Video Article

Measurements of Soil Carbon by Neutron-Gamma Analysis in Static and Scanning Modes

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Abstract

The herein described application of the inelastic neutron scattering (INS) method for soil carbon analysis is based on the registration and analysis of gamma rays created when neutrons interact with soil elements. The main parts of the INS system are a pulsed neutron generator, NaI(Tl) gamma detectors, split electronics to separate gamma spectra due to INS and thermo-neutron capture (TNC) processes, and software for gamma spectra acquisition and data processing. This method has several advantages over other methods in that it is a non-destructive *in situ* method that measures the average carbon content in large soil volumes, is negligibly impacted by local sharp changes in soil carbon, and can be used in stationary or scanning modes. The result of the INS method is the carbon content from a site with a footprint of ~2.5 - 3 m^2 in the stationary regime, or the average carbon content of the traversed area in the scanning regime. The measurement range of the current INS system is >1.5 carbon weight % (standard deviation ± 0.3 w%) in the upper 10 cm soil layer for a 1 hmeasurement.

Video Link

The video component of this article can be found at https://www.jove.com/video/56270/

Introduction

Knowledge of soil carbon content is required for optimization of soil productivity and profitability, understanding the impact of agricultural land use practices on soil resources, and evaluating strategies for carbon sequestration[1,2,3,4]. Soil carbon is a universal indicator of soil quality[5]. Several methods have been developed for soil carbon measurements. Dry combustion (DC) has been the most widely used method for years[6]; this method is based on field sample collection and laboratory processing and measurement that is destructive, labor intensive, and time consuming. Two newer methods are laser-induced breakdown spectroscopy, and near and mid infrared spectroscopy[7]. These methods are also destructive and only analyze the very near-surface soil layer (0.1 - 1 cm soil depth). In addition, these methods only yield point measurements of carbon content for small sample volumes (~60 cm^3 for DC method, and 0.01-10 cm^3 for infrared spectroscopy methods). Such point measurements make it difficult to extrapolate results to field or landscape scales. Since these methods are destructive, recurring measurements are also impossible.

Previous researchers at Brookhaven National Laboratory suggested applying neutron technology for soil carbon analysis (INS method)[7,8,9]. This initial effort developed the theory and practice of using neutron gamma analysis for soil carbon measurement. Starting in 2013, this effort was continued at the USDA-ARS National Soil Dynamics Laboratory (NSDL). The expansion of this technological application over the last 10 years is due to two main factors: the availability of relatively inexpensive commercial neutron generators, gamma detectors, and corresponding electronics with software; and state of the art neutron-nuclei interaction reference databases. This method has several advantages over others. An INS system, placed on a platform, could be maneuvered over any type of field that requires measurement. This non-destructive *in-situ* method can analyze large soils volumes (~300 kg) that can be interpolated to a whole agricultural field using just a few measurements. This INS system is also capable of operating in a scanning mode that determines the average carbon content of an area based on scanning over a predetermine grid of the field or landscape.

Protocol

1. Construction of the INS system

1. Use the general INS system geometry shown in **Figure 1**.

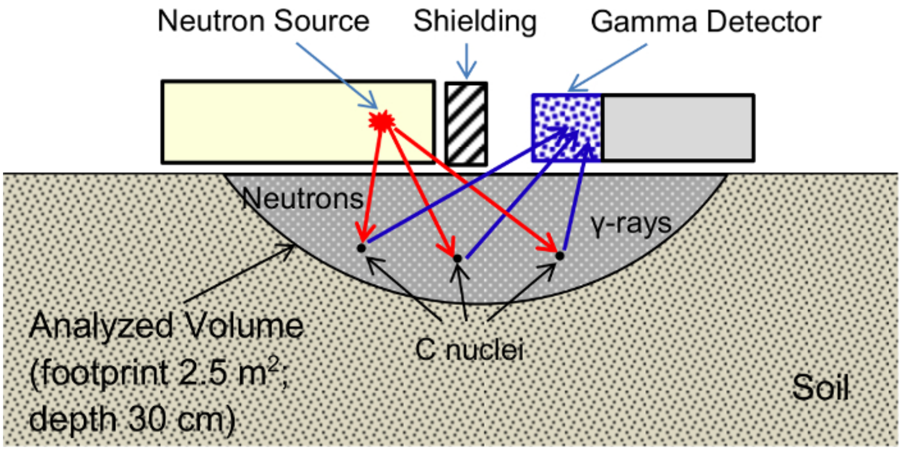

Figure 1. **INS System Geometry.** Please click here to view a larger version of this figure.

2. Use the INS system design shown in **Figure 2**.[10]

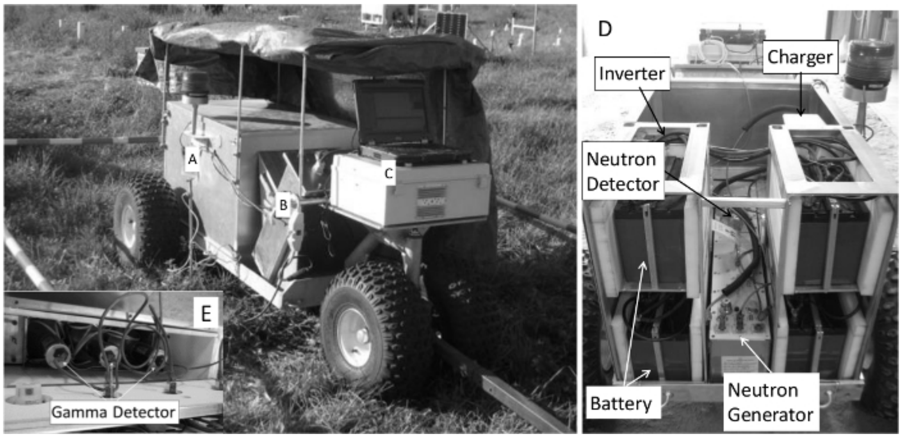

Figure 2. **Overview of the INS System.**
A) first block contains neutron generator, neutron detector, and power system; **B)** second block contains three NaI (Tl) detectors; **C)** third block contains equipment for system operation; **D)** general view of the first block showing individual components; and **E)** close up view of the gamma detectors.[10] Please click here to view a larger version of this figure.

3. Use three blocks in the INS system (see Appendix).
    1. For the first block (A), use a neutron generator (NG) and power system (**Figure 2A** and **2D**). Pulsed neutron output of this generator will be 10^7 - 10^8 n/s with neutron energy of 14 MeV. The power system will consist of four batteries (12 V, 105 Ah), a DC-AC Inverter, and a charger. This block will also contain iron (10 cm x 20 cm x 30 cm) and boric acid (5 cm x 20 cm x 30 cm) shielding to protect the gamma detector from neutron irradiation.
    NOTE: A neutron detector is also included in this block for checking that the NG is functioning properly.
    2. For the second block (B), use gamma-ray measurement equipment (**Figure 2B** and **2E**). This block will contain three 12.7 cm x 12.7 cm x 15.2 cm scintillation NaI(Tl) detectors with corresponding electronics. The exterior size of the detectors with electronics will measure 15.2 cm x 15.2 cm x 46 cm.
    3. For the third block (C), use a laptop computer that controls the neutron generator (with DNC software), detectors, and data acquisition system (**Figure 2C**).

2. Caution and Personal Requirements

1. Have each user of the INS system pass radiological training.
2. Ensure that each person operating the NG carries a radiation monitoring badge. During measurements, the restricted area boundary (>20 µSv/h) around the NG will have the radiation symbol with the words "CAUTION, RADIATION AREA." All edges of the restricted area will be no less than 4 m from the NG.
3. In an emergency, immediately push the "Emergency Interrupt" button on the NG, remove the key from the NG, and unplug the NG from the power source.

3. Preparation of the INS system for Measurement

1. Check the power system. The power level indicator on the charger will be green, or more than 3 red lamps must illuminate. If not, connect the charger to a power outlet and wait until batteries become fully charged (green lamp will illuminate) or until an acceptable power level is reached (≥3 red lamps will illuminate).
2. Turn on the inverter (green lamp illuminates) and laptop.
3. Run the data acquisition program on the laptop to operate the gamma detectors and check the required parameters for each detector. The values of these parameters will be defined and recorded previously at INS system testing.
    1. Place a Cs-137 control source (any type) within 5 - 15 cm from the detectors.
    2. Start spectra acquisition for 1 - 3 min; check the centroids of the 662 keV Cs-137 peak for all detectors. They must be at the same channel. If not, use the Energy Coefficient Scale of thedata acquisition program by changing the value to adjust the 662 keV peak centroids.

4. Turn on the NG by using the special key. The indicator lamp on the NG will illuminate green and yellow.

4. Calibration of the INS System

1. Prepare 4 pits sized 1.5 m x 1.5 m x 0.6 m with homogeneous sand-carbon mixtures (**Figure 3**). Carbon contents is 0, 2.5, 5 and 10 w%.
NOTE: A concrete mixer is used to make synthetic soil composed of construction sand and coconut shell (100% carbon content, average granular diameter < 0.5 mm). Homogeneity of these mixtures is determined visually.

Figure 3. **View of Pit with Sand and Pit with 10 Cw% Sand-carbon Mixture**. Please click here to view a larger version of this figure.

2. Take measurements over the pits using the following steps.
    1. Position the INS system over the pit manually or by towing with a suitable vehicle. Position the INS system such that the projection of the neutron source is centered on the pit.
    2. Run the DNC software on the laptop that operates the NG generator. In the Faults column on the right side of the DNC program screen, all lamps will illuminate green; if not, click the Clear button. Insert the following parameters: for Pulse Parameters - frequency 5 kHz, duty cycle 25%, delay 0 µs, extension 2 µs; for Beam - high voltage 50 kV, beam current 50 µA (note that these parameters can be different depending on the particular INS system setup and task).
        1. Activate the switch on the DNC program screen and wait for the NG to enter the working regime where the High voltage and Beam current will come to stable values corresponding to the entered values; Reservoir current also will come to a stable value.

    3. Run the data acquisition software on the laptop to operate the gamma detectors. Start spectra acquisition by running the data acquisition program for 1 h. The two spectra acquisition processes (INS & TNC and TNC) will appear on the screen.
    4. After 1 h, stop the spectra acquisition and save spectra to the hard drive (File | Save MCA Data | choose the folder and enter the file name.
    NOTE: There will be two saved spectra (TNC and INS) with filename extensions .mca and _gated.mca, respectively).
    5. Select second detector (click the arrow in the top left corner) and save the spectra for this detector. Do the same for third detector.
    6. Click File | Exit to close the software.
    7. Turn off the DNC software by turning off the switch on the DNC program screen.
    8. Repeat steps 4.2.1 - 4.2.7 for the other pits.
    9. Turn off the NG by using the special key. The indicator lamp on the NG will dim.

3. Determine the INS system background spectra by elevating the whole INS system to a distance greater than 4 m above the ground surface and away from any large objects, and repeat data acquisition steps 4.2.2 - 4.2.9.
4. Data processing
    1. Use a spreadsheet program to open data files saved in step 4.2.4. Find values for output and input count rates (OCR and ICR) and real time (RT) in rows 28, 27, and 30, respectively.
    2. Calculate the life time (LT) for INS & TNC and TNC spectra for all measurements as
       $LT_i = OCR_i/ICR_i \cdot RT_i$ (1),
       where OCR_i and ICR_i are the output and input count rates for the i-th measurement and RT_i is real time of the i-th measurement.
    3. Calculate the gamma spectra in counts per second (cps) by dividing the spectra (rows 33-2080 in the spreadsheet) by the corresponding LT.
    4. Calculate the net INS spectra from the corresponding measurements for each pit as
       Net INS Spectrum = $(INS\&TNC - TNC)_{Pit} - (INS\&TNC - TNC)_{Bkg}$ (2)
    5. Find the gamma peaks 1.78 MeV (^{28}Si) and 4.44 MeV (^{12}C) in the Net INS spectrum for each pit, and calculate the peak areas (4.44 MeV C peak area, 1.78 MeV Si peak area) using IGOR software.
        1. Open the software by double clicking the icon. Insert first Net INS Spectrum into the Table.
        2. Click Windows | New Graph | From Target | "FileName" | Do It. The Spectrum appears in the Graph Window. Click Graph | Show Info. The windows with A and B markers appears under the graph window.
        3. Place mouse pointer on sign A, push the left mouse button, and drag the cursor to the spectrum on left side of the 1.78 MeV peak. Place mouse pointer on the sign B, push the left mouse button, and drag the cursor to the spectrum on right side of the 1.78 MeV peak.
        4. Click Analysis | Multi-peak Fit | Start New Multi-peak Fit | From Target | Continue. In the pop-up window marked Use Graph Cursor | Baseline Linear| Auto-locate Peaks Now | Do it | Peak Results. The area of the peak appears in the pop-up window.
        5. Repeat the same operations for 4.44 MeV peak.
        6. Repeat all previous operations with the remaining Net INS Spectra.

    6. Find the Net carbon peak areas for each pit by the equation
       Net C peak area$_i$ = 4.44 MeV C peak area$_i$- 0.058 · 1.78 MeV Si peak area$_i$ (3)
    7. Build the calibration line for the INS system as a direct proportional dependence of the Net carbon peak area vs. carbon concentration expressed in weight percent.
        1. Open new Table in IGOR software: click Window | New Table. Enter pit carbon concentration values in the first column, and the corresponding Net C peak area in the second column.
        2. Plot the Net C peak area vs pit carbon concentration: click Windows | New Graph. Choose Net C peak area as YWave, and carbon concentrations as XWave. Click Do it. The points appear on the Graph.
        3. Build the calibration line: Click Analysis | Curve Fitting | Function - line | From Target | Do it. The calibration line and the calibration coefficient (k) will appear in the window.

5. Conducting Field Soil Measurements in Static Mode

1. Prepare the INS system for measurement according to step 3.
2. Place the system over the site requiring soil carbon content analysis manually or by towing using suitable vehicle. Position the INS system such that the projection of the neutron source is centered over the site being measured.
3. Implement actions following steps 4.2.2 - 4.2.9 and 4.4.1 - 4.4.6 for determining Net C peak areas for the study sites.
4. Calculate the carbon concentration in weight % using the calibration coefficient as

$$Carbon\ concentration = \frac{Net\ C\ peak\ area}{k} \qquad (4)$$

6. Conducting Field Soil Measurements in the Scanning Mode

1. Estimate the path that the INS system will travel over the field while accounting for travel speed (≤ 5 km/h), field size, INS system footprint (radius ~1 m), and measurement time (1 h) such that the moving trajectory eventually covers the whole field area. For convenience, place flags at turn points along the field perimeter.
2. Prepare the INS system for measurement according to step 3.
3. Implement actions following steps 4.2.2 - 4.2.3.
4. Follow the predetermined travel path for 1 h.
5. Implement actions following steps 4.2.4 - 4.2.9 and 4.4.1 - 4.4.6 for determining Net C peak areas for the studied field.
6. Calculate the carbon concentration in weight % using the calibration coefficient by equation 4.

Representative Results

Soil INS & TNC and TNC gamma spectra

A general view of the measured soil gamma spectra is shown in **Figure 4**. The spectra consist of a set of peaks on a continuous background. The main peaks of interest have centroids at 4.44 MeV and 1.78 MeV in the INS & TNC spectra. The second peak can be attributed to silicon nuclei contained in soil, and the first peak is an overlapping peak from carbon and silicon nuclei. The procedure for net carbon peak area extraction from these spectra is described above. This procedure should be used in all cases for determining the net carbon peak area due solely to carbon nuclei.[11]

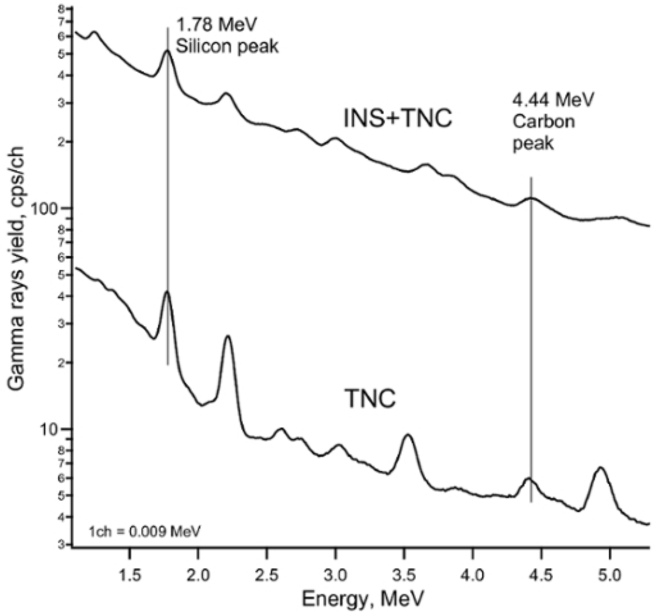

Figure 4. **A Typical Gamma Spectra for Soil Measured by the INS System.** Please click here to view a larger version of this figure.

INS system background measurements

Net INS spectra measured at various system elevation heights above the ground surface are shown in **Figure 5**.[11] Dependencies of the peak areas with centroids at 1.78 MeV, 4.44 MeV, and 6.13 MeV (oxygen peak) with height are illustrated in **Figure 6**. As shown in this Figure, the spectra no longer change at heights greater than 4 m above the ground surface. Accordingly, the spectra at heights greater than 4 m can be attributed to gamma spectra that appear due to the interaction of neutrons with system construction materials. We used one of these spectra (at H=6 m) as the system background spectrum in our data processing.

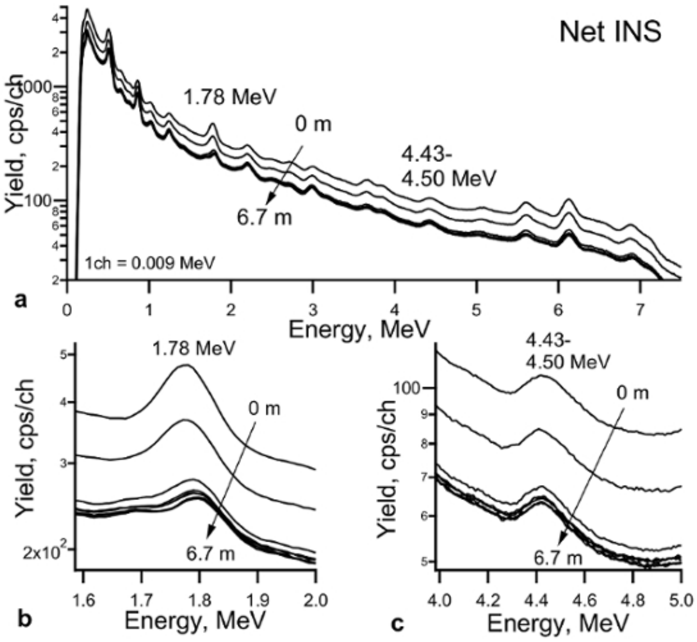

Figure 5. **a)** Net-INS spectra at different INS system heights above the ground; **b)** Fragment of the net-INS spectra around 1.78 MeV; and **c)** Fragment of the net-INS spectra around 4.44 MeV. Arrow designates increasing height.[11] Please click here to view a larger version of this figure.

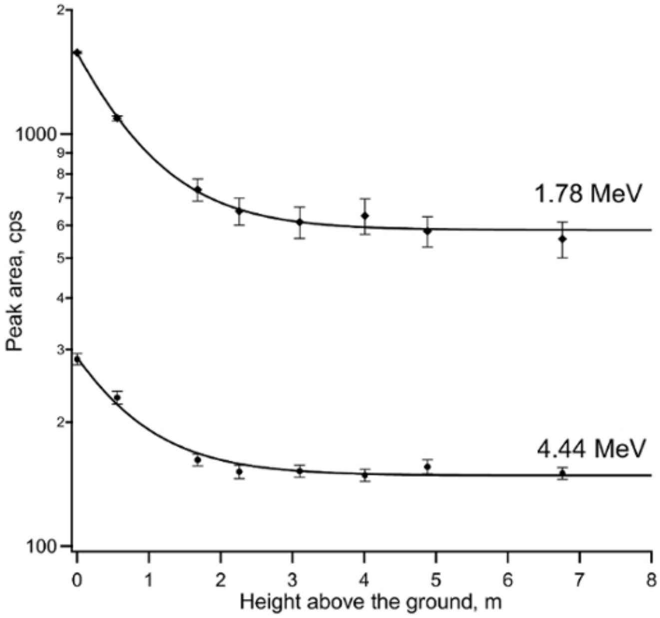

Figure 6. **Dependencies of Peaks Areas with Centroids at 1.78, and 4.44 MeV in the Net-INS Spectra for INS System with Changing Heights Above the Ground**. Please click here to view a larger version of this figure.

Calibration

The net INS spectra generated during INS system calibration are shown in **Figure 7a**.[11] Fragments of the net INS spectra near the 1.78 MeV and 4.44 MeV peaks are shown on a larger scale in **Figures 7b** and **7c**, respectively. As can be seen, the peak with a centroid of 4.44 MeV increases with increasing carbon content in the pit. At the same time, the peak with a centroid at 1.78 MeV slightly decreases as carbon in the pit increases. The dependency of the net carbon peak area (calculated from these spectra) with carbon content in pits (expressed in weight %) is shown in **Figure 8**.[11] As can be seen, this can be represented by a direct proportional dependency passing through the origin (0, 0 point) within experimental error limits. This dependency was used to calibrate further measurements.

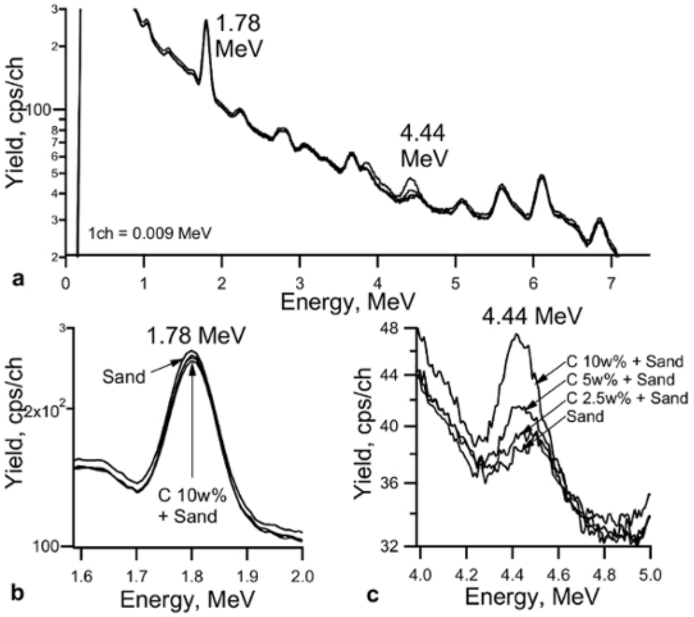

Figure 7. **a)** Net INS spectra for pits with sand-carbon mixtures at 0, 2.5, 5, and 10 carbon w% (uniform mixture); **b)** Fragment of the net INS spectra around 1.78 MeV; **c)** Fragment of the net INS around 4.44 MeV.[11] Please click here to view a larger version of this figure.

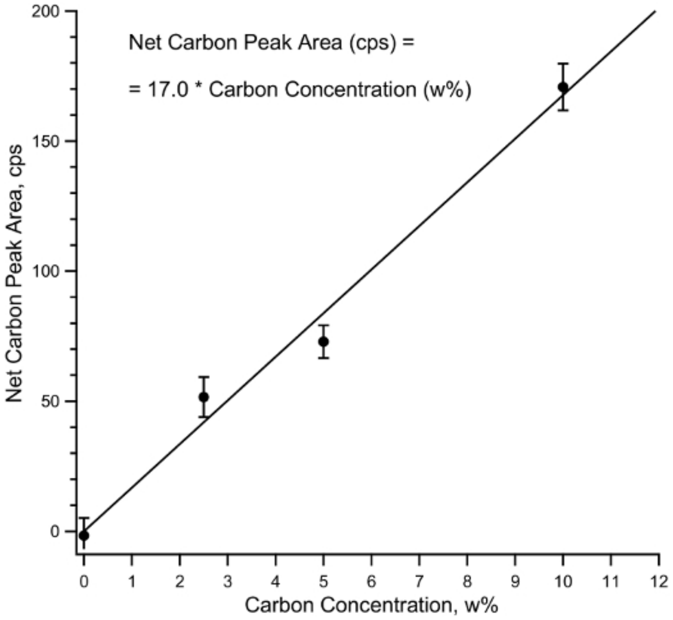

Figure 8. **Dependence of the Net Carbon Peak Area with Carbon Concentration in Pits (Points with Error Bars), and INS System Calibration Line (Solid Line).**[11] Please click here to view a larger version of this figure.

Field measurements of carbon content in static mode

Carbon content measurements in static mode were conducted at several field sites. Results from the Alabama Agricultural Experiment Station Piedmont Research Unit, Camp Hill, AL (110 m x 30 m) are presented in **Table 1**. Field measurements were conducted at the intersections of a 3 by 5 grid with equal distances between gridlines (total 15 sites). As can be seen from the table, the carbon content for individual intersection points varied between 1.4 to 3.1 w% with the standard deviation of all measurements being ~0.3 w%. For comparison, destructive soil samples were also taken at each location for determining soil carbon content using the standard DC method. These data are also presented in **Table 1**. Comparison of the two data sets showed good agreement between both methods for each location and for the average value over the whole field.

Location	Site #	INS measurements			Dry Combustion measurements		
		Carbon, w%	STD, w%	Plot Average ±STD, w%	Carbon, w%	STD, w%	Plot Average ±STD, w%
Camp Hill	OF1	2.2	0.29	2.23±0.45	2.85	0.25	2.25±0.51
	OF2	2.51	0.29		2.54	0.31	
	OF3	1.76	0.22		1.91	0.13	
	OF4	1.88	0.23		2.99	0.94	
	OF5	2.82	0.25		3.03	0.37	
	OF6	2.15	0.21		1.99	0.26	
	OF7	2.77	0.32		1.92	0.41	
	OF8	2.52	0.25		2.44	0.15	
	OF9	2.06	0.26		1.79	0.27	
	OF10	2.17	0.27		2.25	0.45	
	OF11	2.39	0.22		2.23	0.3	
	OF12	3.11	0.31		2.91	0.47	
	OF13	1.44	0.25		1.49	0.42	
	OF14	1.93	0.29		1.8	0.19	
	OF15	1.86	0.27		1.67	0.25	

Table 1. Average Weight Percent in the Upper Soil Layer by Dry Combustion and INS Methods.

It is interesting to compare the carbon distribution maps of the field based on the INS and DC methods (**Figure 9** and **10**). Both maps look very similar, but it should be noted that 2 days were spent on INS mapping, while ~2 months were required to process samples to create the DC map.

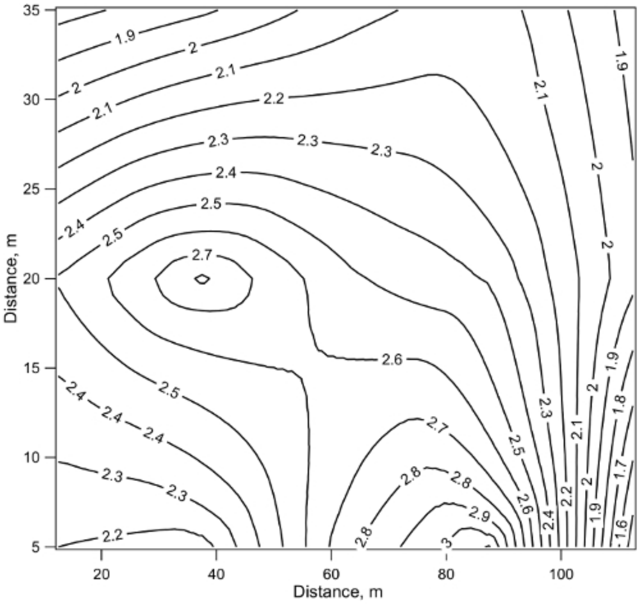

Figure 9. **Carbon Distribution Map of the Camp Hill Field Based on the INS Method.** Please click here to view a larger version of this figure.

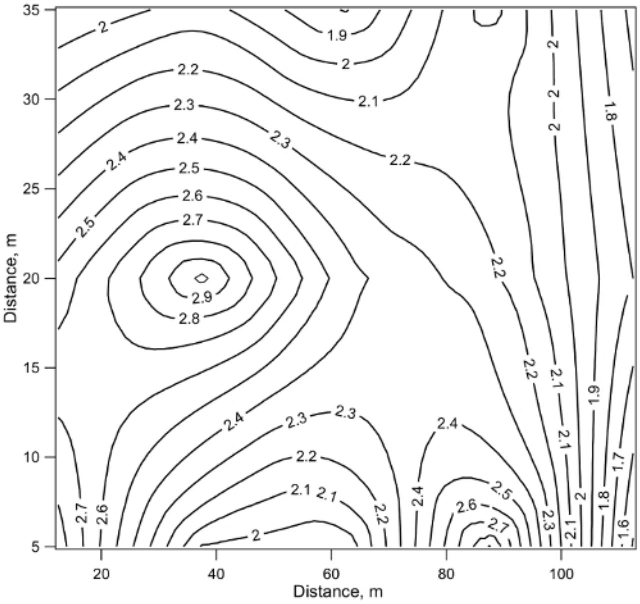

Figure 10. **Carbon Distribution Map of the Camp Hill Field Based on the DC Method.** Please click here to view a larger version of this figure.

Field measurements of carbon content in scanning mode

Soil scientists are often interested in determining carbon content for large areas (*e.g.*, 100 m x 100 m). Instead of determining carbon in locations 10 m apart (requiring 1 h per measurement using INS), it is possible to determine the average carbon content for a 100 m x 100 m field using the INS scanning mode. In scanning mode, it is possible to take INS measurements while passing over the entire field. This scanning measurement can be conducted in the same amount of time needed to measure a single location in static mode (1 h). The proof and principle of the INS scanning mode are demonstrated in this article.

It should be noted that the first attempt for measuring carbon in the scanning mode was less than satisfactory. The acquired scanning spectra were visibly different from the INS & TNC and TNC static mode spectra; the peaks of interest were broader and shorter with peak areas being much less than observed in the static mode. Investigations determined that this distortion was due to the influence of the Earth's magnetic field on the gamma detector's photomultiplier[12]. To resolve this problem, a magnetic screen (mu-metal) was used to shield the gamma-detector. Testing showed that the gamma spectra of a Co-60 control source was nearly identical regardless of the orientation of the screened gamma detector (vertical, horizontal, inclined), while peak centroids and peaks widths changed depending on the orientation of the unscreened detector. These result demonstrated that the effect of Earth's magnetic field on the photomultiplier can be suppressed by using a magnetic screen. Magnetic screening eliminated peak broadening and produced a scanning gamma spectra that looked very similar to the static mode spectra.

To compare static and scanning modes, static measurements of carbon content were performed (1 h each) at 5 random locations within a 15 m x 45 m field and measurement in scanning mode (1 h total) were performed on the same field which had a fairly uniform carbon content. A map of the field showing individual measurement locations and the scanning path is illustrated in **Figure 11**. The net INS spectra of the 5 static mode locations and that of the scanning mode are shown in **Figure 12**. As shown in the **Figure 12**, the scanning mode spectrum looks similar to the static mode spectra and falls in the mid-range of all static spectra.

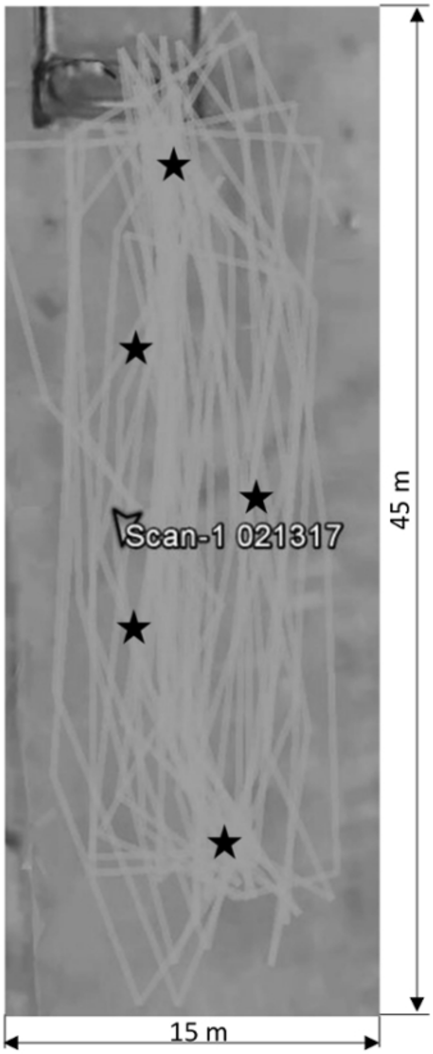

Figure 11. Map of the Field Showing Static Measurement Locations (stars) and the Scanning Path (Lines). Please click here to view a larger version of this figure.

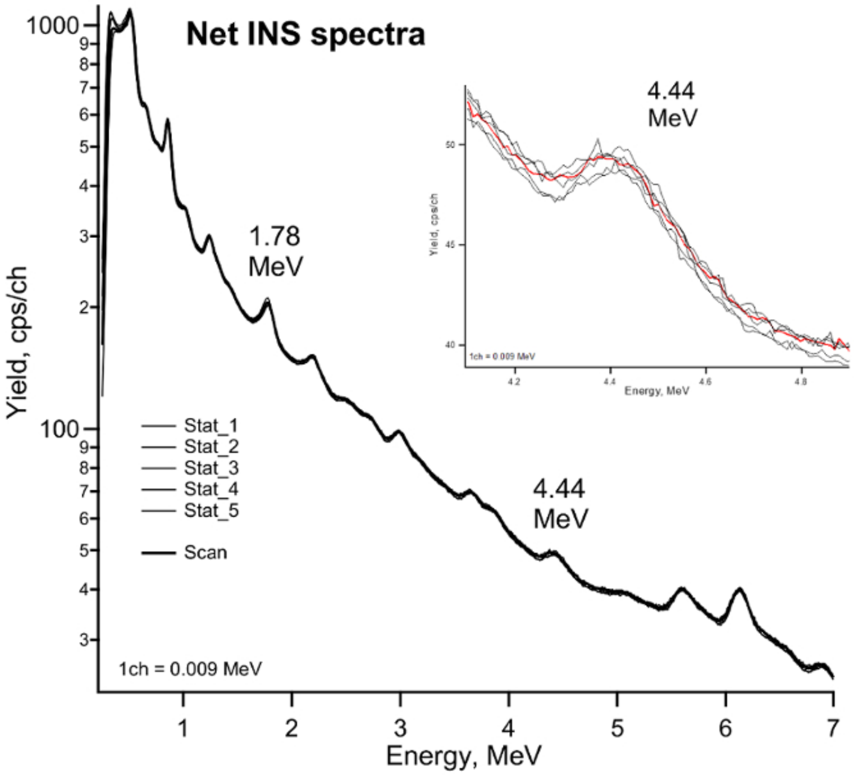

Figure 12. Net INS spectra for static and scanning modes; inset is a fragment of the net INS spectra around 4.44 MeV. Please click here to view a larger version of this figure.

Results of the net carbon peak area calculations are shown in **Table 2**. As can be seen from the presented data, the value of the net carbon peak area measured in scanning mode agrees with the average static mode value within the limits of experimental error. These results prove that INS scanning mode measurements can be used to define the average carbon content in a field. It is important to note that 5 h were spent determining average carbon content in static mode, whereas only 1 h was required in scanning mode.

Mode	Site #	Net Carbon Peak Area, cps	STD, cps	Field Average ±STD, cps
Static	1	64.8	3.9	63.3±3.8
	2	58.1	3.5	
	3	65.4	3.4	
	4	68.9	4.1	
	5	59.4	4.1	
Scanning	over field	64.4	3.3	64.4±3.3

Table 2. Net Carbon Peak Area for Static and Scanning Modes.

Discussion

Building on the foundation established by previous researchers, the NSDL staff addressed questions critical to the practical and successful use of this technology in real world field settings. Initially, NSDL researchers demonstrated the necessity to account for the INS system background signal when determining net carbon peak areas.[11] Another effort showed that the net carbon peak area characterizes the average carbon weight percent in the upper 10 cm soil layer (regardless of carbon depth distribution shape) by direct proportional dependency. In addition, equipment required for INS system calibration (*i.e.*, 1.5 m x 1.5 m x 0.6 m pits with different sand-carbon mixtures) was constructed and calibration procedures necessary for real world applications were developed and performed. The resultant calibration line renders it possible to determine soil carbon content from measured net carbon peak area. While NSDL researchers have incorporated many INS system design improvements, the recent addition of magnetic field shielding of gamma detectors allows for the practical use of the INS system scanning mode for large scale investigations of soil carbon.

Experience in applying the INS method for soil carbon analysis revealed several critical protocol steps. To obtain correct measurement results, it is critical to carefully check and adjust detector parameters using reference sources; this is very important for system stability and reproducing measurement results. The system background and calibration measurements are also critical steps for accurate determination of soil carbon content. Note that the detector parameters should be the same for both system background and calibration measurements. It is expedient to conduct calibration measurements (pits and system background) for several hours to increase accuracy of calibration coefficients. Installing magnetic screens on the detectors is critical for accurate measurement in the scanning mode since unscreened detectors produce very large errors due to the influence of the earth's magnetic field. In addition, magnetic screening improves results in the static mode.

The significance of using the INS method versus the "gold standard" DC method was demonstrated during field mapping. The speed of defining the carbon content by the INS method was ~30 times greater than the DC method. Other advantages of the INS method were discussed in the Introduction section.

Despite the demonstrated agreement between the INS and DC ("gold standard") methods, the current modification of the INS technique has one main limitation which is the minimal detectible level (1.5 w%). Since soil carbon content can be less than this, future efforts will concentrate on improving the sensitivity of the INS system by increasing the number of gamma detectors and optimizing the overall system design or by applying target neutron methods.[13]

Despite this limitation, the current modification of the INS system can be recommended for soil carbon determination of individual locations and for carbon distribution mapping of field terrains. Possible future work using the INS method may explore measuring other soil elements such as nitrogen, iron, and hydrogen.

Disclosures

The authors have nothing to disclose.

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