



**MISSISSIPPI BASIN CARBON PROJECT:  
UPLAND SOIL DATABASE FOR SITES IN YAZOO BASIN,  
NORTHWESTERN MISSISSIPPI**

**U.S. GEOLOGICAL SURVEY**

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MISSISSIPPI BASIN CARBON PROJECT - UPLAND SOIL DATABASE FOR  
SITES IN YAZOO BASIN, NORTHWESTERN MISSISSIPPI

By J.W. Harden<sup>1</sup>, T.L. Fries<sup>1</sup>, and T.G. Huntington<sup>2</sup>

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<sup>1</sup> U.S. Geological Survey, 345 Middlefield Rd., MS 962, Menlo Park, CA 94025

<sup>2</sup> U.S. Geological Survey, 3039 Amwiler Rd., Peachtree Business Center--Suite 130,  
Atlanta, GA 30360

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2

## Table of Contents

1. Background
  - 1.1 Objectives
  - 1.2 Approach
2. Methods
  - 2.1 Field methods
  - 2.2 Sample preparation and drying
  - 2.3 Total carbon , inorganic carbon, organic carbon, total nitrogen,  $^{13}\text{C}$ ,  $^{15}\text{N}$
  - 2.4 Radiocarbon
  - 2.5 Particle size analysis
3. Data-set Identification
  - 3.1 Labeling schemes
  - 3.2 Data set descriptions
    - 3.21 Miss\_Site
    - 3.22 Miss\_Field
    - 3.23 Miss\_Soil
    - 3.24 Miss\_Isotope
    - 3.25 Miss\_Psize
4. Application of the data set
5. References
6. Investigators
7. Acknowledgements

List of tables (as separate downloadable files)

Table 1. Site description--Miss\_Site

Table 2. Descriptive field data--Miss\_Field

Table 3. Soil analytical data--Miss\_Soil

Table 4. Particle size data--Miss\_Psize

Table 5. Soil isotopic data--Miss\_Isotope

## *1. Background*

The conversion of land from its native state to an agricultural use commonly results in a significant loss of soil carbon (Mann, 1985; Davidson and Ackerman, 1993). Globally, this loss is estimated to account for as much as 1/3 of the net CO<sub>2</sub> emissions for the period of 1850 to 1980 (Houghton et al, 1983). Roughly 20 to 40 percent of original soil carbon is estimated to be lost as CO<sub>2</sub> as a result of agricultural conversion, or "decomposition enhancement", and global models use this estimate along with land conversion data to provide agricultural contributions of CO<sub>2</sub> emissions for global carbon budgets (Houghton and others, 1983; Schimel, 1995).

As yet, erosional losses of carbon are not included in global carbon budgets explicitly as a factor in land conversion nor implicitly as a portion of the decomposition enhancement. However, recent work by Lal et al (1995) and by Stallard (1998) suggests that significant amounts of eroded soil may be stored in man-made reservoirs and depositional environments as a result of agricultural conversion. Moreover, Stallard points out that if eroding soils have the potential for replacing part of the carbon trapped in man-made reservoirs, then the global carbon budget may grossly underestimate or ignore a significant sink term resulting from the burial of eroded soil.

Soil erosion rates are significantly (10X) higher on croplands than on their undisturbed equivalents (Dabney et al, 1997). Most of the concern over erosion is related to diminished productivity of the uplands (Stallings, 1957; McGregor et al, 1993; Rhoton and Tyler, 1990) or to increased hazards and navigability of the lowlands in the late 1800's to early 1900's. Yet because soil carbon is concentrated at the soil surface, with an exponential decline in concentration with depth, it is clear that changes in erosion rates seen on croplands must also impact soil carbon storage and terrestrial carbon budgets as well.

## ***1.1 Objectives***

A primary goal of the Mississippi Basin Carbon Project (Sundquist and others, 1998) is to define simple, functional relationships between hillslope erosion/sedimentation and soil organic matter dynamics. To meet this goal, small watersheds were chosen for studies of upland soils in context of and collaboration with ongoing erosion/sedimentation studies. The study sites are located in watersheds in the upland portion of the Yazoo River basin in northeastern Mississippi, where loess soils are known for their high erodibility. A full accounting of hydrology, geography, and site description was reported by Huntington and others (1998) in a companion report, which includes extensive maps, figures, and data pertaining to this report.

In this report we present soil analytical data for forested and agricultural sites managed by the National Sedimentation Laboratory of USDA Agricultural Research Service (Oxford, Miss.) and USDA Forest Service (Holly Springs, Miss.). A descriptive, text format is used to present our theories and strategies, site and field information, methods of measurement, application of data sets, and references. A table format is used to present data for easy downloading from the internet site <http://geochange.er.usgs.gov/pub/carbon/>

## ***1.2 Approach***

Important controls on soil carbon storage include climatic/edaphic controls such as temperature and moisture; plant type; parent material controls such as clay content and soil drainage class. The depth distributions of temperature, moisture, and particle size varies greatly within a soil profile. Therefore potential decomposition and C storage in soils are likely highly sensitive to depth as well. Erosion and burial affect the depth of soil carbon and the potential for organic-matter decomposition through depth-dependent controls on soil temperature, moisture, plant input, and particle size. Therefore, a sampling strategy was designed to allow for stratification by these important variables.

Soil samples were collected to represent soil properties at erosional ("upper") and depositional ("lower") slope positions of small drainage basins for cultivated and uncultivated landuse pairs. Ridgetop positions and slope transects (catenas) were also sampled at some localities. Data from sample analysis are used to determine, compare, or contribute to the determination of (1) inventory of C and N in soil on hillslope positions, (2) turnover times of soil organic matter at these hillslope positions, including assessments of sizes and turnover of fast to slower pools of organic matter, and ultimately, (3) rates of carbon input by net primary production

(NPP) and slope deposition as well as loss by decomposition and erosion at various hillslope positions. Documentation and data sets described in this report include (1) site location, (2) descriptive field data, (3) physical, chemical, and isotopic analysis of (solid phase) soil samples, and (4) isotopic analysis of soil gas collected from static field chambers.

As a strategy for meeting the overall goal of defining simple, functional relationships between hillslope erosion/sedimentation and soil organic matter dynamics, we further refined our goals:

(1) Estimate rates of carbon input, turnover, and accumulation in the soils of ridgetop, eroding "upper" slope and depositional "lower" slope positions. The primary measurements are soil carbon inventories, soil incubations, and measures of  $^{14}\text{C}$  and  $^{13}\text{C}$  content of solid and gas phases.

(2) Relate our estimates of dynamics of soil carbon to slope processes at the hillslope scale.

(3) Help in the development of conceptual models that relate erosional-depositional dynamics to soil-carbon dynamics at various hillslope, watershed, and regional scales.

Parameters can be defined for a simple mass balance equation in which changes in carbon storage over time are defined by inputs and losses of carbon to and from a sampling site. At the sample-site scale, soil carbon is balanced by inputs of net primary production and depositional carbon; losses include loss to decomposition, fire, erosion, and dissolved organic carbon. Each term can be further subdivided into pools of organic carbon that decompose at different rates. For three pools of soil carbon with three different turnover (or replacement) times, inputs to each depositional term are separated and modeled as variables that change over time according to climate and nutrient controls on NPP or to depositional controls on sedimentation rates. Losses of carbon from each pool are modeled as first order or fractional losses, with  $k_1$ ,  $k_2$ , and  $k_3$  indicating the inverse of the turnover time in years.

### Parameterizing Soil Carbon at the Upland Site Scale

$$\begin{aligned}
 dC/dt &= \text{changing C with time} = \text{Inputs} - \text{Losses} \\
 &= I_{\text{production}} + I_{\text{deposit}} - L_{\text{decomp}} - L_{\text{fire}} - L_{\text{eros}} - L_{\text{DOC}} \\
 &= I_{\text{prod}} + I_{\text{dep}} - kC - L_{\text{fire}} - L_{\text{eros}} - L_{\text{DOC}} \\
 &= I_{\text{prod}} + I_{\text{dep}} - (k_1C_1 + k_2C_2 + k_3C_3) - L_{\text{fire}} - L_{\text{eros}} - L_{\text{DOC}} \\
 &= I_{\text{prod}} + I_{\text{dep}} - (k_1C_1 + k_2C_2 + k_3C_3) - L_{\text{fire}} - (L_{\text{er1}} + L_{\text{er2}} + L_{\text{er3}}) - L_{\text{DOC}} \\
 &= I_{\text{prod}} + (I_{\text{dep1}} + I_{\text{dep2}} + I_{\text{dep3}}) - (k_1C_1 + k_2C_2 + k_3C_3) - L_{\text{fire}} - \\
 &\quad (L_{\text{er1}} + L_{\text{er2}} + L_{\text{er3}}) - L_{\text{DOC}}
 \end{aligned}$$

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- Where I = inputs and L = losses
- Production = net primary production
- I<sub>deposit</sub> such as from overland flow or alluviation
- L<sub>decomp</sub> = decomposition
- L<sub>fire</sub> = losses of C to burning
- L<sub>eros</sub> = losses of C to erosion such as overland or rill erosion
- L<sub>DOC</sub> = losses of C from soil layers to leaching of dissolved organic carbon
- $k_1, k_2, k_3$  = decomposition coefficients for pools number 1 (fastest), 2 (intermediate), 3 (slow) of organic carbon
- $C_1, C_2, C_3$  = storage terms for pools # 1, 2, 3 of organic carbon
- $er_1, er_2, er_3, dep_1, dep_2, dep_3$  = erosion and deposition terms for pools #1, 2, 3



The types of data that are collected or estimated for site-specific studies and the model parameters that are estimated from these data can be classified into four types of measurements: (1) carbon and nitrogen inventories (2) decomposition rates (3) erosion rates and (4) deposition rates. Measurements such as the total carbon inventory (C1+C2+C3) can be determined directly. However partitioning the organic carbon into separate terms requires a variety of measurements, calculations, and modeling (Harden and others, in press) or using fractionation techniques for the organic matter (Trumbore, 1994).

#### Model terms (1-4) and types of data collected to define model terms

1. Terms: C, N inventories on landforms and model terms TC, C1, C2, C3

Data:  $^{14}\text{C}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$  measurements of select samples of bulk or fractionated soil carbon.

2. Terms  $k_1, C_1, k_2, C_2, k_3, C_3$  for decomposition coefficients and flux rates

Data: incubation  $\text{CO}_2$  flux,  $^{14}\text{C}$  of incubation  $\text{CO}_2$ ,  $^{13}\text{C}$  of incubation  $\text{CO}_2$ , respiration chamber  $\text{CO}_2$  flux,  $^{14}\text{C}$  of chamber  $\text{CO}_2$ ,  $^{13}\text{C}$  of chamber  $\text{CO}_2$  soil temperature, soil moisture  $^{14}\text{C}$  and  $^{13}\text{C}$  of soil organic matter fractions

3. Terms LTC, Ler1, Ler2, Ler3 for erosional losses of carbon

Data: USDA erosion-plot sediment C, N and organic fraction C, N

USDA weirs (export term) sediment C, N

USDA watersheds and weir sediment  $^{7}\text{Be}$

soil  $^{10}\text{Be}$

soil  $^{7}\text{Be}$

4. Terms  $I_{deTC}, I_{de1}, I_{de2}, I_{de3}$  for depositional carbon

Data: lower slope Cs, Pb, pollen dating

soil  $^{10}\text{Be}$

soil  $^{7}\text{Be}$

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#### Other model parameters derived from the literature and other sources

Terms  $I_{prod}$  and  $L_{fire}$

Information sources Ceres, Century models

## **2. Methods**

### **2.1 Field methods**

Soil profiles were described according to USDA-NRCS methods (Soil Survey Staff, 1951) in which a variety of field properties are recorded for soil horizons at different depths. Properties such as soil color, consistence, texture, structure, root size and density were recorded on field sheets.

The weight of soil organic carbon and total N per land surface area is referred to as the carbon inventory of a site. Measurements of percent organic C, bulk density and depth are included in this data. The calculation is depth-dependent and can be measured or calculated to 0.5m, 1m, or greater depths. Most of MBCP-U measurements include data to 1m depths (see section 9. for data manipulations).

Soil samples were collected in such a way that volumetric data could be combined with gravimetric data to provide measurements in units of volume (3 dimensional), area (2 dimensional), and depth (1 dimensional). Bulk density, field moisture content, and depth increments are included in soil sampling. Bulk density samples were collected with a variety of tools, including cores of known diameter (mineral soil) or boxes (litter and organic horizons) of known area. Samples were collected into the core or box of known volume.

Our most consistent and accurate density measurements for soils were obtained with a coring device by Soil Moisture Corporation ("whomper") in which internal rings can be disassembled for intact samples. Less consistent and less accurate measurements were obtained from a hand-driven soil AMS core with internal sleeves; we found that a slightly crimped tip on the commercial core gave densities comparable to "whomper"; however, in earlier trials where the tip had a straight internal barrel, bulk densities were 30% underestimated. As a result, in most cases for mineral soils, bulk density samples were taken at depth intervals of 0-5, 5-10, at 15 , at 30, at 50, at 70, and at 90 cm with "whomper". In some cases, the revised tip of the AMS probe was used for depths of 0-5, 5-10, 10-20, 20-40, 40-60, 60-80, and 80-100 cm. Gravimetric samples used for analysis of C, N, water content, and isotopic analysis were sampled at depth intervals of 0-5, 5-10, 10-20, 20-40, 40-60, 60-80, and 80-100 cm. Volumetric and gravimetric samples were weighed on collection day on a calibrated balance to 0.01 g.

Samples of soil gas were collected through a soil chamber in line with a LICOR gas analyzer. These sites were monitored seasonally for soil CO<sub>2</sub> flux, by T. Huntington (Huntington and others, in prep). For soil <sup>14</sup>CO<sub>2</sub> and <sup>13</sup>CO<sub>2</sub>, the soil

chamber was placed into a sand ring that was confined between two pieces of PVC rings. The chamber was “scrubbed” by placing a soda lime trap (along with a desiccant to protect the soda lime), which were in line with the circulating air for a period of time that allowed 3 volumes of chamber air to pass through the soda lime. This way, only a small fraction of CO<sub>2</sub> in circulating chamber air was likely to be contributed from the atmosphere, leaving the majority of CO<sub>2</sub> to be respired from roots and soil heterotrophs. After “scrubbing” the chamber gas with soda lime, a valve was used to close the soda lime trap and allow CO<sub>2</sub> to build up in the soil chamber. A valve was then used to trap soil CO<sub>2</sub> onto molecular sieve material for determination of <sup>14</sup>C from the CO<sub>2</sub> and, in some cases, <sup>13</sup>C from the CO<sub>2</sub>.

## ***2.2 Sample preparation and drying***

Field notes were used to inventory all samples entering the laboratory. Samples were visually inspected and weighed as received. Any inconsistency between field descriptions or weights and laboratory observations was resolved before sample preparation began. If samples taken for analytical, moisture or bulk density measurements could not be processed immediately they were stored in the dark, at 4 °C.

Soil and litter samples were laid out on open shelves, in an isolated room, and were allowed to air dry to a constant weight. Temperature in the air drying room ranged from 20-30 °C during this process. Air dry moisture samples, or splits of air dry samples, were then oven dried to constant weight in a forced-draft oven. Litter samples, as well as any other samples that appeared to contain greater than 20 percent organic matter, were oven dried at a temperature of 65 °C to avoid loss of organic matter by oxidation or decomposition. All other samples were oven dried at a temperature of 105 °C. Air-dry and oven dry weights from this procedure were used in the calculations of percent moisture and bulk density.

To prepare air dry soil samples for analysis the samples were first gently crushed using a ceramic mallet and plate. The crushed sample material was thoroughly mixed and then split into analytical and archived portions. The analytical split was weighed and sieved using a 2mm screen. Material not passing the 2mm screen was removed, weighed and transferred to a plastic bag marked with the sample identification, the starting weight of the analytical split and the weight of the material not passing through the 2 mm screen. Material passing the 2mm screen was then ground by hand, using a mortar and pestle, to pass through a 60 mesh (0.246 mm) screen. The ground material was carefully mixed and either the entire sample or a

representative split was placed in a labeled sample container. Archival materials were placed in labelled plastic bags and boxed for storage.

Air dry litter samples were described, photographed, spread onto a work table and then thoroughly mixed using a spatula and a scoop. The well-mixed samples were then split into subsamples for oven drying, analytical chemistry and archiving. Inhomogeneous portions of the sample, such as large sections of bark or large diameter (> 1 cm) material, were placed in separate bags and included with the archive split.

The analytical split of the air dry litter sample was prepared for analysis by first manually chopping or crushing larger material and then milling the entire sample to pass a 0.5 mm screen using a cyclone sample mill. The milled sample was then thoroughly mixed and a representative sample placed in a labelled, glass sample bottle.

The archive split of the litter was placed in a labelled, plastic bag for storage. The bags used for storage were large enough that the entire air-dry archive split could be placed in the bag without crushing the sample. Archive materials were then boxed for storage.

All excess sample materials and waste were oven dried at 120°C for 72 hours prior to disposal.

### ***2.3 Total Carbon, inorganic carbon, organic carbon, total nitrogen, $^{13}\text{C}$ , $^{15}\text{N}$***

Total carbon (TC) was determined by measuring the carbon dioxide ( $\text{CO}_2$ ) produced by combusting the sample in a stream of oxygen ( $\text{O}_2$ ). Total carbon measurements were made using either a LECO carbon determinator (WR-112) or a Fisons NA1500 elemental analyzer(EA)/ Optima isotope ratio mass spectrometer (IRMS). Inorganic carbon (IC) was determined by measuring the  $\text{CO}_2$  generated by heating a sample at 105 °C in acid. A UIC coulometer was used for this measurement. Organic carbon was calculated as the difference between TC and IC. A Fisons NA1500 EA/Optima IRMS was also used for the determination of total nitrogen and for  $^{15}\text{N}$  and  $^{13}\text{C}$  measurements.

Total carbon measurements made using the LECO carbon determinator were carried out by analyzing between 0.1 and 1.0 g of sample, depending on expected carbon concentration. The sample material was mixed with copper metal and iron chip accelerators in a ceramic crucible, the ceramic crucible was placed in a radio frequency furnace and the sample combusted in a stream of carbon dioxide free oxygen. Gases generated by the combustion process were passed through a series of

catalysts, to ensure complete oxidation, and scrubbers, to remove components that would interfere with the CO<sub>2</sub> measurement. The CO<sub>2</sub> was then absorbed onto molecular sieve at room temperature. When sample combustion and CO<sub>2</sub> collection were complete the molecular sieve was heated to 350 °C, releasing the absorbed CO<sub>2</sub>, and the CO<sub>2</sub> measured using a thermal conductivity detector.

The procedure using the Fisons NA1500 EA/Optima IRMS for the determination of total carbon, total nitrogen, <sup>13</sup>C and <sup>15</sup>N employed a Fisons NA1500 elemental analyser for sample combustion and separation of CO<sub>2</sub> and N<sub>2</sub> from other combustion products. The gas stream from the elemental analyser then entered the Optima IRMS which was used to obtain analytical data for total carbon, total nitrogen, <sup>13</sup>C and <sup>15</sup>N. For this analysis between 1 and 30 mg of sample, depending on the estimated carbon concentration, was loaded into a tin capsule and the capsule tightly crimped to exclude atmospheric gases. Samples were then combusted at 1000 °C in a stream of oxygen. The gases generated during combustion then pass through heated combustion and reduction reactors to achieve quantitative conversion of carbon and nitrogen from the sample to CO<sub>2</sub> and nitrogen (N<sub>2</sub>). The combustion products next passed through a chromatographic column where CO<sub>2</sub> and N<sub>2</sub> were separated and then introduced into the mass spectrometer for measurement. Elemental concentrations were calculated based on instrument responses for calibration standards. Isotope ratio measurements were corrected for fractionation effects and calibrated based on materials with known values.

In the early stages of this study, the Fisons NA1500 EA/Optima IRMS was used only to obtain total nitrogen and <sup>15</sup>N data. However, total carbon data for samples analyzed using the LECO instrument, and <sup>13</sup>C data for samples analyzed in a conventional extraction line/mass spectrometer lab, showed excellent agreement with data for the same samples analyzed using the EA/IRMS instrument. As a result of this data comparison the EA/IRMS was used for nearly all TC, IC, <sup>13</sup>C, and <sup>15</sup>N analyses performed on solid samples.

In addition to calibration materials, three standard materials were routinely included in all EA/IRMS sample runs. These materials were a well analyzed sample of ethylenediaminetetracetic acid (EDTA) obtained from Fisons Instruments, S.p.a., a marine sediment (MESS-1) issued by the Chemistry Division of the Canadian National Research Council and a river sediment (NBS1645) issued by the National Bureau of Standards, now known as National Institute of Standards and Technology. Precision estimates, expressed as relative standard deviation, were 3.6-6.5 percent for total carbon, 3.7-6.5 percent for total nitrogen, 1.4-2.9 percent for <sup>13</sup>C, and 20-

47 percent for  $^{15}\text{N}$ , based on results for the standard materials (table below). Approximately 3 percent of all samples were also analyzed in duplicate. For these duplicate runs the range, expressed as a percentage of the average of the duplicate runs, was less than 1 percent for total carbon, less than 2 percent for total nitrogen, less than 1 percent for  $^{13}\text{C}$  and about 15 percent for  $^{15}\text{N}$ .

I.D.	total %C (%rsd)	total %N (%rsd)	delta 13C (%rsd)	delta 15N (%rsd)
EDTA	3.6	3.7	1.5	---
MESS-1	4	3.9	2.9	20.1
NBS-1645	6.5	6.5	1.4	47.2

Inorganic carbon was determined by measuring the  $\text{CO}_2$  generated by treating approximately 100 mg of sample with 2N perchloric acid ( $\text{HClO}_4$ ) and heating the mixture at  $105^\circ\text{C}$ . The evolved gases were first passed through an acidic (pH of 3) saturated silver sulfate ( $\text{Ag}_2\text{SO}_4$ ) solution containing 3percent hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) to remove contaminants and then were bubbled through a partially aqueous solution containing ethanolamine and a colorimetric indicator. The  $\text{CO}_2$  was quantitatively absorbed and converted to a strong, titratable acid by the ethanolamine. The amount of  $\text{CO}_2$  evolved from the sample was measured by integrating the amount of current required to electrically generate enough base to titrate the acid.

Because pH measurements made on soils from both the Goodwin Creek (GC) and Nelson Farm(NF) sites indicated that no inorganic carbon (IC) should be present, inorganic carbon was determined on only a selected set of samples. Results for this set of samples (GCPU1.20i1, GCPU1.40i1, GCPL1.20i1, GCPL1.40i1, NFPU1.20i2, NFPU1.40i3, NFPL1.20i1, NFPL1.40i1) indicated that IC concentrations were less than 0.005 percent. No other IC measurements were made for the GC or NF sites.

The uncertainty in the IC determinations, expressed as relative standard deviation, is approximately 5 percent when IC is present. The range of IC values for standard materials run in duplicate was less than one percent of the carbon value.

## **2.4 Radiocarbon**

The  $^{14}\text{C}$  content of the solid and gas phases of soil is used to calculate overall turnover time or to partition the organic carbon into more labile or stable pools.

(From Trumbore and others, in prep) "  $^{14}\text{C}$  is produced in the stratosphere by the  $^{14}\text{N}(n,p) ^{14}\text{C}$  reaction. The  $^{14}\text{C}$  atom is oxidized rapidly to  $^{14}\text{CO}$ , which has a

lifetime of months before it is oxidized to  $^{14}\text{CO}_2$ . Most  $^{14}\text{C}$  production occurs in the stratosphere, but the long lifetime of  $\text{CO}_2$  enables  $^{14}\text{CO}_2$  to become well mixed throughout the troposphere. The steady state  $^{14}\text{C}$  content of the atmosphere is determined by the exchange of carbon in  $\text{CO}_2$  with that in ocean and biospheric reservoirs. Because of the relatively rapid cycling of carbon between the atmosphere and living biomass, most plants maintain a  $^{14}\text{C}$  specific activity (or  $^{14}\text{C}/^{12}\text{C}$  ratio corrected for mass-dependent isotope fractionation effects) that equals that of atmospheric  $\text{CO}_2$ . Similarly, animals reflect the  $^{14}\text{C}/^{12}\text{C}$  of the plants (or animals) they consume. Upon the death of an organism, the  $^{14}\text{C}$  in its tissues is no longer replenished, and decays with a half life of 5730 years. If the tissue remains intact and isolated from exchange, the  $^{14}\text{C}/^{12}\text{C}$  ratio may be used to indicate the time since the death of the organism. This is the basis for radiocarbon.

Calculation of a radiocarbon age requires the assumption that the  $^{14}\text{C}$  content of the carbon originally fixed in plant tissues equaled that of the atmospheric  $\text{CO}_2$  in 1950 (0.95 times the activity of oxalic acid, or Modern). In fact, the  $^{14}\text{C}$  content of the atmosphere has varied with time because of changes in the production rate of  $^{14}\text{C}$  (cosmic ray flux and magnetic field variations) and because of changes in the distribution of carbon among ocean, biosphere and atmospheric reservoirs. These variations, deduced from the  $^{14}\text{C}$  content of cellulose of known age taken from the annual growth rings of trees, are generally less than 10 percent over the past 7,000 years. More recent changes in the  $^{14}\text{C}$  content of atmospheric  $\text{CO}_2$  have resulted from dilution by  $^{14}\text{C}$ -free fossil-fuel-derived carbon and by the production of  $^{14}\text{C}$  during atmospheric testing of thermonuclear weapons (bomb  $^{14}\text{C}$ ). The latter effect dominates other natural and fossil fuel effects, as the atmospheric burden of  $^{14}\text{C}$  was approximately doubled in the few years preceding the implementation of the Nuclear Test Ban Treaty in 1964. This isotopic spike in the global carbon system provides a means for radiocarbon to be a useful tracer of carbon cycle processes on timescales of decades.

We express  $^{14}\text{C}$  data in the geochemical Delta notation (Delta = capital greek delta), the deviation in parts per thousand (per mil) from an absolute standard (0.95 times the activity of NBS oxalic acid measured in 1950). In this notation, zero equals the  $^{14}\text{C}$  content of 1895 wood, positive values indicate the presence of 'bomb' radiocarbon, and negative values indicate the predominance of C fixed from the atmosphere more than several hundred years ago.

One important correction made in calculating the Delta  $^{14}\text{C}$  value is of note here - the  $^{13}\text{C}$  correct needed to account for isotopic fractionation effects. As an example, consider the  $\delta^{13}\text{C}$  difference between atmospheric  $\text{CO}_2$  and carbon fixed during

photosynthesis by C3 plants, approximately 20%. Assuming the fractionation of  $^{14}\text{C}$  will be roughly twice that of  $^{13}\text{C}$  (since the mass difference between 12 and 14 is twice that between 12 and 13), the  $^{14}\text{C}$  contents of a tree and the  $\text{CO}_2$  which it is fixing through photosynthesis will differ by approximately 40 ‰, even though both  $\text{CO}_2$  and the tree are the same 'age'. To account for fractionation effects, the sample and standard are corrected to a measured value of the same sample, or if not measured, are corrected the value -25 per mil (as noted in parentheses). The standard oxalic acid is corrected in the same way, to -19 per mil.

Unlike the closed systems represented by intact macrofossils, such as seeds or pollen, bulk SOM is a heterogeneous reservoir with a variety of turnover times, to which carbon is continuously added (as new plant matter) and lost (as leached organic carbon or  $\text{CO}_2$ ). The radiocarbon content of SOM can not be interpreted as a 'date', but represents the average age of a carbon atom in this reservoir.

The breakdown of C into faster and slower-cycling pools may be determined by combining several approaches - see the articles in the reference list for more information (this is an evolving research field and no one approach is accepted as valid for all soils).

For soils that are accumulating organic matter, either in upper layers that are recovering from erosion, or in the total soil profile that has evolved since deposition of the loess deposit some 12,000 yr ago, we model the accumulation of carbon (where C is C inventory) as a time sequence described by inputs (I) and decomposition (k) according to the following equation:

$$dC/dt = I - kC \quad (1)$$

$$\text{and } C_t = I/k*(1 - \exp^{-k*t}) \quad (2)$$

where C is carbon mass in units of mass per area, t is time in years, I is input rate in mass per area per year, and k is a decomposition coefficient in units of time<sup>-1</sup>. This approach assumes that decomposition is proportional to total mass. " Time can be modeled over periods of years, decades, centuries or, as for incubations, fractional years.



## ***2.5 Particle-size analysis***

Samples were selected to characterize particle-size distribution of soils. Based on limited size of samples, not all soil horizons and not all sites were fully characterized for particle size. Two preparations, conventional dispersal and water-based dispersal, were used to characterize sand, silt and clay particles.

Conventional dispersal of soil samples is based on the principal that soil particles aggregate to form coarser particles and must be dispersed chemically or physically. Sodium hydroxide, sodium hexametaphosphate, and citrate-bicarbonate were used for dispersal Gee and Bauder (1986, p. 400-401), and samples were sieved (Gee and Bauder, p 401) and analysed by pipet (Gee and Bauder, p. 401-402) for determination of USDA sizes for sand (>.05mm or 50 micrometer), coarse silt (.02 to .05 mm or 20 to 50 micrometer), fine silt (.002 to .02 mm or 2 to 20 micrometer), and clay (<.002mm or <2 micrometer) fractions.

Water dispersal was an experiment on aggregation. Samples were shaken overnight in water, and subjected to the pipet methods (Gee and Bauder, p. 401-402). Sand plus silt in this procedure adds up to >100% because of errors in summing sands and partitioning weights.

## ***3. Data-set identification.***

The data presented represent sampling from the fall of 1996 to the fall of 1997. Four data sets are included for Mississippi sites:

Miss\_Site (site location and explanation)

Miss\_Field (field descriptions of soils)

Miss\_Soil (chemical,physical,isotopic data from soils)

Miss\_Isotope (<sup>14</sup>C of soil samples )

Miss\_Psize

The "Miss\_site" file includes site locations and explanations of site identification, reasons for site choices, and any further information that might help to revisit the site or to find a comparable site for other studies. In essence, the Miss\_Site files are considered to be the "mother of all soil files"; all other data files are coded in a way to tie in specifically to the Site file, which describe location, site conditions, dates, and other pertinent information about the excavation site where most soil samples of this study originate. In practicality, the MBCP-Upland Soils Database is structured as a set of tables in both Microsoft Access (.mdb) files and as tab-delimited ASCII (.rdb) files. Records in each of the four types of files (Miss\_Site, Miss\_Field,

Miss\_Soil, Miss\_Isotope, Miss\_Psize) form a unique file that relate on the fields PROFILE and DEPTH.

The "Miss\_Field" files include those properties described by USDA for field characterization and classification. Soil texture (relative abundance of sand, silt, clay), color (Munsell soil color charts), structure (aggregation), root abundance, and consistence are typically included in field descriptions and provide information on the relative degree of weathering, permeability, and erosion based on comparisons among sites or to published soil descriptions (see for example USDA, 1987).

The "Miss\_Soil" file includes solid phase analyses needed to determine carbon and nitrogen inventory in soils (bulk density, %C %N, C/N ratio in organic matter) and isotopic measurements used for determining decomposition or turnover times (<sup>13</sup>C and <sup>15</sup>N measurements of soil organic matter). Soil moisture data (field moisture content good only for the day of collection; %moisture in air-dry samples) are also included.

The "Miss\_Isotope" file includes radiocarbon analyses on solid and gas-phase samples.

The "Miss\_Psize" file includes particle size analysis of size fractions

### ***3.1 Labeling schemes***

Solid phase soil samples are collected in depth increments (soil horizons) in a vertical array below a primary site location (for example profile number 1 where a core was sampled) within a general site (for example at the upper, erosional site of a hillslope). Gas-phase sampling and in-situ measurements are collected within a hillslope position (upper, erosional) but at separate locations. Whereas soil pits and cores are destructive, gas sampling may re-occupy the same primary site several times over the course of a year. The overall strategy for data collection is replicate measurements at each upper (erosional) and lower (depositional) hillslope positions in each cultivated and uncultivated sites. For Mississippi, the cultivated sites are located within a USDA-ARS research farm, the Nelson Farm, in watershed 2 (Dabney and others, 1997). Samples and observations from that site are coded "NF" followed by a letter designating the overall sample type ("P" for profile; "I" for incubation; "F" for fractionation; "cg" for chamber gas) followed by the slope position "U" (upper) or "L" (lower) , followed by increasing numbers for consecutive samplings. For example NFPU1 and NFPU2, for the first two profiles sampled at the upper hillslope position of the slope at Nelson Farm.

For soil samples, a decimal is used to designate depth increments at the primary site with the basal horizon depth (in cm) listed to the right of the decimal point, for

example NFPU1.20 for 20 cm basal depth or NFPU1.200 for 200 cm basal depth. Lower case letters are used to indicate the intended purpose of the sample and its potential for other uses (a,b,m,f,i for analytical, bulk density, moisture content, fractionation, incubation samples respectively), for example NFPU1.20a,b,m.

### ***3.2 Data set descriptions***

Five categories of data sets are presented, including site and location data (Miss\_Site), field data (Miss\_Field), soil analytical data (Miss\_Soil), soil isotopic data (Miss\_Isotope) and particle size data (Miss\_Psize). Column headings and units for each of these data sets are described herein:

#### ***3.21 Miss\_Site files contain the following information in text format***

Site location, slope description, landuse notes, date of sampling, purpose of sampling, field personnel.

#### ***3.22 Miss\_Field files contain the following information in column format***

PROFILE traces to the \_Site file as discussed above.

DEPTH indicates depth in cm of sampling increment.

DESCRIBE includes a general description of the sampled horizon (A horizon, oxidized B horizon, etc.) (see Soil Survey Staff, 1981).

STRUCTUR includes soil structure following conventions of Soil Survey Staff, 1981.

TEXTURE includes soil texture class following conventions of Soil Survey Staff, 1981.

MCOLOR includes moist soil color following conventions of Soil Survey Staff, 1981.

MCONSIS includes moist consistence following conventions of Soil Survey Staff, 1981.

WCONSIS includes wet consistence following conventions of Soil Survey Staff, 1981.

ROOTS includes root abundance following conventions of Soil Survey Staff, 1981.

***3.23 Miss\_Soil files contain analytical data on the solid phase of soil samples, using numbers assigned in the \_Site file and labling scheme described above.***

PROFILE refers to the profile number in the Miss\_Site file for information regarding location and sampling conditions.

SAMPLE sample identification keys back to \_Site files.

DEPTH is the depth in cm of the base of the soil horizon sample; the top depth is generally the basal depth of the superjacent soil horizon.

THICKNES is horizon thickness in cm and is used to track bulk density and is equal to basal depth minus top depth of sampling increment.

VOLUMEM volumetric moisture content ( $\text{cm}^3$  of water per  $\text{cm}^3$  of soil volume) .

DATEFM is the date of field moisture determination.

AIRDRYM air-dry soil moisture is reported as gravimetric moisture content (grams water per gram oven-dry soil) and can be used to convert other data to the oven-dry basis :  $WW = DW/(1+DW)$  and

$DW = WW/(1-WW)$ , where DW is water content on the dry-weight basis, and WW is water content on the wet-weight basis (Gardner, 1986). Using this relationship, %C or Bulk Density, which are reported per g air-dry soil, can be converted to the more conventional per g oven-dry basis:

$\%C(\text{air dry basis}) * WW/(1-WW) = \%C(\text{oven-dry basis})$ .

BDENSITY bulk density is the grams of solid, air-dry soil material per cubic centimeter of volume, which is measured as the air-dry weight of a known volume of soil.

TOTALC1 total carbon content is expressed as gravimetric percent on an air-dry soil basis. Samples were analyzed on the <2mm soil on a LECO combustion analyser. Inorganic C was content determined for selected samples and was not present in Nelson Farm or Goodwin Creek soils therefore total C is considered organic C.

TOTALC2 total carbon content is expressed as gravimetric percent on an air-dry soil basis. Samples were analyzed on the <2mm soil fractions (once homogenized and ground to <60 mesh) on a Fisons NA1500 elemental analyser. Inorganic C was content determined for selected samples and was not present in Nelson Farm or Goodwin Creek soils therefore total C is considered organic C.

TOTALN total N content is expressed as gravimetric percents on an air-dry soil basis. Samples were analyzed on the <2mm soil fractions (once homogenized and

ground to <60 mesh) on a Fisons NA1500 elemental analyser. We report the C/N ratio.

CNRATIO the C/N ratio is calculated from TOTALC2/TOTALN.

SOILC13 stable isotope  $^{13}\text{C}$  content of the <2mm (bulk) soil is presented in Delta notation SOILN15.

SOILN15 stable isotope  $^{15}\text{N}$  content of the <2mm (bulk) soil is presented in Delta notation.

CDENSITY carbon density (in  $\text{gC}/\text{cm}^3$ ) is calculated from  $(\text{TOTALC2}/100)*\text{BDENSITY}$ .

CSTORAGE carbon storage (in  $\text{gC}/\text{cm}^3$ ) is calculated from  $(\text{TOTALC2}/100)*\text{THICKNES}*\text{BDENSITY}$ .

### ***3.24 Miss\_Isotope files contain the $^{14}\text{C}$ analyses for solid and chamber gas samples.***

PROFILE profile number keys back to \_Site files.

SAMPLE sample identification keys back to \_Site files.

DEPTH indicates depth of sample;NA not applicable usually refers to chamber samples collected at the soil surface.

LABID Laboratory identification numbers are University of California Irvine and Lawrence Livermore Lab numbers.

TYPE sample type analysed.

DEL13C values for Delta $^{13}\text{C}$ .

DEL14C values for Delta $^{14}\text{C}$ .

LABSD values for error in radiocarbon counting.

### ***3.25 Miss\_Psize files contain the following information in column format***

SampleID sample identification keys back to \_Site files.

USDASAND standard dispersant, percent by weight of particles greater than 50 micrometer in size.

USDACOSI standard dispersant, percent by weight of particles between 20 and 50 micrometers in size.

USDAFISI standard dispersant, percent by weight of particles between 2 and 20 micrometers in size.

USDACL standard dispersant, percent by weight of particles less than 2 micrometers in size.

WSAND water dispersant, percent by weight of particles greater than 50 micrometer in size.

WCOSI water dispersant, percent by weight of particles between 20 and 50 micrometers in size.

WFISI water dispersant, percent by weight of particles between 2 and 20 micrometers in size.

WCLAY Water dispersant, percent by weight of particles less than 2 micrometers in size.

#### ***4. Application of the data set***

As stated earlier, the overall intention of the data set is to gain insights into and begin to model the interaction between the carbon cycle and erosion-sedimentation cycle. The data sets are best suited to address C and sedimentation processes on small hillslopes and exiting small hillslopes through runoff.

Models developed for  $^{14}\text{C}$  utilize Equations 1 and 2 for carbon mass balance along with a decay constant for radiocarbon, .0001245. Separate but interactive models are written for 3 pools of soil organic matter that decompose at fast, slow, and extremely slow ("passive") rates. We chose three pools for this stage of research based on various other soil carbon studies (Trumbore, 1994; Parton, 1987), but recognize that soil organic matter is highly dynamic, heterogeneous, and potentially a continuum of sizes and turnover times of carbon pools.

For modeling a sampling site, three pools of carbon are calculated independently according to inputs by NPP (Iprod in Table 2) and by deposition (Idep), losses to decomposition (Ldecomp) by the turnover time of the pool, and losses to erosion (Leros). The three pools are combined for amount and radiocarbon content of the soil gas (sum of  $k1C1$ ,  $k2C2$ , and  $k3C3$  for amount and  $FM1*k1C1...$  for radiocarbon content, where  $FM1$  is Fraction Modern of the  $SOM1$  pool). The erosion term ( $Ler$ ) is modeled separately for each  $SOM$  pool ( $Ler1$ ) by multiplying the sediment loss times the  $SOM$  pools distribution of the topsoil.

Turnover times can be constrained somewhat by the  $^{14}\text{C}$  content of soil gas and bulk soil in models described above and in comparison to data. The turnover time of  $SOM1$  is best constrained by  $^{14}\text{C}$  of soil gas, because soil gas is dominated by respiration of this fast pool. However, the size of the  $SOM1$  pool must be known or estimated in order for the  $^{14}\text{CO}_2$  to be used for turnover times. In one approach, we used the CENTURY ecosystem model (Parton, 1987) to estimate pool sizes. The sizes and turnover times of  $SOM$  pools can also be estimated from incubation fluxes (Fries and others, 1997; see Collins and others, 1997).

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## **6. Investigators**

For information on site location, site selection, temperature and moisture data, and chamber data, please contact:

Tom Huntington

U.S. Geological Survey

3039 Amwiler Rd. Suite 130

Atlanta, GA 30360-2824

770-903-9147

thunting@usgs.gov

For information on sampling strategy, sample submittal, soil characterization and description, analytical data, and isotopic data, and modeling please contact:

Jennifer Harden

U.S. Geological Survey

345 Middlefield Rd. MS 962

Menlo Park, CA 94025

650-329-4949

jharden@usgs.gov

For information on soil analysis, analytical data, and sample tracking, please contact:

Terry Fries

U.S. Geological Survey

345 Middlefield Rd. MS 962

Menlo Park, CA 94025

650-329-5281

tfries@usgs.gov

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