New Analytical Modalities for Soil Carbon Analysis: Overview of the Inelastic Neutron Scattering System

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BNL:

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Inelastic Neutron Scattering

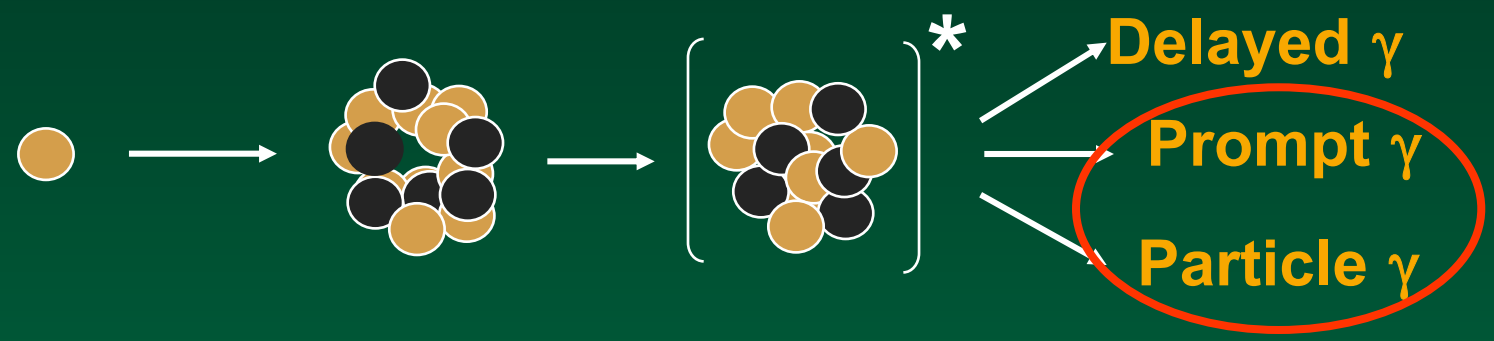
New Modalities for Carbon Analysis

Table 1 Basic Characteristics of the Emerging Technologies						
Instrument	Process	Methodology	Radiation Detected	Depth (cm)	Volume (cm^3)	Measurement Type
LIBS	Atomic	Plasma Induced Emission	Visible	~ 0.1	10^{-2}	Point, Profile, Destructive
NIR	Molecular	Diffuse Reflectance	Near-Infrared	~ 0.2	10^{0}	Surface, Scan, Destructive
MIR	Molecular	Diffuse Reflectance	Infrared	~ 1	10^{1}	Surface, Destructive
Py-MBMS	Molecular	Py-Molecular Beam	m/z	---	10^{1}	Samples Destructive

Inelastic Neutron Scattering

INS Principle Nuclear Reaction

Neutron Interactions With Matter



$$(n) + {}_{z}A^{n+z} \longrightarrow {}_{z}A^{n+z+1}$$

Activation
(n,γ)
(n,x,γ)
,n',γ
,2n,γ
,p,γ
,α,γ

Elements measured in situ:
H, N, C, O, Na, Cl,
Si, Ca, Fe, Ti, Gd, S, Al, K, U, Th

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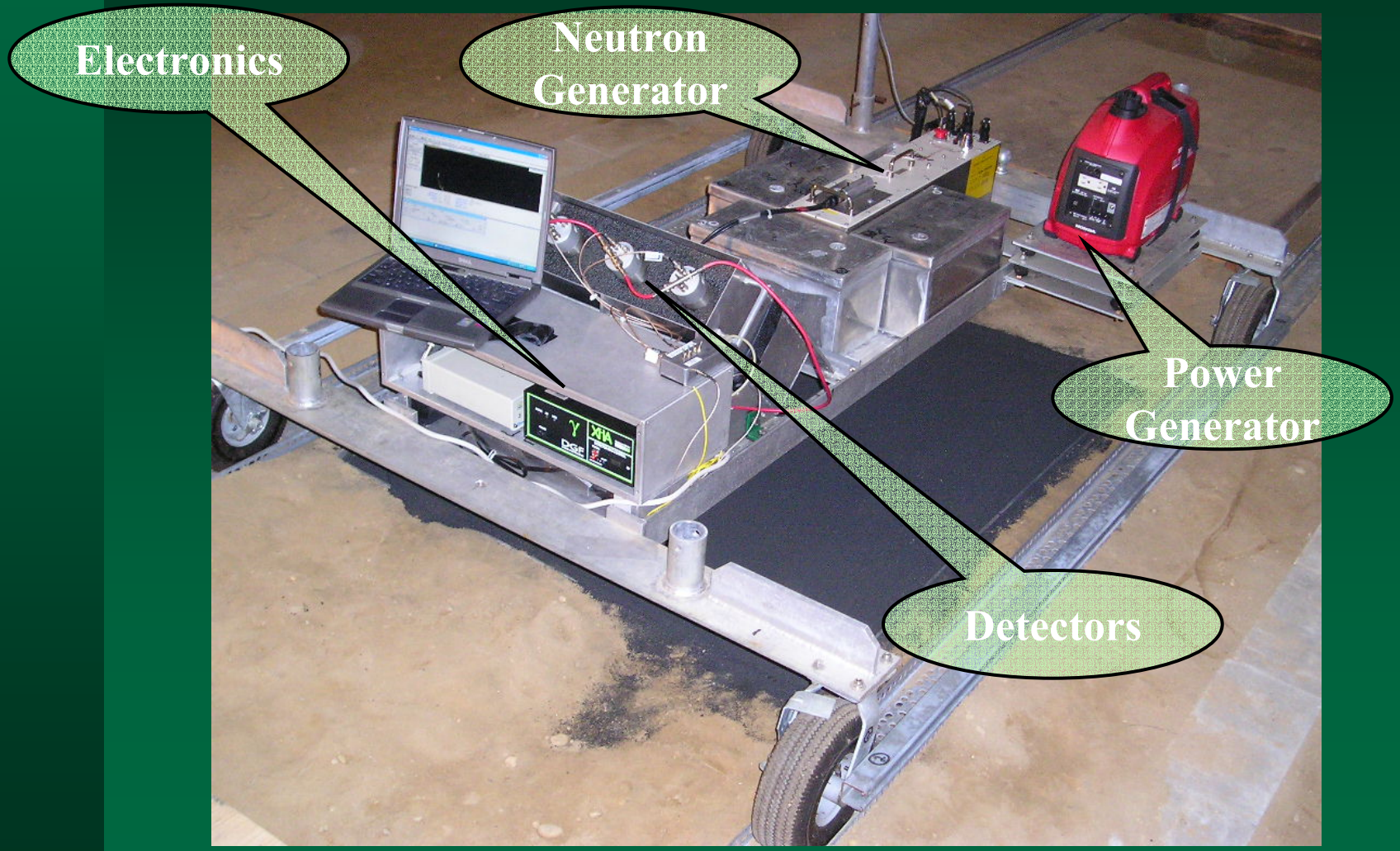
INS System



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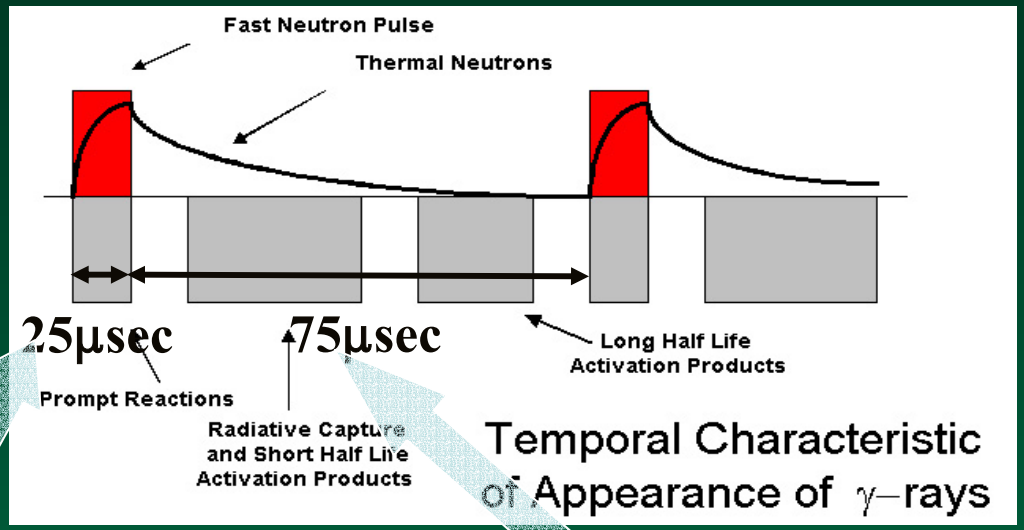
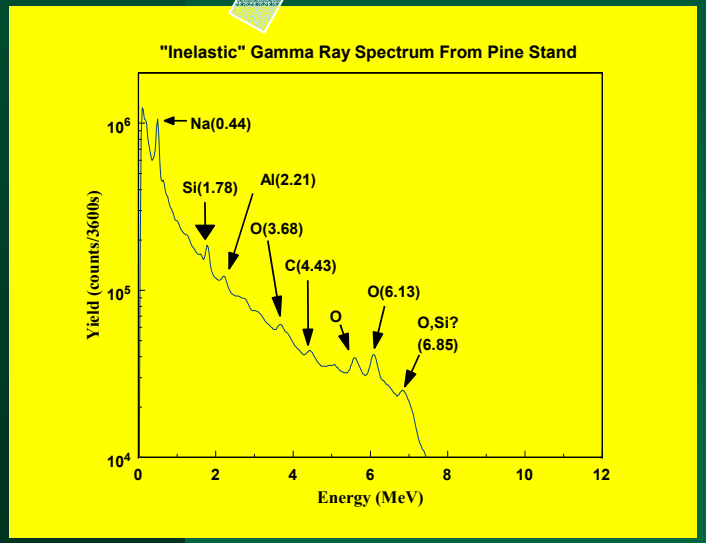
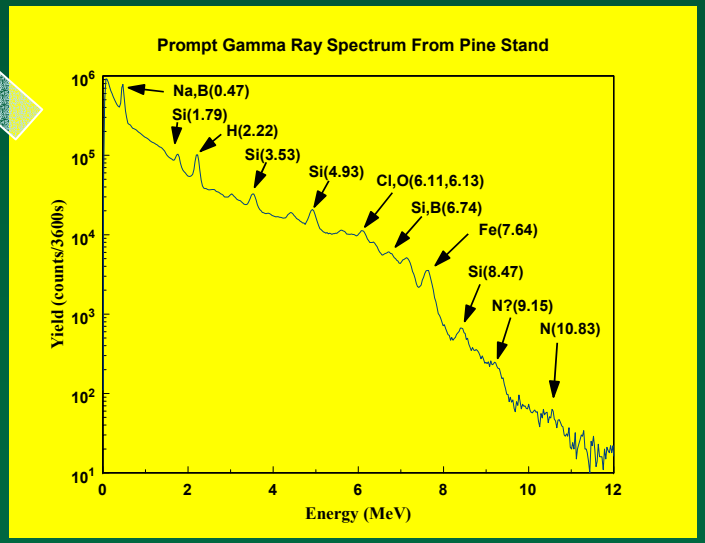
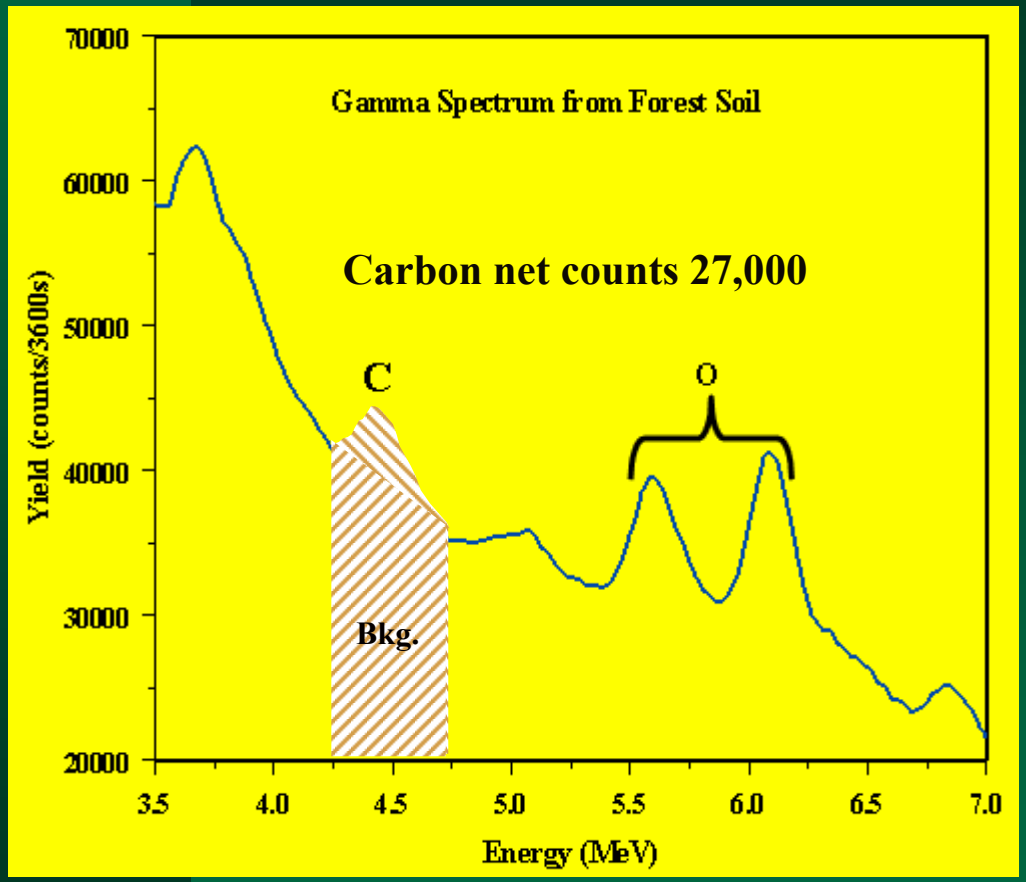
INS Spectra







Peak Analysis



T- Total number of counts

B- Background

N- Net number of counts

$N = T-B \Rightarrow C\ (g) = N/s$

s is sensitivity (count/g)

$\sigma_N = \sqrt{(T+B)}$

Minimum Detection Limit:

$MDL = 3SD = 3\sqrt{B} \Rightarrow$

$C_{MDL} = (3\sqrt{B})/s$

Minimum Detectable Change:

$\Delta_C = (3\ \sigma_N)/s$

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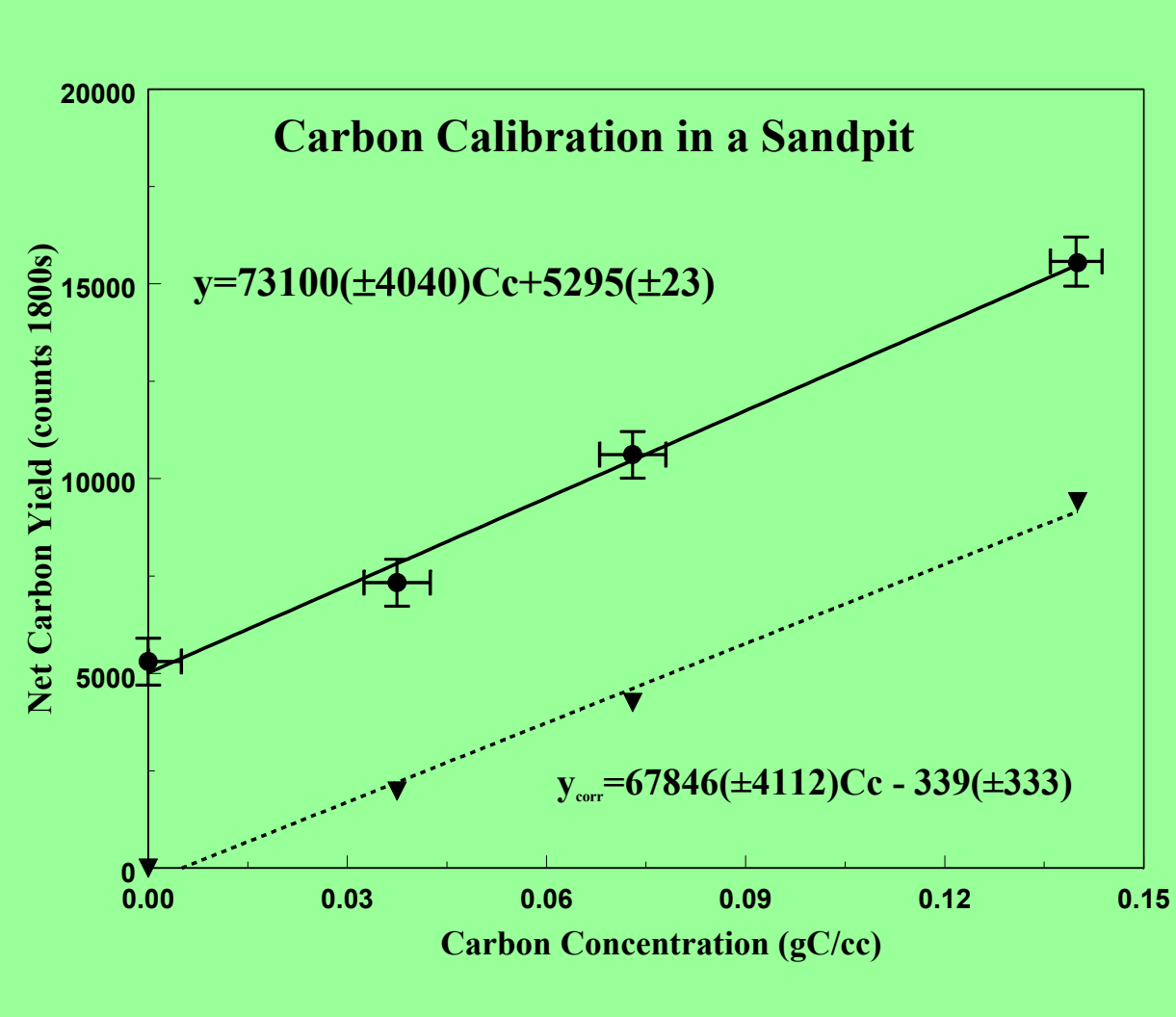
Calibration with Synthetic Soil



Interferences

Two major interferences with the carbon 4.43 MeV photopeak are:

1. $^{28}Si(n,n',\gamma)$ emits a cascade of 4.43 and 1.78 MeV gamma rays that are recorded in the INS spectrum in a fixed ratio.
   However, $^{28}Si(n,p)^{28}Al \rightarrow (\beta^-, T_{1/2}\ 2.2\ min)$ 1.78 MeV and $^{27}Al(n,\gamma)^{28}Al$ recorded in the TNC spectrum interfere with 1.78 gamma ray recorded in the INS spectrum.
2. The single escape peak from the Si 4.93 photopeak in the TNC spectrum overlaps with the 4.43 MeV carbon peak in the INS spectrum.

Thus $N_C = N - \text{Cascade} - \text{SEP}$

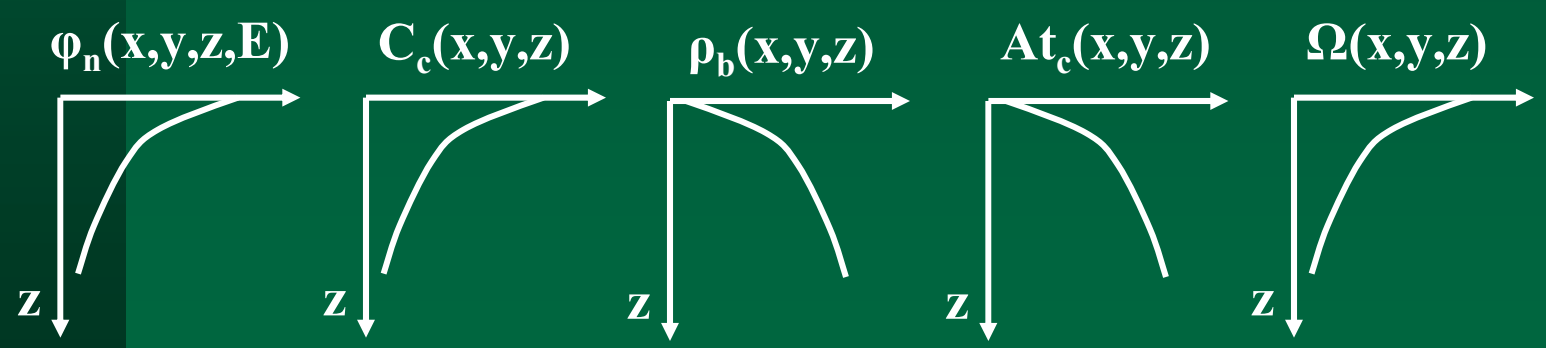
$$N_C = N - [Si(1.78)_{INS} - (Si+Al)(1.78)_{TNC}(LT_{INS}/LT_{TNC})]k_1 - [Si(4.43)_{TNC}(LT_{INS}/LT_{TNC}]$$

System Response Function

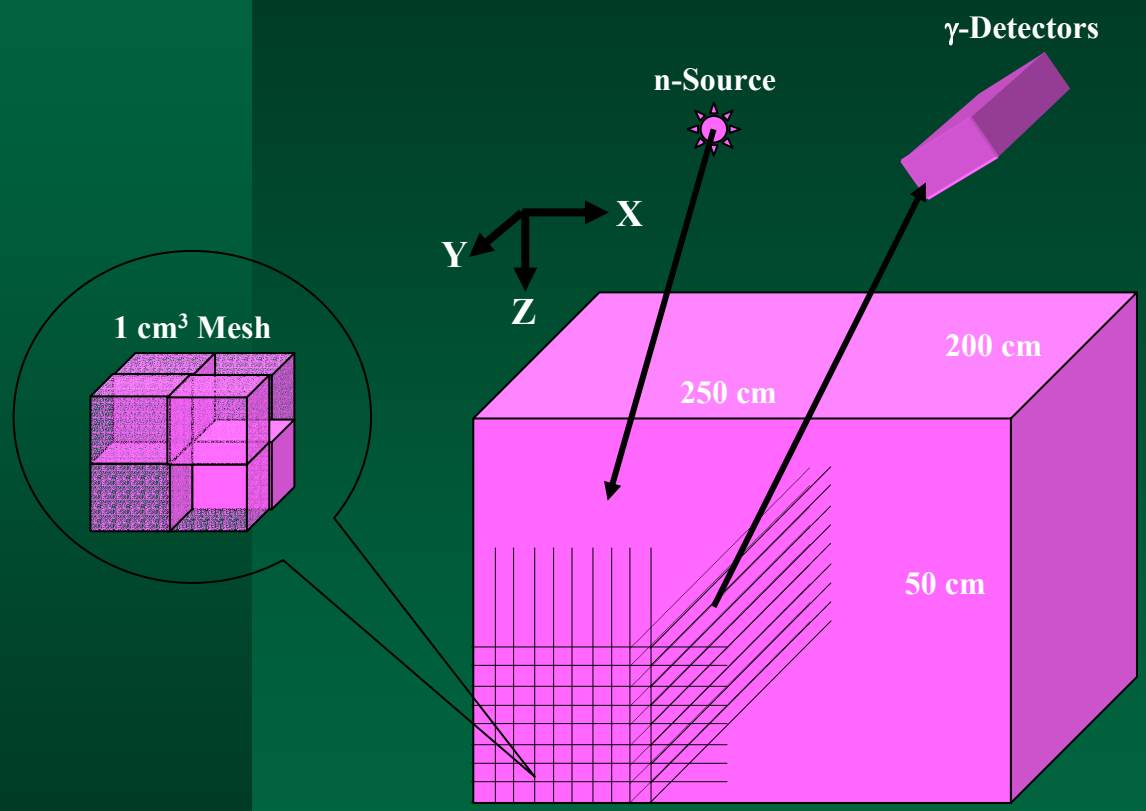
The INS model is derived from basic principles based on fundamental parameters of the entire system

$$R = \int_T dt \int_E \iiint_V \varphi_n(x,y,z,E)\sigma(E)C_c(x,y,z)\rho_b(x,y,z)\Omega(x,y,z)\cdot$$

$$\cdot At(x,y,z)Det(x,y,z)dEdxdydz$$



System Simulation

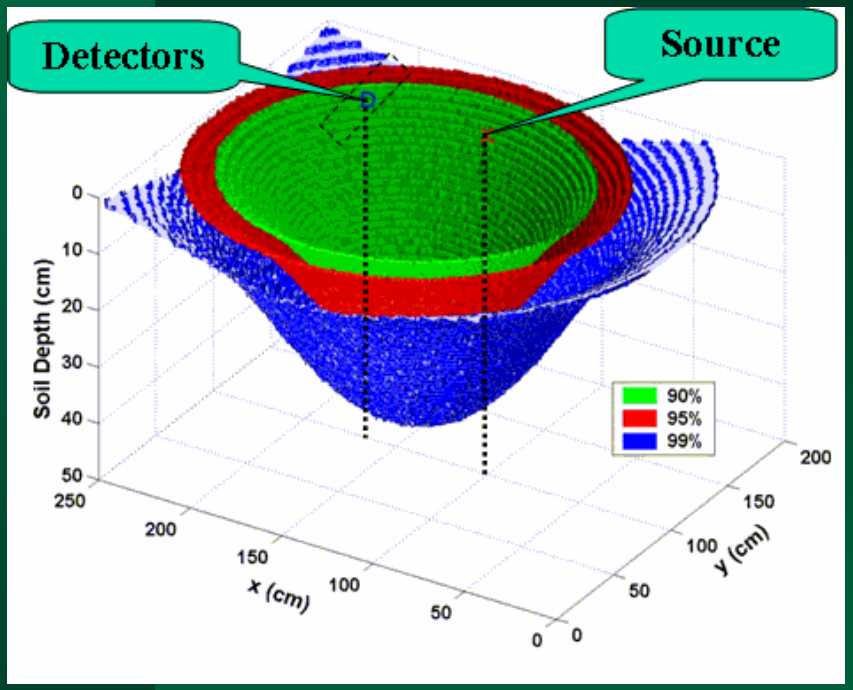


Having a complete simulation model allows

- Studding various parameters of the system
- Perform analytical experiments not possible other way
- Study perturbation effects on the system's response
- System characterization

Conceptual model of the INS system with 2,500,000 voxels. The shadow shielding between the n-source and the detection system is not shown here.

System Simulation



Surfaces describing the volumes from which a 90, 95 and 99% of the carbon gamma rays signal intercepts the detectors.

% of Total Response	Depth (cm)	Footprint (m^2)	Volume (m^3)	Mass (kg)
90	25	2.400	0.233	325.66
95	31	3.345	0.373	522.18
99	44	7.069	0.789	1105.29

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INS versus Chemical Analysis

Pine Stand

Sandy Patch

Oak Forest

	INS gC/cc	Chem. Analysis Mean gC/cc
Pine Stand (w,l)	0.099 ± 0.005	-
Pine Stand (w/o,l)	0.079 ± 0.005	0.073 ± 0.021
Oak Forest (w/o,l)	0.072 ± 0.004	0.085 ± 0.017
Sand Patch	0.026 ± 0.003	0.025 ± 0.002
Sandy Soil	0.091 ± 0.007	0.104 ± 0.019
Sand Pit (Cal.)	0.0	0.0004 ±

A. Torbert, USDA/NSDL, AL

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INS In The Duke Forest

Enon silt loam (a low fertility Hapludalf typical of the SE US piedmont)

Grass Field

Pine Forest

Oak Forest

K. Johnsen, USDA, NC

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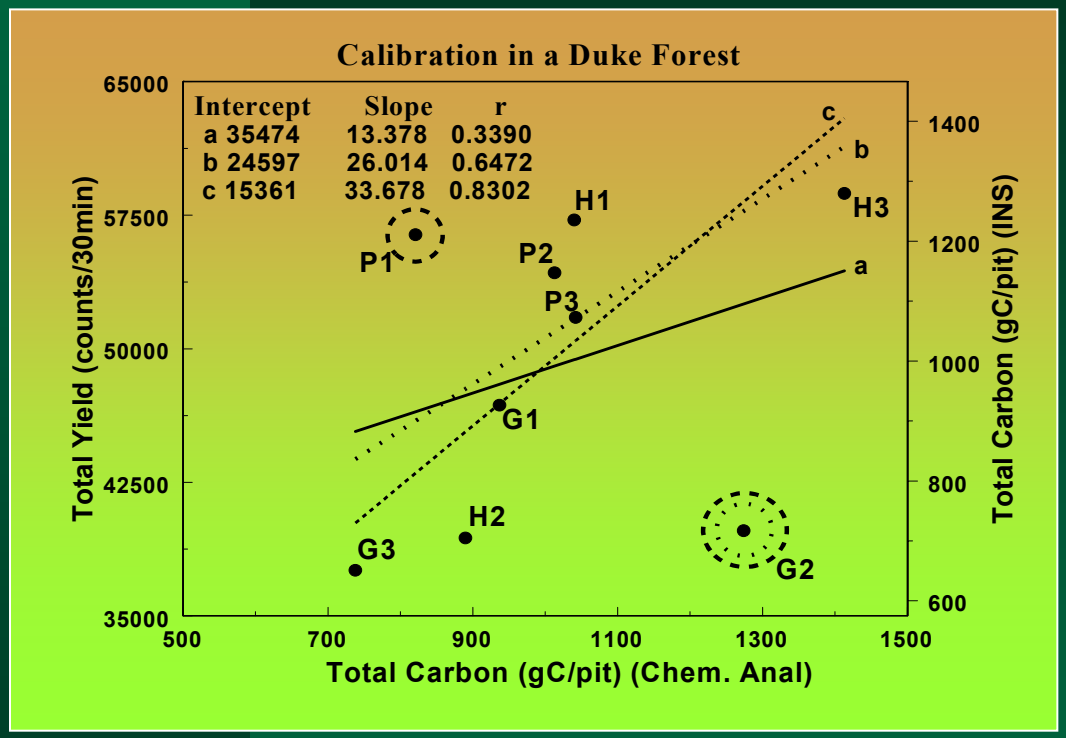
INS Duke Forest Measurements

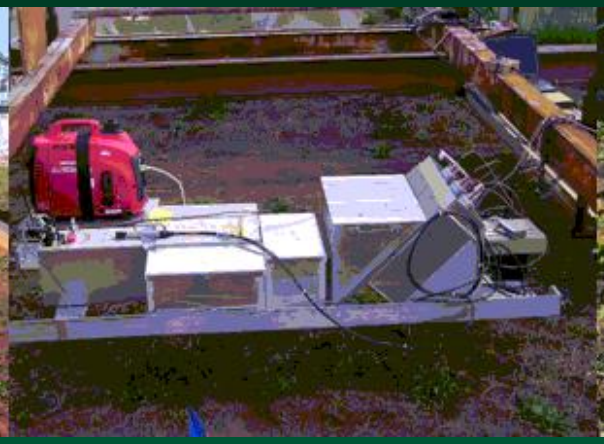


Right axis was created using calibration line "c".
(K. Johnsen, USDA, NC)

Site	C Counts	C Cor.
G1	46,816	20,730
G2	39,786	13,287
G3	37,652	13,641
P1	56,375	28,492
P2	61,286	32,751
P3	51,754	23,362
H1	57,234	33,696
H2	39,374	18,791
H3	58,718	37,358

K. Johnsen, USDA, NC

Scanning Mode

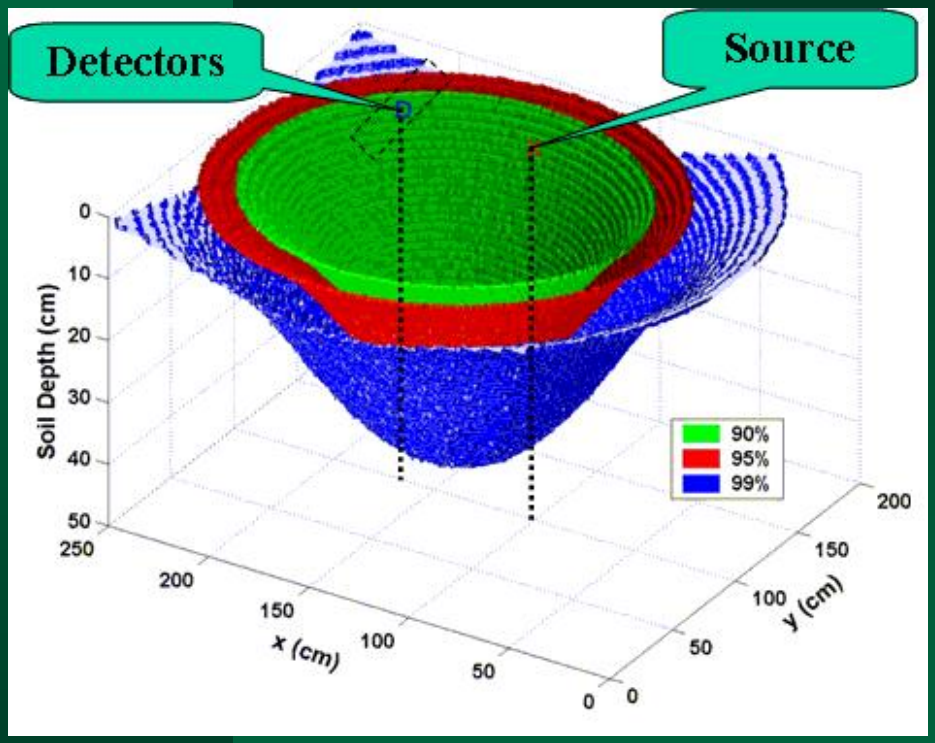


Comparison of static measurements versus scanning for the same counting time.

Uncorrected Net Yield in the C ROI for 1800 s			
	Mean Static Counts	Dynamic Counts	Difference In SD
Hiwassee Clay (V_1)	19,229	19,091	$0.1\sigma_{n-1}$
Vaiden Silty Clay ($2V_1$)	26,783	25,564	$0.8\sigma_{n-1}$
Hiwassee Sandy Loam (Random V)	30,449	31,117	$0.7\sigma_{n-1}$

A. Torbert, USDA/NSDL, AL

MCNP Simulation



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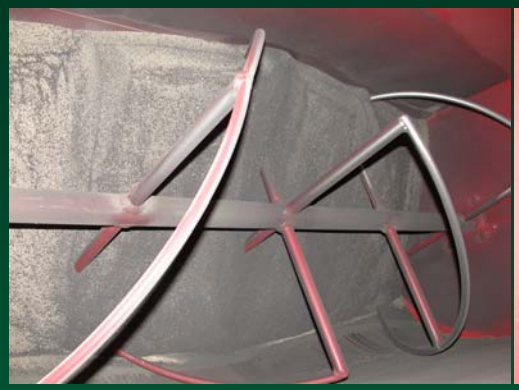
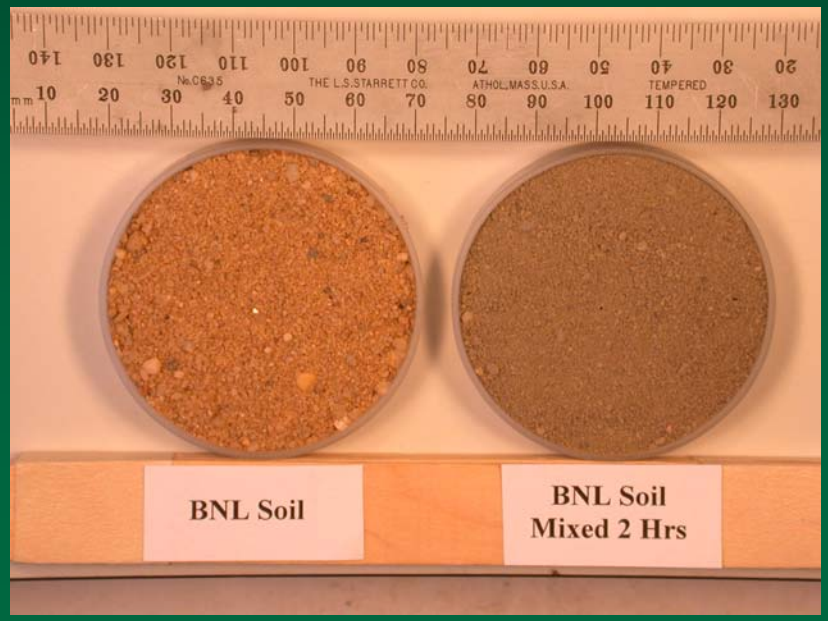
Soil Mixer for Synthetic Soils





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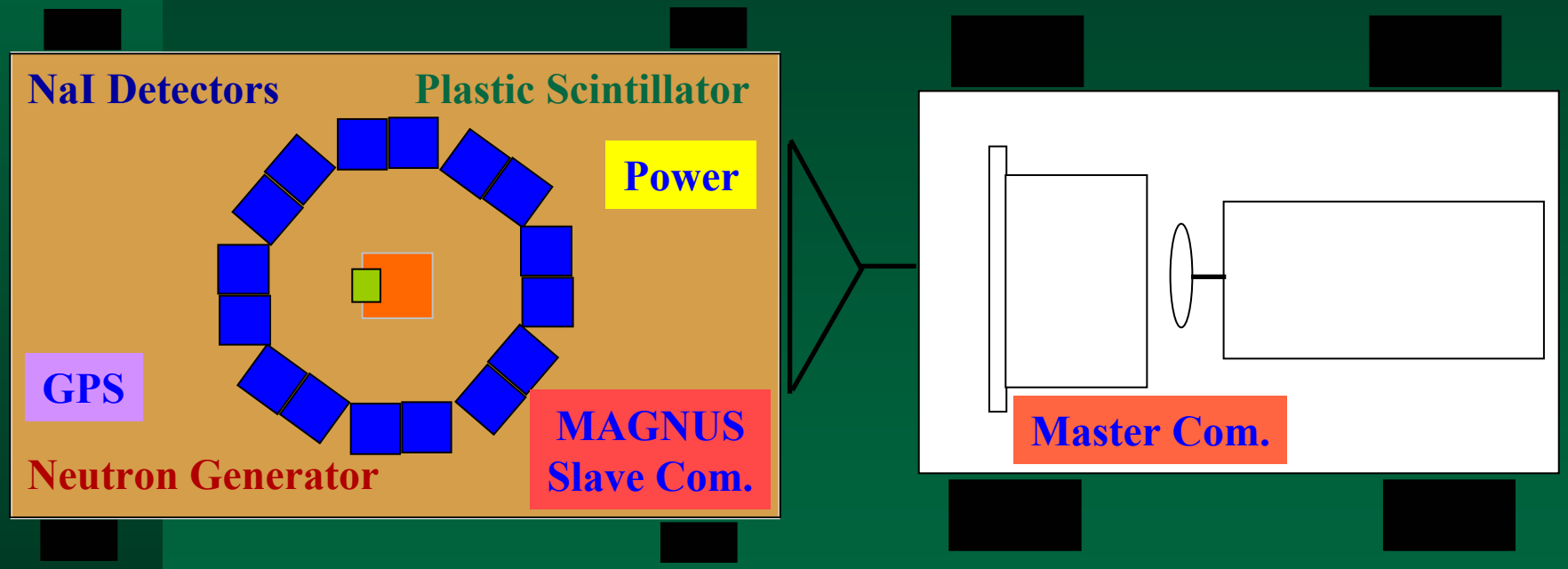
Beta Prototype



MCNP calculations indicate that transport of the high energy neutron group depends weakly on the soil moisture and density.

Summary

- INS is a non-destructive method for fast quantification of soil C in large (~0.3m^3) volumes or over large areas.
- Since the system integrates carbon content over constant volume it can be calibrated in terms of carbon concentration (gC/cc) or total carbon per unit area.
- The INS system can be deployed in either static or dynamic mode.
- INS response function and MCNP simulations enable system characterization and interrogation not possible by any other means.
- A high efficiency INS beta prototype is being prepared.

New Technologies for Environmental Research and Field Ecology!

Brookhaven National Laboratory