



# Soils Data Related to the 1999 FROSTFIRE Burn

By K.L. Manies, J.W. Harden, and R. Ottmar

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## Abstract

This report describes the sample collection and processing for U.S. Geological Survey efforts at FROSTFIRE, an experimental burn that occurred in Alaska in 1999. Data regarding carbon, water, and energy dynamics pre-fire, during, and post-fire were obtained in this landscape-scale prescribed burn. U.S. Geological Survey investigators measured changes in the stocks of carbon (C), nitrogen (N), mercury (Hg), and other components in pre- and post-burn soils of this watershed.

## Introduction

FROSTFIRE was a landscape-scale prescribed burn in Alaska, the goal of which was to study the large-scale consequences of fire on the C, water, and energy pools in boreal forests both during and after the fire. Of the 2,500 acres prescribed to burn, approximately 900 acres were consumed, the majority of which was black spruce (*Picea mariana* (Mill.) BSP) forest. More than 50 research teams studied a variety of topics, including the impacts of weather and vegetation on fire, the impacts of fire on CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub>O fluxes, as well as the impacts of fire on stream chemistry. FROSTFIRE was developed, in part, because boreal forests contain the majority of the world's soil organic C (McGuire and others, 2009; Schuur and others, 2008; Tarnocai and others, 2009) and play an important role in the global C cycle. Fire affects C storage of the boreal forest both directly (fire emissions, loss of vegetation) and indirectly. Indirect effects include altering the soil-temperature regime through changes in albedo and the loss of organic layers, which play an important role in soil insulation (O'Neill and others, 2002). Changes in soil temperature in turn affect decomposition rates (Wickland and others, 2010) and vegetative regrowth (Johnstone and others, 2010). Therefore, a large-scale burn was needed to permit accurate analysis of the short- and long-term impact of fire on these ecosystems.

As a part of the FROSTFIRE project, U.S. Geological Survey (USGS) investigators measured changes in the stocks of C, nitrogen (N), mercury (Hg), and other components in pre- and post-burn soils. The data presented here represent the USGS sampling effort, as well as data from organic soils sampled by the U.S. Forest Service (USFS) Fire and Environmental Research Applications (FERA) team that were shared with USGS. Results from this study were published in Harden and others (2004) and Turetsky and others (2006).

## Site Descriptions

The sites were located within the Caribou-Poker Creeks Research Watershed (CPCRW), which is ~45 km north of Fairbanks (fig. 1), Alaska, and managed by the Bonanza Creek Long-Term Ecological Research (LTER) program. CPCRW is comprised of birch and aspen stands on south-facing slopes and black-spruce dominated stands on north-facing slopes. The soils have a thin cap of loess over discontinuous permafrost.

Four USFS sites were used to characterize the pre- and post-burn organic soils (table 1). Within each site, eighteen plots were positioned 20.1 m (2 chains) apart along two transects established perpendicular to the slope. Sixteen depth-of-burn or consumption rods were systematically positioned around each plot to mark the surface of the organic soils (fig. 2). These pins were remeasured following the fire to determine organic soil-depth reduction. Pre-burn soil samples were located randomly along these transects. The burning of FROSTFIRE occurred July 8–15, 1999. Initial post-burn soil sampling occurred once the watershed was open to scientists at locations adjacent to the severity pins. Additional post-burn sampling of the lower black spruce (LBS) site occurred in May of 2000.

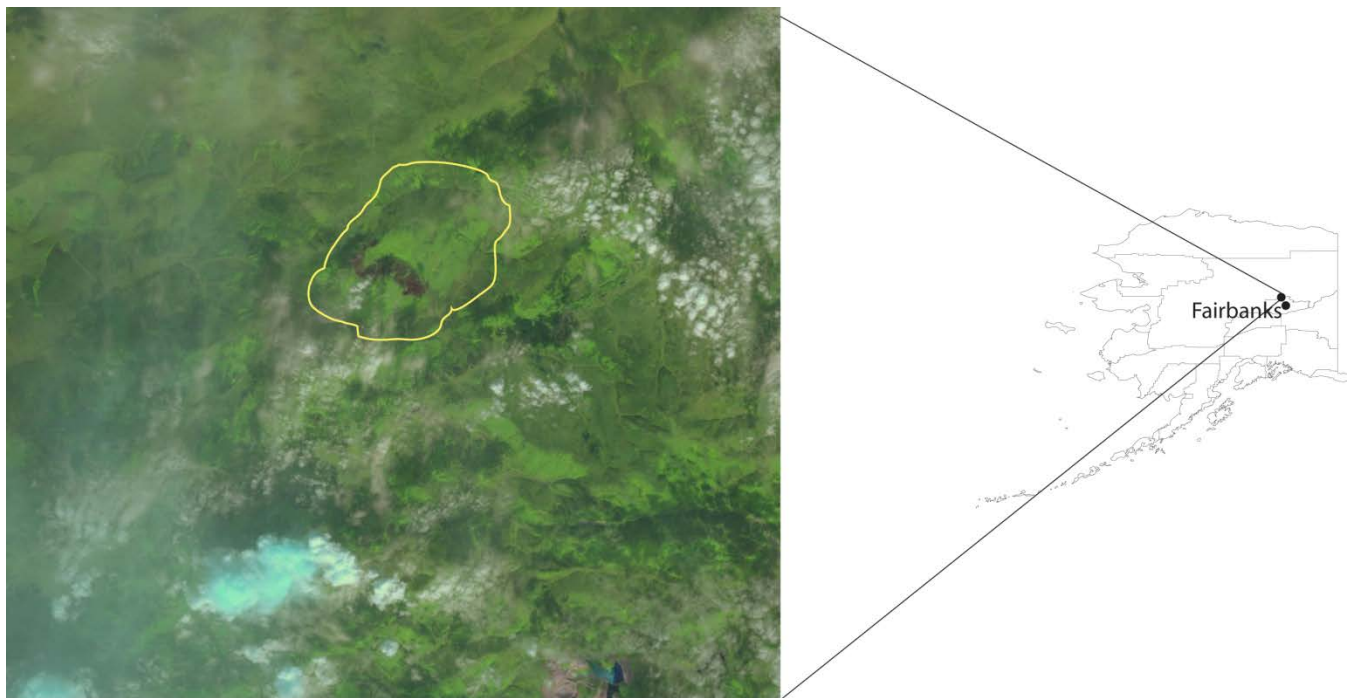


Figure 1. Landsat image of the area that burned during FROSTFIRE (red part of image) within the approximate boundary of the Caribou-Poker Creeks Research Watershed (CPCRW). The CPCRW is found within interior Alaska.

Table 1. List of sites located within the FROSTFIRE watershed.

USFS Code	USFS name	General location
FFS2	Lower black spruce (LBS)	Located at the base of the watershed along Poker Creek
FFS3	Birch #2 (BIR)	Located on a south facing hillslope above Poker Creek
FFS4	Sloped black spruce (SLBS)	Located along a north facing hillslope below FFS5
FFS5	Upper black spruce (UPBS)	Located at the top of the hillslope near Helmer's Ridge

[USFS, U.S. Forest Service

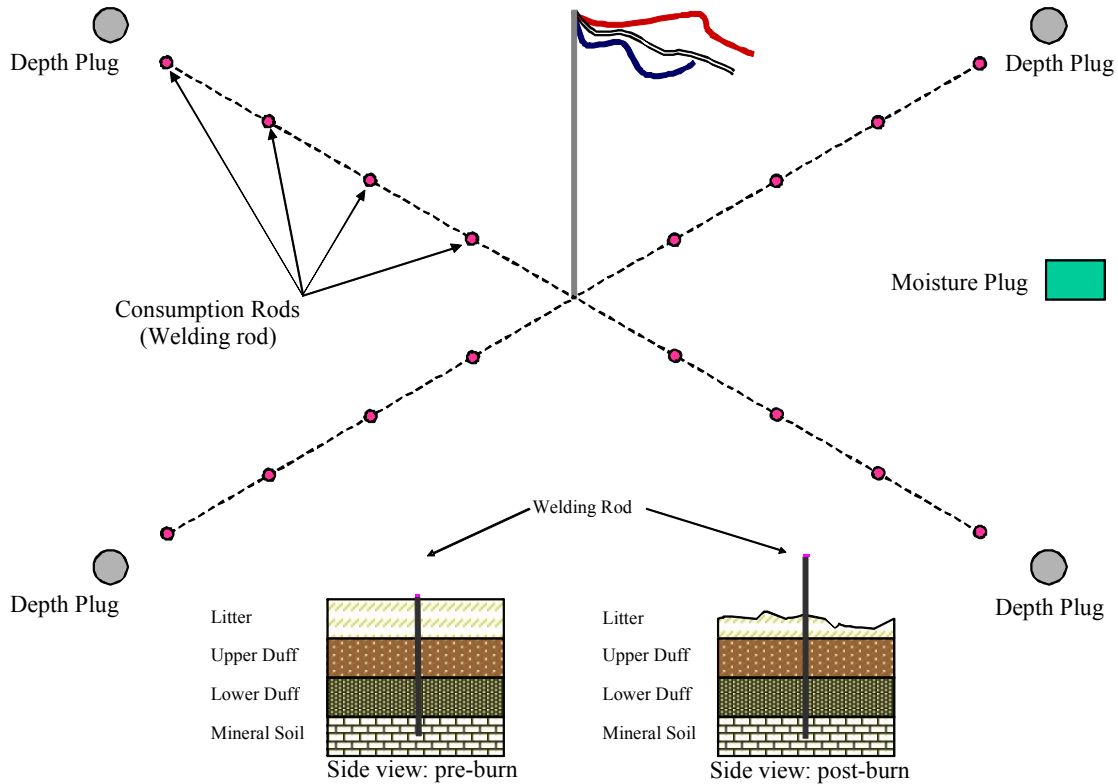


Figure 2. A schematic showing the layout of the U.S. Forest Service (USFS) sites, which were used in this study by both USFS and U.S. Geological Survey (USGS) investigators for their pre- and post-burn sampling strategy.

## Sample Collection & Processing

Pre-burn soil samples collected by USGS personnel were characterized by describing the individual horizons, or layers, to the depth at which the soil was frozen. (Since these sites were initially sampled in May, frozen soil was encountered before mineral soil was reached.) Bulk soil (or combined horizons) was then sampled volumetrically to a depth greater than that which the fire was predicted to burn. Each of these samples was divided into two subsamples: a moisture sample and a bulk-density/analytical sample. Post-burn samples collected in 1999 and 2000 were obtained by cutting blocks of organic soil of known dimensions from the forest floor and subdividing these blocks into distinct soil horizons. The entire sample was used for moisture, bulk density, and analytical purposes for the 1999 post-burn samples. In 2000,

these samples were only used for bulk density and analytical purposes and a separate moisture subsample was taken adjacent to the initial sampling location.

Regardless of sampling method, all samples were air dried to a constant mass. Next, a split of the sample or the moisture subsample was oven dried at 65°C. The analytical subsample/split was not oven dried, but these data can be converted to an oven-dry basis using gravimetric moisture content in the air dried sample. Analytical splits were weighed and roots wider than 1 cm in diameter were removed, weighed, and saved separately. The remaining sample was then milled in an Udy Corp. Cyclone mill (Fort Collins, Colorado) to pass through a 0.5-mm screen. The milled sample was thoroughly mixed and a representative sample was placed in a labeled, glass sample bottle for chemical analysis. Archive fractions of most of the samples are available by contacting J.W. Harden at the USGS Menlo Park, California office.

Table 2. Organic soil horizon codes and descriptions, FROSTFIRE burn, Alaska.

Horizon code	General description
LM	Live moss: green moss layers, leaf and needle litter.
DM	Dead moss: undecomposed or very slightly decomposed moss. Few roots.
UD	Upper duff: fibric layers in which roots are more abundant than recognizable moss parts. They would be classified as fibric layers (Canadian system) or Oi layers (U.S. system).
LD	Lower duff: well decomposed organics with no or little recognizable plant parts. They would be mesic or humic layers (Canadian system) or Oa layers (U.S. system).
LT	Litter: hardwood leaf and twig litter.
LDF	Combined horizon of live moss, dead moss, and upper duff.
LTF	Combined horizon of litter, dead moss, and upper duff.
L	Live moss, generally green. May contain leaf and needle litter.
D	Dead moss. Not decomposed or slightly decomposed. Few, if any, roots.
F	Fibric organics, with roots often more abundant than plant parts.
M	Mesic, or moderately decomposed, organics. Amorphous material present.
H	Humic, or highly decomposed, organics. Smears upon squeezing.
X	Unknown
Ash	Ash
A	Mineral soil with less than 20% organic matter, as judged in the field.
bX	A lower case b before a horizon code indicates that the layer has been burned.
fX	A lower case f before a horizon code indicates that the layer was frozen when sampled.

[These descriptions are modified from Canadian (Committee, 1998) and U.S. Department of Agriculture-Natural Resources Conservation Service (USDA-NRCS; Staff, 1998) methodologies.]

The week before the fire USFS personnel also collected pre-burn samples for determining water content. Some of these samples were obtained volumetrically by inserting a square metal box of a known volume into the forest floor. Different soil horizons (table 2) within the profile were separated and described. Horizons thicker than 5 cm were also divided. These samples were oven dried at 65°C and then sent to the USGS to be processed for chemistry in the same manner as the USGS pre-burn samples.

## Laboratory Methods

### Total Carbon, Total Nitrogen, $\delta^{13}\text{C}$ , $\delta^{15}\text{N}$

A Carlo Erba NA1500 elemental analyzer coupled to a Micromass Optima isotope ratio mass spectrometer (IRMS) in continuous flow mode was used to determine total C, total N,  $\delta^{13}\text{C}$ , and  $\delta^{15}\text{N}$ . Because carbonates are rare in this area and mineral-soil

pH values from a previous study were <7.0 (Rieger and others, 1972), nearly all of this C can be considered organic. Samples were combusted in the presence of excess oxygen. The resulting sample gasses were swept in a continuous flow of helium through an oxidation furnace, followed by a reduction furnace, to yield CO<sub>2</sub>, N<sub>2</sub>, and water vapor. Water was removed by a chemical trap, and CO<sub>2</sub> and N<sub>2</sub> were chromatographically separated before entering the Micromass Optima IRMS for the measurement of C, N, δ<sup>13</sup>C and δ<sup>15</sup>N. Carbon and N were determined by integrating the major ion peaks (mass 44 for CO<sub>2</sub>, and mass 28 for N<sub>2</sub>). For reliable quantification of δ<sup>15</sup>N, 15 to 30 μg N generally are needed, and few samples met this criterion. Thus, the δ<sup>15</sup>N data are not reported here; these data are available from the authors by request. The δ<sup>13</sup>C data are reported as deviations in parts per thousand (‰) relative to a standard, here Vienna Pee Dee Belemnite (V-PDB). δ<sup>13</sup>C was calculated as:

$$\delta^{13}\text{C} \text{ ‰} = \left( \left( \frac{{}^{13}\text{C sample}/{}^{12}\text{C sample}}{{}^{13}\text{C standard}/{}^{12}\text{C standard}} \right) - 1 \right) * 1000. \quad (1)$$

Table 3. Statistics of analyses for working standards run on the IRMS from 2001 through 2008.

Standard	%C			%N			<sup>13</sup> C		
	Avg.	Stdev.	N	Avg.	Stdev.	N	Avg.	Stdev.	N
MESS-1	3.02	0.15	49	0.18	0.01	49	-25.64	0.14	49
NBS-1645	5.21	0.39	53	0.09	0.01	55	-22.29	0.27	53

[Certified values: MESS-1 = 2.99 percent C; NBS-1645 = 0.08 percent.]

All samples were compared to a main working standard, ethylenediamine tetra-acetic acid (EDTA). The chemical formula for this compound corresponds to a C content of 41.09 percent and a N content of 9.59 percent. Two working standards were analyzed as samples in all runs to check consistency and overall precision: a marine sediment standard (MESS-1), issued by the Chemistry Division of the Canadian National Research Council, and a river sediment standard (NBS 1645), issued by the National Bureau of Standards, now the National Institute of Standards and Technology (NIST). Certified values were obtained from Govindaraju (1989). Measured values of these working standards for runs including FROSTFIRE and other samples can be found in table 3.

### <sup>14</sup>C Analyses

A subset of samples were run for <sup>14</sup>C activity. Most of these samples were chosen to aid in modeling soil turnover and(or) to partition carbon into more labile versus stable pools. The <sup>14</sup>C content of ground, untreated soil was measured by vacuum sealing a homogenized sample containing ~1 mg C with cupric oxide and elemental silver in a quartz tube. The sample was then combusted at 850°C, and the resulting CO<sub>2</sub> was purified cryogenically and reduced to graphite using a modified reduction method with titanium hydride, zinc, and cobalt catalyst (Vogel, 1992). The graphite target was measured directly for <sup>14</sup>C at W. M. Keck C Cycle Accelerator Mass Spectrometer (AMS) Laboratory at UC Irvine.

The <sup>14</sup>C data are expressed in Delta notation (Δ<sup>14</sup>C), which, similar to δ<sup>13</sup>C, expresses the deviation in the <sup>14</sup>C/C in parts per thousand (‰) as compared to the standard NIST Oxalic Acid I (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>), with additional correction for fractionation, based on generalized <sup>13</sup>C values (see Stuiver, 1980; Stuiver and Polach, 1977). Δ<sup>14</sup>C values also are presented as Fraction Modern (FM) values, calculated using the following equation:



$$FM = [(S - B) / (M - B)], \quad (2)$$

where B, S, and M represent the  $^{14}\text{C}/^{12}\text{C}$  ratios of the blank, the sample, and the modern reference, respectively. The modern reference is defined at 95 percent of the radiocarbon concentration (in AD 1950) of NBS Oxalic Acid I normalized to  $\delta^{13}\text{C}_{\text{VPDB}} = -19 \text{ ‰}$  (Olsson, 1970).

### Loss on Ignition, Elemental Analysis, and Mercury

Loss on Ignition (LOI) was measured by the USGS for the majority of samples. Air-dried, ground samples were heated at  $550^{\circ}\text{C}$  in a muffle furnace for five hours. The change in weight pre- and post-heating was used to calculate LOI. For 28 samples, LOI was measured by XRAL Laboratories (SGS Group, Toronto, Canada).

Elemental concentrations were analyzed using inductively coupled plasma (ICP) procedures at XRAL Laboratories. Samples analyzed for LOI at the USGS were submitted to XRAL as ash. These samples were analyzed using the 95 element ICP method (also known as ICP-95 and WR ICP). The remaining samples were submitted as ground soil and analyzed using the 40 element ICP (ICP-40) method. The two ICP procedures differ in that the ICP-95 method uses a lithium metaborate fusion followed by dissolution, while the ICP-40 method uses an acid dissolution. Therefore, if samples contain refractory minerals impervious to strong acid, the ICP-40 data will be lower than the ICP-95 data. We do not expect there to be a large difference between the two processes for our samples. Phosphorus data from the ICP-40 procedure were reported as percent P, while the ICP-95 data were reported as percent  $\text{P}_2\text{O}_5$ . Therefore, the percent P data was converted to percent  $\text{P}_2\text{O}_5$  data by multiplying percent P values by 2.2912. More information regarding the ICP-40 procedure, calibration techniques, and its detection limits can be found in Briggs (2002). More information regarding the ICP-95 procedure can be found in Marabini and others (1992).

Hg was analyzed by XRAL Laboratories. Samples were digested with mixtures of  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ ,  $\text{HCL}$ ,  $\text{KMnO}_4$  (5 percent), and  $\text{K}_2\text{S}_2\text{O}_8$  (5 percent). Excess  $\text{KMnO}_4$  was reduced using a hydroxylamine sulfate solution, and the resulting Hg (II) was reduced by a solution of  $\text{SnCl}_2$ . The Hg vapor was then separated and measured using a LEEMAN PS200 Automated Mercury Analyzer (H. De Souza, XRAL Laboratories, oral commun.).

### Sample Nomenclature

All samples are labeled with a code describing the site and location from which the samples were obtained. Pre-burn USGS samples have the site represented by the site code (table 1) followed by a “U”, indicating that the sample is unburned. A space separates the site information from further information. This space is followed by the USFS plot number nearest to the sample location. The number after the decimal point represents the basal depth (cm) of the sample. Pre-burn USFS samples have the site represented by the site name (table 1) followed by a space. This space is followed by the USFS sample number (based on the number of samples taken that day). The number after the decimal point is the basal depth (cm) of the sample.

Post-burn samples from 1999 were all taken at site FFS5 (also referred to as UPBS). This notation was shortened to FS5. The letter “A” after the site name indicates the sample was obtained post-burn. This information is followed by a space. The number after the space represents the plot number from which the sample was taken.

Within each plot were sixteen fire-severity pins. The number in the parentheses represents the pin number next to which the sample was taken. The number after the decimal point is the basal depth (cm) of the sample. The letter “R” after the sample means this sample is a duplicate taken post-rain. This extra sampling effort occurred because it rained lightly during the night between the two days of post-burn sampling. Some upper-surface profiles were resampled post-rain to test whether significant mixing or leaching of ash occurred during the rain.

## Dataset Descriptions

Four separate downloadable files contain the soil data collected from the sites described in this report. The first file, *1\_FROSTFIRE\_File\_Descriptions*, which is a PDF, describes in detail the data within the following three workbook files (in several file formats). *of2011-1216\_FROSTFIRE\_megaPhysical* contains physical descriptions of the samples, such as volumetric field moisture, bulk density, and height above mineral soil. *of2011-1216\_FROSTFIRE\_megaChemistry* contains elemental C, N,  $\delta^{13}\text{C}$ ,  $^{14}\text{C}$ , elements from ICP analysis, and Hg for samples on which these analyses were run. The last file, *of2011-1216\_FROSTFIRE\_Transects*, contains field descriptions for individual horizons that make up the pre-burn USGS soil profiles.

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