	44	6.2	0.14

#### Depth Profiles of $^{14}\text{C}$ in Different Density Fractions

Depth profiles of  $^{14}\text{C}$  associated with the three density fractions are shown in Figures 6a,b. Summing the product of carbon content times specific activity at each depth has provided bulk profile values within 10–15% of those previously reported in Figure 3a, providing substantial credibility for our analytical methods and measurements. At CRL, the trends for all three fractions are very similar. Unfortunately, an organic content <0.3% below 20 cm has made it extremely difficult to measure  $^{14}\text{C}$  below these depths.

Quite different trends are visible in the PNGS data. No significant change in specific activity is seen throughout the profile in the very light material. However, specific activities of the carbon associated with the intermediate and heavy fractions decrease progressively down to  $\approx 15$  cm, and then start to rise with increasing depth. No accompanying rise in stable carbon or bulk  $^{14}\text{C}$  had been noted (Figs. 2b and 3a, respectively). In the case of the bulk profile it is possible that pretreatment of these samples had been inadequate to remove all the inorganic carbon present at these depths.

To explain the increase in specific activity observed with depth in the fractionated samples, a mechanism must be invoked whereby high specific activity  $^{14}\text{C}$  can build up at depth. Two possible hypotheses have been considered. The first suggests that a small percentage of the fresh near-surface carbon is being leached, and is transported downward as dissolved organic carbon (DOC) with recharging precipitation containing dissolved  $\text{CO}_2$ , as bicarbonate ion. (The pH of this soil is 7.7.) The water table is known to be close to the surface in the region from which the cores were obtained. As it approaches this depth, the solution becomes saturated with bicarbonate, causing the precipitation of  $\text{CaCO}_3$  which could provide a nucleus for flocculation of the DOC. The dilute acid treatment given all samples prior to analysis may dissolve this bicarbonate but leave the very small quantities of re-agglomerated organic carbon still attached to soil particles. As mentioned earlier, very low organic

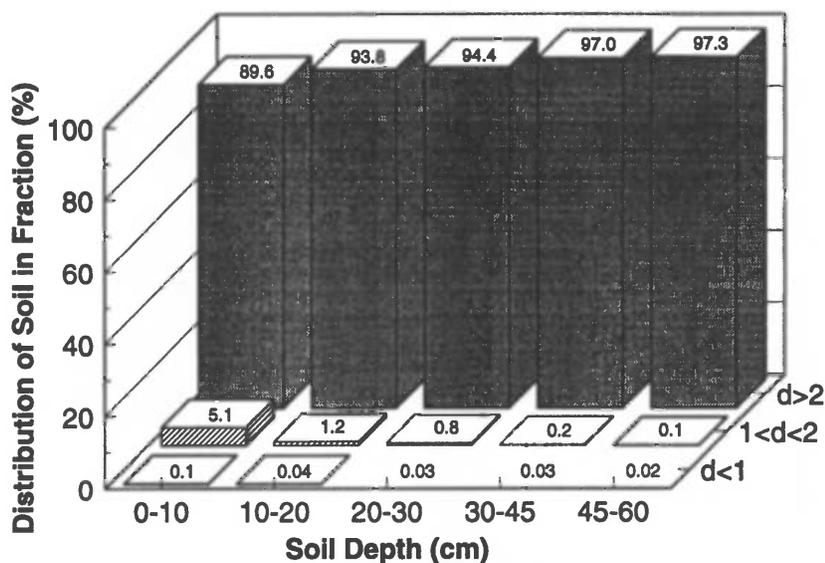


Fig 4a. 3-D depth profiles of soil density distribution at CRL

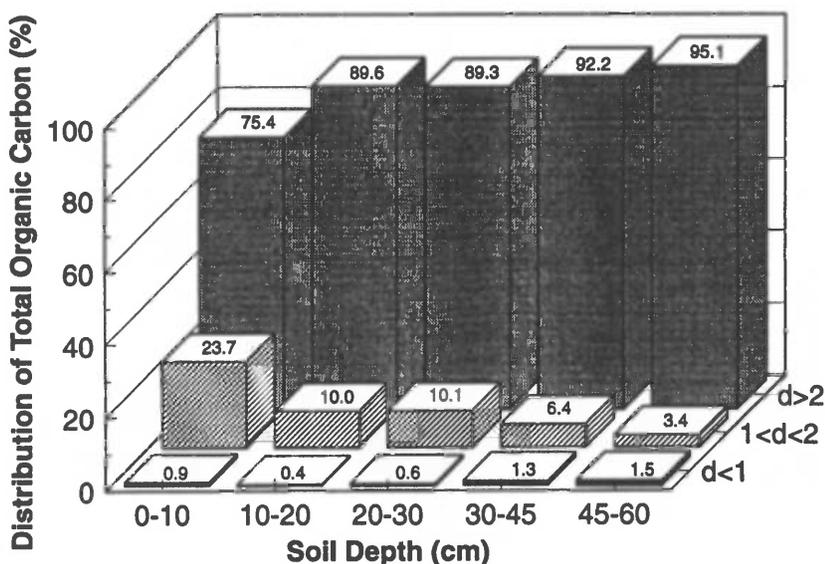


Fig. 4b. 3-D depth profiles illustrating the distribution of organic carbon in three density fractions at CRL, as a percentage of the total carbon recorded at each depth

carbon content made it impossible to look for a similar phenomenon at depth in the CRL profile; however, the low pH of this soil ( $<4$ ) would preclude bicarbonate ion formation in soil pore water. This would also be true for the anoxic wetland soil. In order to test this hypothesis, we sent a suite of core samples for x-ray diffraction measurements to identify any changes in concentrations of carbonate and other accessory minerals with depth. Preliminary results have not indicated significant support for this hypothesis.

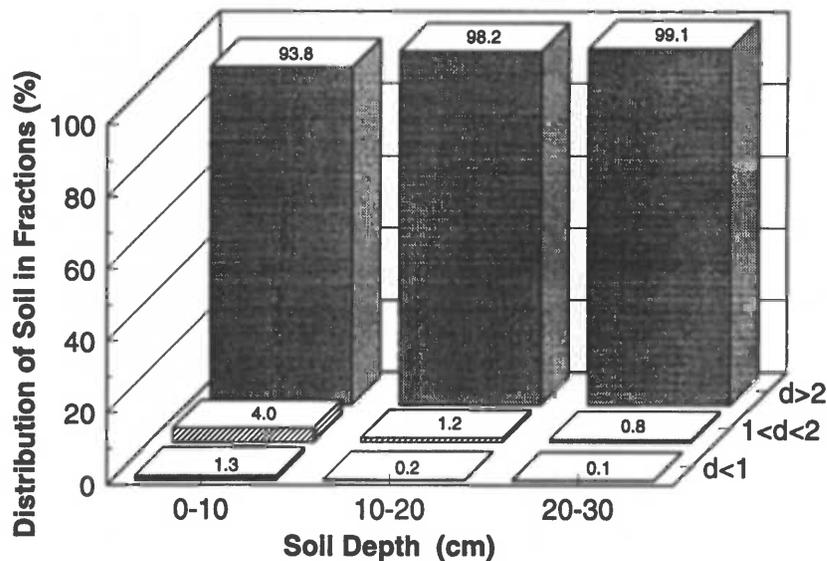


Fig. 5a. 3-D depth profiles of soil density distribution at PNGS

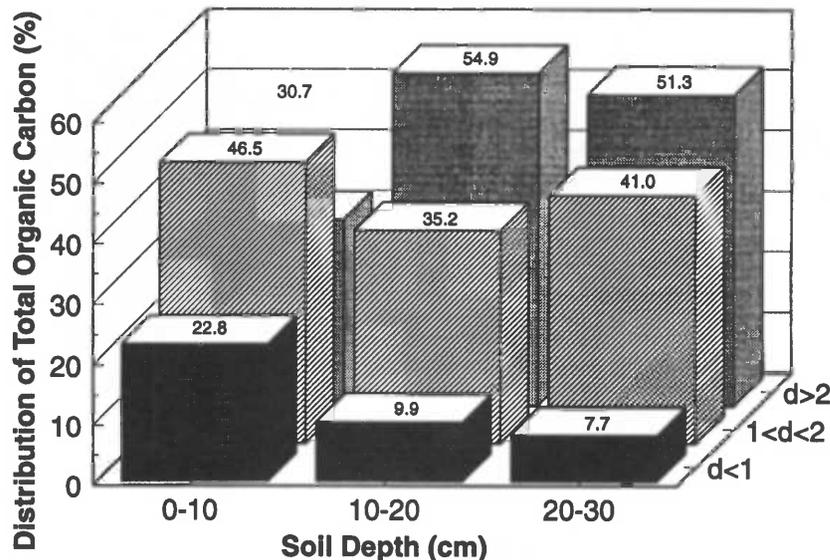


Fig. 5b. 3-D depth profiles illustrating the distribution of organic carbon in three density fractions at PNGS, as a percentage of the total carbon received at each depth

Our second hypothesis postulates that the newly formed high specific activity carbon is being moved down in the profile by root action, and is deposited there primarily as protein exudates.  $\delta^{13}\text{C}$  measurements of bulk and fractionated core samples indicate a decrease of  $\approx 1\%$  in the intermediate and heavy fractions below 30 cm, the depth at which  $^{14}\text{C}$  values increase. This would suggest the involvement of a metabolic process such as protein production causing isotopic fractionation. Transformation into other products by microbial biomass might cause further fractionation. However, additional work is required to validate this argument.

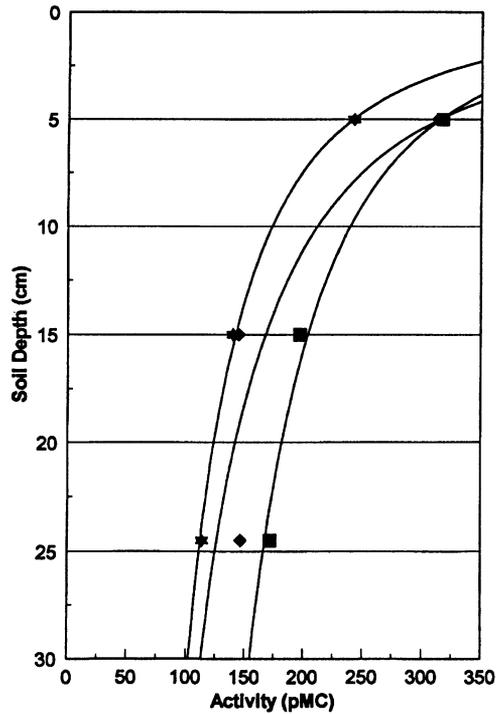


Fig. 6a. Depth profiles of <sup>14</sup>C in three density fractions at CRL. ■ = light fraction (d<1), ◆ = intermediate fraction (1<d<2), \* = heavy fraction (d>2). Statistical errors ≤10%.

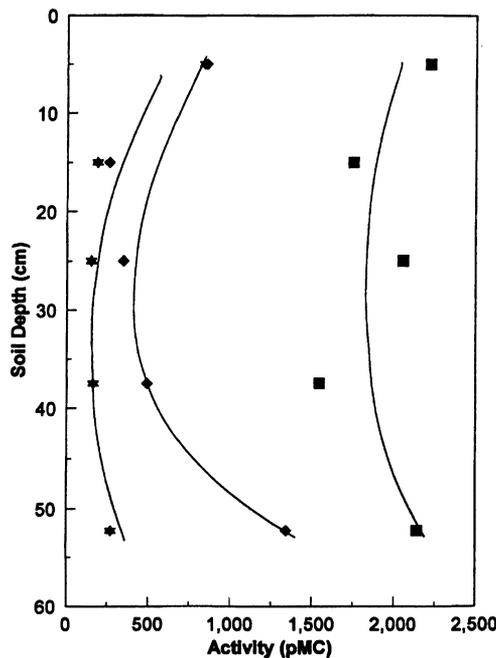


Fig. 6b. Depth profiles of <sup>14</sup>C in three density fractions at PNGS. ■ = light fraction, ◆ = intermediate fraction, \* = heavy fraction. Statistical errors ≤5% for light and intermediate fractions, ≤10% for heavy fraction.

Changes in  $\delta^{13}\text{C}$  have been used previously to investigate the turnover of microbial biomass and active fractions of soil organic matter (Ryan, Aravena and Gilham 1995). Use of the two isotopes in experiments performed in an area of heightened atmospheric <sup>14</sup>C signal might provide valuable insights on this topic.

### Acid Hydrolysis of the Heavy Fraction Prior to "Age" Estimation

It has been pointed out recently (Harrison 1996) that treatment of a soil sample with 6N HCL prior to analysis will remove the less recalcitrant fractions of carbon, and hence provide a more accurate value for the residence time of this material. We have analyzed two samples from one PNGS core, with and without this acid treatment (Table 5). The first sample came from the  $d>2$  fraction in the 0–10 cm slice, and both treatments gave essentially the same result. However, a similar treatment of a sample from the  $d>2$  fraction in the  $>45$  cm section gave two very different values, indicating that some of this material is more easily solubilized and is younger than the more recalcitrant carbon. These data support the earlier indication of "new" carbon being moved to depth in the profile.

TABLE 5. Effect of Acid Hydrolysis on Specific Activity in the Heavy Fraction of the PNGS Core

Fraction	$^{14}\text{C}$ (acid)	$^{14}\text{C}$ (no acid)	Errors
	Bq kgC <sup>-1</sup>	Bq kgC <sup>-1</sup>	
0–10 cm	1770	1892	±5%
>45 cm	437	612	±10%

### CONCLUSION

Soil profiles studied show increasing percentages of organic carbon as follows: (an Ottawa Valley upland forest site) < (a clay soil from the St. Lawrence Lowlands) < (an anoxic wetland site north of Lake Huron). Although concomitant increasing bulk turnover times are to be expected, estimates based on both literature values and our own soil degassing measurements indicate very similar turnover times in the Ottawa Valley and the St. Lawrence Lowlands ( $\approx 20$  yr). However, estimates based both on the percentage of the weapons-testing pulse remaining in background cores, and on the percentage of reactor  $^{14}\text{C}$  remaining in cores taken near reactor sites, have indicated that bulk turnover times are closer to 14 yr at CRL. Depth profiles of three density fractions in two of these soils support the site-specific estimates based on  $^{14}\text{C}$  measurements. They indicate that in the sandy matrix, the bulk of the organic carbon is in the top 10 cm, in which the light and intermediate fractions predominate. A low pH supports the microbial population required for rapid turnover. In contrast, most of the organic carbon in the clay soil is in the intermediate and heavy fractions even in the top 10 cm. Higher pH levels slow down microbial actions, permitting some of the carbon to be altered to more resistant forms that do not break down so rapidly. Bulk turnover times are consequently somewhat longer in this matrix. The much longer bulk turnover times estimated for the seasonally anoxic soils indicate that oxygen is an even more important parameter for microbial growth than is pH.

Profiles of reactor  $^{14}\text{C}$  stored in two of these soils indicate that these additions mirror the behavior of stable carbon. Depths of penetration of the "new"  $^{14}\text{C}$  parallel increasing carbon turnover times. Residence times for the heavy fraction of the small organic carbon component below the depth of anthropogenic inputs are  $>1000$  yr at all three sites.

A small buildup of high specific activity  $^{14}\text{C}$  observed at *ca.* 50 cm in a clay soil near one reactor site may lead to a new approach to studying the mechanisms involved in the cycling of the active fractions of soil organic matter (SOM). At both reactor sites, the potential for significant long-term buildup of  $^{14}\text{C}$  is very low.

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