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AMS ^{14}C MEASUREMENTS OF FRACTIONATED SOIL ORGANIC MATTER: AN APPROACH TO DECIPHERING THE SOIL CARBON CYCLE

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ABSTRACT. ^{14}C measurements are reported for fractionated soil organic matter from a genetic soil sequence which was sampled several times during the period of atmospheric nuclear weapons testing. Fractionation of the soils by density followed by acid hydrolysis was successful in separating the organic matter into components with mean residence times for carbon ranging from 5 to 20 years (low density fraction) to several thousand years (residue after acid hydrolysis). Comparison of the infiltration of bomb ^{14}C into the vertical soil profile with the distribution of ^{137}Cs, gives clues as to the mechanism (most probably dissolved transport) for importing carbon into deeper soil layers.

INTRODUCTION

An estimated 1300 to 1500 $\times$ 10^{15}g of carbon is sequestered as organic matter in soils (Schlesinger, 1977; Post *et al,* 1982). This is roughly twice the amount of carbon present either in the atmosphere as CO_2 or as land biosphere (Whittaker & Likens, 1973), and 200 times the amount of CO_2 added to the atmosphere annually by fossil fuel burning (Rotty, 1977). To understand the evolution of soil humic material, and to better incorporate the organic matter in soils into models of the global carbon cycle, it is necessary to quantify the relative amounts and turnover times of labile and refractory carbon in soil organic matter, and how these vary with parameters such as climate, vegetation and geography.

^{14}C dating of bulk soil organic matter gives ^{14}C ages which range from over-modern (containing ^{14}C produced by atmospheric nuclear weapons testing) to > 10,000 yr. Because many of the published ^{14}C measurements of soil organic material were made since the 1960s, the reported ages reflect varying degrees of bomb ^{14}C contamination. To correctly interpret the radiocarbon age of soil organic matter as a mean residence time, ^{14}C measurements must be made of pre-bomb soils.

The rate of infiltration of the ^{14}C produced by atmospheric nuclear weapons testing into the soil carbon reservoir can be a useful tool for deciphering carbon turnover in soils on time scales of decades to hundreds of years. O'Brien & Stout (1978), O'Brien (1984, 1986) and Harkness, Harrison & Bacon (1986) have shown that the increase in ^{14}C content in bulk soil organic matter can only be explained if soil organic matter is a mixture of components that accumulate and decay at different rates.

Fractionation of soil organic matter by chemical or physical means separates soil organic matter into components with different ages (eg, Campbell *et al,* 1967; Scharpenseel, Ronzani & Pietig, 1968; Martel & Paul,

1974; Goh *et al,* 1976). Goh *et al* (1984) and Martel and Paul (1974) attempted to trace the amount of infiltration of bomb ^{14}C into chemically fractionated soil organic matter, but were hampered by the prohibitively large amounts of soil needed to supply sufficient carbon for liquid scintillation counting, and by the lack of pre-bomb soils for comparison with modern equivalents.

Our approach to quantifying the soil carbon cycle has been to combine the information available from measurements of the natural and bomb ^{14}C in soil organic matter using Accelerator Mass Spectrometry. AMS reduces the sample size necessary for ^{14}C measurements by a factor of 1000 over conventional counting methods, thus allowing analysis of fractionated soil organic matter from small quantites (25 to 100g) of stored pre-bomb soil. We compare the ^{14}C content of fractionated organic matter in soils collected from the same location before and after the period of atmospheric nuclear weapons testing. Information on several time scales is derived from this procedure: the ^{14}C determinations of the pre-bomb fractions give the mean residence time for carbon in each fraction on time scales of hundreds to tens of thousands of years; the infiltration of bomb ^{14}C into the fractions provides information on the cycling of carbon on time scales of years to decades.

This paper reports the results of monitoring the increase in bomb ^{14}C in a genetic soil sequence (where a soil is being developed), sampled several times from 1937 to 1986.

EXPERIMENTAL METHODS

Samples

The samples are from the San Dimas Experimental Forest (SDEF), located in the San Gabriel Mountains east of Los Angeles, California. This area of chaparral vegetation was set aside for research in the 1930s by the US Department of Forestry. The lysimeters were dug as pits 5.3 m^2 by 2.1m deep, then refilled with homogenized local soil. After a three-year interval during which the soil was allowed to compact and settle, each lysimeter was planted with a specific type of shrub vegetation typical of the native chaparral. With the exception of a fire that swept the area in 1960, the lysimeters have remained undisturbed since the 1940s.

Stored samples of the original homogeneous fill soil, litter and mineral soil from one of the lysimeters for the years 1958, 1960, 1961 and 1975 were obtained from P Zinke, University of California, Berkeley (Zinke & Stangenberger, 1975; Zinke, 1977). Additional samples were collected from the same lysimeter in March 1986. The lysimeter sampled was planted with chamise *(Adenostoma fasticulites),* an evergreen schlerophyllous shrub extremely common in the California chaparral. The soil is a reddish sandy clay loam developed on the diorite which forms the San Gabriel Mountains. The climate is Mediterranean.

Sample Treatment

All soil samples were oven dried (60°C) to constant weight, then ground and sieved. After initial addition of 0.1N HCl to remove soil carbonates (none present in the SDEF soils), the samples were fractionated

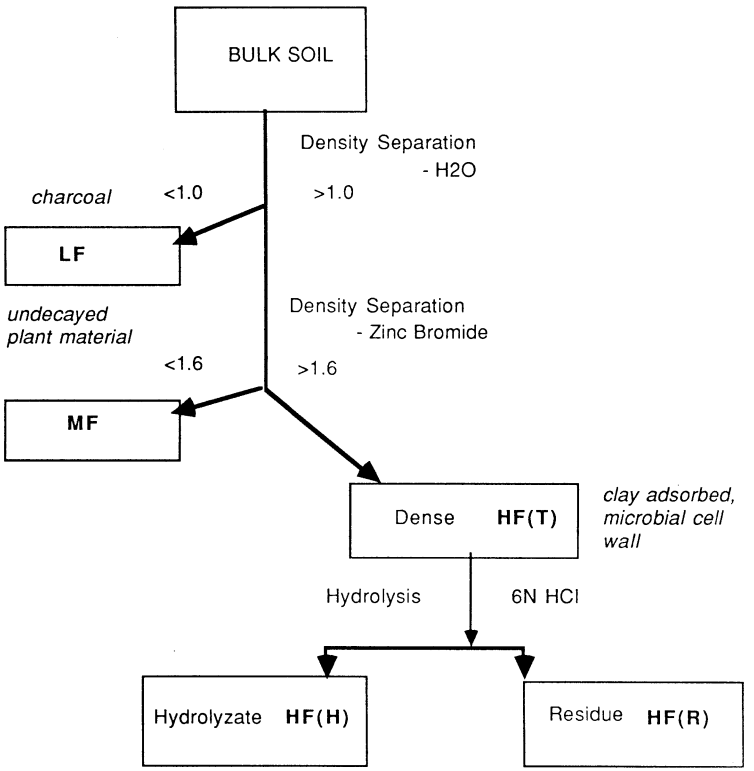

Fig 1. Method used to fractionate the San Dimas soil samples. LF = low density (<1.0g/cc); MF = moderate density (1.0g/cc <MF<1.6g/cc); HF (T) = total heavy fraction (>1.6g/cc); HF(H) = hydrolyzable dense fraction; HF(R) = residue after hydrolysis of the dense fraction

according to the procedure shown in Figure 1, with separation by density into three fractions (<1.0g/cc; LF, between 1.0 and 1.6g/cc; MF, and > 1.6g/cc; HF(T)), followed by acid hydrolysis (in 6N HCl) of the densest fraction. Details of the fractionation procedure are given in Trumbore (1988). Soil and soil fraction samples were combusted by sealing the sample with CuO wire in an evacuated quartz tube, then placing the tube in a 950°C oven for 2 hr. The resulting CO_2 was purified and reduced to graphite targets for AMS using the apparatus and methods described in Vogel *et al* (1984, 1987a, 1987b). $^{14}C/^{12}C$ ratios were measured using the Simon Fraser AMS Facility at the McMaster University Tandem Laboratory. In addition to the C and ^{14}C measurements, 50 to 100g of sample were sealed in aluminum cans and gamma-counted to determine ^{137}Cs activity. The data for all measurements can be found in Trumbore (1988).

TABLE 1

Results of fractionating the 1937 fill soil for the San Dimas lysimeters. Numbers in italics were calculated using mass balance considerations. The %C in each fraction was determined as CO_2 evolved on combustion. $\delta^{13}C$ was assumed to be −25‰.

Fraction	%C in fraction	% of C in soil	^{14}C age (yr)	±
Unfractionated soil	0.23	100	924	105
ρ<1.5g/cc, LF + MF	5.6	18	M	
ρ>1.5/cc, HF(T)	0.19	82	1280	160
Hydrolyzable, HF(H)	*0.23*	*61*	*635*	
Nonhydrolyzable, HF(R)	0.66	21	3530	220

Table 1 shows the distribution of C and ^{14}C in the various fractions in the 1937 fill soil. Separation of material with densities of <1.6g/cc from the 1937 San Dimas fill soil removed ca 20% of the soil carbon, and increased the ^{14}C mean residence time from 925 yr in the unfractionated soil to 1280 yr in the dense residue (HF(T)). Relatively fresh, unaltered vascular plant material partitions into the low density fractions, while the higher density residue contains more altered plant matter, microbial cell wall debris and organic matter adsorbed to clays (Ertel & Hedges, 1985). Charcoal, potentially an 'old' contaminant of the low density material, should be in the< 1.0 g/cc (LF) fraction.

The heavy residue of the density separation procedure (HF(T)) was further fractionated by hydrolysis in 6N HCl at 100°C for 24 hr. The most easily hydrolyzed compounds (amino acids, proteins, carbohydrates, simple sugars) should also be those most readily attacked by micro-organisms (Riffaldi & Schnitzer, 1973; Jawson & Elliott, 1986). The results in Table 1 demonstrate that 60% of the original soil carbon (75% of the carbon in the dense fraction) was hydrolyzed. The residue after acid hydrolysis (HF(R)) had a ^{14}C age of 3530 yr; the age of the hydrolyzed dense material (HF(H)) was calculated by mass balance to be 660 yr.

RESULTS AND DISCUSSION

Evolution of C and ^{14}C Profiles

Figure 2 (A-C) shows the evolution of the carbon inventory with time and depth for the bulk soil (A), the total dense (HF(T); B) and dense-nonhydrolyzable (HF(R); C) fractions. The inventory (in gC/m^2 per cm depth in soil) is given for the 1937 homogeneous soil, and the 1958 and 1986 profiles. The points are located at the midpoint of the sampling depth in the soil; the "error bar" shows the depth range homogenized by the sampling process.

The lysimeter soil accumulated carbon throughout the period 1937 to 1986. Most of the accumulation took place in the upper 18 cm of the soil. Increases were observed in all fractions, but the majority of carbon for any

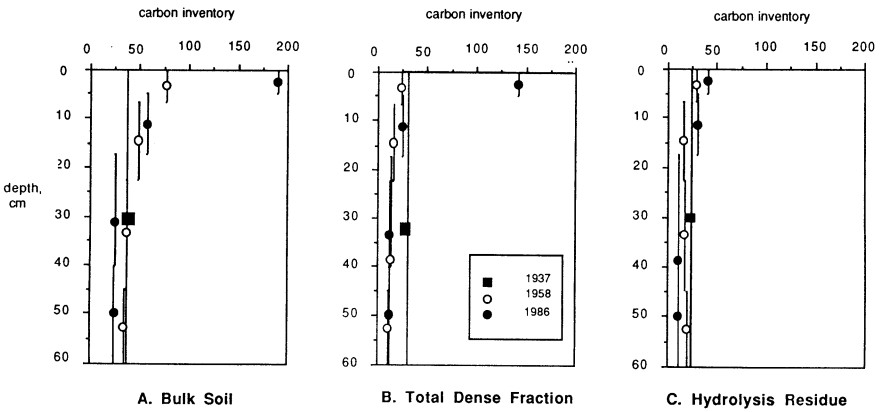

Fig 2. Carbon inventory (in gC/m^2 per cm depth in the soil) for the 1937 fill soil and the 1958 and 1986 profiles. The points show the mid-depth and the 'error bars', the extent of the sampling interval. Litter, which contains up to 6 times more C than the upper mineral soil, is not shown. A, B and C show the results for the unfractionated mineral soil, the total dense fraction, and the residue after acid hydrolysis, respectively.

given depth interval in the mineral soil is contained in the dense fraction (HF(T)). The relative abundances of the fractions change with depth in the soil. The proportion of low density material (LF + MF) decreases with depth, making up 20% or more of the total carbon in the litter and uppermost soil layers, but <10% of the carbon in the deeper portions of the soil. The ratio of hydrolyzable (HF(H)) to nonhydrolyzable (HF(R)) carbon decreases from ca 2 in the upper soil layers to 1 in the deeper soil.

Changes in the ^{14}C content of the soil organic matter also involve increases in all fractions at all depths. Figure 3A,B shows the changes in $\Delta^{14}C$ with depth and time for the total dense fraction (HF(T); A) and residue left after acid hydrolysis (HF(R); B) for litter and mineral soil. Carbon labeled with ^{14}C produced by atmospheric nuclear weapons testing has penetrated to the 30 – 60cm depth interval in the 1986 profile in both fractions.

The distribution of ^{137}Cs in the San Dimas soil samples is shown in Figure 3C. ^{137}Cs, with a half-life of 30 yr, was produced during atmospheric nuclear weapons testing and should not be present in pre-bomb soils. As much of the ^{137}Cs in soils is associated with organic particulate material (Monaghan, 1984), its distribution can tell in particular of the vertical transport of particulate carbon. The inventory of ^{137}Cs in the soil in 1986 shows no significant difference from the 1969 inventory, indicating cesium has not been lost from the soil profile. Since ^{137}Cs has not penetrated as deeply into the soil profile as bomb ^{14}C, mechanical mixing of carbon (eg, by soil organisms) from the litter layer into the underlying mineral soil is not an important process for the lysimeter soil. The source of the bomb ^{14}C at depth must be either from root turnover or transport of dissolved material from the overlying litter layer.

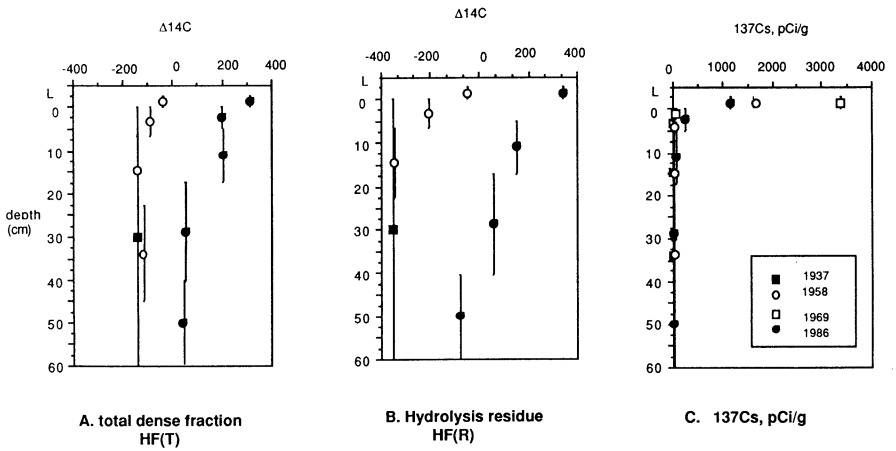

Fig 3. $\Delta^{14}C$ and ^{137}Cs in the San Dimas lysimeter soil for 1937, 1958 and 1956. Same display conventions as Figure 2. The litter layer (L) is shown. A and B show $\Delta^{14}C$ for the total dense and hydrolyzable fractions; C shows ^{137}Cs, decay corrected to 1986.

Modeling

The residence times of the rapidly cycling low density fractions (LM + MF) can be deduced from the observed infiltration of bomb ^{14}C between 1960 to 1986. Because the soil, accumulating carbon, is not at steady state, a time-dependent box model is needed to track the changes in carbon inventory and ^{14}C within the litter and mineral soil. A simple, one-box model which keeps track of the carbon inventory and ^{14}C content for each fraction was used to determine the fluxes of carbon and ^{14}C into and out of the soil for the 1960 to 1986 period. The prescribed inputs for each soil fraction are 1) the original inventory of C and ^{14}C in 1960 (deeper mineral soil values used 1958 data), 2) the $\Delta^{14}C$ of fresh plant material added to the soil fraction each year, assumed to equal the average atmospheric $\Delta^{14}C$ for a particular year (from Tans, 1981), and 3) the trend in amount of plant matter added to the soil in the years following a fire event (assumed, based on observation of litter fall and accumulation rates after a fire event by Kittredge (1955), to increase linearly from 1960 to 1974, then be constant). The adjustable parameters, which were varied to produce the best fit to the observed increases in ^{14}C and C inventory, are the amount of carbon input each year to each fraction and the fraction of carbon lost each year (the reciprocal of the mean residence time). The soil was split into three layers, the litter, upper mineral soil and the deeper mineral soil, each modeled independently. Since any carbon added to a fraction with a residence time > ca 200 yr will accumulate (decay will be negligible), these models cannot distinguish between fractions with residence times >200 yr.

Figure 4 shows the model results (lines) for the LF + MF (low density), HF(T) (total dense) and HF(R) (hydrolysis residue) fractions for the top 7cm of mineral soil as an example. Figure 4A shows the ^{14}C results, 4B, the

carbon inventory data. The model parameters used to generate the model fits shown are given in Table 2.

In general, the increases in carbon inventory and ^{14}C observed in the 1960 to 1986 period are reproduced quite well with the simple one-box model. (An additional test, which used the same model parameters applied to the 1937 initial conditions also did a reasonable job of reproducing the 1958 observations; see Trumbore (1988) for more details of the modeling).

The model-derived mean residence time for low density material in the litter layer is 20 yr; in the upper mineral soil it is 5 yr. The litter layer in the chaparral soils appears to act as a relatively passive accumulator of carbon; periodic fires are necessary to recycle nutrients into usable form for plants (Zinke, 1977). An additional parameter was found necessary in modeling the low density fraction in both litter and mineral soil. This reflected the necessity of having some small (<5%) noncycling component of the low density material which dilutes the ^{14}C addition by new material. Physically, this could represent charcoal.

Assuming that the 1937 fill soil represented a steady-state condition for carbon in chaparral soils, the ^{14}C ages of the pre-bomb soil organic matter should represent the mean residence times for carbon in each fraction: for the low density (LF + MF), dense-hydrolyzable (HF(H)), and dense-nonhydrolyzable (HF(R)) fractions, these ages were ca 0, 650 and 3500 yr, respectively. Table 2 summarizes the model-derived annual input rates of new carbon to each fraction in each depth interval. Also shown are the mean residence times used in the modeling process and those obtained from the 1937 fractionated fill soil. When the soil reaches steady state, the annual rate of input of carbon to the soil will equal the loss of carbon (the decay rate × the amount of carbon present). The final two columns in Table 2 give the extrapolated steady-state inventory of carbon in each depth interval assuming the mean residence times used in modeling and the measured mean residence times. The total amount of carbon in the soil (to 1m depth, assuming that 0–15cm has the characteristics of 'upper' mineral soil and 15–85cm is 'deep' mineral soil), is between 18.7 and 132kgC/m^2. Post *et al* (1982) estimate the amount of carbon in Mediterranean scrub ecosystems (to 1m depth, excluding detritus) as only 7.2kgC/m^2. The carbon inventory in the 1937 fill soil (also to 1m depth) was 3.6kgC/m^2. Thus, the model parameters used, when extrapolated to steady state, overestimate the soil carbon inventory by at best a factor of two. Several explanations can be offered.

1) The fractionation scheme is not entirely successful at separating soil organic matter into components characterized by different mean residence times. If this is true, the dense fractions (HF(H), HF(R)) contain both rapidly and slowly cycling fractions, the mean residence time representing an average of these two components.

2) Some of the carbon inventory estimates for the deep, dense soil organic matter fractions are in error. Overestimation by as little as a factor of two of the carbon inventory in the 1937 dense fractions could reduce the estimated steady-state inventory of carbon in the deep soil by nearly a factor of five.

3) All of the bomb radiocarbon in the deeper portion of the soil could be due to the accumulation of hydrophobic compounds in the lower layers

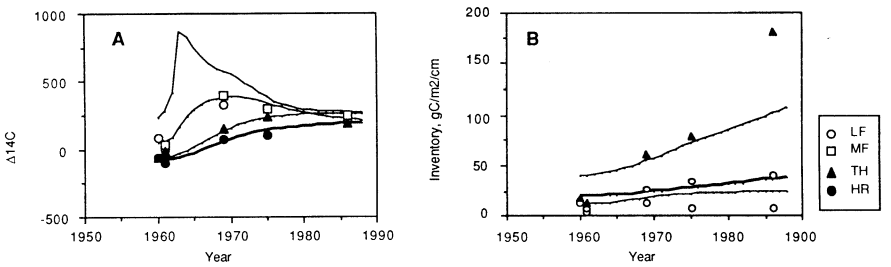

Fig 4. Model results (lines) for bomb ^{14}C incorporation into the LF + MF, HF(T), and HF(R) fractions for the upper 7cm of mineral soil. Measurements of ^{14}C and carbon inventory are also shown (points). A shows the changes in $\Delta^{14}C$ for the period 1960 to 1986; B shows the carbon inventory change for the same period.

TABLE 2

Best fit model parameters for the different depth intervals and fractions. Also calculated are the steady-state inputs of carbon to each fraction, the calculated inventory of carbon in each fraction at steady state and the inventory of carbon in the total soil to 1m depth at steady state.

Depth	Fraction	Input gC/m²cm/yr	Residence time (yr) (model)	(meas)	Inventory at steady state gC/m²/cm (model MRT)	(meas MRT)
Litter	LF+MF	8.9	10	10		
	HF(H) (hydrolyzable)	5.5	>200	?		
	HF(R) (nonhydrolyzable)	5.5	>200	?		
Upper	LF+MF	3.3	5	5	16	16
soil	HF(H) (hydrolyzable)	2.2	>200	660	>440	1452
	HF(R) (nonhydrolyzable)	0.9	>200	3300	>180	2904
Deep	LF+MF	?	5	5		
soil	HF(H) (hydrolyzable)	0.33	>200	660	>66	218
	HF(R) (nonhydrolyzable)	0.17	>200	3300	>34	561
Total carbon inventory					>17,800	132,000

(to 1m depth, assuming 15cm Upper and 85cm Deep), in gC/m²

during the 1960 fire event. During high-temperature fires in chaparral environments, these hydrophobic compounds are formed in the upper soil layers and deposited deeper in the soil where they can act as a barrier to soil water loss. In this case, accumulation of carbon in the deep soil occurs in events; net inputs, and, therefore, the increase in inventory through time and the steady-state inventory, are smaller than estimated by the models.

4) The assumption that the 1937 fill soil represents steady state is in error. This is obviously true, since the soil began to accumulate carbon when vegetation was planted on it. Thus, the application of mean residence times for what is probably quite deep mineral soil (where annual inputs of new carbon are small) to the upper layers of mineral soil might easily overestimate the steady-state carbon inventory.

One assumption in the model is that the only loss of carbon is through 'decay' described using a first-order rate constant. Other processes by which carbon may be lost to the soil profile include loss by erosion or as dissolved organic carbon that percolates out of the soil. Chaparral soils, which develop in mountainous regions, are perhaps eroded so fast that they are not in place the several thousand years necessary to achieve steady state. Until a means of estimating erosion rates, and estimates of what portion of the mineral soil erodes are available, this hypothesis is difficult to test. An erosion rate of 200mg soil/cm^2 yr (if distributed evenly through a 1m soil column) results in a loss of carbon each year equivalent to loss by decay with a mean residence time of 3000 yr. The Tanbark Flats area on which the lysimeters were installed was specially graded to ensure no loss of soil by erosion, while erosion rates in the San Dimas region may actually be >200mg/cm^2 yr. Losses of organic material by dissolved transport, although also difficult to estimate, can be of the same order of magnitude. Thus, the mean residence times observed in the 1937 fill soil may reflect erosion rates, or dissolved transport time scales. In this case, the effect of this soil on atmospheric CO_2 concentrations for a given perturbation is overestimated, since the material lost is not decayed.

CONCLUSIONS

Chaparral soils appear to be efficient accumulators of carbon, both as plant detritus and its alteration products. To a first order, soil organic matter can be modeled as a two-component system: a rapidly (5 – 20 yr) cycling low density component consisting of relatively unaltered vascular plant material, and a slowly cycling, dense fraction which constitutes the majority of soil carbon and has a mean residence time >200 yr. This dense component can be further broken down into fractions by acid hydrolysis. The hydrolyzable portion has a shorter mean residence time than the nonhydrolyzable residue in the pre-bomb soil; it also takes up more bomb ^{14}C in the 1960 to 1986 period.

Questions remain as to the mechanisms of introduction of new carbon into deep soil layers, the vertical transport of carbon within soil profiles, and the relative importance of loss of carbon by soil weathering or dissolution compared to the decay process. To help answer these questions, information from other tracers, such as ^{137}Cs, are necessary to studies of this kind.

The organic matter in soil in other climate regimes, particularly more humid ones, may behave quite differently from the chaparral soils discussed here. More measurements of pre- and post-bomb fractionated soil organic matter samples from widely different climatic regimes are needed in order to better understand the nature of the carbon cycle in soils.

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