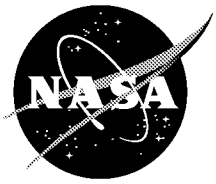
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Technical Report Series on the Boreal Ecosystem-Atmosphere Study (BOREAS)

Forrest G. Hall and Sara K. Conrad, Editors

Volume 233

BOREAS TGB-6 Soil Methane Oxidation and Production from NSA BP and Fen Sites

Bruce Deck and Martin Wahlen
UCSD, Scripps Institute of Oceanography, LaJolla

National Aeronautics and
Space Administration

Goddard Space Flight Center
Greenbelt, Maryland 20771

November 2000

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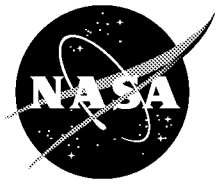
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BOREAS TGB-6 Soil Methane Oxidation and Production from NSA BP and Fen Sites

Bruce Deck, Martin Wahlen

Summary

The BOREAS TGB-6 team collected soil methane measurements at several sites in the SSA and NSA. This data set contains soil methane consumption (bacterial CH_4 oxidation) and associated ^{13}C fractionation effects in samples that were collected at various sites in 1994 and 1996 from enclosures (chambers). Methane ^{13}C data in soil gas samples from the NSA YJP and OJP sites for 1994 and 1996 are also given. Additional data on the isotopic composition of methane (carbon and hydrogen isotopes) produced in the NSA beaver ponds and fen bog in 1993 and 1994 are given as well. The data are stored in tabular ASCII files.

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1. Data Set Overview

1.1 Data Set Identification

BOREAS TGB-06 Soil Methane Oxidation and Production from NSA BP and Fen Sites

1.2 Data Set Introduction

Destruction of methane in the troposphere by chemical reaction with OH- is the primary sink for this gas. As much as 10% (Born et al., 1990) of the total destruction of atmospheric methane may be due, however, to bacterial oxidation on relatively dry soils. In determining the global isotopic budget for methane, the isotopic fractionation and enrichment due to soil oxidation must be considered in comparison to that resulting from the reaction with the OH- in the troposphere. Boreal forest soils have a large aerial extent, and adequate measures of the fractionation occurring in the methane oxidation process must be determined for inclusion in any global budget.

Methane emissions from ponds and fens are a significant source in the methane budget of the boreal region. An initial study on the isotopic composition of this methane source and the isotopic composition in relation to oxidation of methane at the sediment surface of the ponds or fen was conducted. Information on the isotopic composition of the methane is important in both understanding the biogeochemistry of the system and determining the regional and global methane isotope budget.

Soil methane oxidation is a net consumption of methane from the overlying atmosphere by bacterial processes in aerobic soils, with an accompanying isotope fractionation. The flux will be a function of the microbiological activity of the methanotrophs, and in dependent on the microbiology, soil character, temperature, and moisture content.

The isotopic composition of methane emitted by saturated anoxic sediment is dependent on the sediment composition and geochemistry, but it is influenced by oxidation, which is in part a function of rooted plant activity. The dependence of the isotopic composition on rooted plant activities is not well known and will depend on the plant type, sediment temperature, and numerous other variables.

Air samples indicating methane consumption were obtained from sealed enclosures placed over soil sites for several hours. Soil gas samples taken by subsurface probes were analyzed for the isotopic carbon composition in methane. Gas samples of varying methane concentration from bubbles and equilibrated water samples taken from beaver ponds and fens were analyzed for the carbon and hydrogen isotope composition.

1.3 Objective/Purpose

The objective of this study was to determine the isotopic composition of methane released from the BOReal Ecosystem-Atmosphere Study (BOREAS) Northern Study Area (NSA) Beaver Pond and Fen sites.

1.4 Summary of Parameters and Variables

The main variables measured in this study were soil depth, CH_4 concentration, ^{13}C isotope concentration, and H isotope concentration.

1.5 Discussion

Dislodged bubbles from anoxic sediments from five beaver ponds were sampled in 1993, and bubble and sediment/water samples from the NSA Tower Beaver Pond (TBP) were taken in 1994 to determine the carbon and hydrogen isotope values of methane emitted from these sites.

Gas samples from flux enclosures (chambers) over the soil and subsurface soil gas samples were taken and returned to the lab for analysis. Data were collected once in 1994 and at four time periods in 1996 from two sites near the towers at the Old Jack Pine (OJP) and Young Jack Pine (YJP) sites in the NSA. A set of chamber measurements from the Southern Study Area (SSA) OJP and YJP sites along with soil gas samples was obtained in 1994. While some concentration measurements were made in the field in conjunction with other investigators, these data sets consist of methane concentration and carbon isotope ($^{13}C/^{12}C$) ratios for each analyzed sample.

The sites sampled in 1996 duplicated the NSA sites that were sampled in 1994. At OJP, the Crill (Trace Gas Biogeochemistry [TGB]-01) Lichen chamber #4 and the TGB-01 Moss chamber #7 were sampled. For the YJP, Moore (TGB-05) chambers #4 and #6 were sampled. Two-liter samples were taken from each of the chambers, drawn over a 1-min sampling period, at times of 0.75 to 7 hr depending on estimated methane uptake. The chambers at the OJP were approximately 20 m apart, while those of the YJP were within 5 m of each other. Soil probe samples were taken at shallow depths in the immediate (1 m) vicinity of the chambers. Samples were drawn slowly (5 min for 2 liters) to avoid contamination from gas sucked in from intermediate levels. A summary description of the data collection is given in Section 6.2.

1.6 Related Data Sets

BOREAS TGB-01 Soil CH4 and CO2 Profile Data from NSA Tower Sites
BOREAS TGB-01 CH4 and CO2 Chamber Flux Data
BOREAS TGB-01 CH4 Concentration and Flux Data from NSA Tower Sites
BOREAS TGB-03 CH4 and CO2 Chamber Flux Data over NSA Upland Sites
BOREAS TGB-03 Soil CO2 and CH4 Profile Data over the NSA

2. Investigator(s)

2.1 Investigator(s) Name and Title

Dr. Martin Wahlen
Professor of Physics, PI

Dr. Bruce Deck
Research Specialist, Assoc. PI

2.2 Title of Investigation

Isotopic Composition of Methane Produced and Consumed in Boreal Ecosystems

2.3 Contact Information

Contact 1:
Bruce Deck
UCSD, Scripps Institution of Oceanography
La Jolla, CA, 92093-0220,
(619) 534-6248
(619) 534-0967 (fax)
bdeck@UCSD.edu

Contact 2:
Jeffrey A. Newcomer
Raytheon ITSS
Code 923
NASA GSFC
Greenbelt, MD 20771
(301) 286-7858
(301) 286-0239 (fax)
Jeffrey.Newcomer@gsfc.nasa.gov

3. Theory of Measurements

In order to determine the effect the land surface has on the overlying atmospheric concentration, the flux of any gas through the various pathways into or out of the soil sediment or biota must be determined. In the case of methane in boreal forests, the interaction between the atmosphere and forest soils is direct and not mediated to any major extent by trees or woody plants. Thus, the flux of methane into or out of the soil can be determined, and the isotopic fractionation examined, by placing closed chambers on the surface for a period of time. In addition, samples of soil gas taken below the soil surface will provide information on the rate of methane consumption and the isotopic fractionation. In the case of ponds or bogs, funnels at the water surface or monitoring of the overlying water can be used to determine sediment gas exchange. In either situation, changes in concentration and isotopic composition of methane over time will indicate the effect the soil or sediment will have on the overlying atmosphere (source/sink).

The basic problem with this sampling approach is to avoid having chamber placement cause perturbations of the naturally occurring gas exchange either through biological or physical changes in the system. To this end, the temperature, humidity, air circulation, light, and other parameters inside the chamber can be controlled to mimic those outside the chamber. In many cases, controls on chamber parameters are not adequate for extended periods of time and the best sampling strategy is to leave the chambers enclosed for the minimum consumption of methane to ensure the necessary accuracy in the determinations. For concentration changes in methane, the high degree of precision attainable allows

for short (0.3-1 hr) maximum enclosure times. In the determination of isotopic methane composition, the times necessary for measurable changes to be determined with reasonable precision in the analysis are a factor of 2 to 4 longer. In addition, care must be taken at all times to ensure that the sampling and storage procedures do not introduce additional errors. Finally, it must be realized that chamber, soil probe samples, and/or any discreet measurements at a finite number of sites can only approximate the estimates of actual average soil-atmosphere interaction at any one site.

Samples taken from ponds and fens show a net flux of methane into the atmosphere. The investigations started here were not meant to quantify the net flux, but to allow a determination of the isotopic composition of the emitted methane.

Because of the large amount of time required for the processing of the isotope samples, a limit on the number of samples taken in any field season was necessary. The fewer the number of samples or the greater the variability in site characteristics, the poorer the data approximation. The limitations on the number of samples taken imposed by isotopic analysis procedures are significant in this study, but are balanced in part by the uniformity in microbial biochemistry, which drives the methane production and consumption in all areas.

4. Equipment

4.1 Sensor/Instrument Description

- Chambers (funnels)/Soil Probes
- Sampling Flasks/Manifold
- Gas Chromatography Analysis
- Separation Chromatography/Combustion
- Mass Spectrometric Analysis

4.1.1 Collection Environment

Samples were taken at specific sampling periods, the majority from sites at tower locations. Sites for 1993 were chosen to determine (preliminary) methane production and included the NSA Fen site and several beaver ponds. It was proposed that 1994 data be taken at different times to cover a seasonal cycle, but only samples from late August-September were collected. These included chamber gas and soil gas sampling at the SSA YJP and OJP sites 04-Aug and NSA YJP and OJP sites 23-24-Aug. Sets of methane production samples from the TBP site were taken 25-26-Aug, 31-Aug, and 09-Sep-1994. Samples for 1996 were taken in the NSA only from the YJP and OJP sites at four sampling times, 03-Jun, 26-Jun, 02-03 Aug, and 08-11-Sep. Most samples were taken in good weather. Samples taken on 03-Jun-1996 were made following several days of rain and while the ground was frozen below approximately 10 to 15 cm. Also, the final samples on 11-Sep-1996, from the NSA OJP were made following a heavy cold rain period.

4.1.2 Source/Platform

Samples from the wet production areas were taken from the shore or walkways. Soil flux measurements were made utilizing fixed collars left in the soil from year to year. Tube and sample probes were inserted into the soils for each soil gas collection.

4.1.3 Source/Platform Mission Objectives

The purpose of the collars was to support the chamber and allow measurements to be made.

4.1.4 Key Variables

Isotopic composition of methane (carbon, $^{13}CH_4$ and hydrogen, CH3D isotopes) in samples from sediment production, and carbon isotope fractio nation in the methane consumption by bacteria in soils.

4.1.5 Principles of Operation

Samples were taken in the field and returned to Scripps Institute of Oceanography (SIO) for methane concentration analysis, after which the methane was quantitatively separated from the bulk air sample. Following combustion with pure oxygen, the methane sample was separated into fractions for carbon and (if possible) hydrogen isotope analysis on a high-precision duel inlet isotope ratio mass spectrometer (VG Prism II).

4.1.6 Sensor/Instrument Measurement Geometry

Discrete sample sites was selected; there were no field sensors as such. The actual nature of the soil surface or pond sites, with relationship to rock, litter cover, and vegetation, was of importance in determining sampling sites, and soil depth in conjunction with soil probe measurements. Samples were analyzed with a GOW-MAC flame ionization gas chromatograph (GC) and a VG Prism II isotope ratio mass spectrometer.

4.1.7 Manufacturer of Sensor/Instrument

Chambers as made by Crill TGB-01, Moore TGB-03. Concentration analysis GOW-MAC flame ionization GC (GOW-MAC Inst. Co. Lehigh Valley, PA 18002). Separation and methane combustion on custom high vacuum lines (M. Wahlen). Mass spectrometric determinations on a VG Prism II isotope ratio mass spectrometer (Micro Mass, Beverly, MA 01915).

4.2 Calibration

4.2.1 Specifications

Calibrations were performed using methane standard concentration determinations and isotopic analysis conducted at SIO during the lab analysis. Methane determination on field samples was made with a manometric manifold inlet loop on a flame ionization GC system with a 2-meter, 1/8-inch i.d., 60/80 mesh Poropack Q column, using nitrogen carrier gas. Calibration was performed with a set of five working standards covering the range of 0.8 to 2.8 ppm previously calibrated against SIO/Commonwealth Scientific and Industrial Research Organization (CSIRO) international standards (R. Weiss) to better than 0.2%. Overall precision and accuracy of the system in this range was approximately 0.5%. Calibration against the BOREAS working methane standards (0.9 and 2.0 ppbv) also showed this agreement within experimental errors. Samples with higher than 3-ppm methane concentration were diluted and/or injected at reduced pressure and calibrated against volumetric standards up to 10% methane. Uncertainty for these samples was approximately 5%.

For the 2-liter soil chamber and probe sample runs on the extraction line, pure nitrogen blank determinations, synthetic air standards, and calibrated isotopic methane standards passed through the lines and combustion systems during development provided the calibration of the system. For calibration during normal run, clean SIO air was used as a standard, with $^{12/13}C$ methane values of -47.2 permil Pee Dee Belemnite (PDB). Approximately 1 standard per 4 samples was run. Based on replicate SIO air standards (2 liters) and replicate 2-liter samples taken in the field, the uncertainty in analysis was found to be primarily dependent on the uncertainty introduced in the separation procedure. This was determined to be approximately +/- 1 permil. The mass spectrometric determination error was far smaller than this value and is included in the +/- 1 permil value. Actual runs of the mass spectrometer using the working standard (-43.40 +/- 0.05 permil), measured in the size range of the samples, yielded a running precision of better than +/- 0.1 permil.

For samples of higher methane concentration from production sites, the variability for the sample size processed again limited the error to approximately +/- 2 permil (PDB) for carbon and +/- 5 permil Standard Measure Ocean Water (SMOW) for hydrogen isotopic composition.

4.2.1.1 Tolerance

Samples with a methane concentration less than 0.75 µl methane/sample were not reliable for carbon isotopes measurements. Samples with less then 250 µl methane/sample did not produce reliable hydrogen isotope results.

4.2.2 Frequency of Calibration

Individual GC systems and the mass spectrometer were calibrated daily using additional standards in the middle of the GC runs. Separation and combustion procedures were calibrated biweekly or more often if necessary.

4.2.3 Other Calibration Information

The mass spectrometric standards are directly traceable to the National Institute of Standards Technology (NIST) primary isotope standards: National Bureau of Standards (NBS)-19, NBS-16, and NBS-17 for ^{13}C, SMOW, Standard Light Antarctic Precipitation (SLAP), Greenland Ice Sheet Precipitation (GISP) for D. Methane concentrations were measured against SIO/CSIRO (R. Weiss) substandards.

5. Data Acquisition Methods

Soil gas exchange at the NSA was measured using either aluminum enclosures at OJP or plastic enclosures at YJP of 0.4 m^2 and 0.075 m^3 or 0.05 m^2 and 0.018 m^3, respectively. The chambers rested on skirts permanently placed 10 cm deep in the soil in 1993 or 1994 and were equipped with a water seal to prevent air leakage. Once the chambers were in place, the air was mixed approximately every 5 minutes either with a fan or by rapidly drawing and returning 50 cc of air several times with a syringe. After a suitable time period (0.75 to 2 hr, in most cases), air samples were withdrawn through a restricting manifold, to prevent a sudden vacuum in the chamber, into 2-liter evacuated glass flasks with high vacuum valves. After the sampling manifold was attached to the sample flask, it was evacuated to remove extra air and to check for leaks, before the sample was taken. Subsurface gas samples were taken using a pointed 3/8-inch steel probe inside of which was a 1/8-inch tube connected to a nose chamber open to the soil through several small holes. This probe was pushed into the soil to the desired sampling depth, the manifold was attached and evacuated, and the sample flask (as above) was filled. For the subsurface samples, the manifold was set to allow approximately 3 minutes for the flask to fill. For a few of the 1994 soil gas samples, horizontal 1/8-inch sample tubes (TGB-03) previously placed at several depths in the soil were used following the same procedures.

Samples taken for methane production from submerged sediments were primarily obtained by capturing bubbles that had either been released over time or were dislodged by motion from the surface of the sediment. The gas from these samples (20 to 100 cc) was transferred to evacuated serum bottles with rubber septum caps. Several sets of samples equilibrated with helium gas from water samples above the sediments or piezometers in the sediments were also collected.

Methane concentration analyses were performed with a flame ionization GC equipped with a 2-m, 1/8-inch Porapack Q (60-80 mesh) column using nitrogen as a carrier gas. Replicate peak height measurements from a chart recorder were used to quantify the results. The inlet line to the sample loop was equipped with a precision pressure gauge calibrated to 0.5%. Standards at 959, 1649, and 2722 ppb were run at several pressures, several times during sample analyses, to allow correction for any deviation in linearity as a function of pressure or concentration.

Separation and combustion of the methane in the chamber and soil gas samples to CO_2 for the mass spectrometric analysis was performed on an extraction line specifically designed for this type of sample. The 2-liter (air) sample was passed at 50 cc/min or less through two liquid nitrogen (LN_2)-cooled traps to remove water, CO_2, and other condensable gases, and then through a trap containing several grams of 10 mesh activated charcoal. This charcoal column retains 500-800 cc of air (N_2, O_2) at any time, with methane and other trace components totally retained. After the entire sample was processed (down to <10 torr) and pumped at vacuum for 15 minutes, the charcoal was warmed from LN_2 temperature to approximately -150 °C over a period of 5 minutes. During this time, the majority of the air retained on the charcoal was allowed to bleed off at a carefully controlled rate, while the methane was retained. Then the charcoal column was immersed in a -90 °C acetone bath and the air allowed to continue to bleed off at a controlled rate, again retaining the methane. A low flow of helium is passed through the column for 1 min to dislodge additional air. The gas on the charcoal was then

transferred to a smaller charcoal column (approx. 1 g) in LN_2 by first warming it to room temperature for 10 minutes and then heating it 240 °C for 10 minutes.

In a similar manner, the second charcoal column was warmed and again any remaining excess air was removed. Helium was passed through the first column onto the second to maximize the transfer. At this stage, 1 to 2 cc of total gas containing approximately 3.7 microliters (or less) of methane (corresponding to the methane in 2 liters at ambient air) remained. This sample was transferred to an inlet loop of a thermal conductivity GC fitted with a special low flow MS5a column using helium carrier gas. The column and detector were modified so that a nearly complete separation of methane from nitrogen could be obtained with an elution time of 25 minutes at room temperature. The methane eluting from the column was captured on a short MS5a column and transferred, following removal of the residual helium, with an MS5a finger to the combustion system. The combustion of the methane was done by condensing the methane sample at LN_2 temperature onto two aluminum oxide pellets coated with platinum (Alfa #89106), and then condensing a tenfold excess of pure oxygen onto the pellets, followed by a combustion of the mixture at 650 °C for 1 hour. The CO_2 was then separated from the water produced in a trap at -70 °C and sealed in a 6-mm glass break tube for later mass spectrometric analysis. Extensive work was necessary to initially clean the pellets, and then keep them clean, resulting in a low CO_2 blank for this step. Samples with methane concentration higher than 10 ppm required proportionately smaller than 2-liter air samples to be analyzed. Samples in the percent methane range were able to be processed using similar GC steps and combustion; however, when more then 250 µl of water were generated, the water was saved for D isotope analysis.

6. Observations

6.1 Data Notes

None given.

6.2 Field Notes

Sampling NSA 1993

24-Aug-93	Bubbles from dislodgment Fen Tower walkways 1, 2, and 3.
25-Aug-93	Bubbles, Gilliam Rd. beaver pond (14.5 km N of 391) west shore.
26-Aug-93	Bubbles, OBS beaver ponds #3, 4, and 1, TBP, south shore. All samples from near shore,water depths 10-50 cm, various degrees of plants and algal growth.

Sampling SSA 1994

4-Aug-94	YJP Tower Flux (TF)-04 chambers A, B, and D soil probes A/B 15 cm, 20 cm. OJP TF-4chambers G, H, and J probes G/H 15 cm, 20 cm. Samples taken by R. Strieglís TF-04group, chamber times long (12+ hr).

Sampling NSA 1994

24-Aug-94	OJP, three chambers at TGB-01 lichen site, no collars, three soil gas probes. YJP,three chambers TGB-03 with collars #4, 5, and 6, five soil probes 1-2 meters southof TBG-03 soil tubes.
25-Aug-94	TBP samples from funnels, piezometers, water profile. BOREAS AES 2-ppm and 0.9-ppmstandards sampled.
26-Aug-94	YJP TGB-03 soil tubes site 1c and 3a sampled at 20 and 40 cm depth. Ambient air sampledeach day.
31-Aug-94 through 13-Sep-94	TBP samples, gross bubbles, funnel, piezometer, and water samples. Taken by A. DoveTGB-03.

Sampling NSA 1996

29-May-96 to 3-Jun-96	Ground frozen below 15 cm, surface soil <10 °C with a high degree of watersaturation. One sample taken at each chamber OJP 4 and 7, YJP 4 and 6, OJP at approximately4.5-hr sampling time, YJP at approximately 6 hr. No soil probe samples.
28 and 29-Jun-96	One sample collected from each chamber (above) over 1.5 to 2 hr. No soil gas samples.
1-Aug-96 to 4-Aug-96	General surface temperatures approximately 25 °C, weather good and dry. Duplicatesamples at 45 and 90 min for both OJP chambers, four soil probe samples, two neareach chamber at approximately 20 cm depth. Sampling the YJP site chambers was identicalexcept for collection times of 1 and 2 hr.
8-Sep-96 to 12-Sep-96	OJP sampled 8-Sep, light showers two days prior, chamber samples collected in duplicateat 1 and 2 hr. Soil samples, two near each chamber at approximately 15-cm depth.Cold heavy rain for two days. OJP samples taken on 11-Sep, temperature <9 °C.Single samples at 1 hr, duplicate samples collected each chamber 1 and 2 hr. Twosoil probe samples taken 15 and 17-cm depth. Two ambient air samples collected.

7. Data Description

7.1 Spatial Characteristics

7.1.1 Spatial Coverage

The data for 1993 were sampled as a preliminary examination of production from the available pond and fen locations. This set of analyses (dislodged bubbles) was not repeated in subsequent years. The data for 1994 and 1996 were collected from specific soil sites at the tower locations in the NSA and SSA, OJP and YJP, and the NSA. The North American Datum of 1983 (NAD83) coordinates for the sites are:

NSA Tower Fen (55.91481N, 98.42072W)
NSA Beaver Pond Gillam Road 14.5-km N of Highway 391 on Gillam Road
NSA OBS Beaver ponds (55.88007N, 98.48139W)
NSA TBP (55.84225N, 98.02747W)
NSA OJP (55.92847N, 98.62396W)
NSA YJP (55.89575N, 98.28706W)
NSA TBP (55.84225N, 98.02747W)
SSA OJP (53.91634N, 104.69203W)
SSA YJP (53.87581N, 104.64529W)

7.1.2 Spatial Coverage Map

Not available.

7.1.3 Spatial Resolution

These data represent point measurements from the 2-m x 2-m sample areas.

7.1.4 Projection

Not applicable.

7.1.5 Grid Description

Not applicable.

7.2 Temporal Characteristics

7.2.1 Temporal Coverage

Data were collected over the SSA sites in August 1994 only. Data were collected over the NSA sites at various times in 1993, 1994, and 1996.

7.2.2 Temporal Coverage Map

Site Name	Date
NSA Tower Fen	24-Aug-93
NSA Beaver Pond Gillam Road	25-Aug-93
NSA-TBP	26-Aug-93
	31-Aug-94
	13-Aug-94
	25-Aug-94
SSA-OJP	4-Aug-94
SSA-YJP	4-Aug-94
NSA-OJP	23-Aug-94
	30-May-96
	2-Aug-96
	11-Sep-96
	28-Jun-96
NSA YJP	24-Aug-94
	26-Aug-94
	3-Jun-96
	3-Aug-96
	8-Sep-96
	28-Jun-96

7.2.3 Temporal Resolution

Individual samples were taken over several hours of field time at each site. Production samples from funnels and piezometers represent collection or equilibration over several days.

7.3 Data Characteristics

7.3.1 Parameter/Variable

The parameters contained in the data files on the CD-ROM are:

Column Name
SITE_NAME
SUB_SITE
DATE_OBS
SAMPLE_TYPE
SAMPLE_LOCN
MEASUREMENT_DURATION
SOIL_DEPTH
CH4_CONC
ISOTOPE_13C
ISOTOPE_H
CRTFCN_CODE
REVISION_DATE

7.3.2 Variable Description/Definition

The descriptions of the parameters contained in the data files on the CD-ROM are:

Column Name	Description
SITE_NAME	The identifier assigned to the site by BOREAS, in the format SSS-TTT-CCCCC, where SSS identifies the portion of the study area: NSA, SSA, REG, TRN, and TTT identifies the cover type for the site, 999 if unknown, and CCCCC is the identifier for site, exactly what it means will vary with site type.
SUB_SITE	The identifier assigned to the sub-site by BOREAS, in the format GGGGG-IIIII, where GGGGG is the group associated with the sub-site instrument, e.g. HYD06 or STAFF, and IIIII is the identifier for sub-site, often this will refer to an instrument.
DATE_OBS	The date on which the data were collected.
SAMPLE_TYPE	Type of medium for the sample.
SAMPLE_LOCN	Specific location where the sample was measured.
MEASUREMENT_DURATION	Length of time for the measurement.
SOIL_DEPTH	The depth below the soil surface at which the measurement was taken.
CH4_CONC	CH4 concentration.
ISOTOPE_13C	Carbon 13/12 isotope ratio.
ISOTOPE_H	Hydrogen D/H isotope value of methane.
CRTFCN_CODE	The BOREAS certification level of the data. Examples are CPI (Checked by PI), CGR (Certified by Group), PRE (Preliminary), and CPI-??? (CPI but questionable).
REVISION_DATE	The most recent date when the information in the referenced data base table record was revised.

7.3.3 Unit of Measurement

The measurement units for the parameters contained in the data files on the CD-ROM are:

Column Name	Units
SITE_NAME	[none]
SUB_SITE	[none]
DATE_OBS	[DD-MON-YY]
SAMPLE_TYPE	[none]
SAMPLE_LOCN	[none]
MEASUREMENT_DURATION	[minutes]
SOIL_DEPTH	[millimeters]
CH4_CONC	[parts per million]
ISOTOPE_13C	[permil]
ISOTOPE_H	[permil]
CRTFCN_CODE	[none]
REVISION_DATE	[DD-MON-YY]

7.3.4 Data Source

The sources of the parameter values contained in the data files on the CD-ROM are:

Column Name	Data Source
SITE_NAME	Investigator
SUB_SITE	Investigator
DATE_OBS	Investigator
SAMPLE_TYPE	Investigator
SAMPLE_LOCN	Investigator
MEASUREMENT_DURATION	Investigator
SOIL_DEPTH	Investigator
CH4_CONC	gas chromatograph
ISOTOPE_13C	VG Prism II
ISOTOPE_H	VG Prism II
CRTFCN_CODE	Assigned by BORIS
REVISION_DATE	Assigned by BORIS

7.3.5 Data Range

The following table gives information about the parameter values found in the data files on the CD-ROM.

Column Name	Minimum Data Value	Maximum Data Value	Missng Data Value	Unrel Data Value	Below Detect Limit	Data Not Cllctd
SITE_NAME	NSA-999-T06GR	SSA-YJP-FLXTR	None	None	None	None
SUB_SITE	TGB06-CON01	TGB06-CON01	None	None	None	None
DATE_OBS	24-AUG-93	11-SEP-96	None	None	None	None
SAMPLE_TYPE	N/A	N/A	None	None	None	None
SAMPLE_LOCN	N/A	N/A	None	None	None	None
MEASUREMENT_DURATION	0	960	None	None	None	None
SOIL_DEPTH	0	10000	None	None	None	None
CH4_CONC	-197.98	3.3	None	None	None	None
ISOTOPE_13C	-68.3	-30.8	-999	None	None	None
ISOTOPE_H	-384	-224	-999	None	None	Blank
CRTFCN_CODE	CPI	CPI	None	None	None	None
REVISION_DATE	12-JUN-97	12-JUN-97	None	None	None	None

Minimum Data Value -- The minimum value found in the column.
Maximum Data Value -- The maximum value found in the column.
Missng Data Value -- The value that indicates missing data. This is used to indicate that an attempt was made to determine the parameter value, but the attempt was unsuccessful.
Unrel Data Value -- The value that indicates unreliable data. This is used to indicate an attempt was made to determine the parameter value, but the value was deemed to be unreliable by the analysis personnel.
Below Detect Limit -- The value that indicates parameter values below the instruments detection limits. This is used to indicate that an attempt was made to determine the parameter value, but the analysis personnel determined that the parameter value was below the detection limit of the instrumentation.
Data Not Cllctd -- This value indicates that no attempt was made to

```
                    determine the parameter value.  This usually
                    indicates that BORIS combined several similar but
                    not identical data sets into the same data base table
                    measure that parameter.

Blank -- Indicates that blank spaces are used to denote that type of value.
N/A   -- Indicates that the value is not applicable to the respective column.
None  -- Indicates that no values of that sort were found in the column.
------------------------------------------------------------------------------
```

7.4 Sample Data Record

The following are wrapped versions of sample data records from a selected file on the CD-ROM:

```
SITE_NAME,SUB_SITE,DATE_OBS,SAMPLE_TYPE,SAMPLE_LOCN,MEASUREMENT_DURATION,
SOIL_DEPTH,CH4_CONC,ISOTOPE_13C,ISOTOPE_H,CRTFCN_CODE,REVISION_DATE
'NSA-OJP-FLXTR','TGB06-CON01',24-AUG-94,'chamber_flux','OJP_4-1',82,0.0,1.838,
-46.1,,'CPI',12-JUN-97
'NSA-OJP-FLXTR','TGB06-CON01',24-AUG-94,'chamber_flux','OJP_4-1',147,0.0,1.798,
-46.7,,'CPI',12-JUN-97
```

8. Data Organization

8.1 Data Granularity

The smallest unit of data tracked by the BOREAS Information System (BORIS) is the measurements made for a given site on a given day.

8.2 Data Format(s)

The Compact Disk-Read-Only Memory (CD-ROM) files contain American Standard Code for Information Interchange (ASCII) numerical and character fields of varying length separated by commas. The character fields are enclosed with single apostrophe marks. There are no spaces between the fields.

Each data file on the CD-ROM has four header lines of Hyper-Text Markup Language (HTML) code at the top. When viewed with a Web browser, this code displays header information (data set title, location, date, acknowledgments, etc.) and a series of HTML links to associated data files and related data sets. Line 5 of each data file is a list of the column names, and line 6 and following lines contain the actual data.

9. Data Manipulations

9.1 Formulae

None.

9.1.1 Derivation Techniques and Algorithms

None.

9.2 Data Processing Sequence

The data, after calibration of the measurement, were not modified, except for the Craig correction applied by the mass spectrometer data calculation of d ^{13}C.

9.2.1 Processing Steps

None.

9.2.2 Processing Changes
None.

9.3 Calculations

9.3.1 Special Corrections/Adjustments
None.

9.3.2 Calculated Variables
None.

9.4 Graphs and Plots
None.

10. Errors

10.1 Sources of Error
Data quality appears to be high for most of the data set, although the chambers from the 1994 SSA and May/June 1996 were left closed far longer than the normal 2 hours. Low consumption of methane in the soils resulted in final concentrations of methane in the chambers somewhat higher than desired. The 1994 OJP samples were not tightly sealed to the ground and probably exchanged with the outside air, resulting in low apparent consumption. The duplicate samples in 1996 were used to determine the analytical error. Soil methane flux measurements using these data are lower than the values determined by other groups that sampled for shorter periods of time.

Errors in these data are a combination of sample transport, storage, processing, and analyses. The greatest uncertainty in any of the data results from the synoptic nature of the discrete sampling. Samples taken in proximity to one another varied widely in their data values, often with results differing by more than any combination of experimental errors would allow. In this study, sample storage, processing, and analysis procedures were checked before and during the field work using standards and blank determinations, and the associated error was determined. Errors introduced during sampling could be inferred from several conditions, such as lack of good seals on chambers or long chamber deployments, which did not reproduce the normal planned conditions of sampling. Where there are indications that this happened, it has been noted. Analysis of some of the data, such as the methane production samples, shows an enormous range of values, in part because they were sampled in several different ways at many different sites. Also, questions such as dislodged bubbles representing the release of methane are not considered. Lacking a more complete data set describing many additional parameters influencing methane production/consumption, analysis of even the error from this type of sample is difficult.

10.2 Quality Assessment

10.2.1 Data Validation by Source
Prior studies and controls introduced during this work show that the sample storage, concentration analysis, and the mass spectrometric determinations introduced relatively small errors into the determinations. The separation and combustion procedure were the most important steps in the error determination and accounted for the majority of error in the analysis.

10.2.2 Confidence Level/Accuracy Judgment
For all the production samples, the error in analysis is +/- 2 permil vs. PDB for d ^{13}C in carbon and +/- 5 permil in dD in hydrogen, based on replicates and standards. No estimate of the errors introduced by the sampling procedure is made.

For the chamber and soil gas samples, the error in carbon isotope determination is +/- 1 permil from the total analysis procedure, over 80% of which is related to the separation step. Sampling "errors" for chamber samples were probably minimal for enclosure times less than 2 hr., but this is only a comparison to a nondisturbed system. Soil gas samples had a real error associated with the sample depth determination, +/- 5 cm, because of the soil character.

10.2.3 Measurement Error for Parameters

Production samples, 2 permil (PDB) carbon, 5 permil (SMOW) hydrogen. Chamber and soil gas samples, 1 permil (PDB) carbon; except samples with concentrations below 500 ppbv, 2 permil (PDB) d ^{13}C in carbon. Soil depth for probe +/- 5 cm.

10.2.4 Additional Quality Assessments

None given.

10.2.5 Data Verification by Data Center

Data were examined for general consistency and clarity.

11. Notes

11.1 Limitations of the Data

The data as presented are not correlated with many of the other parameters necessary for evaluation. These parameters were collected by other investigators and must be processed and included with these data. The synoptic nature of the data must be understood. Only the chamber and soil gas data in 1996 cover any part of the seasonal cycle.

11.2 Known Problems with the Data

The production data from 1993 do not have much supporting data from other investigators, and several of the sites were not sampled at later times. Almost all of the prodded/dislodged bubble samples were taken near the shore, where plant root activity would be a major variable, but a quantification of this variable has not been made. The samples from the SSA 1994 chambers were left down far longer than would have been desired, and the results were most probably influenced by this. The May 1996 chambers were sampled when the ground was still frozen and saturated with water, and were in place longer than desired. This may have resulted in unusual conditions.

11.3 Usage Guidance

None given.

11.4 Other Relevant Information

In a large project like this, the number of sites that needed to be covered was greater than the group's ability and funding. Promised help in field sampling was good at times and lacking in other cases. Coordination of this effort, which required primarily lab time, with those of the other investigators, who worked primarily in the field, proved difficult.

12. Application of the Data Set

The data, while still under analysis, demonstrate the methane fractionation occurring with soil oxidation/consumption. When the approximate methane consumption flux is calculated for the representative soil types, the effect on the atmospheric methane isotope composition can be determined. The probability of a concurrent methane production occurring with methane consumption in the soils is suggested by some of the data.

13. Future Modifications and Plans

The data presented here need to be combined with other available parameters that were measured by other investigators.

14. Software

14.1 Software Description

Not applicable.

14.2 Software Access

Not applicable.

15. Data Access

The TGB-06 soil methane data are available from the Earth Observing System Data and Information System (EOSDIS) Oak Ridge National Laboratory (ORNL) Distributed Active Archive Center (DAAC).

15.1 Contact Information

For BOREAS data and documentation please contact:

ORNL DAAC User Services
Oak Ridge National Laboratory
P.O. Box 2008 MS-6407
Oak Ridge, TN 37831-6407
Phone: (423) 241-3952
Fax: (423) 574-4665
E-mail: ornldaac@ornl.gov or ornl@eos.nasa.gov

15.2 Data Center Identification

Earth Observing System Data and Information System (EOSDIS) Oak Ridge National Laboratory (ORNL) Distributed Active Archive Center (DAAC) for Biogeochemical Dynamics http://www-eosdis.ornl.gov.

15.3 Procedures for Obtaining Data

Users may obtain data directly through the ORNL DAAC online search and order system [http://www-eosdis.ornl.gov/] and the anonymous FTP site [ftp://www-eosdis.ornl.gov/data/] or by contacting User Services by electronic mail, telephone, fax, letter, or personal visit using the contact information in Section 15.1.

15.4 Data Center Status/Plans

The ORNL DAAC is the primary source for BOREAS field measurement, image, GIS, and hardcopy data products. The BOREAS CD-ROM and data referenced or listed in inventories on the CD-ROM are available from the ORNL DAAC.

16. Output Products and Availability

16.1 Tape Products

None.

16.2 Film Products

None.

16.3 Other Products

These data are available on the BOREAS CD-ROM series.

17. References

17.1 Platform/Sensor/Instrument/Data Processing Documentation

Instrument manual, VG Prism II, Micro Mass, Beverly, MA.

17.2 Journal Articles and Study Reports

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Wahlen, M., N. Tanaka, B. Deck, and R. Henry. 1990. dD in Methane; Additional constraints of a global CH_4 Budget. Trans. Am. Geo. Union 71, 43, pg. 1249.

Wahlen, M., N. Tanaka, R. Henry, B. Deck, J. Zeglen, J.S. Vogel, J. Southon, A. Shemesh, R. Fairbanks, and W. Broecker. 1989. Carbon-14 in methane sources and in atmospheric methane: The contribution from fossil carbon. Science, 245, 286-290.

17.3 Archive/DBMS Usage Documentation

None.

18. Glossary of Terms

- Chamber/Enclosure - A sealed space in contact with the soil. Usually mounted on a skirt dug into the soil.
- LN_2 - Liquid nitrogen.
- NBS 19, NBS 16, NBS 17 - Reference standards for d ^{13}C in carbon provided by NIST, formerly known as NBS.
- PDB - The reference standard for ^{13}C isotope of carbon. 0 permil.
- Permil - Description of the isotopic composition of a sample relative to a standard.
- SMOW, SLAP, GISP - Reference standards for dD in hydrogen provided by NIST.
- Soil probe - A metal tube pushed to depth in the soil allowing soil gas samples to be taken.

19. List of Acronyms

```
AES      - Atmospheric and Environment Service
ASCII    - American Standard Code for Information Interchange
BOREAS   - BOReal Ecosystem-Atmosphere Study
BORIS    - BOREAS Information System
CD-ROM   - Compact Disk-Read-Only Memory
CSIRO    - Commonwealth Scientific and Industrial Research Organization
           (Division of Atmospheric Research, Australia)
DAAC     - Distributed Active Archive Center
EOS      - Earth Observing System
EOSDIS   - EOS Data and Information System
GC       - Gas Chromatograph
GIS      - Geographic Information System
GISP     - Greenland Ice Sheet Precipitation
GSFC     - Goddard Space Flight Center
HTML     - HyperText Markup Language
NAD83    - North American Datum of 1983
NASA     - National Aeronautics and Space Administration
NBS      - National Bureau of Standards (now NIST)
NIST     - National Institute for Standards and Technology
NSA      - Northern Study Area
OA       - Old Aspen
OBS      - Old Black Spruce
OJP      - Old Jack Pine
ORNL     - Oak Ridge National Laboratory
PANP     - Prince Albert National park
PDP      - Pee Dee Belemnite
SIO      - Scripps Institution of Oceanography
SLAP     - Standard Light Antarctic Precipitation
SMOW     - Standard Measure Ocean Water
SSA      - Southern Study Area
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TBP       - Tower Beaver Pond (NSA)
TF        - Tower Flux
TFEN      - Tower Fen (NSA)
TGB       - Trace Gas Biogeochemistry
UCSD      - University of California, San Diego
URL       - Uniform Resource Locator
YJP       - Young Jack Pine
```

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M. Wahlen and B. Deck, Scripps Institution of Oceanography, University of California San Diego, La Jolla CA 92093-0220

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Bruce Deck and Martin Wahlen
Forrest G. Hall and Sara K. Conrad, Editors

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13. ABSTRACT *(Maximum 200 words)*

The BOREAS TGB-6 team collected soil methane measurements at several sites in the SSA and NSA. This data set contains soil methane consumption (bacterial CH_4 oxidation) and associated ^{13}C fractionation effects in samples that were collected at various sites in 1994 and 1996 from enclosures (chambers). Methane ^{13}C data in soil gas samples from the NSA YJP and OJP sites for 1994 and 1996 are also given. Additional data on the isotopic composition of methane (carbon and hydrogen isotopes) produced in the NSA beaver ponds and fen bog in 1993 and 1994 are given as well. The data are stored in tabular ASCII files.

14. SUBJECT TERMS
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