

LBA-ECO TG-09 SOIL ISOTOPIC C, N, H₂O, AND N₂O DATA, TAPAJOS NATIONAL FOREST, BRAZIL

Revision date: May 19, 2011

Summary:

This data set reports the results of carbon, nitrogen, and oxygen isotopic analyses of soil, soil water, and N₂O soil gas samples; total soil carbon and nitrogen concentrations; and soil texture and bulk density. Samples were collected from the km 83 Logged Forest Tower Site and the km 67 Seca-Floresta Site in the Tapajos National Forest (TNF) near Santarem, Para, Brazil. Soil samples were collected in July of 2000 and soil gas samples were collected in 2001 and 2002. Soil and gas samples were collected from various soil types at each site and from several depths in specially constructed pits. There is one comma-delimited ASCII data file with this data set.

Data Citation:

Cite this data set as follows:

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This data set was archived in May of 2011. Users who download the data between May 2011 and April 2016 must comply with the LBA Data and Publication Policy.

Data users should use the investigator contact information in this document to communicate with the data provider. Alternatively, the LBA Web Site [<http://lba.inpa.gov.br/lba/>] in Brazil will have current contact information.

Data users should use the Data Set Citation and other applicable references provided in this document to acknowledge use of the data.

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1. Data Set Overview:

Project: LBA (Large-Scale Biosphere-Atmosphere Experiment in the Amazon)

Activity: LBA-ECO

LBA Science Component: Trace Gas and Aerosol Fluxes

Team ID: TG-09 (Trumbore / Perez / Camargo)

The investigators were Trumbore, Susan E.; Perez Acosta, Tibisay Josefina ; Camargo, Plinio B. de ; Costa, Enir Salazar da; Silva, Daniela M. L. da and Tyler, Stanley C. You may contact Tibisay Perez (tperez@ivic.ve)

LBA Data Set Inventory ID: TG09_N2O_Soils

This data set reports the results of carbon, nitrogen, and oxygen isotopic analyses of soil, soil water, and N2O soil gas samples; total soil carbon and nitrogen concentrations; and soil texture and bulk density. Samples were collected from the km 83 Logged Forest Tower Site and the km 67 Seca-Floresta Site in the Tapajos National Forest (TNF) near Santarem, Para, Brazil. Soil samples were collected in July of 2000 and soil gas samples were collected in 2001 and 2002. Soil and gas samples were collected from various soil types at each site and from several depths in specially constructed pits.

Related Data Sets:

- [LBA-ECO CD-04 SOIL MOISTURE DATA, KM 83 TOWER SITE, TAPAJOS NATIONAL FOREST, BRAZIL](#) (soil moisture study from the same km 83 site at which this soils isotope study was done)

2. Data Characteristics:

Data are presented in one comma separated ASCII file: TG09_Isotopes_in_Soil_Pools_Tapajos.csv

Column	Heading	Units/format	Description
1	ID		Sample ID, replicate samples are given the same number and are distinguished by A or B
2	Site		Sampling location within the Tapajos National Forest, south of Santarem Para
3	Pit_ID		At km 83: C = Clay soil, SC = sand/clay transition soil, S = sand soil. A and B are replicate pits. At the Seca Floresta plots, km 67: CF = control plot, A = pit 1, B= pit 2; SF = dry down plot A=pit 4, B=pit 6)

4	Depth	cm	Sample depth (cm)
5	Collection_date_soils	yyyy/mm/dd	Collection date (yyyy/mm/dd) for soil samples (data in columns 6-14)
6	Bulk_density	g cm ⁻³	Dry weight of soil per unit volume reported as grams per cubic centimeter (g cm ⁻³)
7	delta_13C_soils	per mil	Isotopic ratio of ¹³ C/ ¹² C in carbon dioxide referenced to PDB, measured with continuous flow on Finigan Delta Plus at CENA (per mil)
8	C_concentration_soils	%	Carbon content of soils in percent (%) analyzed by dry combustion at CENA (Centro de Energia Nuclear na Agricultura at the University of Sao Paulo)
9	delta_15N_soils	per mil	Isotopic ratio of ¹⁵ N/ ¹⁴ N in carbon dioxide referenced to N ₂ , measured with continuous flow on Finigan Delta Plus at CENA (per mil)
10	N_concentration_soils	%	Nitrogen content of soils in percent (%) analyzed by dry combustion at CENA (Centro de Energia Nuclear na Agricultura at the University of Sao Paulo)
11	CN_ratio_soils		Mass based ratio of soil carbon to nitrogen
12	Sand	%	Percent sand from soil texture analysis
13	Silt	%	Percent silt from soil texture analysis
14	Clay	%	Percent clay from soil texture analysis
15	Collection_date_soil_water_1	yyyy/mm/dd	First sampling date (yyyy/mm/dd) for measurement of stable oxygen isotopes in soil water
16	delta_18O_soil_water_1	per mil	delta ¹⁸ O of the first set of extracted soil water relative to standard mean ocean water (SMOW) (per mil)
17	Collection_date_soil_water_2	yyyy/mm/dd	Second sampling date (yyyy/mm/dd) for measurement of stable oxygen isotopes in soil water. (Provided 01-Sep (yy-mmm) date changed to 2001/09/01)
18	delta_18O_soil_water_2	per mil	delta ¹⁸ O of the second set of extracted soil water relative to SMOW (per mil)
19	Collection_date_soil_gas_1	yyyy/mm/dd	First sampling date (yyyy/mm/dd) for measurement of stable oxygen and nitrogen isotopes in soil gases
20	N2O_mixing_ratio_soil_gas_1	ppb	N ₂ O mixing ratio (samples collected on date in column 19) for soil gases sampled using tubes installed at the various depths (ppb)
21	delta_15N_N2O	per mil	delta ¹⁵ N compared to atmospheric N ₂ of N ₂ O sampled from pore space (samples collected on date in column 19) (per mil)
22	Error_delta_15N_N2O	per mil	Precision of N ₂ O delta ¹⁵ N isotope measurement (per mil)
23	delta_18O_N2O	per mil	delta ¹⁸ O compared to atmospheric N ₂ of N ₂ O sampled from pore space (samples collected on date in column 19)

24	Error_delta_18O_N2O	per mil	Precision of N2O delta 18O isotope measurement (per mil)
25	Collection_date_soil_gas_2	yyyy/mm/dd	Second sampling date (yyyy/mm/dd) for measurement of stable oxygen and nitrogen isotopes in soil gases
26	N2O_mixing_ratio_soil_gas_2	ppb	N2O mixing ratio (samples collected on date in column 25) gases sampled using tubes installed at the various depths reported in parts per billion (ppb)
Missing data are represented by -9999			

Example Data Records:

ID,Site,Pit_ID,Depth
,Collection_date_soils,Bulk_density,delta_13C_soils,C_concentration_soils,delta_15N_soils,
N_concentration_soils,CN_ratio_soils,Sand,Silt,Clay,Collection_date_soil_water_1,
delta_18O_soil_water_1,
Collection_date_soil_water_2,delta_18O_soil_water_2,Collection_date_soil_gas_1,N2O_mixing_r
atio_soil_gas_1,
delta_15N_N2O,Error_delta_15N_N2O,delta_18O_N2O,Error_delta_18O_N2O,Collection_date_s
oil_gas_2,
N2O_mixing_ratio_soil_gas_2

1A,km 83,C A,0-5,2000/07/21,-9999,-28.64,4.33,10.2,
0.34,12.9,-9999,-9999,-9999,2000/07/08,-9999,
2001/09/01,-9999,2001/11/16,-9999,
-9999,-9999,-9999,-9999,2002/03/15,
-9999

1B,km 83,C A,0-5,2000/07/21,0.81,-28.45,4.56,9.92,
0.36,12.59,31,8,61,2000/07/08,-5.13,
2001/09/01,-1.94,2001/11/16,353.1,
4.91,0.06,19.88,0.83,2002/03/15,
-9999

2,km 83,C A,10,2000/07/21,0.9,-27.4,1.79,11.77,
0.16,11.04,18,7,75,2000/07/08,-4.18,
2001/09/01,-2.77,2001/11/16,440.34,
3.92,0.006,19.8,0.24,2002/03/15,
648.39

...

13,km 67 Seca Floresta control,CF A,0,2000/07/21,0.96,-28.89,4.52,8.45,
0.33,13.75,12,10,78,2000/07/08,-4.55,
2001/09/01,-0.61,2001/11/22,392.6,
-0.64,0.24,17.61,0.36,2002/03/19,
-9999

14,km 67 Seca Floresta control,CF A,10,2000/07/21,0.96,-27.5,2.17,9.71,
0.18,11.82,12,14,74,2000/07/08,-3.83,
2001/09/01,-1.54,2001/11/22,425.3,
3.61,0.18,19.92,0.36,2002/03/19,
808.37

...

Site boundaries: (All latitude and longitude given in decimal degrees)

Site (Region)	Westernmost Longitude	Easternmost Longitude	Northernmost Latitude	Southernmost Latitude	Geodetic Datum
Para Western (Santarem) - km 83 Logged Forest Tower (Para Western (Santarem))	-54.9707	-54.9707	-3.017	-3.017	World Geodetic System, 1984 (WGS-84)
Para Western (Santarem) - km 67 Seca-Floresta Site	-55.0	-55.0	-2.750	-2.750	World Geodetic System, 1984 (WGS-84)

Time period:

- The data set covers the period 2000/07/01 to 2002/03/23
- Temporal Resolution: daily

Platform/Sensor/Parameters measured include:

- LABORATORY / IRMS (Isotope Ratio Mass Spectrometer) / NITROUS OXIDE
- LABORATORY / WEIGHING BALANCE / SOIL BULK DENSITY
- LABORATORY / ANALYSIS / SOIL TEXTURE
- LABORATORY / IRMS (Isotope Ratio Mass Spectrometer) / CARBON
- LABORATORY / IRMS (Isotope Ratio Mass Spectrometer) / NITROGEN
- LABORATORY / IRMS (Isotope Ratio Mass Spectrometer) / RADIOISOTOPES

3. Data Application and Derivation:

Tropical forest soils are the largest natural source of N₂O to the atmosphere (Matson and Vitousek 1990). The use of stable isotope composition of N and O in atmospheric N₂O and its sources has been proposed as a way to better constrain the global N₂O budget. Variations in the flux and isotopic signatures of N₂O from tropical soils reflect the microbiological processes that produce and consume N₂O as well as the physical controls of the rate of N₂O movement from soil to the atmosphere (Perez et al. 2000, 2001). Understanding the relative contribution of mechanisms responsible for such variability will provide new tools for extrapolating stable isotope results into large scales.

4. Quality Assessment:

Isotopic composition of nitrous oxide

The uncertainty in N₂O isotopic signature, determined by repeated measurements of an N₂O isotope standard, was plus/minus 0.2 per mil and plus/minus 0.3 per mil for ¹⁵N and ¹⁸O, respectively. The delta ¹⁵N and delta ¹⁸O values are expressed relative to atmospheric N₂ and Vienna-standard mean ocean water (V-SMOW), respectively.

Nitrous oxide concentration

N₂O mixing ratios were determined by collection of 20 mL nylon syringes in the soil depth probes and measurement by electron capture detector (ECD) gas chromatography. Calibration curves with two N₂O standards (400 and 800 ppb) were made each sampling day. The uncertainty of the method based in the calibration curves with n=3 for each standard is plus/minus 5 ppb.

Amount and isotopic composition of soil carbon and nitrogen

The errors of %C, %N, delta 13C and delta 15N are calculated based on repeated analysis of standards of known isotopic composition. Their values are plus/minus 0.05%, plus/minus 0.1%, plus/minus 0.1 per mil and plus/minus 0.2 per mil for %C, %N, delta 13C, and delta 15N, respectively.

18O isotopic composition of soil water

The delta 18O values are expressed relative to the Vienna-standard mean ocean water (V-SMOW). The method uncertainty was plus/minus 0.1 per mil based on repeated analysis of standard materials.

5. Data Acquisition Materials and Methods:

Soil collection

Soils were collected from the Tapajos National Forest (TNF) near Santarem, Para state, Brazil. Soils from three primary forest sites were collected: a clay-rich (Oxisol), a sandy clay loam (Ultisol) and sandy loam (Ultisol) (described in Silver et al., 2000; Telles et al., 2003), and one set of samples from an Oxisol soil located in a forest dry down experiment (Seca Floresta) located in the km 67 site of the TNF (for this site details see Nepstad et al. 2002). The soil texture was determined at 0-5, 10, 25, 75, 100 and 200 cm of depth at the km 83 site. The texture classification at deeper layers shows that the sandy clay loam soil become a clay textured soil.

Amount and isotopic composition of TN

The fresh soils samples stored at 4 degrees C were taken to the Laboratorio de Ecologia Isotopica at CENA where they were dried at 60 degrees C for 24 hours. Samples were sieved (2 mm) and milled, and total carbon and total nitrogen content and 15N isotopic composition were determined by CF-EA-IRMS. The nitrogen content analyzed this way is the sum of organic and inorganic N and is reported as percentage of total soil mass.

Soil depth N₂O mixing ratio sampling collection

For the Brazilian clay, sandy clay loam, and sandy loam, we dug a pit of 2 meters depth where we inserted 2 m of 1/8 inch (outer diameter) stainless steel tube probes at 10, 25, 75, 100, and 200 cm on one wall of the pit. One end of the probe had holes to allow soil air flow in and the other end had a 1/8 inch Swagelok fitting with a septum to collect the air samples with a syringe. We used one of the pit walls to determine physical parameters (bulk density and water content) and soil samples for nutrient soil analysis. We used a 20 ml syringe to collect soil air at each depth to determine N₂O concentration. We flushed the first 20 ml of air to avoid possible N₂O dilution within the probe (due to the probe length). At this site we sampled soil air for N₂O mixing ratios and N₂O stable isotope determinations at 0, 10, 25, 75, 100, and 200 cm of depth, as well as for water content, concentrations and isotopic composition of total carbon (TC) and nitrogen (TN), and soil nutrient (NH₄⁺ and NO₃⁻) concentrations. At the Seca Floresta site the same parameters were measured at 10, 25, 50, 100, 200, 300, 700, and 1,100 cm depth in a pit that was already being monitored monthly for trace gases concentrations (Davidson et al., unpublished results).

Stable isotope sampling collection

The samples for stable isotope analysis were collected using a N₂O collection system that consisted of an evacuated stainless steel canister attached to a tee with a septum in one end and a drierite/ascarite trap (for removal of CO₂ and H₂O) at the other end. The other end of the drierite/ascarite trap was connected to either the chamber placed on the soil surface, or the soil probe at each particular depth. The evacuated can was filled with the gas sample following the procedure described elsewhere [Perez

et al., 2006]. After a 2-minute equilibration period, the canister valve was closed and the sample stored until it was analyzed at UC Irvine using a Finnigan MAT 252 isotope ratio mass spectrometer operated in continuous flow mode coupled with an online custom-made preconcentrator and gas chromatograph.

Isotopic composition of nitrous oxide

The samples stored in stainless steel canisters were transported to the University of California Irvine. In S. Tyler's lab, we used a custom-built gas pre-concentrator for N₂O stable isotope analysis attached to a isotope ratio mass spectrometer. The procedure was as follows: We transferred the samples into glass bulbs (either 100 mL or 250 mL in volume) equipped with two valves. The smaller glass bulbs were chosen for the samples with the highest N₂O concentration. A sample placed in a bulb was connected to the inlet of the pre-concentrator. High-flow ultra-high purity helium (25 mL/min) carried the sample first to an ascarite and then to a MgClO₄ trap (to remove CO₂ and H₂O), then to a Nafion dryer (to further remove H₂O; Perma Pure, Toms River, New Jersey, USA), and finally to the next trap in line where the N₂O was condensed cryogenically (195 degrees K) and the other non-condensable gases were removed (N₂, O₂, CH₄, CO). Enough helium was used to flush the sample bulb volume three times and ensure that all the sample was extracted from the bulb. The N₂O on the first trap was released by warming it to room temperature and transferring it cryogenically to a Porapak Q trap (Alltech, Deerfield, Illinois, USA) at room temperature to remove the hydrocarbons remaining in the sample. Finally, the sample was transferred into a cryofocusing trap (Poraplot Q, Alltech) before its injection into a gas chromatograph (GC). The sample was transferred to the GC by a stream of low-flow UHP helium (3 mL/min) and the N₂O was separated from remaining traces of CO₂ by a 25-m Poraplot Q capillary column (Alltech). Finally the bulk delta 18O and delta 15N isotopic compositions were measured by the Finnigan MAT model Delta XL isotope ratio mass spectrometer (ThermoElectron, Waltham, Massachusetts, USA) connected to the GC via an open split.

18O isotopic composition of soil H₂O

The delta 18O values of soil water were determined by means of a CO₂ microequilibration method (Moreira et al. 1997).

6. Data Access:

This data is available through the Oak Ridge National Laboratory (ORNL) Distributed Active Archive Center (DAAC).

Data Archive Center:

Contact for Data Center Access Information:

E-mail: uso@daac.ornl.gov

Telephone: +1 (865) 241-3952

7. References:

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Related Publications

- Perez, T. 2005. Factors that Control the Isotopic Composition of N₂O from Soil Emissions. In *Stable Isotopes and Biosphere-Atmosphere Interactions: Processes and Biological Controls*. L.B. Flanagan, J.R. Ehleringer, D.E. Pataki, Eds. A Volume in the *Physiological Ecology Series*, Elsevier Press. 69-84, 2005.