

Radiocarbon Dating for Determination of Soil Organic Matter Pool Sizes and Dynamics

E. A. Paul,* R. F. Follett, S. W. Leavitt, A. Halvorson, G. A. Peterson, and D. J. Lyon

ABSTRACT

The size and turnover rate of the resistant soil organic matter (SOM) fractions were measured by ^{14}C dating and $^{13}\text{C}/^{12}\text{C}$ measurements. This involved soils archived in 1948, and recent samples, from a series of long-term sites in the North American Great Plains. A reevaluation of C dates obtained in the 1960s expanded the study scope. The ^{14}C ages of surface soils were modern in some native sites and near modern in the low, moist areas of the landscape. They were much older at the catena summits. The ^{14}C ages were not related to latitude although this strongly influenced the total SOM content. Cultivation resulted in lower C contents and increased the ^{14}C age by an average of 900 yr. The 10- to 20-cm depths from both cultivated and native sites were 1200 yr older than the 0- to 10-cm depth. The 90- to 120-cm depth of a cultivated site at 7015 yr before present (BP) was 6000 yr older than the surface. The nonhydrolyzable C of this depth dated 9035 yr BP. The residue of 6 M HCl hydrolysis comprised 23 to 70% of the total soil C and was, on the average, 1500 yr older. The percentage of nonhydrolyzable C and its ^{14}C age analytically identify the amount and turnover rate of the old resistant soil C.

SOIL ORGANIC MATTER is the primary storehouse of soil C and the source of available N and to some extent P, S, and minor elements of agricultural, forest, and grassland soils (Townsend et al., 1996). It reflects the vegetation, soil biota, climate, parent material, time to steady state, and natural and human disturbances. Thus SOM acts as a signature, as well as a controller, of ecosystem functioning (Paul, 1984). It is one of the primary factors that affect soil quality, water infiltration, erosion resistance, tillage, and adsorption and degradation of pollutants (Doran et al., 1994). Microbial decomposition of SOM at times when there is no concurrent uptake of nutrients by plants can lead to extensive losses of nutrients, and pollution of both groundwater and the atmosphere. The dynamics of SOM must be known for its proper management.

The recent interest in SOM as a source-sink in global C budgets results from the assessment that 25% of the radiative climate forcing in global change is attributable to agriculture (Tans et al., 1990; Schimel, 1995). Further, SOM is a major component of the terrestrial sink necessary to balance present global CO_2 budgets (Lugo, 1992; Lal et al., 1995). The concept that SOM contains fractions with varying decomposition rates predates the availability of tracers. In the 1930s, German workers talked of *Nahr* or nutritive humus. The first tracer utilized involved ^{13}C , followed by ^{15}N and ^{14}C (Coleman and Fry, 1991; Knowles and Blackburn, 1993). Jansson

(1958), working with ^{15}N added to soils, conceptualized an active fraction and estimated it to have a turnover time of 15 to 20 yr.

Bombardment of atmospheric N_2 with cosmic rays produces ^{14}C with a half-life of 5568 yr. This and the ^{14}C artificially produced by thermonuclear (H) bomb testing, started in the 1950s, is oxidized to $^{14}\text{CO}_2$ and assimilated by plants. Plant residues are transformed into SOM with little isotopic discrimination. As ^{14}C -dating techniques that measure these very low levels of ^{14}C became available, they were used to date buried soil horizons in archaeological work and occasional surface soils. The ^{14}C -dating nomenclature assigns samples with ^{14}C contents equivalent to 95% of a 1950 oxalic acid standard as modern. Counts are expressed as percentage modern C (pmC). Higher counts can come from bomb ^{14}C or by contamination from other ^{14}C sources (Harkness et al., 1991). Ages are expressed as BP.

The first ^{14}C age determined for a North American grassland soil was that of Simonson (1959). He reported a soil from North Dakota to have an age of 1300 yr. Other early ^{14}C ages included Swedish and American podzols at 50 to 370 yr BP (Broecker and Olson, 1959; Tamm and Ostlund, 1960). Broecker and Olson (1960) suggested that ^{14}C dating, especially bomb ^{14}C , should be useful for the investigation of soil characteristics. Paul et al. (1964) demonstrated that ^{14}C dating together with organic-C fractionation could be used to determine the dynamics of SOM. Their work, with Canadian grassland soils, showed that the mean residence time (MRT) of soils was dependent on soil type, landscape position, soil depth, length of cultivation, vegetation, and soil particulates (Campbell et al., 1967a; Martel and Paul, 1974a; Anderson and Paul, 1984).

The SOM of a 1889 Rothamsted, England, sample, dated in 1970, was 1000 yr BP in age (Jenkinson and Rayner, 1977). This was much greater than would be expected from calculations based on rates of SOM accumulation and plant residue inputs. Jenkinson and Rayner (1977) combined ^{14}C dating with studies involving added ^{14}C residues and knowledge of plant residue inputs in long-term experiments to develop a realistic model of C turnover. Subsequent models (van Veen and Paul, 1981; Parton et al., 1987; Paustian et al., 1992) utilized the available ^{14}C dates to characterize SOM dynamics. Three SOM pools, decaying according to first-order reaction kinetics, have been shown to best describe C dynamics (Parton et al., 1993; Paul et al., 1995). Interpretations have also been made using two pools (Hsieh, 1993; Harrison et al., 1993).

Campbell et al. (1967b) used ^{14}C dating to measure the ^{14}C age of the fulvic, humic, and humin fractions of SOM. These were interpreted relative to turnover times

E.A. Paul, Crop and Soil Sciences, Michigan State Univ., East Lansing, MI; R.F. Follett, USDA-ARS, Ft. Collins, CO; S.W. Leavitt, Lab. of Tree Ring Research, Univ. of Arizona, Tucson, AZ; A. Halvorson, USDA-ARS, Mandan, ND; G.A. Peterson, Dep. of Soil and Crop Sciences, Colorado State Univ., Ft. Collins, CO; and D.J. Lyon, Panhandle Research and Extension Center, Univ. of Nebraska, Scottsbluff, NE. Received 15 Dec. 1995. *Corresponding author (eapaul@msu.edu).

Abbreviations: SOM, soil organic matter; BP, before present; MRT, mean residence time; TAMS, tandem accelerator mass spectrometer; FACE, free atmosphere carbon enrichment experiment; IRMS, isotope ratio mass spectrometer.

by expressing the ^{14}C ages as MRT. Humic acids were the oldest materials, especially when humics associated with the metal ions in soil were isolated. Fulvics were much younger and humins were intermediate in age. Martel and Paul (1974a) dated nine fractions of the SOM of a grassland (Sceptre Association, Aridic Boroll). The light fraction obtained by flotation in a heavy liquid contained much of the bomb ^{14}C ; its removal changed the soil ^{14}C age from 350 to 500 yr BP. Hydrolysis with 6 M HCl produced a nonhydrolyzable residue that dated 1760 yr BP. Further extractions of this residue with NaOH resulted in only small increases in the ^{14}C age of the remaining residue to 1965 yr.

Cultivation increased the ^{14}C age of a northern grassland soil (Oxbow Association, Udic Boroll) from 250 to 295 yr BP after 15 yr of cultivation. The age increased to 710 yr BP after 60 yr of cultivation. Hydrolysis with 6 M HCl was used to fractionate the organic soil components. One would expect that, as the soil lost its C during cultivation, the percentage of nonhydrolyzable C would increase. However, the nonhydrolyzable C from both the native and the cultivated soil represented approximately one-half of the soil C (Martel and Paul, 1974b). A Udic Haploboroll soil (Indian Head), had a ^{14}C age of 1910 yr in 1963 and 1515 yr in 1978. This showed the effect of bomb ^{14}C incorporation (Anderson and Paul, 1984). The residue of hydrolysis of the 1963 sample dated 2820 yr and that of the 1978 sample dated 2095 yr. Humic acids were the next oldest, dating 2455 yr in the 1963 and 1720 yr in the 1978 samples. The SOM of the coarse clay fraction of another Mollisol at 1255 yr was shown to be the oldest of the particulate fractions isolated from that soil. This was not as old as the humic acids (1425 yr). The Canadian soils were developed during the 12 000 to 14 000 yr since their glaciation. The ^{14}C ages of the pedogenically much older soils found in unglaciated regions of North America are not known.

Trumbore (1993) and Trumbore et al. (1996) confirmed the observations of Anderson and Paul (1984) that acid hydrolysis was effective in differentiating old C of temperate soils. They did not find this to be true for tropical sites. Acid hydrolysis does not solubilize the lignin, phenols, and some cellulose of plant residues. Hydrolysis left 42% of the C of wheat (*Triticum aestivum* L.) straw and 34% of the C of maize (*Zea mays* L.) residues undissolved (Follett et al., 1997). It also has differential effects on soil fulvics and humics (Schnitzer and Khan, 1972). Although most of the plant residue can be removed by density flotation and microscopic picking prior to hydrolysis, experiments with ^{14}C -depleted CO_2 in the Arizona Free-Air CO_2 Enrichment (FACE) study showed incorporation of plant-residue C into the acid-resistant fraction after 2 yr exposure to ^{14}C -depleted CO_2 (Leavitt et al., 1994).

The pool sizes and fluxes of SOM are best interpreted relative to ecosystem dynamics by using soils from well-characterized long-term sites with a known history of management and known residue inputs (Paul et al., 1995). Such sites are available in a range of grassland soils from the Canadian Prairies south to Texas (Fig. 1). Haas et al. (1957) characterized the C and N levels,

plant yields, and climate of many of these sites. They archived samples of both native and cultivated soil. Paul et al. (1997) have provided more recent documentation for many of these sites. The availability of tandem accelerator mass spectrometers (TAMS) that analyze milligram-sized samples makes it possible to utilize archived samples by ^{14}C dating. Present samples from those sites, under the same management, make the archived samples especially valuable. This is the first of a series of related studies that use acid hydrolysis, ^{14}C dating, $^{13}\text{C}/^{12}\text{C}$ ratios, and extended incubation of soils from long-term plots to obtain analytically derived estimates of the pools and fluxes of soil organic C. Here we report results on ^{14}C ages for a range of sites in the North American grasslands that include samples to depth. Acid hydrolysis was used to determine both the size and ^{14}C age of the old resistant fraction.

METHODS

Archived soil samples, collected in 1947–1949 from Akron, CO; Mandan, ND; Hayes, KS; and Big Springs and Dallas, TX (Haas et al., 1957) were obtained from the Northern Great Plains Research Center in Mandan, ND. Field collection of soils from Akron, CO (Halvorson et al., 1997) and Sidney, NE (Peterson et al., 1994) research sites (native grassland vs. long-term winter wheat–fallow rotation) were accomplished by use of a hydraulic corer using 3.5- and 3.8-cm-diam. tubes, respectively. Each sample was a composite of three soil cores per replicate for each treatment. Soil cores were collected along the length of each plot. In addition, soil samples from a replicated, uncultivated sod-plot treatment, with a grass-species shift to predominantly cool-season species, were obtained at Sidney, NE. The Lethbridge site represented samples collected by Campbell et al. (1967a,b).

Composited soil samples from the Sterling, CO, site (two cores from each of two replications of the cultivated wheat–fallow system) were collected from summit, sideslope, and toeslope positions using a hand probe. The 300-m-long catenary sequence at the Sterling site descends 5.5 m from the summit position to the toeslope. The steepest part of the sequence occurs in the sideslope position. Water runs off both summit and sideslope positions when rainfall intensities exceed soil infiltration rates. The toeslope catches most of this water and is more productive than either the summit or sideslope positions. Soil samples from the Maricopa, AZ, enriched CO_2 site were obtained with two soil cores from each of two replicates of the ambient treatment of the cotton (*Gossypium hirsutum* L.) field associated with the FACE experiment (Leavitt et al., 1994).

Soil locations are shown in Fig. 1; selected soil characteristics are given in Table 1. Plant-free soil and the nonhydrolyzable C after hydrolysis with hot, 6 M HCl were analyzed for C content, $^{13}\text{C}/^{12}\text{C}$, and ^{14}C age. Soil samples were passed through a 2-mm sieve to remove large plant fragments and pebbles. A 5- to 20-g subsample of the soil was acidified with 1 M HCl to remove carbonates. After decanting the HCl, the subsamples were immersed in a 1.2 g cm^{-3} NaCl solution and stirred. The heavier solids were allowed to settle and floating plant fragments were skimmed off. This process was repeated until no more material floated to the surface. Subsamples were rinsed free of salt, dried, and pulverized with a mortar and pestle. Next, 1 to 5 g of the processed subsamples were picked free of recognizable plant and root fragments under 20 \times magnification. Organic-C content and ^{13}C were determined after

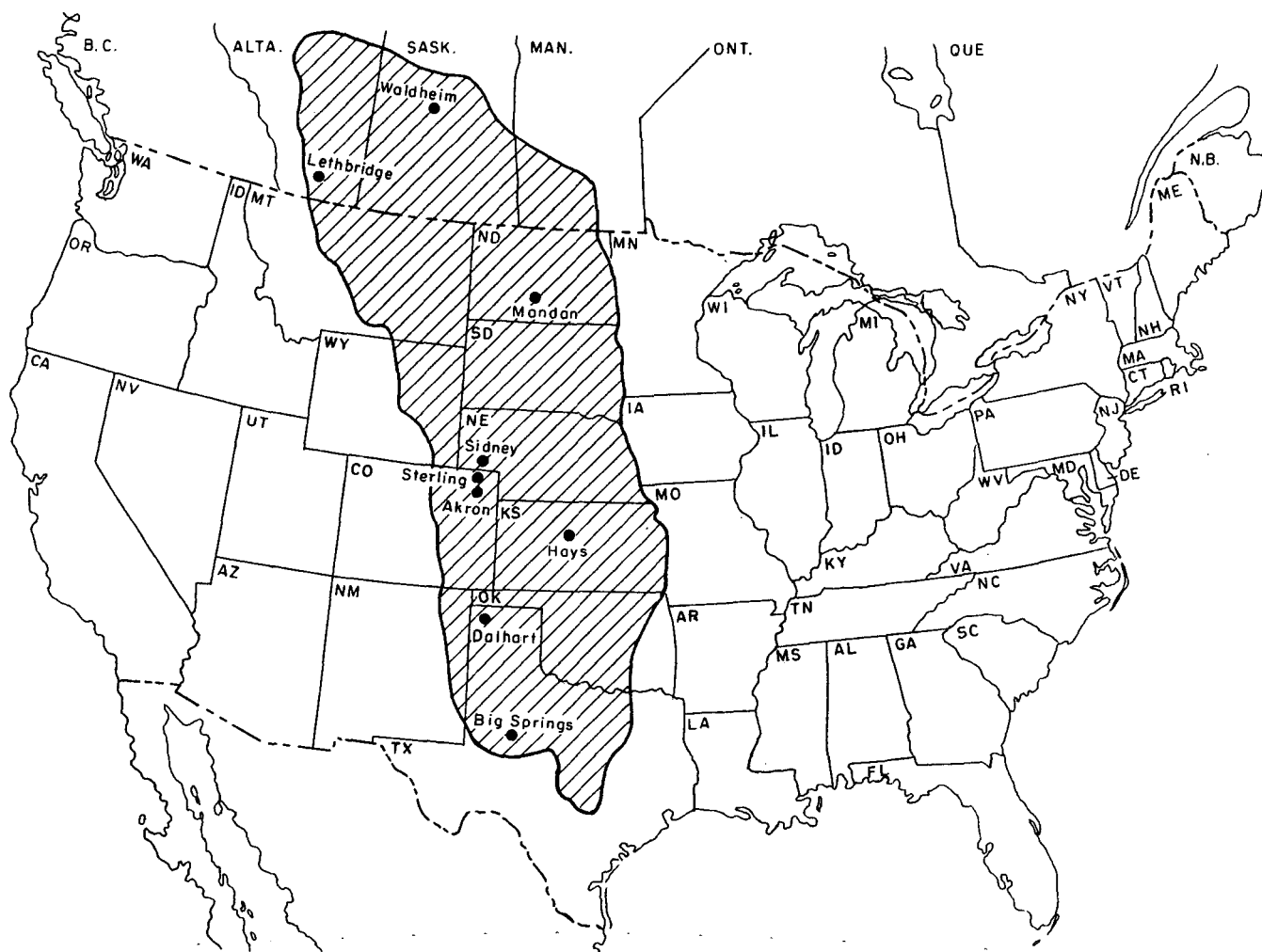


Fig. 1. The location of sampling sites in the North American grasslands.

combustion of 0.10 to 0.25 g of processed-soil subsamples in quartz tubes at 900°C in the presence of copper oxide, silver foil, and copper turnings (Coleman and Fry, 1991). Carbon yield was determined manometrically from the CO₂ combustion product, and CO₂ was analyzed mass spectrometrically to determine $\delta^{13}\text{C}$. Isotope ratio mass spectrometer (IRMS) precision is about 0.05‰ for samples of the same gas. Seven separate combustions and analyses of one soil produced a standard deviation of 0.25‰ in the $\delta^{13}\text{C}$ content and a precision in C content of 0.03%.

Acid hydrolysis was performed using 1 to 3 g of soil refluxed in hot, 6 M HCl for 18 h. The soluble materials were separated by filtration followed by repeated evaporation to remove the HCl. The residue of hydrolysis was rinsed with deionized water and dried. Soil and nonhydrolyzable fractions were combusted as described above. Soil C age, based on ¹⁴C activity, was determined on a TAMS at the University of Arizona Accelerator Facility after reducing the CO₂ to CO on hot zinc and subsequently to graphite on hot iron. The accelerator measurements of ¹⁴C activity are expressed as percentage modern C (pmC), employing a $\delta^{13}\text{C}$ correction for isotope fractionation (Goh, 1991). The ¹⁴C content was converted into age (years BP) with A.D. 1950 being considered 100 pmC and 0 yr BP.

Organic-C content measurements were made using flotation procedures not normally used when determining total organic C in soil. Because of the need for comparison of organic-C content measurements reported by Haas et al.

(1957) to our measurements for both archived and recently collected soil samples, we also utilized the following organic-C determination procedure for all samples. Total organic C and $\delta^{13}\text{C}$ were determined on air-dried subsamples of the soils by an automated CHN analyzer interfaced with an IRMS. Soil carbonates were removed by addition of 100 mL of 0.03 M H₃PO₄ to 5 g soil and shaking for 1 h. The procedure was repeated until the pH of the soil solution remained within 0.2 pH unit of that of the original acid solution. Regression of the manometer and CHN analyzer procedures for determination of total organic C resulted in a correlation (R^2) of 0.98. The slope of the linear regression line and its intercept were not significantly different ($P < 0.01$) from 1.0 and 0.0, respectively. The reported organic-C contents are the averages for the two procedures when data from both were available.

RESULTS

The loess soil site at Akron, CO, (Aridic Paleustoll) has crop and soil management treatments initiated in 1908. Comparison of the total C of the 1947 samples, as determined by Haas et al. (1957) and in two of our laboratories, shows good agreement (Table 2). Haas et al. (1957) reported that cultivation for 50 yr produced a 46% decrease in soil C in the 0- to 15-cm depth and an 18% decrease in the 15- to 30-cm layer. The 1992

native site was very similar in soil C to the 1947 native sample. The adjacent cultivated site after 90 yr of cultivation had 35% less total organic C in the 0- to 10-cm depth and 21% less in both the 10- to 20- and 20- to 30-cm depths.

There was a general decrease in the percentage of nonhydrolyzable C with depth in the native Akron site. The deeper sampling of the cultivated site showed a decrease followed by an increase in the deepest layer. Sand content did not change with depth, but silt increased as the clay content decreased (Table 2). The ¹⁴C age of the 1947 native sample was very similar to the 1992 sample, especially when one considers the greater depth of the 1947 sample (Table 3). There is a problem in the interpretation of the 1947 cultivated, stored sample in that the deeper horizon is younger in its ¹⁴C age than the surface sample.

Analyses of ¹⁴C by TAMS are restricted by high costs and limited accelerator accessibility. The 1947 and 1992 native samples were analyzed in duplicate for surface horizons and singly for subsurface soils. Errors shown represent field variability where analyses were obtained on more than a single composite sample. The ¹⁴C age (Table 3) of the total organic C of the native sites was modern in 1947 and 193 yr in 1992. Acid hydrolysis confirmed that the 1947 sample, whose total C was 107 pmC, was contaminated. The pmC of 153 calculated for the acid hydrolyzable C would not be found in bomb-affected plant residues until 1970. The source of this contamination is not known. We had problems with contamination of archived samples but not those collected in 1992.

The large increase in ¹⁴C age with depth, from 193 yr at 0 to 10 cm to 4068 yr BP at 30 to 45 cm, shows major changes in the factors controlling the deposition and turnover of the SOM within the soil profile. Similar ages were found in the cultivated site; the total soil C reached an age of 7000 yr BP at the 90- to 120-cm depth (Table 3). Acid hydrolysis of soil from the native site increased the ¹⁴C age of the residue of hydrolysis by 1800 yr at the surface and 3500 yr at the 30- to 45-cm depth. Nonhydrolyzable C ages parallel those of the total C in the native and cultivated sites. It is difficult to explain ages of 7000 yr in total C and 9000 yr for the 50% of the C that is nonhydrolyzable at soil depths that are still in the crop rooting zone. The samples from the cultivated Akron plots showed a wide range in the ¹⁴C age of the nonhydrolyzable C at the 10- to 20-cm depth. Two plots (205 and 307) with slightly different management histories were used to obtain replication on this site. Plot 205 had a ¹⁴C age of 3925 yr BP, Plot 307 dated at 7240 yr.

The inverse relationship between C content (Fig. 2A) and the ¹⁴C age of both the total soil C and the nonhydrolyzable C (Fig. 2B) is reminiscent of data from an isotope dilution experiment where tracer contents and the level of materials in the pool are inversely related. The age progression at lower depths does not reflect changes in the soil texture. These soils become more silty at 90 to 120 cm (Table 2) and very sandy at depths beyond which they were C dated. A decomposition rate constant *k* (yr⁻¹) can be calculated if the ¹⁴C age is taken as the

Table 1. Soil factors and properties by location.

Soil series Texture	Taxonomic classification	Location										
		Lethbridge, Alberta	Mandan, ND	Sidney, NE	Akron, CO	Sterling, CO	Sterling, CO	Sterling, CO	Hays, KS	Dalhart, TX	Big Springs, TX	Mariopca, AZ
		Lethbridge clay	Temvik silt loam	Duroc loam	Weid loam	Weid loam	Satanta loam	Ablinas loam	Munjour sandy loam	Dalhart fine sandy loam	Amarillo fine sandy loam	Trix clay loam
		fine-loamy, mixed Aridic Argiboroll	fine-silty, mixed, Typic Haploboroll	fine-silty, mixed, mesic Pachic Haplustoll	fine, montmoril- lonitic, mesic Aridic Paleustoll	fine, montmoril- lonitic, mesic Aridic Paleustoll	fine- loamy, mixed, mesic Pachic Argustoll	fine- loamy, mixed, mesic Pachic Argustoll	coarse- loamy, mixed (calc.) mesic Typic Ustifluvent	fine- loamy, mixed, thermic Aridic Haplustalf	fine- loamy, mixed, thermic Aridic Paleustoll	fine-loamy, mixed (calc.), hyperthermic Typic Torrifluvent
Soil horizon Depth, cm		AP	Apt† 0-18	A 0-15	Apt† 0-8	Ap 0-8	Ap1 0-8	Ap1 0-8	A† 0-18	Ap† 0-13	A† 0-28	- 0-30
Fraction		-	-	-	-	45.0	42.3	-	-	-	-	35.0
Sand (0.05-2.00 mm), %		48.7	-	-	-	34.2	30.3	-	-	-	-	31.5
Silt (0.002-0.05 mm), %		24.1	-	-	-	25.4	18.4	-	-	-	-	33.5
Clay (<0.002 mm), %		27.2	20.0	20.0	22.0	20.8	18.4	11.0	11.0	14.0	14.0	-
pH (1:1)		-	6.8	7.5	7.1	7.1	7.6	7.9	7.9	7.2	7.2	-
Cation-exchange capacity, cmol kg ⁻¹		-	-	16.0	-	14.2	15.4	-	-	-	-	25.5
Electrical conductivity, S m ⁻¹		-	0.0	0.0	-	0.01	0.02	-	-	-	-	-

† Established soil series description using mean values from soil interpretation records.

Table 2. Characteristics of the Akron, CO, site.

Soil treatment	Depth	Organic C†	Total organic C‡ ± SE	Non-hydrolyzable C ± SE	Soil texture		
					Sand	Silt	Clay
	cm		g kg ⁻¹		%		
Native 1992	0–10		13.6 ± 1.45	55.9 ± 0.3			
	10–20		9.5 ± 0.6	45.3¶			
	20–30		7.9 ± 0.7	44.5 ± 0.9			
	30–45		5.9 ± 0.2	39.4 ± 2.9			
Native 1947	0–15	14.2	13.6 ± 0.56	54.4 ± 1.8			
	15–30	7.9	8.25 ± 0.36	28.0¶			
Cultivated 1947	0–15	7.7	9.3¶	–			
	15–30	6.5	8.1	–			
Cultivated 1992	0–10		8.9 ± 1	59.1 ± 2.5	36	31	33
	10–20		7.5 ± 0.4	54.2 ± 3.8	29	40	31
	20–30		6.2 ± 0.4	48.5 ± 1.8	29	41	30
	30–60		6.1 ± 0.6	49.9 ± 3.9	36	39	25
	60–90		3.5 ± 0.3	45.3 ± 0.4	33	49	18
	90–120		2.2 ± 0.2	51.1 ± 1.5	29	52	19

† Data from Haas et al. (1957).

‡ Data from this study.

§ Interpolated from Halvorson et al. (1995).

¶ Single-sample analyses—instrument replication only.

mean residence time and one assumes first-order kinetics. The value of k in first-order reactions at steady state is $1/\text{MRT}$. Figure 2C shows the decomposition rate constant of the nonhydrolyzable C to be relatively uniform with depth. The total soil C shows a much higher decomposition rate at the surface. This reflects the effect of addition of materials with high decomposition rates.

The Colorado catenary site, cultivated for 50 yr, had organic C contents that ranged from 6.9 g kg⁻¹ in the 0- to 10-cm layer of the summit to 9.8 g kg⁻¹ in the surface layer of the toeslope position (Table 4). This was accompanied by a decrease in age from 1600 to 700 yr BP. The aboveground biomass in this wheat–fallow system, since 1986, has averaged 5070, 5540, and 6350 kg ha⁻¹ yr⁻¹ for summit, sideslope, and toeslope positions, respectively. The straw that remained on the soil surface of the corresponding sites after grain harvest averaged 3400, 3900, and 4300 kg ha⁻¹ (Peterson et al., 1994). One would expect equivalent differences in root inputs. Differences in residue input affect the ¹⁴C age, but the

amount of residue cannot account for all of the age differences on these sites. Data from another catenary site in Saskatchewan (Martel and Paul, 1974a) are shown for comparison. The Saskatchewan site, cultivated for 30 yr at the time of sampling, shows its northern location. It had three times as much C as the Colorado site, but no major C content changes along the catena. However, the ¹⁴C age decreased from 575 yr at the summit to modern in the depression. This site shows the effects of differing water-use efficiencies on the ¹³C content of plants with a common C₃ heritage. The upper slopes in the catena had a δ¹³C of -25.1‰. The change to -27.2‰ at the lower, wetter sites is consistent with known effects of plant water-use efficiency on the δ¹³C content (Farquhar et al., 1982). The ¹³C contents are used in the calculation of the C dates and elsewhere (Follett et al., 1997) where mixtures of C₃–C₄ plants provide a powerful additional tracer.

The Sidney, NE, site (Table 5) represents a silty, mixed, mesic Pachic Haplustoll. Soil from adjacent native pasture has an average C content of 21.0 g kg⁻¹.

Table 3. The ¹⁴C activity and ¹⁴C age of the total soil organic matter and hydrolysis fractions of the Akron, CO site.

Soil	Depth	¹⁴ C activity			¹⁴ C age		
		Total organic C ± SE	Nonhydrolyzable C ± SE	Hydrolyzable C	Total org C ± SE	Nonhydrolyzable C ± SE	Hydrolyzable C†
	cm		pmC		yr BP		
Native 1992	0–10	97.6 ± 1.4	78.0 ± 1.3	123	193 ± 118	1994 ± 134	Bomb
	10–20	82.7 ± 1.3	66.5‡	98	1528 ± 128	3260‡	130
	20–30	74.2 ± 0.2	51.4 ± 1.4	93	2398 ± 22	5355 ± 220	625
	30–45	60.4 ± 4.0	39.0 ± 3.7	72	4068 ± 528	7600 ± 775	2480
Native 1947	0–15	107.5 ± 2.9	69.6 ± 0.8	153	Modern	2915 ± 85	Contam.
	15–30	72.2‡	57.6‡	78	2611‡	4431‡	1990
Cultivated 1947	0–15	82.4‡	–	–	1560‡	–	–
	15–30	87.8‡	–	–	1040‡	–	–
Cultivated 1992	0–10	85.2 ± 2.7	66.7 ± 4.9	114	1296 ± 262	3326 ± 616	Bomb
	10–20	74.5 ± 0.9	61.4‡	106	2368 ± 104	5736 ± 472	Bomb
	20–30	64.4 ± 2.1	53.4 ± 4.8	75	3540 ± 265	5070 ± 730	2355
	30–60	52.8 ± 2.2	41.5 ± 3.8	64	5143 ± 333	7187 ± 658	3540
	60–90	46.5 ± 1.6	37.0 ± 3.4	54	6160 ± 285	8013 ± 742	4905
	90–120	41.8 ± 2.3	32.5 ± 2.5	51	7015 ± 450	9035 ± 440	5420

† Calculated by difference.

‡ Single-sample analyses—instrument replication only.

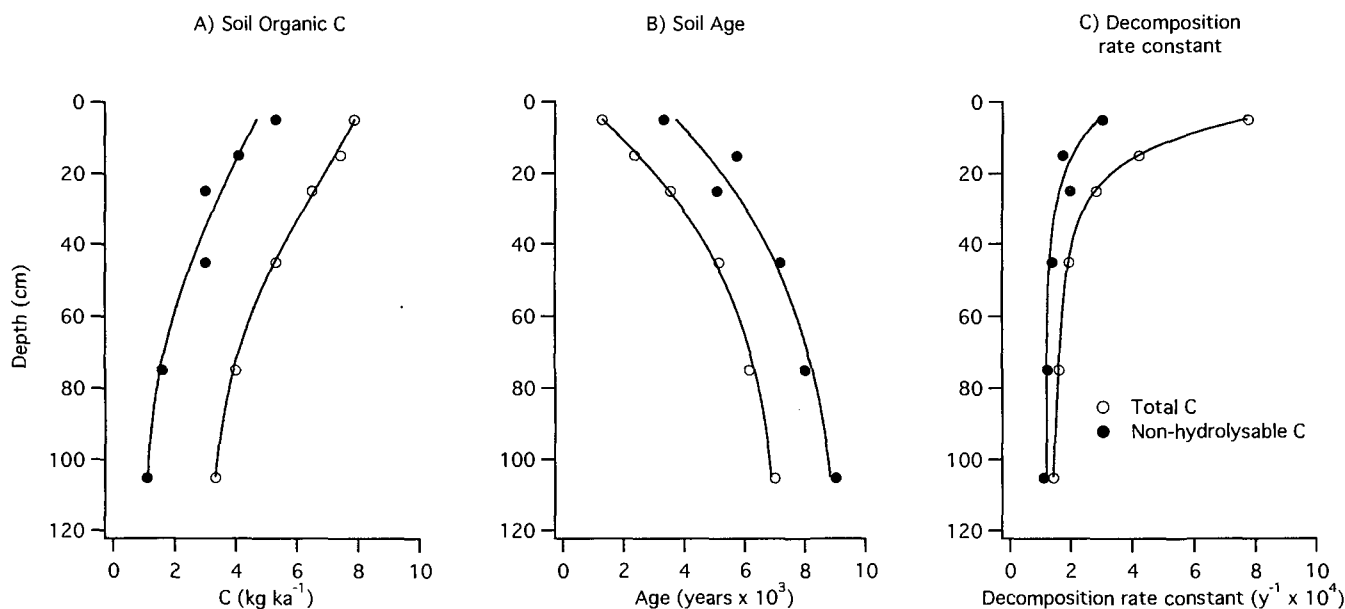


Fig. 2. The relationship of soil organic C content, ^{14}C age and decomposition rate constant with depth in the Akron, CO, cultivated soil.

This is 50% greater than the nearby, native Akron site at 13.6 g kg^{-1} but 20% lower than a native, mixed prairie site in Saskatchewan. The Sidney experimental plots, when established in 1970, were fenced. Three replicated strips of native vegetation remained as part of the rotation. These were not grazed nor did they undergo prairie fires. The vegetation in these strips changed to primarily a C_3 sod during the 20 yr after fencing. The C content in the 0- to 10-cm depth increased from 23.3 g kg^{-1} in 1970 (Fenster and Peterson, 1979) to 30 g kg^{-1} in 1992. One could argue that the sod collected wind-blown soil from adjacent cultivated strips, but eroded soil would be lower in C content. The strong influence of unharvested vegetation in the sod soils is shown by its ^{14}C signature of 112 vs. 105 pmC for the native. The 112 pmC of the total soil C represents the mixture of old material and materials >112 pmC derived from bomb-affected ^{14}C containing residues added between 1972 and 1992. The 0 to 10 cm of the plow layer with less C than the native,

and only one-half that of the sod, had a ^{14}C age of 490 yr. The sod sites had 61% of its C present as nonhydrolyzable residue with a pmC activity of 87.1 and a ^{14}C age of 1110 yr. The plowed soil had a hydrolyzability similar to that of sod but did not have the usual large increase in age for the nonhydrolyzable residue; its age was 905 yr with a large error figure. The 10- to 20-cm layer of the plowed soil dated 900 yr older than the 0- to 10-cm layer, even though one would expect mixing of layers in a cultivated soil. This reflects the shallow depth of plowing (12 cm) at this site.

The wheat-fallow and wheat-hay-fallow rotations at Lethbridge, Alberta, (Table 6) were sampled in 1962. The rotations at that time had been established for 30 yr. Carbon contents vary within the different entry points of the wheat-fallow rotation, showing the soil variability encountered in the field (Campbell, 1964). The ^{14}C ages are the oldest obtained for unfractionated, surface soils in either the earlier Canadian studies or this study. They

Table 4. The tracer-C signatures in the catenary sequence of native soil at Sterling, CO, and Waldheim, Saskatchewan.†

Position	Depth cm	Total organic C \pm SE g kg^{-1}	^{14}C activity	^{14}C age	$^{13}\text{C} \pm \text{SE}$
			pmC	yr BP	‰
Sterling, CO					
Summit	0-10	6.9 ± 0.4	81.9 ± 0.7	1602 ± 68	-19.9 ± 0.06
Sideslope	0-10	6.4‡	86.3‡	1182‡	-20.5‡
	10-20	5.8‡	72.6‡	2571‡	-18.9‡
Toeslope	0-10	9.8 ± 0.2	91.2 ± 0.6	742 ± 53	-20.1 ± 0.02
Waldheim, Saskatchewan					
Summit	0-10	24	-	575 ± 80	-
Sideslope	0-15	21	-	270 ± 45	-25.0
Toeslope	0-15	27	-	216 ± 45	-25.1
	15-22	6.2	-	635 ± 55	-26.4
	22-52	4.2	-	930 ± 55	-29.2
Depression	0-18	24	-	Modern	-27.2
	22-35	5.2	-	700 ± 55	-27.9
	60-70	4.5	-	4870 ± 60	-

† The Martel and Paul (1974a) site at Waldheim, Saskatchewan, is classified in the Oxbow Association in the Canadian classification with the upper-slope soils being classified as Udic Haploborolls.

‡ Analysis of only one field sample was obtained.

Table 5. The organic C content and ^{14}C signatures of the Sidney, NE, site.

Treatment	Depth	Organic C \pm SE	Nonhydrolyzable C \pm SE	^{14}C activity		^{14}C age	
				Total organic C \pm SE	Residue C \pm SE	Total organic C \pm SE	Nonhydrolyzable C \pm SE
				pmC		yr BP	
	cm	g kg ⁻¹	%				
Native	0-10	21.0 \pm 0.85	70 [†]	105.5 [†]	94.4 [†]	Modern	462 [†]
Sod	0-10	30.0 \pm 0.71	61 \pm 3	112.0 \pm 7.0	87.1 \pm 6.7	Modern	1110 \pm 648
Plow	0-10	14.0 \pm 0.34	62 \pm 1	94.1 \pm 1.0	89.6 \pm 6.0	490 \pm 100	905 \pm 550
	10-20	12.2 \pm 1.10	-	84.6 [†]	-	1338 [†]	-

[†] Analysis of only one field sample was obtained.

show large treatment effects. An average age of 1950 yr was found in the wheat-fallow. In the wheat-hay-fallow cropping sequence, it was 1500 yr. Haas et al. (1957) found a significant relationship between the percentage of soil particles by weight that were <0.005 mm and the total C and N of their soils. Our study contained a large number of variables such as landscape position, management, and latitude. This, together with the low number of samples, did not allow us to obtain a relationship between ^{14}C age and silt plus clay. Table 6 shows a 10% range in silt plus clay in the soils of the different entry points of the Lethbridge wheat-fallow rotation. If the results of three points can be believed, these show that the ^{14}C age was least at the lowest silt plus clay content and highest at the high silt plus clay content.

The Mandan, ND, 1947 native site (Table 7) had C contents similar to those of the more northern Waldheim sites in Saskatchewan. These occurred in a soil where silt plus clay accounted for only 12 to 18% of the total soil particles and were part of the reason for the poor overall relationship between texture, soil C content, and ^{14}C age. The Mandan site had an age of 1200 yr BP for the surface 0 to 15 cm and 2150 yr BP for the 15- to 30-cm depth. Acid hydrolysis left a residue accounting for 58% of the original SOM with an age of 2245 yr. The 1947 cultivated sample showed an incongruity with depth. The surface and subsurface samples were similar in soil C and ^{14}C age at both depths, being very similar to the 15- to 30-cm depth of the native site. Explanations for this could involve erosion of the surface layer or the effects of deep plowing. Sampling the Mandan native site in 1992 showed the surface to be near modern. This would be expected from bomb-C effects. The 15- to 30-cm depth was unaffected; it was similar to the 1947 sample from the same site.

The 1947 Hays, KS, silty clay soil dated 645 yr BP at the surface and 1215 yr BP for the 15- to 30-cm depth. This small difference between the surface and subsurface relative to other sites was associated with one of

the smallest age differences between total soil and non-acid-hydrolyzable C (1055 yr). The Dalhart, TX, native loam to fine sandy loam showed the low C content found in drier southern USA soils. Of special interest is the 77% hydrolyzability of the soil. The surface soil at 930 yr BP produced a 1380-yr difference for the nonhydrolyzable C. This represents only 23% of the total C. The 29% nonhydrolyzable C of the subsurface was 1330 yr older than the total C of this soil.

The soil from Big Springs, TX, shows one problem associated with ^{14}C dating. Many laboratories utilize enriched tracer ^{14}C in experiments that range from crop residues to pesticides. Soil samples from Big Springs, archived for 45 yr, were contaminated at 680 and 560 pmC for the surface and subsurface, respectively. The source of this contamination is unknown. The samples are now stored in a well-isolated archival site. The contamination was removed by acid hydrolysis of the surface soil to obtain a residue with a count of 72.4 pmC. This is equivalent to a ^{14}C age of 2600 yr BP and indicates that the contaminant was associated with a protein or sugar rather than a ligniferous plant residue that would not have been solubilized by hydrolysis. The subsurface soil was not hydrolyzed.

The data from Maricopa, AZ, (Table 7) come from a previously arid site that now is irrigated. This Typic Torrifluent has a C content very similar to that of native Big Springs and Dalhart, TX, sites at 5 to 6 g C kg⁻¹ soil. The ^{14}C age of the residue of hydrolysis also is similar at 2400 yr BP. This soil is now under irrigation with relatively high substrate inputs; the percentage of nonhydrolyzable C and the great age were unexpected. One would expect a more significant contribution from the active fraction. However, under irrigation the active fraction could rapidly decompose leaving only the old resistant fraction. Other reasons could involve the fluvial origin and the deposition of older C. The site, although having four replicates rather than two like the other sites, showed the highest field variability in ^{14}C

Table 6. The effect of cultural practices on the ^{14}C content of soil organic matter at Lethbridge, Alberta (from Campbell, 1964).

Crop sequence	Rotation entry [†]	Yield	Depth	Total organic C	^{14}C age \pm SE	Silt	Clay	Silt plus Clay
Wheat-fallow-wheat	Wheat yr2	1030	0-15	13	2400 \pm 80	25	28	53
	Fallow	-	0-15	11	1515 \pm 95	21	22	43
	Wheat yr1	1525	0-15	10	1930 \pm 70	23	25	48
Wheat-Hay-Fallow-Wheat	Wheat yr2	1293	0-15	14	1430 \pm 75	24	27	51
	-	-	0-15	15	1560 \pm 75	26	27	53
	Wheat yr1	1095	-	-	-	-	-	-

[†] Crop in place the year sampled.

Table 7. The ^{14}C signature of historical and modern soil samples in the Great Plains.

Site	Site and year sampled	Depth	Total organic C	C in hydrolysis residue	^{14}C activity		^{14}C age	
					Total C \pm SE	Residue C \pm SE	Total organic C \pm SE	Residue C \pm SE
					pmC		yr BP	
Mandan, ND	Native 1947	0-15	25.6 \pm 1.1	58 \pm 3	86.2 \pm 0.8	75.6 \pm 1.6	1200 \pm 75	2245 \pm 175
		15-30	17.3 \pm 0.8	-	76.45 \dagger	-	2155 \dagger	-
Mandan, ND	Cultivated 1947	0-15	16.5	-	78.5 \pm 0.5	-	1945 \dagger	-
		15-30	17.5	-	77.4 \pm 0.5	-	2060 \dagger	-
Mandan, ND	Native 1992	0-15	28	-	99.3	-	56	-
		15-30	17	-	80.0	-	1825	-
Hays, KS	Native 1947	0-15	23.2 \pm 0.8	56 \pm 0.2	92.3 \pm 0.1	80.9 \pm 0.2	645 \pm 5	1700 \pm 25
		15-30	14.0 \pm 0.6	-	86.0 \dagger	-	1215 \pm 55	-
Big Spring, TX	Native 1947	0-15	5.9 \pm 0.2	43 \pm 1.4	680	72.4 \pm 2.7	contam.	2600 \pm 300
		15-30	5.2 \pm 0.2	-	560	-	contam.	-
Dalhart, TX	Native 1947	0-15	6.0 \pm 0.2	23 \dagger	89.1 \pm 0.6	75.0	931 \dagger	2314 \dagger
		15-30	4.7 \pm 0.4	29 \pm 11	82.1 \pm 1.2	69.6 \pm 0.6	1588 \pm 120	2918 \pm 68
Maricopa, AZ	Cultivated 1947	0-30	5.8	54 \dagger	92.8 \pm 3.6	72.0 \pm 3.7	400	2400

\dagger Analysis of only one field sample was obtained.

that we have encountered to date. Leveling of sites prior to irrigation can expose varying amounts of subsurface soil. These have very different ages than the surface.

DISCUSSION

The data showing similar to possibly slightly increased SOM levels in recently sampled sites relative to those sampled in 1947 do not represent a large enough sample for us to say that SOM levels have increased since 1947. They, however, point out that the cultivation-induced, drastic decrease in SOM levels that has mesmerized soil scientists for so long should be an issue of the past as new steady-state levels are reached. The Sidney site converted from a grazed C_4 to an ungrazed C_3 sod actually showed a SOM increase above native levels. This series of sites with a large range of climate, landscape, and management effects was not designed to specifically measure clay effects. It is not surprising that the influence of silt plus clay could not be ascertained. The effect of the north-south gradient on the content of total C is demonstrated. The latitude effect is not noticeable in soil ^{14}C ages.

The use of ^{14}C -dating techniques made it possible to characterize the ^{14}C age of a cross section of North American Great Plains soils. In relating these ages to MRT, we realize that the soil has a continuum of different-aged materials (Goh, 1991). The interpretation of ^{14}C ages in the past led to the concept of a physically active, relatively biologically inactive pool of C that constitutes approximately one-half of the soil C (Paul and van Veen, 1978; Paul, 1984). The ^{14}C ages and resultant MRT were used to determine pool sizes and fluxes in the mathematical models of Jenkinson and Rayner (1977) and van Veen and Paul (1981). They were indirectly used in later models such as CENTURY (Parton et al., 1987, 1993). Our data from a more extensive range of sites support the original concepts.

The ^{14}C ages of surface soil represented some with high concentrations of bomb ^{14}C (Sidney, NE, sod). The 1676 yr BP of the summit of the Sterling, CO, catena shows the influence of erosion. The 2400 yr BP age of the wheat-fallow rotation at the Lethbridge, Alberta, site is the oldest age for the total C of surface horizons in our studies. Depth effects were consistent and strik-

ing. The 10- to 20-cm depth was an average of 1200 yr older than the 0- to 10-cm depths of both cultivated and native sites sampled in 1992. The archived samples from the 1947-1949 study of Haas et al. (1957) were obtained before bomb ^{14}C production; they had been influenced by the dilution of the atmospheric $^{14}\text{CO}_2$ by industrial CO_2 with no radioactivity in what is known as the Suess effect (Goh, 1991). These soils showed a 1200 yr age increase between the 0- to 15- and the 15- to 30-cm depths. We must conclude that generally there cannot have been a large amount of mixing of surface and subsurface soils by either soil fauna or by human activities such as cultivation.

We do not have a good explanation for the 7000 yr BP age of the 90- to 120-cm depth of the Akron soil. The ^{14}C age differences between the surface and subsurface samples from the other soils indicate that they are likely to be similarly old at depth once they are ^{14}C dated. Such differences with depth have been previously noted for other geographical locations (Goh et al., 1984; Scharpenseel, 1993, 1994). The total SOM at depths of 30 cm and deeper had ^{14}C ages as old and older than the paleosols collected from Colorado by Kelly and Yonkers (1993). They attributed the 6000-yr age of the deep soil samples to Holocene sand dune formation rather than to ongoing pedogenic processes. The Weld soil series such as that at Akron is known to contain paleosols. The increase in the silt content and decrease in clay at depths below 60 cm at this site show a difference in soil particle deposition patterns. The ^{14}C age, however, increased uniformly at the various depths (Fig. 2). The possibility of a paleosolic profile is not an easy explanation for our results. Old, inert soluble C diffusing down the profile has been considered as a cause of the old age with depth. This, however, should be minimal in the dry, high base exchange soils in this study. Inorganic carbonates that occur at depth in grassland soils were removed prior to analysis. They should not affect the age of the SOM. The carbonates of the Maricopa, AZ, soil comprised 2.5 g $\text{CO}_3\text{-C kg}^{-1}$ with a ^{14}C age of 2420 \pm 170 yr (Leavitt et al., 1994). These were similar in age to the acid-insoluble C. Root CO_2 fixation is known to occur to some extent. The possibility that root fixation of C from carbonates at depth must be considered. How-

ever the ^{14}C content of soil CO_2 has been shown not to differ greatly from that fixed by the aboveground plant parts (Broecker and Olson, 1959).

The dilution of old geological organic C in the parent material with modern C must be considered. The close inverse relation between the C content and ^{14}C age with depth as in Fig. 2 is reminiscent of an isotope dilution experiment. Small amounts of very old or dead C can greatly influence the ^{14}C age. One could envision a loess soil such as the one at Akron, CO, receiving deposited material with significant contents of old C. This was found to be the case in a lacustrine site studied by Haas et al. (1986). However, similar increases in age with depth also were found in glacial till sites such as those of Saskatchewan (Martel and Paul, 1974b) and in other Mollisols (Scharpenseel, 1993). One must seek a pedological-biological explanation with the interesting question of what the decomposition dynamics (CO_2 evolution) would be of the 7000-yr-old Akron, 90- to 120-cm soil sample under laboratory conditions.

The residue of hydrolysis from the various soils contained 23 to 70% of the total organic soil C. Hydrolysis produced an average age difference of 1400 yr in surface soils and 1700 yr in subsurface samples where a larger portion of the C appeared to be hydrolyzable. The C content and ^{14}C activity of the hydrolyzable fraction can be calculated from the data for the whole soil and the nonhydrolyzable fractions. Acid hydrolysis removes proteins, nucleic acids, and polysaccharides of soil (Schnitzer and Khan, 1972). It does not solubilize all of the cellulose of plant residues. Schnitzer and Khan (1972) measured the effects of 6 M HCl on the characteristics of isolated humic and fulvic acids. Hydrolysis resulted in a weight loss of approximately 40% for both fractions. The C content of humic acids remained unchanged at 57%; that of fulvics was 53% in the residue relative to 49% in the total fulvics. This is related to the observation that the H content and O-containing functional groups remained unchanged as a percentage of the residue in the humic acids, whereas the fulvics were drastically decarboxylated with an evolution of CO_2 -C.

We conclude that acid hydrolysis, if interpreted relative to its action on both plant residues and SOM, gives meaningful pool size estimates for temperate soils. Some of our archived samples were suspected to be contaminated. Hydrolysis helped verify the contamination. It also provided the basis for determining the decomposition rates of the soil-C pools. The great age of both the total soil and the nonhydrolyzable fraction at depth is not yet fully explained. Martel and Paul (1974b) exhaustively treated the nonhydrolyzable residue with NaOH to peptize and solubilize humics. Hydrofluoric acid was then used to remove clays. This resulted in the deflocculation or solubilization of 93% of total soil C. The C remaining was not as old as nonhydrolyzable C. The NaOH extract of the 6 M HCl residue of hydrolysis gave the oldest fraction.

Soil depth, catenary position, and management all affected the soil ^{14}C age. The position in the catena was especially notable. Lower slopes in the catenary sequences not only had lower ^{14}C ages, they also had much different ^{13}C contents. The explanation for this is

not clear. Erosion of the upper slopes would expose older subsurface organic matter. The lower slopes should contain some of the transported C but should also contain a larger fraction of young, active material. The $\delta^{13}\text{C}$ values of plants become less negative as water-use efficiency of plants increases on upper slopes. This signature is reproduced in the SOM. The $\delta^{13}\text{C}$ values were essentially not different in the catenary sequence in Colorado. This catena did not contain the moist depressional site equivalent to that in Saskatchewan. The effect of the plant water-use efficiency in the lower, wetter sites could be offset by CH_4 formation during decomposition under anaerobic conditions. The CH_4 evolved (Boutton, 1991) can have a signature (δ) of -56% . The C remaining in the soil would be more positive than that of the incoming plant residues. That this did not occur on these two sites gives an indication that CH_4 formation is not a major process in these moist but not often flooded areas.

Our results show the applicability of both ^{14}C dating and soil hydrolysis (Leavitt et al., 1997) to analytically establish the pool size and flux rates of the resistant, soil organic C. Plant-residue lignin and related compounds are known to be included in the nonhydrolyzable residue; the nonhydrolyzable residue may overestimate the size of the old, resistant fraction. Sodium hydroxide peptization could further separate these materials. The material further removed from the residue by NaOH would be only slightly older than the residue of hydrolysis (Martel and Paul, 1974b). The resistant fractions are so old that, although of great importance from a structure, water and nutrient, and pesticide adsorption standpoint, they play a small role in nutrient cycling. Global C cycle questions must, however, consider both the size and the turnover rate of these older fractions (Paul et al., 1995).

This study is the first of a series that will incorporate ^{14}C ages and $\delta^{13}\text{C}$ contents of soil fractions as affected by parent material, vegetation, management, location, and depth. Also being conducted are long-term incubations with measurement of the $\delta^{13}\text{C}$ of the evolved CO_2 on sites with a known signature of different $\delta^{13}\text{C}$ contents. This will allow us to model the data relative to plant residue inputs on sites with a long management history. Leavitt et al. (1994) showed that $\delta^{13}\text{C}$ was more consistent and easier to measure than ^{14}C ages. Carbon-14 dating gives us pool sizes and fluxes of the resistant fractions as well as an indication of the overall pedogenic characteristics; $\delta^{13}\text{C}$ and long-term incubation will measure the dynamics of the more active fraction(s).

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