**Title:** Soil and pyrogenic carbon in an old-growth coast-redwood forest (working title)

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**Abstract:** (172/300 words)

Fire is an important ecological feature in temperate forests. Fire regimes are often best described and informed by long-term records yet determining past fire regimes may be difficult in ecosystems with limited available fire proxies or histories. We use radiocarbon dating and quantification of both soil macro-charcoal and soil and pyrogenic carbon in an old growth redwood stand to examine fire events in redwood forests, an ecosystem with few fire records. We sampled charcoal fragments, soil carbon and soil pyrogenic carbon of soils in the Headwaters Forest Reserve, a protected fragment of old growth redwood in Humboldt County, California to investigate whether prehistoric fire events were detectible in soil archives. Radiocarbon dates from macro-charcoal indicate fire events occurring a maximum of 6,840 calibrated years BP, predating existing records. Composite 14C dates show increased fire activity within the last 1,000 years in synchrony with existing dendrochronological records. Soil C averaged 928 g/m2, of which a high proportion was pyrogenic C (15-30%). Information from this multi-proxy reconstruction clarifies our understanding of the nature of coast redwood fires, contributing to ongoing discussions of coast redwood fire regimes.

# Introduction

Contextualizing fire in modern ecosystems under changing climatic and fire conditions requires the spatial and temporal insights provided by long-term (millennial-scale or longer) records of fire (Whitlock et al. 2003, Gavin et al. 2007). Paleoecological records of fire provide insights into the interactions of fire, vegetation and climate over longer time scales, enabling investigation of the functional mechanisms and relationships driving changes in fire regimes (Conedera et al. 2009). Additionally, millennial-scale fire records clarify historic and pre-historic ranges of fire variability, providing context for modern shifts in variability. Methods such as radiocarbon dating and pyrogenic carbon estimates may enable reconstructing fire activity in locations without reliable lacustrine depositional environments or suitable tree ring records, but also depend on access to an appropriate physical archive of soil charcoal. Clarifying uncertainties in millennial-scale fire patterns in ecosystems without reliable, appropriate, or accessible paleoecological archives will require insight from novel fire proxies and archives.

The distribution and abundance of pyrogenic carbon and charcoal stored within soil can provide insight into local long-term fire dynamics. Pyrogenic carbon (henceforth referred to as PyC) is the physical residue and productions of fire and includes soot, char, partially charred material and any individual compounds altered on a molecular level by combustion (Bird et al. 2015). Here, we use PyC to capture the full spectrum of combusted material present in soil and reserve the term ‘charcoal’ specifically to refer to macroscopic fragments of partially combusted material (Knicker 2001, Bird et al. 2015, Schmidt and Noack 2000). The presence and distribution of soil charcoal and pyrogenic carbon within soil is a meaningful proxy of fire history since it communicates the age and spatial location of past fires (Gavin et al. 2007, Ohlson and Tryterud 2000). Soil charcoal is often both spatially constrained and temporally persistent (Schmidt et al. 2011): in systems without substantial soil movement, the presence of soil charcoal reflects the specific location of a fire (Gavin et al. 2007, Clark 1988). Furthermore, charcoal incorporated into forest soils may reside for centuries to millennia (Hammes et al. 2008, Lehmann et al. 2008), allowing charcoal to serve as an archive of fire history that both overlaps and predates tree-ring and anthropogenic records (Bird et al. 2015).

Using soil charcoal as a proxy of fire relies on radiocarbon dating charcoal fragments found within specific soil contexts and depths (i.e., Gavin et al. 2007). Radiocarbon dating, however, is an imperfect approach: the technique is expensive, and may overestimate the realistic age of a fire if the wood material itself is old enough at the time of burning (referred to as the inbuilt age error) (Gavin et al. 2003, Harmon et al. 1986). Despite those limitations, the use of radiocarbon dated charcoal material as a proxy of fire activity persists, particularly in systems without alternative reliable archives (Gavin et al. 2003).

The characteristics and dynamics of the coast redwood fire regime across millennium remains uncertain (Varner and Jules 2017). The mesic nature of redwood forest structure and the frequent precipitation in Northern California suggest infrequent burning, yet existing tree ring records reveal 30-year intervals between fire prior to Euro-American settlement (Stuart 1987, Brown and Swetnam 1994, Brown et al. 1999, Brown and Baxter 2003, Stephens and Fry 2005, Norman 2007). Some studies suggest that the decadal fire intervals prior to Euro-American settlement are the result of First Nations burning habits (Sawyer et al. 2000). While native burning may certainly have contributed to frequent fire, coast redwoods possess fire-adapted traits such as basal and epicormic sprouting and thick bark that suggest a much longer co-existence with frequent fire (Sawyer et al. 2000). Current increases in the intensity and frequency of wildfires across the western United States have sparked concern about the consequences of frequent burning in coast redwood stands (Westerling et al. 2006, Fried at al. 2004). Despite their importance for contextualizing modern trends in variability, few traditional paleoecological records of fire are available. Additionally, redwood tree rings can be difficult to date (Fritz 1940), and few lakes exist in the region with adequate sediment deposition (citation: me) highlighting the importance of using alternative techniques to accurately reconstruct stand fire dynamics.

No previous research has evaluated either the carbon or pyrogenic carbon content of coast redwood mineral soils. Advances in charcoal and carbon quantification methods present a unique opportunity to investigate how fire has influenced characteristics of soil and carbon cycles within old growth coast redwood forests. To establish a baseline understanding of soil carbon and charcoal dynamics within coast redwoods, we asked the following research questions: 1) what is the abundance, distribution and age of pyrogenic carbon deposited within old growth coast redwood soils? 2) does the abundance, distribution or age of pyrogenic carbon differ according to landscape position or depth of soil? 3) How does the abundance, distribution, or age of pyrogenic carbon in redwood soils compare to similar ecosystem types? To address those questions, we used radiocarbon dating and elemental analysis to evaluate the ages and distribution of soil charcoal and PyC across a gradient of landscape positions in an old growth coast redwood forest in Northern California.

# Methods

## Study Area

We sampled organic and mineral soils in old growth coast redwood forests within the Elk River and Salmon Creek watersheds at the Headwaters Forest Reserve in Humboldt County, California. Elevation ranges from 100 to 2,000 feet. Soils are mostly shallow (>1m) and are a mix of alfisols and ultisols (BLM data, unpublished). The climate is maritime: cool and wet winters are followed by warm, cloudy summers. We sampled only old-growth stands of coast redwoods, an ecosystem dominated by coast redwood in the overstory, but with occasional Douglas-fir (*Pseudotsuga menziesii*), grand fir (*Abies grandis*) and western red cedar (*Thuja plicata*) individuals.

The Headwaters Forest Reserve has a history of disturbance: remaining old growth is protected, but edges of both second and old growth stands are in close proximity to timber roads and the reserve boundaries themselves. Unpublished tree ring data shows that fires occurred in the reserve between every 10-42 years since the 1760s and suggests fire activity increased after 1850 until 1936 when fire suppression management went into effect. (Norman and Jennings, unpublished).

Map

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**Figure 1. Map of study sites within Headwaters Forest Reserve.**

Sites were selected based on access to old growth stands but at a minimum 50-m distance from past disturbances such as logging or road construction. We established 20 sampling sites in total, primarily on ridgetops (n =16), excluding steep slopes or valley bottoms to sample specifically for spatially constrained charcoal with minimal depositional movement via erosion, runoff, or debris flows (Fig 1). This exclusion intentionally provided a more conservative estimate of charcoal presence and abundance. One valley-bottom site (EELS\_01) was sampled opportunistically for comparison.

We sampled charcoal fragments at exact depths from a clean soil profile between 0 and 45 cm deep using soil pits dug at each site. At two sites (GOV\_01 and WORM\_03), debris-flow deposits were exposed by stream or road cuts, allowing for deeper sampling. We took soil cores at debris-flow sites vertically from the face of the soil profile at 10 cm increments. Samples from these sites are displayed separately, to account for the distinct depositional nature of charcoal found within debris flows.

To estimate the abundance of total PyC and overall carbon content of redwood mineral soils, we took multiple volumetric soil cores at each site using a 5-cm split core sampler with a known volume driven into the soil up to 30 cm deep. We removed coarse litter prior to sampling, but did not distinguish between O, A, and B horizons due to a lack of distinct boundaries. We measured bulk density as the total dry weight of the sample divided by the volume of the soil corer (98.2 cm3).

## PyC Quantification

To quantify the abundance of PyC within redwood soils, we relied on two known methods of soil PyC and charcoal quantification methods: physical charcoal quantification (Clark 1988) and acid-peroxide digestion. Physical quantification is a more traditional approach but requires much more time and labor (Clark 1988), while acid-peroxide digestion, established by Kurth et al. 2006 and others (Pingree et al. 2012), requires no physical counting of particles, and potentially captures a greater range of pyrogenic materials. We used both methods to compare and report any difference in results between the two. Prior to both quantification procedures, we dried bulk soil samples in an oven at 60°C for 24 hours (Pingree et al. 2012).

### Physical Charcoal Quantification

We soaked soil-core samples overnight in a 10% KOH solution to disperse organic clumps, and then rinsed through 2 mm and 0.5 mm test sieves. We removed material from both size classes and treated each with 3% H2O2 for over 24 hours. Once oven-dried, we counted charcoal fragments under a microscope, and weighed the total mass of identifiable charcoal within each soil-core section for each site. Charcoal concentration from sieved samples was calculated by dividing mass of charcoal by the dry weight of the sample.

### Chemical Charcoal Quantification

To estimate PyC concentrations chemically, we completed an acid-peroxide digestion following the methods of Kurth et al. (2006) as modified by Pingree et al. (2012). We ground samples in a ball mill to <0.76 μm before adding 1.0 gram to a 50 ml flask with 20 ml of 30% H2O2 and 10 ml of 1 M HNO3. We swirled samples by hand to promote effervescence at room temperature across a 30-minute period, before heating in a water bath to 90°C for 16 hours. After digestion, we filtered samples through pre-weighed filter papers and dried at 60°C for over 24 hours before weighing to obtain the mass of residual material after digestion and filtration.

To constrain PyC estimates produced by acid-peroxide digestion, we created charcoal standards by combusting dry western red cedar samples (*Thuja plicata*) wrapped in aluminum foil at 450°C. We ground a 1:9 ratio of charcoal and charcoal-free rock (Condrey Mountain Schist from Southwestern Oregon) to <0.76 μm in a ball mill, creating a 10% charcoal standard.

We determined the percent of carbon present in soil and digested soil samples using a Mass Spectrometer at the Laboratory of Stable Isotope Ecology, University of Miami. We report Total C following digestion as charcoal C and assume that all non-charcoal organic C was consumed during peroxide-acid digestion.

## Radiocarbon Dating

To establish whether pyrogenic carbon is distributed stratigraphically and to estimate the range of ages of charcoal in redwood soils, we selected charcoal samples with known depths at each sample site for accelerator mass spectrometry (AMS) radiocarbon dating based on depth, size, and quality of sample. We cleaned samples with alternating heated 10% KOH and 10% HCL rinses prior to radiocarbon dating at the Center for Accelerator Mass Spectrometry at Lawrence Livermore National Laboratory in Livermore, California (Table S1).

## Data Analysis

We determined charcoal C concentration (PyC) of digested soil samples using )\* 1000, where M1 = mass of digested sample, C1 = C concentration of digested sample, and M2 = mass of original sample. We estimated PyC mass per square meter as the product of PyC concentrations and bulk density summed over all depth increments ( \* 10,000, where BD = bulk density).

We calibrated radiocarbon dates using the CALIB 5.0.1 program based on the INTCAL13 calibration curve (Reimer et al. 2013) and produced calendar age estimates of modern dates using Oxcal (version 4.4, Ramsey 2009).

To investigate the presence of a stratigraphy of charcoal dates within redwood soils, we compared radiocarbon date estimates of charcoal fragments from the same soil pit but different depths where such pairings existed (n = 21). A stratigraphy existed if the fragment from the deeper depth had an older calibrated radiocarbon age.

# Results

## PyC Quantification

Bulk density of soil samples increases on average with depth from 0.3 g/cm3 at the soil surface to 1 g/cm3 at 30 cm (Appendix 1: Figure S1).

Total C of undigested bulk soil samples decreases with depth (Fig 6A). The ratio of PyC to total C slightly increases with depth, indicating the effect of preservation ability (Figure 6B). Average proportions of PyC to total C per depth increment range from 0.089 – 0.199. The total average ratio of PyC was 0.159. The average mass of PyC was highest in hillslope sites, though not enough valley sites may have been sampled for adequate comparison. The higher levels of hillslope PyC compared to that found on ridgetops is a strong indication of PyC transportation through erosion (Abney and Berhe 2018).

Total PyC in grams per square meter ranged from 620 to 1,488 g/m2 per site across all depths, with an average of 928 g/m2 across all sites (Fig. 7).

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**Fig. 4. Total soil carbon (mg) and the ratio of soil carbon to pyrogenic carbon across depth (0-35 cm) in old growth redwood soils.** Results from elemental analysis of undigested soil and digested soil samples. A) Total C levels for undigested soil samples across depth (0-35 cm). B). Ratios of PyC to total C across depth (0-35 cm).

## Charcoal stratigraphy

All adjacent dates were compared pairwise for a total of 21 comparisons. 13 out of the 22 paired dates display an age reversal (older samples at shallower depths), indicating a lack of stratigraphy within those sites (Fig 3).

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**Fig. 3. Age-depth relationships of paired soil-charcoal radiocarbon dates**.

Stratigraphic relationship between calibrated radiocarbon age and depth in soil across soil-charcoal radiocarbon dates from individual sites with at least two radiocarbon dates. Line at y = 0 represents no difference in age between paired samples. Points above y = 0 display no age reversal, indicating charcoal samples at deeper depths produced older calibrated radiocarbon ages, while points below y = 0 indicate charcoal samples at deeper depths were found to be younger, indicating a lack of stratigraphy.

## Radiocarbon Dates

Charcoal deposited within the debris flow displayed an older range of calibrated radiocarbon dates (931 to 6,839 years BP) than charcoal deposited within mineral soils on ridgetops, hillslopes, and valleys (modern to 3,805 calibrated median years BP). Due to the distinct difference in age between landscape types, results from the debris flow are reported separately. Two charcoal fragments from the WORM\_03 site dated at 6,666- and 6,839-year BP, though they were located a meter apart in depth (Fig 2). Due to the nature of the site and the unusual age of the samples, these dates were not included in subsequent analysis, but their presence indicates the potential longevity of charcoal within redwood soils.

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**Fig. 2. Radiocarbon dates of charcoal samples according to depth of sample**. [add error bars]

43 dates are shown from 20 sites (average 2 dates per site), plotted according to site type and against depth within soil profile. Site type indicated with color and shape of point.

# Discussion

The abundance, distribution, and age of soil charcoal within old growth coast redwood soil differs across landscape position. Charcoal and carbon concentrations are greatest in the top 10 cm of redwood soils and decline with depth, though charcoal is still found 30-35 cm deep. Radiocarbon dates demonstrate charcoal has the capacity to preserve over thousands of years across different depths within redwood soils, though not always within a stratigraphy. However, the soil charcoal record in coast redwood mineral soils contains evidence of fire history that precede tree-ring records of fire.

The age reversals of radiocarbon dated soil profiles indicate stratigraphy within mineral soils within coast redwood forests is relatively arbitrary, signaling that redwood soils experience mixing across a variety of spatial scales. On a fine-scale, bioturbation driven by earthworms and other creatures mix soils consistently over time (Gerbert et al. 2003). On a larger scale, tree tip-ups upturn large amounts of soil, creating mounds up to 1-meter high (Norman et al. 1995), that can take several thousand years to completely disappear (Schaetzel and Follmer 1990). Given the size of redwood root systems (Phillips et al. 2013: total structural root length can be 0-700 m in trees 1-4 years old), this process may be even more exaggerated. Across greater spatial scales, soil mixing may be driven by erosion or depositional events: the ages and depths of charcoal fragments from the alluvial fan indicates that material in the alluvial fan sampled was likely transported by a depositional event (Fig 2). While alluvial fan dates were not included in analysis because of their distinct age and source, these fragments are evidence that charcoal can persist in coast redwood soils for thousands of years, perhaps especially when buried deeply in depositional events.

The highest density of radiocarbon dates from mineral soils occurred within the last 1,000 years, suggesting a number of things: either a shift in the frequency of fire, optimized charcoal preservation or a bias in sampling. Two soil charcoal radiocarbon dates registered as “modern” (~1950 or more recent) and may be from settler/logger-induced broadcast burns that took place in the region in the 1900s and earlier in order to expose adjacent landscapes for easier harvesting (Table S1). Overlap exists between the fire scars dated by unpublished Norman and Jennings work and the soil charcoal radiocarbon dates that occur within the last few hundred years. Specifically, fire events at ca. 100 and ca. 175 years BP registered in both the soil charcoal and tree ring records, emphasizing that the higher density of radiocarbon density may reflect fire frequency. While greater investigation is needed to fully constrain fire activity across the last millennium in coast redwood forests, dated fires from the Norman and Jennings project only extend back to 250 years BP, again emphasizing the value of the temporal extent of soil charcoal.

## Pyrogenic Carbon

The average proportion of PyC relative to total soil C in redwood ecosystems was comparable to estimates for ecosystems with frequent fire (15%), and proportions at deeper depths were distinctively higher (20%). Specifically, the average g/m2 of PyC across sites was higher than estimates produced for coastal Douglas-fir forests in Southwest Oregon, boreal forest soils, Sierra Nevada soils and dry Ponderosa Pine forest soils, all systems that undergo regular fire at different temporal intervals (Ball et a. 2010, Kurth et al. 2006, Pingree et al. 2012, Bélanger and Pinno 2008, Mackenzie et al. 2008). Pingree 2012 found ca. 700 g/m2 of PyC in the Siskiyou Mountains, constituting nearly 20% of total C in surface and subsurface mineral soils. Comparing across ecosystems, the system with the most similar mass of PyC is a boreal forest in Saskatchewan with a reported 4,000-11,000 kg charcoal C ha−1 (Bélanger and Pinno 2008). Mass of PyC across redwood soil sites ranged from 6,000-14,000 kg per hectare, suggesting similarities in the processes leading to charcoal production and preservation. Our results suggest redwood ecosystems contain greater pyrogenic carbon in mineral soils than comparable sites with frequent fire, which may be a result of the abnormally high aboveground biomass in redwood forests and a testament to efficient burial and preservation of charcoal into mineral soils.

Our estimates of pyrogenic carbon abundance suggest that fire may have an impact on carbon cycling within coast redwood forests comparable to ecosystems with frequent fire regimes (fires occurring somewhere between 5 to 30 years). High levels of biomass in redwood forests may contribute to the large quantity of charcoal produced, and hillslope erosion and tree-tip-up bioturbation may promote the burial and preservation of charcoal. Acid digestion may underestimate PyC soil content, as charcoal standards lost 34% of PyC during digestion. However, the extremely young age (weeks-months) of standards means they contain components that will be lost through normal soil-respiration activity while in a soil environment for decades or longer. Decomposition rates for younger, artificially made charcoal are often higher and Kurth et al. (2006) reported PyC loss of less than 10% (Douglas-fir charcoal) using the same acid-peroxide digestion. Therefore, it is not clear to what degree the acid-digestion underestimates PyC concentrations, meaning more work is needed to clarify the effect of charcoal age on digestion estimates.

Furthermore, interpreting past fire activity from soil charcoal requires a series of assumptions about the various processes impacting soil charcoal deposition and persistence: charcoal degradation within soil may occur at different rates between systems or across depth (), subsequent fires may consume charcoal in upper soil layers () and erosion may transport particles across large distances (Conedera et al. 2009, Doetterl et al. 2016). Due to the myriad of factors that induce soil mixing and movement (erosion, cryoturbation, tree tip ups, bioturbation, etc), few soil systems display a stratigraphy signaling a consistent age-depth relationship (older soil at deeper depths), limiting our ability to extrapolate results (Conedera et al. 2009, Carcaillet et al. 2000). However, recovering individual dates within mixed soils still allows for individual records of fire across a longer time scale than that available currently in redwood fire proxies.

Soil charcoal in old growth redwood forests provide a record of fire activity inaccessible through tree-ring or anthropogenic records. High levels of mixing may improve the capacity of charcoal to persist within soil, allowing for preservation of fire history records, though not in sequence. Levels of PyC in old growth coast redwood forests are comparable to other fire-prone ecosystems, suggesting fire is a feature of coast redwood ecosystems over thousand-year time scales.

**Data Availability:** The data and code used in this manuscript have been made available for reproducibility purposes and are accessible at the following DOI: [10.5281/zenodo.4455777](https://doi.org/10.5281/zenodo.4455777).

# Acknowledgements

This research was supported by funding provided by the Bureau of Land Management National Landscape Conservation System research support program (). Fieldwork was supported by the BLM Arcata field office. We thank Alexia Gee for assistance in the field and Kergis Hiebert for assistance in the laboratory. We thank Lukas Silva and Toby Maxwell for valuable support and advice. Finally, we thank Rosemary Sheriff for providing friendly review which significantly contributed to the quality of the paper.

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# Supplement:

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| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Table S1. Radiocarbon dates of individual pieces of soil charcoal from Headwaters Forest Reserve, California. MS = Forest mineral soil. | | | | | | | | |
| Site | Site Type | CAMS # | Sample ID | Depth (cm) | C14 Age | | Error | Cal yr BP |
| Governor’s Grove | Colluvial Hollow | 175998  177582  177078  177079  177080  177081  175999 | GOV-01  GOV-01  GOV-01  GOV-01  GOV-01  GOV-01  GOV-01 | 17  23  31  38  50  52  67 | 1010  1050  4490  4185  4155  4355  2615 | 35  30  60  30  30  35  40 | | 798-1040  924-1050  4891-5313  4618-4837  4580-4826  4848-5036  2543-2844 |
| Ridge MS | 177460  177461 | GOG-01  GOG-01 | 1  18 | Modern  445 | 30 | | 465-534 |
| Ridge MS | 177462  177463 | GOG-02  GOG-02 | 4  16 | 90  155 | 30  35 | | 22-265  1-285 |
| Worm Trail | Ridge MS | 177084 | WORM-01 | 11 | 2285 | 35 | | 2315 |
| Alluv. Fan | 175996  175997 | WORM-03  WORM-03 | 12  110 | 6000  5845 | 30  30 | | 6839  6666 |
| Salmon Creek Trail | Ridge MS | 177082  177083 | SCT-01  SCT-01 | 16  20 | 680  1255 | 30  30 | | 561-680  1083-1278 |
| Ridge MS | 177458  177459 | SALM-01  SALM-01 | 0  13 | 365  175 | 30  40 | | 316-501  1-298 |
| Ridge MS | 177580  177581 | SALM-03  SALM-03 | 3  22 | 840  90 | 30  30 | | 686-891  22-265 |
| Worm Trail | Ridge MS | 177453  177454 | WOMT-01  WOMT-01 | 5  19 | 480  680 | 30  40 | | 499-542  556-687 |
| Ridge MS | 177455  177455 | WOMT-02  WOMT-02 | 2  15 | 575  365 | 30  30 | | 503-648  316-501 |
| Ridge Trail | Ridge MS | 177577 | RIDG-01 | 19 | 830 | 30 | | 688-789 |
| Ridge MS | 177465  177457 | RIDG-02  RIDG-02 | 2  11 | 875  2245 | 30  35 | | 709-907  2153-2343 |
| Elk River | Valley MS | 177578  177579 | EELS-01  EELS-01 | 6  13 | 305  75 | 30  30 | | 299-462  27-259 |
| Worm Trail (Left fork) | Hill. MS | 177586  177587 | WOLF-01  WOLF-01 | 5  16 | 580  Modern | 30 | | 533-649 |
| Hill. MS | 177588 | WOLF-02 | 3 | 1420 | 35 | | 1287-1377 |
| Ridge MS | 177589  177590 | WOLF-03  WOLF-03 | 10  45 | 3615  265 | 30  30 | | 3841-4062  1-433 |
| Ridge MS | 177591 | WOLF-04 | 10 | 420 | 30 | | 333-523 |
| Worm Trail (Right fork) | Hill. MS | 177592  177593 | WORF-02  WORF-02 | 3  20 | 1155  985 | 30  30 | | 982-1175  797-959 |
| Ridge MS | 177594 | WORF-03 | 8 | 3805 | 30 | | 4089-4291 |
| Ridge MS | 177595 | WORF-05 | 7 | 200 | 30 | | 1-303 |

Chart, box and whisker chart

Description automatically generated

**Figure S1: Bulk density of soil samples across depths.**

**Chart, box and whisker chart

Description automatically generated**

**Figure S2. Comparing charcoal concentration estimates produced by physical and chemical quantification methods.**