

# Global Biogeochemical Cycles

## RESEARCH ARTICLE

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### Key Points:

- A new analytical technique, based on C-14, is presented for assessing the distribution of labile organic carbon in marine sediments
- Based on samples from the Antarctic shelf and San Clemente Basin, seabed LOC distributions yielded  $\tau$  values ranging from 0.1 to 60 years
- The agreement between our LOC data and those reported in the literature provides confidence that the new technique is sound and justified

### Supporting Information:

Supporting Information may be found in the online version of this article.

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## Using Radiocarbon to Assess the Abundance, Distribution, and Nature of Labile Organic Carbon in Marine Sediments

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**Abstract** Positive  $^{14}\text{C}$  gradients have recently been observed within the surface mixed layer of several continental-margin sediments. The best explanation for these positive  $^{14}\text{C}$  gradients is the occurrence and rapid degradation of labile organic carbon (LOC) in the upper 5–10 cm of the seabed. Based on a two-component model for sedimentary organic matter (i.e., a planktonic labile component and an older refractory component), bulk  $^{14}\text{C}_{\text{org}}$  data were used to determine the abundances of LOC within the surface mixed layers of three cores from the West Antarctic Peninsula (WAP) shelf and one core from San Clemente Basin (California Borderland). LOC contents in surface samples from the four stations varied from 0.5 to 1.1 mg/cm<sup>3</sup>, comprising 20% (San Clemente Basin) to 80% (WAP, Sta. G) of the total organic carbon. By incorporating a steady state diagenetic model and particle-mixing bioturbation coefficients, the LOC profiles were used to determine LOC turnover times (LOC  $\tau$ ) and LOC e-folding depths. The LOC  $\tau$  values for the West Antarctic Peninsula sediments varied from 0.09 to 0.59 years, whereas the LOC  $\tau$  value from the San Clemente Basin core was 63 years. The LOC e-folding depths for the WAP stations varied from 0.8 to 3.4 cm, in contrast to the LOC e-folding depth in San Clemente Basin, which was 4.0 cm. LOC characteristics from the four cores examined in this study were compared to LOC data in the literature as a means of substantiating the overall  $^{14}\text{C}_{\text{org}}$ -based approach and justifying model assumptions.

**Plain Language Summary** A new C-14 based technique for measuring the abundance of Labile (or reactive) Organic Carbon in marine sediments is presented. Based on this new approach and a model for sedimentary organic matter, Labile Organic Carbon depth profiles from West Antarctic Peninsula and California Borderland sediment cores were used to determine Labile Organic Carbon turnover times, which ranged from 0.1 to 0.6 years (Antarctic shelf sediments) to 63 years (California Borderland deposits). Within the bioturbated surface layer of the seabed, Labile Organic Carbon makes up only 6%–12% of the Total Organic Carbon, however, the Labile Organic Carbon is the source of nearly all nutrition and energy for bottom-dwelling fauna and bacteria. The Labile Organic Carbon abundances and turnover times are in good agreement with values reported in the literature, confirming the efficacy of the new C-14 based technique.

## 1. Introduction

The dominant source of nutrition to most marine benthic ecosystems is the flux of particulate organic matter from surface ocean waters. The nature of this organic carbon flux becomes much more complex as the organic material, primarily produced by phytoplankton, settles through the water column and undergoes aggregation (e.g., formation of marine snow), disaggregation, degradation reactions, macromolecule formation, selective preservation processes, as well as being attacked by a variety of grazers and microbes (Fabiano et al., 2001; Lee et al., 2004). In addition to these transformations in the water column, the organic material accumulating in the seabed, originating from marine sources and terrestrial soils, can be degraded via benthic faunal ingestion and microbial attack. Consequently, marine sedimentary organic matter is a complex mixture of organic compounds, exhibiting varying degrees of degradation and reactivities as well as providing a spectrum of nutritional value to benthos.

Berner (1980) was one of the first to describe the nature of marine sedimentary organic matter as a continuum of components with varying reactivity, forming a complex pool of undefined and diverse organic

components. The fundamental equation describing the change in sedimentary organic carbon ( $G$ ) over time ( $t$ ) was:

$$\frac{dG}{dt} = \sum \frac{dG_i}{dt} \text{ where } \frac{dG_i}{dt} = -k_i G_i \quad (1)$$

where “ $k_i$ ” is the degradation constant for a specific component, ( $G_i$ ), of the bulk organic matter.

This equation was based on the assumption that the pool of sedimentary organic matter is composed of organic compounds, all of which may have different reactivities, but all follow first-order reaction kinetics during their decomposition in the seabed (Boudreau & Ruddick, 1991; Westrich & Berner, 1984). In these early studies of marine organic carbon diagenesis, the term “Labile Organic Carbon,” or LOC, usually referred to the reactive organic carbon that was remineralized in the near-surface sediment as a result of microbial and benthic faunal activity. In this sense, LOC is used as a relative term describing the organic matter that is most readily bioavailable. Middelburg (1989) took this idea further and documented a log/log relationship between organic carbon reactivity (i.e., “ $k$ ” in Equation 1) and sediment age, noting that the organic carbon degradation rate decreased as the age of the sediment increased. The organic geochemistry literature is full of research examining the composition and structure of organic matter as a function of degradation rate, or reactivity (see Benner & Amon, 2015; Burdige, 2007; Hedges et al., 2001; Hedges & Keil, 1995; Lee et al., 2004; Middelburg et al., 1993; Soetaert et al., 1996; Wakeham et al., 1997). Arndt et al. (2013) present one of the most comprehensive discussions for quantifying marine organic matter degradation in the recent literature.

Hammond et al. (1996) were one of the first to quantify marine sediment organic carbon degradation rates in the field and consider the effects of bioturbation on organic matter degradation rate calculations. The researchers used oxygen porewater profiles to estimate labile organic carbon degradation rates in Equatorial Pacific deep-sea sediments. They reported that there was a dominant labile organic carbon phase with a turnover time of 0.02–0.4 years, which explained 70–90% of the labile organic carbon degradation in the upper 1–2 cm of the seabed. A second, more refractory organic phase occurred at some stations, and its turnover time was on the order of 40–300 years. Sayles et al. (2001) also reported LOC degradation rates in their study of Southern Ocean deep-sea sediments (AESOPS Transect), based on the curvature of porewater nitrate profiles. They found that in sediments south of the Polar Front (i.e., areas of relatively high organic carbon flux), the LOC turnover times varied from 0.2 to 3.4 years, whereas north of the Sub-Antarctic Front (i.e., areas of reduced organic carbon flux), the LOC turnover time was ~40 years. One difficulty in using the porewater oxidant technique for estimating LOC degradation is that the stoichiometric relationship between the oxidant (or metabolite) and LOC often must be assumed, leading to uncertainty in the LOC values.

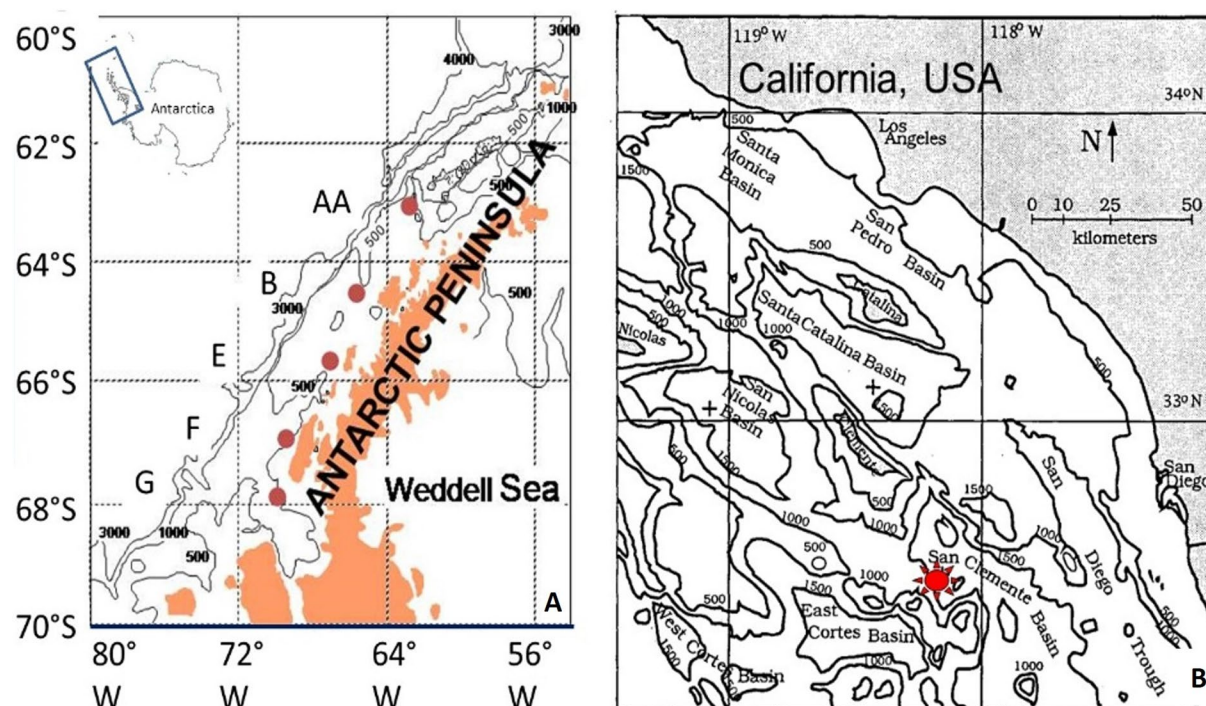
Another technique for estimating the distribution of reactive organic matter (or LOC) in surface sediments is the determination of biopolymeric organic carbon, which is equal to the sum of lipid, protein, and carbohydrate abundances in the seabed (Navarro et al., 1993). This approach has been used to estimate bioavailable organic carbon abundances in sediments from the Ross Sea (Fabiano & Pusceddu, 1998), the Gulf of Lion (Grémare et al., 2003), the Weddell Sea (Isla et al., 2006), and the Larsen Shelf on the eastside of the Antarctic Peninsula (Isla & DeMaster, 2018). The main issue affecting this technique is that some of the organic compound classes (such as certain lipids) resist attack by benthic faunal enzymatic systems and by microbes, persisting deep into the seabed well below the zone of LOC (Wang & Druffel, 2001; Wang et al., 1998).

The distribution of LOC in marine sediments also has been estimated by measuring enzymatically hydrolyzable amino acids (EHAA) as a proxy for reactive bioavailable organic carbon. Field examples of EHAA usage include: Dauwe et al. (1999b) in the North Sea, Mayer et al. (2002) on the North Carolina continental slope, Mincks et al. (2005) on the West Antarctic Peninsula, and Garcia et al. (2010) on the Western Iberian continental margin. Some of the difficulties in using this proxy to represent LOC abundance are that not all LOC is EHAA and some EHAA persists (as a background signal) long after the LOC has been biologically consumed (Sañe et al., 2013).

Lastly, reactive organic carbon abundance and distribution have been estimated in marine sediments by measuring Chlorophyll-a. One of the best examples of research using chlorophyll-a as a proxy for LOC comes from the work of Stephens et al. (1997), who measured chlorophyll-a in central Equatorial Pacific deep-sea sediments. They observed a chlorophyll-a turnover time ranging from 11 days to 2.6 years. Chlorophyll-a certainly is labile organic carbon, but it represents a very small fraction of the labile organic carbon pool and its turnover time is typically shorter than that of most LOC. Consequently, chlorophyll-a's use as a proxy, representing all of the LOC in surface sediments, is limited, and a technique that measures the abundance of bioavailable organic matter in the entire LOC pool in surface sediments is needed. As a result of these analytical shortcomings and proxy inadequacies, degradation rates of labile organic carbon in surface marine sediments have been difficult to quantify and only determined infrequently.

DeMaster et al. (2002) reported that the  $^{14}\text{C}_{\text{org}}$  content of bulk surface sediments from the North Carolina continental slope corresponded to a  $^{14}\text{C}$  age of 350–2000 years. Surprisingly, the organic tissues from bottom-dwelling infauna (mainly polychetes) living in those surface sediments had negative  $^{14}\text{C}$  ages (as a result of bomb  $^{14}\text{C}$ ), matching the values of surface algae. Clearly, the benthic infauna were not receiving their nutrition from bulk organic matter, but rather were assimilating mainly planktonic organic carbon from the surface sediments. As a result of this finding, when  $^{14}\text{C}_{\text{org}}$  measurements were made on West Antarctic Peninsula megacore samples and a strong positive gradient in bulk  $^{14}\text{C}_{\text{org}}$  activity was observed in the upper 3–8 cm of the seabed (DeMaster et al., 2016), it was reasonable to presume that much of the gradient in  $^{14}\text{C}_{\text{org}}$  activity was likely to result from decreasing LOC, deposited recently on the seafloor as plankton/phytodetritus. These findings were similar to those reported by Wang et al. (1998) and Wang and Druffel (2001), who observed a distinct positive  $^{14}\text{C}$  gradient in the total organic carbon (TOC) fraction in two cores from the northeastern Pacific Ocean and one from the Southern Ocean. Consistent with Middelburg's (1989) correlation between organic carbon reactivity and sediment age, our approach to understanding reactive organic carbon cycling in surface sediments is based on the assumption that to a first approximation there are two pools of organic matter in most marine sediments: a labile (young) component (which we presume has properties equivalent to those of surface planktonic) and a more refractory (old) component. Our two-component system for marine sediments follows directly from the research of Hammond et al. (1996), who calculated the reactive organic carbon profile, based on dissolved oxygen porewater gradients (with the most labile component explaining nearly all of the reactive organic carbon degradation in central Equatorial Pacific surface sediments).

In this paper, a new analytical technique is described for estimating the abundance and distribution of LOC in marine sediments. Naturally occurring  $^{14}\text{C}$  (radiocarbon) is used as a tracer to resolve the relative abundances of the two predominant forms of organic carbon in the seabed: (1) reactive/bioavailable LOC; and (2) more refractory organic carbon that is not readily degraded during early diagenesis. This two-component system certainly is a simplistic model for describing the complex mixture of organic compounds in marine sediments. However, the efficacy of the two-component approach for determining LOC in the seabed can be assessed by comparing the  $^{14}\text{C}$ -based LOC abundances and turnover times to those values from previous studies in the literature as an independent corroboration. In addition, incorporating this approach for evaluating LOC characteristics enables assessment of whether the gradient in bulk  $^{14}\text{C}_{\text{org}}$  activity observed in the upper 10 cm of some marine sediments (e.g., DeMaster et al., 2016; Isla & DeMaster, 2018; Wang et al., 1998) is a result of the addition of planktonic material to the sediment-water interface that undergoes rapid degradation in surface sediments. Bioturbation in surface marine sediments affects the abundance, distribution, and nature of LOC. In this research, the intensity of bioturbation was quantified using two naturally occurring radioisotopes,  $^{234}\text{Th}$  and  $^{210}\text{Pb}$ . This is a commonly used approach described extensively in the literature (e.g., Lecroart et al., 2010). Once the  $^{14}\text{C}$ -based LOC technique is substantiated by corroborating our LOC abundances and distributions with those from the literature, the physical and biological factors affecting LOC degradation can be examined and understood better (such as decreased reactivity with depth in the seabed and the effect of benthic faunal ingestion on LOC turnover times).



**Figure 1.** Locations of sediment cores used in this study. Field areas included the West Antarctic Peninsula (Figure 1a stations “B” and “G”) and San Clemente Basin (Figure 1b, station shown as the red star) in the California Borderland.

## 2. Methods and Materials

### 2.1. Sample Collection

The initial cores that were analyzed to test the  $^{14}\text{C}$  approach for assessing marine sedimentary LOC came from the West Antarctic Peninsula (FOODBANCS Project; Smith & DeMaster, 2008; Smith et al., 2008, 2012; see Table S1 for core locations) and from San Clemente Basin in the California Borderland (Age-Dependent Mixing Project; Fornes et al., 2001; Smith et al., 2001; see Supplemental Data Table S1 for core location). The West Antarctic Peninsula (WAP) study (Figure 1), called Food for Benthos along the Antarctic Continental Shelf (or FOODBANCS Project), examined the fate of reactive organic carbon reaching surface sediments, tracking its assimilation by benthic fauna, its bioturbation down into the seabed, its microbial degradation, as well as its accumulation in the sediment column. The San Clemente Basin study (Figure 1), called the Age-Dependent Mixing Project (or ADM), examined variations in bioturbation intensity as a function of nutritional value for benthic fauna as well as characteristic timescale. Cores from two West Antarctic Peninsula sites (Sta. B and Sta. G, with water depths of ~675 and 672 m, respectively) were used in this research as well as a single core from the San Clemente Basin site (water depth = 1,885 m). At Sta. B (WAP), samples were collected using a box corer in 2001 and a megacorer in 2009, whereas at Sta. G, samples were collected using a megacorer in 2009. Samples from the San Clemente Basin core were collected using a multicorer in 1998. These sediment cores were sampled at intervals ranging from 0.5 cm near the core surface to 1–2 cm intervals at depth. During the multicorer and megacorer subsampling, which occurred within hours of sample collection, the outer cm of sediment (in contact with the plastic core liner) was discarded to avoid contamination from smearing during core tube penetration into the seabed. Sediment samples for  $^{14}\text{C}_{\text{org}}$  analyses were frozen in sealed plastic bags, whereas samples for  $^{234}\text{Th}$  and  $^{210}\text{Pb}$  analyses were stored as wet sediment in glass porosity vials, prior to being shipped back from the field to the North Carolina State University laboratory. Phytoplankton samples on the West Antarctic Peninsula shelf were collected using a 0.5 m diameter hoop net (20- $\mu\text{m}$  mesh size) and were stored frozen until analysis. The plankton samples were collected at the same station and on the same cruise as the corresponding sediment sample. For the California Borderland plankton sample, a large piece of macroalgae (kelp) was collected using the



submersible, ALVIN, in San Clemente Basin. After collection, the macroalgae sample was kept frozen until laboratory analysis.

## 2.2. Analytical Methods

$^{14}\text{C}$  activity was measured on the sediment organic carbon fraction, after freeze drying and removing the inorganic carbon ( $\text{CaCO}_3$ ) using a 1N HCl pretreatment (25 °C) for a 24-h period. Thirty to 90 mg of carbonate-free dried sediment from the West Antarctic Peninsula were placed in tin boats, which were loaded into an elemental analyzer (Flash EA 1112) for the measurement of carbon and nitrogen abundance. As the  $\text{CO}_2$  gas was emitted from the elemental analyzer, it was collected using cryogenic techniques, then purified and transferred into a glass ampule, which was sent to the NOSAMS facility (National Ocean Sciences Accelerator Mass Spectrometry at Woods Hole Oceanographic Institution) for measurement of  $^{14}\text{C}$  and  $^{13}\text{C}$  abundances using accelerator mass spectrometry (Isla & DeMaster, 2018; Purinton et al., 2008). Total Organic Carbon (TOC) contents were reported on a calcium carbonate-free basis ( $\text{TOC}_{\text{CCF}}$ ), however, there is so little calcium carbonate in these sediments that  $\text{TOC}_{\text{CCF}}$  is approximately equal to  $\text{TOC}_{\text{SED}}$ . The  $^{14}\text{C}$  activity of the organic carbon from the San Clemente Basin sediments was determined by liquid scintillation counting of benzene (DeMaster et al., 2002) produced from the  $\text{CO}_2$  released during the combustion of 80–100 g of dried, carbonate-free sediment.

$^{234}\text{Th}$  and  $^{210}\text{Pb}$  samples were weighed wet, dried at 60°C for several days, reweighed (to determine water loss), and then homogenized using a mortar and pestle. Typically, 5–10 g of dried homogenized sediment were used for the  $^{234}\text{Th}$  and  $^{210}\text{Pb}$  analyses. After adding the appropriate radiochemical spikes ( $^{230}\text{Th}$  for  $^{234}\text{Th}$ ,  $^{232}\text{U}$  for  $^{238}\text{U}$  ( $^{234}\text{Th}$ 's parent), and  $^{209}\text{Po}$  for  $^{210}\text{Po}$  (the granddaughter of  $^{210}\text{Pb}$ )), the naturally occurring, particle-reactive radionuclides were extracted into near-boiling 6N HCl and 7N  $\text{HNO}_3$ , purified, and then plated, following the methods of Aller and DeMaster (1984). The thorium and uranium isotopes were plated from TTA (thenoyltrifluoroacetone) solutions onto stainless steel planchets, whereas the polonium isotopes were spontaneously plated onto silver squares.

## 2.3. Determining Labile Organic Carbon Abundance and Turnover Time in Marine Sediments

$^{14}\text{C}_{\text{org}}$  profiles were used to resolve the abundances of the two predominant types of organic matter in the seabed: a labile/bioavailable component (i.e., LOC), and a more refractory organic component that is not readily available to benthic fauna or seabed microbes. In our two-component model, the  $^{14}\text{C}$  content of the labile organic carbon (LOC) is set equal to that of surface plankton, because it is presumed that the reactive organic carbon in the surface sediments is primarily composed of recently deposited planktonic material. The  $^{14}\text{C}$  activity of the more refractory organic carbon component is set equal to the asymptotic  $^{14}\text{C}$  activity value at the base of the bioturbated layer, which is equivalent to the  $^{14}\text{C}$  activity of the bulk sedimentary organic matter once all of the labile planktonic material has been degraded. Based on these assumptions, the equation used to calculate the abundance of LOC in the seabed is:

$$\text{LOC} \left( \text{g of C/cm}^3 \right) = \frac{\left( \text{Fm}_s - \text{Fm}_{\text{asym}} \right)}{\left( \text{Fm}_{\text{pl}} - \text{Fm}_{\text{asym}} \right)} \left( \frac{\text{Wt. \% Org. C}}{100} \right) \left( \rho_{\text{dbd}} \right) \quad (2)$$

where LOC is the labile organic carbon abundance (expressed in g of labile C/cm<sup>3</sup> of wet sediment);  $\text{Fm}_s$ ,  $\text{Fm}_{\text{asym}}$ , and  $\text{Fm}_{\text{pl}}$  are the  $^{14}\text{C}$  contents (expressed as fraction modern of the  $^{14}\text{C}$  standard) in the bulk sediment sample, in the asymptotic end member at depth, and in surface plankton, respectively; Wt. % Org. C is the weight percent organic carbon content of the sediment sample (expressed in g of total organic C/g of dry sediment times 100); and  $\rho_{\text{dbd}}$  is the sediment sample dry bulk density (expressed in g of dry sediment/cm<sup>3</sup> of wet sediment). The first term on the right-hand side of the equation (i.e., the Fm term) calculates the fraction of LOC (or plankton) in a sample relative to the total organic carbon content. This two-component sedimentary organic carbon model essentially equates labile organic carbon (LOC) to the planktonic organic carbon that is regenerated in the surface mixed layer. Several previous investigators (e.g., Hammond et al., 1996; Sayles et al., 2001) have used a three-component model to describe the diagenesis of marine sedimentary organic matter. In these studies, the most reactive of the labile phases accounted for as much as 90% of the organic matter degradation in surface sediments. In addition, the  $^{14}\text{C}$  approach for assessing

LOC, described in this manuscript, can be applied to different depth intervals in the sediment column (see Section 4 and Taylor et al., 2020) to generate depth ranges with different LOC turnover time values (comparable to the three-component models used by Hammond et al. (1996) and Sayles et al. (2001)).

The total inventory of LOC in the seabed can be calculated using the following equation:

$$\text{LOC Inventory} = \int_0^{z^*} C^* \rho_{\text{dbd}} \partial z \quad (3)$$

where LOC Inventory has units of g LOC/cm<sup>2</sup> of seabed, C\* is the LOC content of the sediment (expressed in g LOC/g dry sediment),  $\rho_{\text{dbd}}$  is the dry bulk density,  $z$  is depth (cm), and  $z^*$  is the depth of LOC penetration into the seabed. For comparative purposes the inventory of Total Organic Carbon (TOC) also was integrated down to the  $z^*$  depth, so the integrated fraction (LOC/TOC) could be calculated for the sediments of the surface mixed layer.

Based on the distribution of LOC in the seabed, a diagenetic model (see Equation 4) can be constructed describing the dominant processes controlling reactive sedimentary organic matter (Berner, 1980):

$$\frac{\partial(\rho_{\text{dbd}} C^*)}{\partial t} = 0 = D_b \left( \frac{\partial^2(\rho_{\text{dbd}} C^*)}{\partial z^2} \right) - S \frac{\partial(\rho_{\text{dbd}} C^*)}{\partial z} - k \rho_{\text{dbd}} C^* \quad (4)$$

In this equation,  $t$  is time (yr),  $D_b$  is the bioturbation coefficient (with units of cm<sup>2</sup>/yr, simulated as a diffusive process),  $S$  is the sediment accumulation rate (cm/yr), and  $k$  is the degradation rate constant for LOC in the sediment mixed layer (yr<sup>-1</sup>). The reciprocal of  $k$  is equal to the LOC turnover time, commonly referred to as LOC  $\tau$ . This equation presumes that the distribution of LOC in the seabed is at steady state (i.e.,  $\partial C^* / \partial t = 0$ ). If the penetration of LOC and naturally occurring radioactive tracers into the seabed occurs primarily as a result of bioturbation and not sediment accumulation, the second term on the right side of Equation 4 can be considered negligible and dropped from the equation. In the development of this diagenetic model, dry bulk density is assumed to be constant in time and space, so it factors out of Equation 4. The reason for this simplification is that bioturbation is believed to be an exchange process that moves sediment through an infinite number of small, random transport events (simulated by diffusion). The benthos in these two field areas (and most other marine sediments) exchange sediment particles during their feeding and locomotion activities rather than sediment volumes, so the diffusive bioturbation process acts on the gradient in particle concentration (or mass) rather than on wet sediment volume, as suggested in Equation 4 (Fornes, 1999; McClintic et al., 2008; Smith et al., 2001, 2006). Based on these assumptions, Equation 4 can be simplified to:

$$\frac{\partial C^*}{\partial t} = 0 = D_b \left( \frac{\partial^2 C^*}{\partial z^2} \right) - k C^* \quad (5)$$

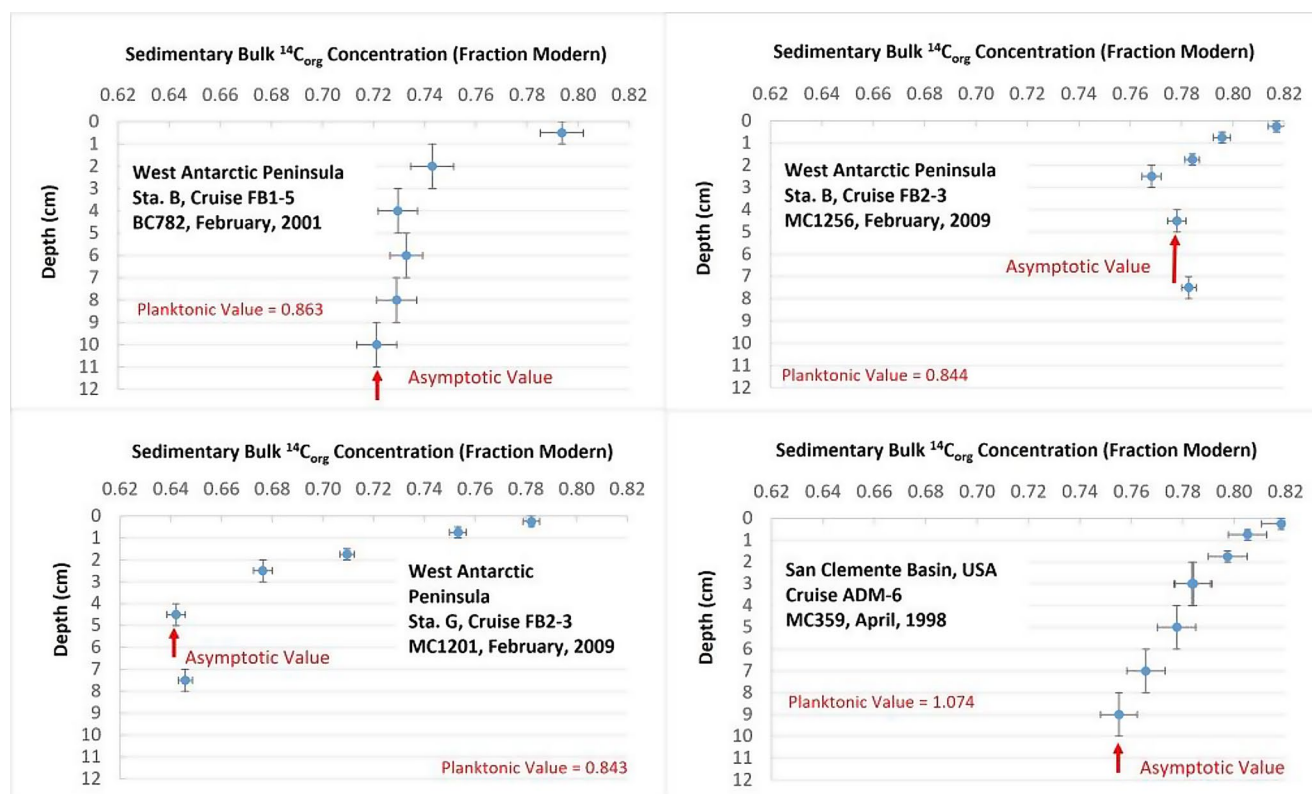
whose steady state (linear) analytical solution is:

$$\text{Ln}[C^*(z)] = -\left(\sqrt{k / D_b}\right)(z) + \text{Ln}[C_o^*] \quad (6)$$

Consequently, if the LOC distribution in a core is known and the bioturbation intensity can be quantified (using naturally occurring radioactive tracers), the LOC degradation rate ( $k$ ) can be determined from the diagenetic model, as well as the LOC turnover time ( $\tau$ ). The standard error in the slope of the  $\text{Ln}(\text{LOC})$  versus depth plot was assessed using EXCEL LINEST software, which provides the least squares slope, its standard error, as well as the  $R^2$  and  $p$  values for the data set.

#### 2.4. Determining Bioturbation Intensities in Marine Sediments

Particle-reactive, naturally occurring radionuclides have been used to establish bioturbation intensities (as characterized by the bioturbation coefficient,  $D_b$ ) in continental-shelf (e.g., DeMaster et al., 1985), continental-margin (e.g., Fornes et al., 1999), and in deep-sea sediments (e.g., DeMaster & Cochran, 1982) for many decades. An important aspect of this radiochemical approach is that the radiochemical tracer, used to



**Figure 2.** Bulk  $^{14}\text{C}_{\text{org}}$  profiles from three West Antarctic Peninsula cores and one San Clemente Basin core. The relative amounts of labile organic carbon and more refractory organic carbon were resolved using  $^{14}\text{C}$  by assuming the sedimentary organic carbon pool was comprised of two components: labile/bioavailable organic matter (set equal to the planktonic  $^{14}\text{C}$  activity) and refractory organic matter that is not degraded during early diagenesis (set equal to the  $^{14}\text{C}$  asymptotic value at the base of the particle mixed layer). Both of the  $^{14}\text{C}$  endmember values are provided in the figure for each core location and cruise.

characterize the bioturbation coefficient, must represent a timescale commensurate with the process being studied (in this case degradation of LOC). In all three study sites discussed in this research (two from the West Antarctic Peninsula and one from San Clemente Basin), bioturbation coefficients were established based on profiles of excess  $^{234}\text{Th}$  (half-life of 24 days, characterizing a 100-day timescale) as well as profiles of excess  $^{210}\text{Pb}$  (half-life of 22 years, characterizing a 100-year timescale).  $^{234}\text{Th}$  supported activities were determined by direct  $^{238}\text{U}$  measurements, whereas  $^{210}\text{Pb}$  supported activities were determined by averaging total  $^{210}\text{Pb}$  values deep in the sediment column (well below the depths of excess activity). The excess  $^{234}\text{Th}$  and  $^{210}\text{Pb}$  profiles were modeled using the same diagenetic equation (Equation 5) as used for LOC, only instead of the LOC concentration ( $C^*$ ), excess activity ( $A^*$ , units of dpm of tracer/g of dry sediment) was used. In addition, the LOC degradation rate constant ( $k$ ), was replaced by the appropriate radioactive decay constant ( $=\ln(2)/\text{tracer half-life}$ , units of  $\text{yr}^{-1}$ ). The slope and standard error of the  $\ln(A^*)$  versus depth plot was assessed using EXCEL LINEST software, which provides the least squares slope, its standard error, as well as the  $R^2$  and  $p$  values for the data set (see Figure S1). The  $D_b$  values used in the calculation of the LOC turnover times ( $\tau$ ) for these study sites were the ones whose characteristic timescales matched the resulting LOC  $\tau$  values most closely.

### 3. Results

#### 3.1. $^{14}\text{C}_{\text{org}}$ Profiles and LOC Distributions

The profiles of  $^{14}\text{C}_{\text{org}}$  from the four cores examined in this study (Figure 2), all showed a positive gradient in the upper 3–9 cm of the seabed. In the West Antarctic Peninsula cores the high  $^{14}\text{C}$  activities extended down to 3–9 cm, whereas in the San Clemente Basin core (California Borderland) the high values extended down to 8 cm. Surface sediment  $^{14}\text{C}_{\text{org}}$  activities (expressed as Fm) varied from 0.794 to 0.817 (corresponding

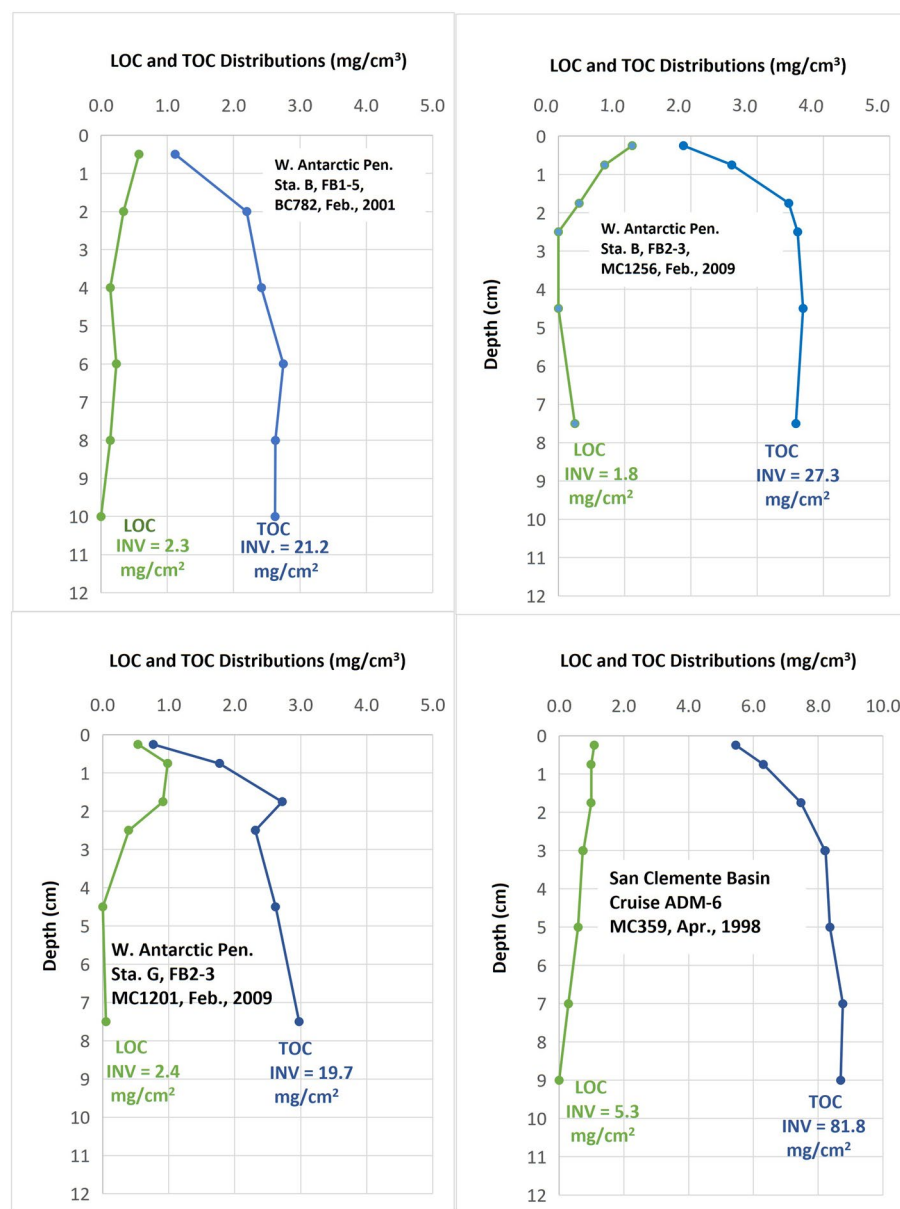
to radiocarbon ages of 1,850 to 1,620 years) in the West Antarctic Peninsula (WAP) cores, with a similar Fm value in San Clemente Basin of 0.818 (corresponding to a radiocarbon age of 1,610 years). The  $^{14}\text{C}_{\text{org}}$  activities of all four surface sediments were consistent with literature values (see Griffith et al., 2010 for discussion of associated processes). Planktonic  $^{14}\text{C}_{\text{org}}$  activities varied from 0.843 to 0.863 (corresponding to radiocarbon ages of 1,370 to 1,180 years) for the WAP stations, increasing to 1.074 (corresponding to a negative radiocarbon age of 570 years, i.e., in the future) for the San Clemente Basin station, as a result of the presence of bomb  $^{14}\text{C}$ . The  $^{14}\text{C}$  planktonic activities observed in WAP surface waters were consistent with literature values (Berkman & Forman, 1996; Key et al., 2004) and were considerably less than atmospheric values (prebomb and postbomb), primarily as a result of regional upwelling. Relative to the WAP planktonic values, the San Clemente Basin  $^{14}\text{C}_{\text{org}}$  planktonic value was enriched in  $^{14}\text{C}$  activity, but significantly lower than postbomb atmospheric values, primarily because of the upwelling off the California coast (although diminished relative to that occurring in the Antarctic). In all four cores studied, the surface sediment  $^{14}\text{C}_{\text{org}}$  content was considerably lower than the corresponding planktonic activity for that station and time, consistent with the two-component approach for modeling sedimentary organic carbon. The down-core differences in  $^{14}\text{C}$  Fm, ranging from 0.049 at Sta. B (WAP) to 0.14 at Sta. G (WAP), were readily resolved relative to the analytical uncertainty in the  $^{14}\text{C}_{\text{org}}$  measurement (typically 0.003–0.007 for Fm or 30–70 radiocarbon years for  $^{14}\text{C}$  age).

Based on these  $^{14}\text{C}_{\text{org}}$  data, the associated Wt. % total organic carbon values, sediment dry bulk density measurements and Equation 2, the LOC abundances were calculated for each of the four sediment cores (see Figure 3 and Table S2). Total organic carbon (TOC) distributions (Calcium Carbonate-Free TOC abundances were ~bulk TOC abundances) were also presented for the four cores for comparative purposes with the LOC data. The LOC contents of the surface samples from the four stations varied from 0.5 to 1.1 mg/cm<sup>3</sup> and comprised from 20% (San Clemente Basin) to 80% (WAP Sta. G) of the sample TOC. The depth of penetration of the LOC into the seabed (both diffusive and advective) varied from 3 to 9 cm in the four cores studied. Equation 3 was used to calculate the sediment column inventories for LOC and TOC (Figure 3), which varied from 1.8 to 5.3 mg/cm<sup>2</sup> for LOC and from 20 to 82 mg/cm<sup>2</sup> for TOC. The TOC profile was only integrated down to the depth of LOC penetration for comparative purposes. The LOC inventories at the WAP stations (1.8–2.4 mg/cm<sup>2</sup>) were less than half of the LOC inventory in San Clemente Basin (5.3 mg/cm<sup>2</sup>), whereas the TOC inventories at the WAP stations (20–27 mg/cm<sup>2</sup>) were less than 1/3 of the TOC inventory at the San Clemente Basin station (82 mg/cm<sup>2</sup>). The percentage of the TOC inventory that was composed of LOC material varied from 6.4% (San Clemente Basin, 1998) to 12.1% (WAP Sta. G, 2009) in the surface mixed layer sediments of the two study areas. Dauwe et al. (1999b) reported comparable percentages of reactive organic matter (5–17%) in North Sea sediments, based on EHAA-N:Total-N data. Clearly, the  $^{14}\text{C}$ -based LOC abundances make up a relatively small amount (<12%) of the TOC within the surface mixed layers of our cores. However, this reactive/labile organic matter provides nearly all of the nutrition/energy to the benthic community (DeMaster et al., 2002; Purinton et al., 2008).

### 3.2. Sediment Bioturbation Coefficients

Excess  $^{234}\text{Th}$  profiles were used to characterize bioturbation intensities for the West Antarctic Peninsula (WAP) stations [i.e., Stations B (2001), B (2009) and G (2009)], whereas an excess  $^{210}\text{Pb}$  profile was used to characterize the bioturbation intensity at the San Clemente Basin (SCIB) station (see Figure S1). The longer-lived tracer was used at the SCIB station because the LOC turnover time at the SCIB Station was on the order of decades (see Section 4), whereas the LOC turnover times at the WAP stations were on the order of a year or less (better represented by excess  $^{234}\text{Th}$  mixing coefficients. The bioturbation coefficients ( $D_b$  values) for the WAP stations and the SCIB station were determined from the excess activity profiles (see Figure S1) and the activity-transformed Equation 6. The  $^{234}\text{Th}$   $D_b$  values for the WAP stations (characterizing a 100-day time timescale) ranged from 3.0 cm<sup>2</sup>/yr (Sta. G, 2009) to 22 cm<sup>2</sup>/yr (Sta. B, 2001). In contrast, the excess  $^{210}\text{Pb}$   $D_b$  value from the SCIB station (characterizing a 100-year timescale) was 0.26 cm<sup>2</sup>/yr. The depth of diffusive penetration of LOC is shown in Figure S1, so that a comparison can be made of the bioturbation tracer distribution and the LOC distribution. These bioturbation coefficients were plugged into the LOC diagenetic model (Equations 5 and 6), enabling calculation of the LOC  $\tau$  values.





**Figure 3.** Labile Organic Carbon (LOC) and Total Organic Carbon (TOC) distributions (in  $\text{mg C/cm}^3$ ) from three West Antarctic Peninsula cores and 1 San Clemente Basin core. The LOC and TOC inventories are shown for each core, integrated down to the depth at which there is no LOC. The fraction that the LOC inventory comprised of the TOC inventory (integrated to the same depth) was always very low; varying from 6.4% in the San Clemente core (1998), to 6.7% in the West Antarctic Peninsula (WAP) core at Sta. B (2009), to 10.8% in the WAP core at Sta. B (2001), to 12.1% in the WAP core at Sta. G (2009).

## 4. Discussion

### 4.1. Determination of LOC Turnover Times

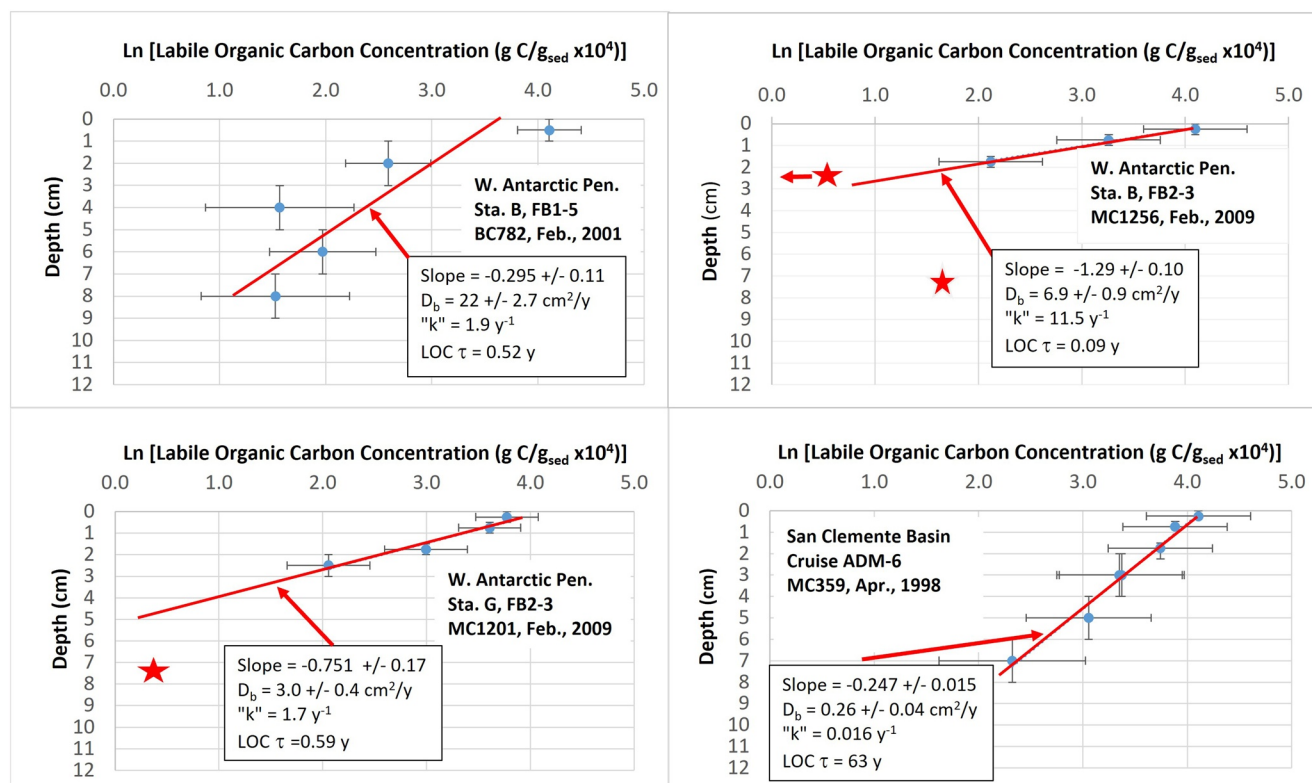
The linear least squares slope of the  $\ln(\text{LOC})$  versus depth ( $z$ ) plot (Figure 4), along with the bioturbation coefficient ( $D_b$ ), were used to calculate the LOC degradation constant ( $k$ ) for the surface mixed layer of each core. Based on Equation 6, “ $k$ ” is equal to the slope of the  $\ln(\text{LOC})$  versus depth plot squared times the  $D_b$  value. The LOC turnover time (LOC  $\tau$ ) is equal to the reciprocal of the LOC degradation rate (i.e.,  $1/k$ ). The slope of the  $\ln(\text{LOC})$  versus depth plot, the  $D_b$  coefficient, and the calculated LOC  $k$  and LOC  $\tau$  values are shown in Figure 4 for each of the four cores examined in this study. In this figure, depth in the sediment

**Table 1**  
Labile Organic Carbon (LOC) Data From Marine Sediments

Location/Core	$D_b$ (cm <sup>2</sup> /yr)	$D_b$ Tracer	LOC Tracer	LOC " $k$ " <sup>a</sup> (yr <sup>-1</sup> )	LOC $\tau$ <sup>a</sup> (yr)	LOC Inventory (mg C/cm <sup>2</sup> )	Reference
AA WAP Sta. B (2001)	22 ± 5.5	<sup>234</sup> Th	<sup>14</sup> C	1.9 ± 2.0	0.52 ± 0.54	2.3 ± 0.8	This Study
AA WAP Sta. B (2009)	6.9 ± 1.7	<sup>234</sup> Th	<sup>14</sup> C	12 ± 5	0.087 ± 0.039	1.8 ± 0.5	This Study
AA WAP Sta. G (2009)	3.0 ± 0.7	<sup>234</sup> Th	<sup>14</sup> C	1.7 ± 1.0	0.59 ± 0.33	2.4 ± 0.2	This Study
Calif. Borderland SCIB (1998)	0.26 ± 0.07	<sup>210</sup> Pb	<sup>14</sup> C	0.016 ± 0.006	63 ± 25	5.3 ± 1.1	This Study
WAP Surf Sed Range	1.5–36	<sup>234</sup> Th	<sup>14</sup> C	0.1–1.2	0.8–10	<sup>b</sup>	Taylor et al. (2020)
WAP Subsurf Range	0.2–1.8	<sup>210</sup> Pb	<sup>14</sup> C	0.0002–0.031	32–>600	17–70 <sup>b</sup>	Taylor et al. 2020
LIS A + B Range	0.09–1.6	NSS <sup>210</sup> Pb <sup>a</sup>	<sup>14</sup> C	<0.02–0.3	3.2–>60	1.5–22	Isla and DeMaster (2018)
LIS A Range	0.42–2.7	NSS <sup>210</sup> Pb <sup>a</sup>	<sup>14</sup> C	0.008–0.17	6–130	9.2–75	Taylor (2018)
Eq. Pacific Deep-Sea Range	0.21–1.4	<sup>234</sup> Th + <sup>210</sup> Pb	O <sub>2</sub>	2–43	0.02–0.43	–	Hammond et al. (1996)
Eq. Pacific DS (Chl-a) Range	0.6–9.1	<sup>234</sup> Th	Chl-a	0.27–22	0.045–3.7	1.6–6.5 × 10 <sup>-6</sup>	Stephens et al. (1997)
So. Ocean (S. Sub AA Ft) Range	0.06–0.16	<sup>210</sup> Pb	NO <sub>3</sub> <sup>-</sup>	0.29–5.1	0.20–3.4	–	Sayles et al. (2001)
So. Ocean (N. Sub AA Front)	0.16	<sup>210</sup> Pb	NO <sub>3</sub> <sup>-</sup>	0.024	42	–	Sayles et al. (2001)

<sup>a</sup>When two LOC components were calculated, the data show the values corresponding to the most reactive carbon. <sup>b</sup>Inventories shown represent both LOC fractions ( $k_1$  and  $k_2$ ).

<sup>c</sup>Nonsteady state <sup>210</sup>Pb profiles (characteristic timescale of years).



**Figure 4.** Natural Log of Labile Organic Carbon concentration versus depth plots for three cores from the West Antarctic Peninsula and one from San Clemente Basin. The least squares slope from each panel, along with the bioturbation coefficient, was used to calculate the labile organic carbon degradation rate constant ( $k$ ), whose reciprocal is equal to the LOC turnover time ( $\tau$ ). The LOC e-folding depth for each core was calculated by determining the reciprocal of the slope of the  $\ln(\text{LOC})$  versus Depth plot. The corresponding depths were:  $3.4 \pm 0.4$  cm (Sta. B, 2001),  $0.8 \pm 0.1$  cm (Sta. B, 2009),  $1.3 \pm 0.2$  cm (Sta. G, 2009), and  $4.0 \pm 0.1$  cm (SCIB, 1998).  $R^2$  values for these linear regressions varied from 0.69 to 0.99. The red stars indicate data that were not used in the linear regression analysis. Most of these points probably originated as a result of nondiffusive bioturbation.

column is plotted on the vertical axis for intuitive purposes, with  $\ln(\text{LOC})$  plotted on the horizontal axis; however, the slopes shown in Figure 4 data boxes and the ones used to calculate the LOC “ $k$ ” value were the reciprocal of the slope as plotted. For the West Antarctic Peninsula (WAP) stations the LOC  $\tau$  values varied from 0.09 years at Sta. B (2009), to 0.52 years at Sta. B (2001) to 0.59 years at Sta. G (2009) (see Table 1). The LOC e-folding depth (i.e., the depth over which the LOC concentration decreased by a factor of  $1/e$ ; Hammond et al., 1996) for each of these cores was:  $3.4 \pm 0.4$  cm,  $0.8 \pm 0.1$  cm, and  $1.3 \pm 0.2$  cm, respectively. The e-folding depth was determined by taking the reciprocal of the slope shown for each core in Figure 4. Based on the e-folding data, the LOC abundance at Sta. B (2009) drops off 4 times faster than at the same station during 2001. This difference in e-folding depth (or slope), coupled with the difference in bioturbation intensity between 2001 and 2009 ( $22 \text{ cm}^2/\text{yr}$  versus  $6.9 \text{ cm}^2/\text{yr}$ , respectively) yielded an LOC  $\tau$  value that was 6 times longer in 2001 (0.52 years) than in 2009 (0.09 years). At Sta. G the e-folding depth was intermediate (1.3 cm) relative to those at Sta. B, however, the bioturbation coefficient ( $3.0 \text{ cm}^2/\text{yr}$ ) was smaller than either value at Sta. B, which yielded a smaller LOC degradation rate ( $k$ ) and a longer LOC  $\tau$  value (0.59 years) at Sta. G.

In contrast to the WAP LOC  $\tau$  values (0.1–0.6 years), the LOC turnover time at the San Clemente Basin (SCIB) station (in the California Borderland) was considerably longer (63 years) (Table 1, Figure 4). The e-folding depth was substantially greater in the San Clemente Basin core ( $4.0 \pm 0.1$  cm) as compared to the WAP cores (0.8–3.4 cm). In addition, the longer LOC  $\tau$  value (timescale of decades) at the SCIB station required a commensurate timescale bioturbation tracer, so the  $^{210}\text{Pb}$   $D_b$  value ( $0.26 \text{ cm}^2/\text{yr}$ , characterizing a 100-year timescale) was used to calculate the LOC degradation rate, instead of the  $^{234}\text{Th}$   $D_b$  value ( $0.8 \text{ cm}^2/\text{y}$ , for a 100-day timescale). The San Clemente Basin core had the smallest  $\ln(\text{LOC})$  versus depth slope (i.e., the largest e-folding depth) and it also had the slowest bioturbation intensity of all four cores examined in this study, which led to a much smaller degradation rate constant ( $k$ ) and by far the longest LOC  $\tau$  value (63 years at SCIB versus 0.1–0.6 years on the WAP shelf). Possible explanations for the longer LOC  $\tau$  values in SCIB sediments include the presence of brown macroalgae (i.e., kelp) in the surface sediments (Hardison et al., 2013) as well as increased amounts of moderately reactive terrestrial organic matter (Bourgeois, et al., 2011) in these deposits (as compared to the WAP stations). Dethier et al. (2014) reported high levels of polyphenolic compounds in certain species of kelp, which can deter both bacterial colonization and herbivory. The labile organic carbon in SCIB sediments is likely to be predominantly of marine origin, because the terrestrial carbon in these deposits is probably older and less reactive (Blair & Aller, 2012) and has been through multiple microbial cycles on land (e.g., in soils and freshwater dispersal systems), making it less labile (Burdige, 2005).

#### 4.2. Comparison of $^{14}\text{C}$ -Based LOC Measurements with LOC Values From the Literature

As described in Section 1, an important aspect of substantiating the utility of this new LOC analytical technique is to compare the  $^{14}\text{C}$ -based LOC  $\tau$  values and LOC abundances with LOC data from the literature. There are many research papers that report organic carbon degradation fluxes for surface sediments (e.g., Hales et al., 1994), however, there are far fewer studies that couple measurements of LOC degradation rate and bioturbation coefficient, such that LOC  $\tau$  values can be calculated accurately. For comparison with our LOC data, we chose the Hammond et al. (1996) study and the Stephens et al. (1997) study from the deep-sea Equatorial Pacific, as well as the Sayles et al. (2001) study from the Southern Ocean ( $170^\circ\text{W}$ ). Table 1 provides a direct comparison of LOC  $\tau$  values, LOC inventories, and bioturbation information between this  $^{14}\text{C}$ -based LOC study and other LOC studies from the literature. In all three of the articles from the literature, a second degradation rate constant ( $k_2$ ) for a more refractory phase also was calculated (representing a relatively small fraction of the total LOC). In this study, we have chosen to contrast our  $^{14}\text{C}$ -based LOC data, which assumes a single labile phase and degradation rate, with the  $k_1$  values (and associated LOC  $\tau$  values) reported in the literature.

The agreement between the  $^{14}\text{C}$ -based LOC  $\tau$  values from the WAP sediments (0.1–0.6 years) and the LOC  $\tau$  data from the literature (0.02–0.4 years from Hammond et al., 1996; typically 0.2–3.4 years from Sayles et al., 2001) is very good, corroborating the overall efficacy of the  $^{14}\text{C}$ -based approach for measuring LOC abundance, inventory, and turnover time. The  $^{14}\text{C}$ -based LOC profiles from the WAP stations yielded LOC e-folding depths that varied from 0.8 to 3.4 cm, which is in good agreement with the e-folding depths from

Hammond et al. (1996) [typical values of 1.1–3.3 cm], from Sayles et al. (2001) [typical values 0.4–2.4 cm], and from Stephens et al. (1997) [typical values 0.2–2 cm for chlorophyll-a]. Sayles et al. (2001) reported one high LOC  $\tau$  value from a low organic carbon flux station located just north of the Sub-Antarctic Front that was equal to 42 years, which is in reasonable agreement with our San Clemente Basin LOC  $\tau$  value of 63 years. Further study is needed to explain the primary causes of these relatively high LOC  $\tau$  values (42–63 years), however, most of the initially studied surface sediments have LOC  $\tau$  values on the order of a few years or less, comparable to the planktonic  $\tau$  values (0.05–0.5 years) reported by Westrich and Berner (1984). Chl-a  $\tau$  values, ranging from 0.05 to 4 years, were reported by Stephens et al. (1997) for Equatorial Pacific sediments located between 0°N and 9°N (including many of the same stations as Hammond et al., 1996). The contribution of chlorophyll-a to the bulk LOC abundance is probably limited because the Equatorial Pacific Chl-a inventories ranged from 2 to 7 ng C/cm<sup>2</sup> (Stephens et al., 1997).

Table 1 also compares the LOC  $\tau$  values, LOC inventories, and bioturbation data from this study to that of other <sup>14</sup>C-based LOC measurements recently added to the literature (Isla & DeMaster, 2018; Taylor, 2018; Taylor et al., 2020). Isla and DeMaster (2018) used the same <sup>14</sup>C approach as this study for resolving LOC from TOC in collapsed ice shelf Larsen B and Larsen A sediments (eastside of Antarctic Peninsula). <sup>14</sup>C-based LOC  $\tau$  values ranged from 3.2 to >60 years in these Larsen A and B cores, with typical values between 4 and 40 years. Thus, the results from the Isla and DeMaster (2018) study are in good agreement with the range of <sup>14</sup>C-based LOC  $\tau$  values reported in this study (0.1–60 years). The Isla and DeMaster (2018) LOC study of the Larsen A and Larsen B region used nonsteady-state <sup>210</sup>Pb profiles (characteristic timescale of years in contrast to this study that used <sup>234</sup>Th profiles, which have a characteristic timescale of months). The longer-lived particle tracers tend to have smaller bioturbation coefficients (Smith et al., 2001), which leads to smaller LOC degradation constants ( $k$ ) and larger LOC  $\tau$  values. The LOC inventories observed in the Larsen A and B sediment cores typically ranged from 2 to 9 mg C/cm<sup>2</sup>, which are somewhat higher than those observed in our study of the WAP shelf (1.8–2.4 mg C/cm<sup>2</sup>; Table 1).

Taylor (2018) also measured LOC characteristics in the Larsen A region using the <sup>14</sup>C-based LOC technique, only the field sites from the Larsen A shelf were selected to represent various times since the Larsen A ice shelf had retreated over the core location (15–17 years in the western Larsen A to 44–170 years in the eastern stations). Nonsteady-state excess <sup>210</sup>Pb profiles were used in this study to establish bioturbation coefficients. The LOC  $\tau$  values for the cores from the western Larsen A (15–17 years since ice shelf collapse) ranged from 6 to 16 years, whereas the LOC  $\tau$  values from the eastern Larsen A had LOC  $\tau$  values that ranged from 34 to 127 years. The western Larsen A cores, dominated by recent accumulation of planktonic organic carbon had lower LOC  $\tau$  values than the LOC  $\tau$  values from the cores with longer duration of ice-free conditions (eastern Larsen A). The LOC  $\tau$  values from the western Larsen A (6–16 years) were somewhat higher than those observed in this study for the WAP shelf (0.1–0.6 years). In contrast, the cores from the eastern Larsen A (LOC  $\tau$  values of 34–127 years) were more comparable to our LOC  $\tau$  value from San Clemente Basin (63 years). Taylor (2018) states that the primary reason for the increase in LOC  $\tau$  values from the western Larsen A to the eastern Larsen A is the increased exposure time to benthic faunal and microbial processing of the seabed, causing an “aging” of the LOC in the sedimentary mixed layer. The LOC inventories in the western Larsen A (9–19 mg C/cm<sup>2</sup>) were higher than the LOC inventories in our current WAP study (1.8–2.4 mg C/cm<sup>2</sup>), whereas the LOC inventories in the eastern Larsen A (27–75 mg C/cm<sup>2</sup>) were much higher than any of the observed LOC inventories in this study, even that from the SCIB core (5.3 mg C/cm<sup>2</sup>).

Taylor et al. (2020) reported <sup>14</sup>C-based LOC data from a variety of depositional environments on the West Antarctic Peninsula shelf, including the Palmer Deep and Andvord Bay fjord. LOC  $\tau$  values ranged from 0.8 to 10 years in surface sediments (in reasonable agreement with the Antarctic data from this study; 0.1–0.6 years), whereas the LOC inventories ranged from 17 to 70 mg C/cm<sup>2</sup>. These Taylor et al. (2020) LOC inventories are an order of magnitude higher than those observed in our study. The main reasons for the difference in these inventories are that Taylor et al. (2020) measured <sup>210</sup>Pb as well as <sup>234</sup>Th as bioturbation tracers, and <sup>210</sup>Pb has a characteristic timescale of 100 years (as compared to the 100-day characteristic timescale of <sup>234</sup>Th), so the bioturbation was characterized to a much greater depth (25–30 cm instead of 3–9 cm for our stations B and G). Along with measuring bioturbation to greater depths, the Taylor et al. (2020) reported much deeper <sup>14</sup>C asymptotes, 25–30 cm, as compared to our West Antarctic Peninsula study that



had 4–10 cm  $^{14}\text{C}$  asymptotes. The deeper in the  $^{14}\text{C}$  profile the asymptotes are, the smaller the  $^{14}\text{C}$  asymptotic values, which leads to higher concentrations of LOC and higher LOC inventories (see Section 4.5).

### 4.3. Substantiating Assumptions Used in the LOC Modeling

The primary assumption of the  $^{14}\text{C}$ -based LOC approach is that sedimentary organic carbon within the surface mixed layer can be adequately represented as a two-component system, comprised of a labile organic carbon phase and a refractory organic phase. Support for this assumption comes from ramped pyrolysis studies (e.g., Rosenheim et al. 2008), in which the amount of carbon dioxide generated during TOC combustion is continuously monitored as the temperature is ramped from 250 to 900 °C. In this technique 4–6 integrated gas samples are collected over the ramped temperature sequence for  $^{13}\text{C}$  and  $^{14}\text{C}$  analysis. Taylor (2018) reported ramped pyrolysis data from a megacore collected in the western Larsen A (Sta. G). The  $\text{CO}_2$  release pattern as a function of combustion temperature showed two dominant peaks for all three samples analyzed (depths of 0–0.5, 4–5, and 10–12 cm). The low temperature peak (resulting from burning the reactive/labile organic matter) exhibited relatively high  $^{14}\text{C}$  abundances (consistent with the results of Komado et al. 2012), whereas the high temperature peak (resulting from burning the more refractory organic matter) exhibited diminished levels of  $^{14}\text{C}$  activity. Taylor (2018) showed that with depth the magnitude of the low temperature (labile) peak diminished, in contrast to the high temperature (refractory) peak whose relative abundance increased consistently. In addition, Taylor (2018) measured the  $^{14}\text{C}$ -based LOC abundances in the same western Larsen A megacore as the pyrolysis measurements and found that they correlated well ( $R^2 = 0.999$ ) with the down-core distribution of the low temperature/labile organic carbon peak area, quantitatively confirming the relationship between these two approaches for characterizing labile organic matter. Rosenheim et al. (2013) also used the pyrolysis approach and measured the  $^{14}\text{C}$  content of the labile, low temperature peak down-core as a means of improving  $^{14}\text{C}$  chronologies in Antarctic marine sediments. The two-peak distribution in the  $\text{CO}_2$  release pattern during ramped pyrolysis provides qualitative support for the two-component model used in this study to represent the reactivity of marine sedimentary organic matter. Lastly, the curvatures in porewater  $\text{O}_2$  profiles (Hammond et al., 1996) and porewater  $\text{NO}_3^-$  profiles (Sayles et al., 2001) in marine sediments are consistent with a two-component organic carbon system, although a three-component regime cannot be ruled out.

The second assumption used to develop the C-14 LOC approach is that the C-14 content (or age) of the LOC is relatively uniform within the bioturbated layer. Corroboration of this assumption comes from Wang et al. (1998), who reported the C-14 contents of THAA (Total Hydrolyzable Amino Acids), carbohydrates, and lipids with depth within the 3-cm bioturbated layer of a deep-sea core from the northeast Pacific Ocean (Sta. M). The C-14 contents of all three biopolymeric phases were fairly constant (no consistent trends) within the upper 3 cm of the seabed. These data corroborate our assumption that the C-14 content of the LOC is relatively uniform within the zone of bioturbation.

An additional assumption in our diagenetic model for labile organic matter was that the effects of sediment accumulation were negligible relative to bio-diffusive particle transport. Long-term sediment accumulation rates were determined below the zone of bioturbation using  $^{14}\text{C}$  geochronology at each of the three sites examined in this study. The sediment accumulation rates (1,000-year timescale) were as follows: WAP (Sta. B: 28 cm/kyr), WAP (Sta. G: 5.6 cm/kyr), and SCIB (14 cm/kyr). Based on these sediment accumulation rates, LOC would be buried a maximum distance of 0.02 cm during the LOC turnover times in WAP sediments and <0.9 cm in SCIB sediments. Both of these distances are small relative to the total penetration depths of LOC into the seabed at these stations.

The diagenetic model used in this study (Berner, 1980) assumes steady-state distributions for the labile organic carbon on a timescale commensurate with that of the characteristic bioturbation tracer (typically five half-lives) and with the LOC  $\tau$  value. In all three of the West Antarctic shelf cores examined in this study the LOC  $\tau$  values were on the order of months (range 1–7 months). LOC inventories at Sta. B varied from  $2.3 \pm 0.8 \text{ mg C/cm}^2$  in 2001 to  $1.8 \pm 0.5 \text{ mg C/cm}^2$  in 2009 (agreeing well within their analytical uncertainties, Table 1). In addition, two Antarctic stations (B and G), separated by a distance of nearly 400 km, exhibited surface sediment LOC  $\tau$  values within this same range. These data support the occurrence of at least a pseudoconstant regime with regard to LOC systematics in both time and space.

Another assumption incorporated into the LOC modeling is that the  $^{14}\text{C}$  activities of surface plankton are representative of the  $^{14}\text{C}$  contents of the organic carbon bioturbated down into the sediment column.  $^{14}\text{C}$  measurements of surface plankton made as part of this study are in good agreement with dissolved inorganic carbon (DIC)  $^{14}\text{C}$  measurements from nearby surface waters reported in the literature (e.g., Berkman & Forman, 1996; Key et al., 2004). More importantly, measurements of the  $^{14}\text{C}$  contents of benthic megafaunal tissue from the same West Antarctic stations are statistically indistinguishable from the  $^{14}\text{C}$  contents of surface phytoplankton (Purinton et al., 2008). Certainly, as planktonic organic matter settles through the water column, the nature of the organic matter may change as a result of zooplankton grazing and ingestion as well as microbial organic carbon remineralization. However, the bulk planktonic organic carbon appears not to change its  $^{14}\text{C}$  activity during transport through the water column (Fabiano et al., 2001). In addition,  $^{14}\text{C}$  Fm values are corrected for compositional fractionation processes based on their  $^{13}\text{C}/^{12}\text{C}$  variations.

In this study bioturbation is treated as a diffusive process, acting on particle concentration gradients (i.e., abundance/g of dry sediment) rather than on a concentration per volume of wet sediment (Fornes, 1999; McClintic et al., 2008; Smith et al., 2001, 2006). Certainly, there is evidence within our field data showing that not all bio-transport is diffusive (e.g., see  $^{14}\text{C}$  profile from Sta. B (2009) in this paper). However, the radiochemical specific activity data (abundance/g dry sediment) and the LOC concentrations (g C/g dry sediment), in general, exhibited exponentially decreasing profiles with depth, consistent with diffusive particle mixing. When evidence of nonlocal advective mixing occurred, the corresponding data were not incorporated into the modeling of the LOC data (see points represented by red stars in Figure 4).

#### 4.4. Comparison of the $^{14}\text{C}$ -Based LOC Determinations With Other Techniques

Throughout this article, the presumption has been made that the  $^{14}\text{C}$ -based approach for determining labile organic carbon abundances and turnover times is as good or better than other proxies and tracers of reactive organic matter (e.g., biopolymeric organic carbon, enzymatically hydrolyzable amino acids (EHAA), or porewater oxidant/metabolite profiles). Consequently, a comparative discussion of the various analytical techniques is warranted. Biopolymeric organic carbon (i.e., the sum of lipid, protein, and carbohydrate abundances) has been used by several researchers to estimate reactive organic carbon concentrations in marine surface sediments (Fabiano & Pusceddu, 1998; Grémare et al., 2003; Isla et al., 2006; Navarro et al., 1993). Isla and DeMaster (2018) provide a direct comparison of the  $^{14}\text{C}$ -based LOC approach with biopolymeric organic carbon assessments in five cores from the Larsen B region. In three of the five cores, a statistically significant ( $p < 0.05$ ) correlation was observed between  $^{14}\text{C}$ -based LOC abundances and biopolymeric organic carbon abundances. Tselepidis et al. (2000), Danovaro et al. (2001), and Lee et al. (2004) reported many inconsistencies in reactivities among organic compound classes, comprising biopolymeric organic carbon. Wang et al. (1998) and Wang and Druffel (2001) measured the  $^{14}\text{C}$  content of organic compound classes with depth in two cores from the northeastern Pacific Ocean and one from the Southern Ocean (Pacific sector). They found that the carbohydrate fraction had the highest  $^{14}\text{C}$  abundances (i.e., youngest), the total hydrolyzable amino acid fraction had intermediate  $^{14}\text{C}$  abundances, and the lipid fraction had the lowest  $^{14}\text{C}$  abundances.

Another approach for assessing bioavailable/reactive organic matter in marine sediments has been the measurement of EHAA (Dauwe et al., 1999a, 1999b; Garcia et al., 2010; Mayer et al., 2002; Mincks et al., 2005). Dauwe et al. (1999a) established a degradation index, based on the relative amounts of selected protein amino acids in a sample, as a way of characterizing organic matter bioavailability. However, when EHAA abundances were measured in North Sea sediments (Dauwe et al., 1999b), three of the five sediment cores showed little or no down-core variation in EHAA abundance in the upper 15–20 cm of the seabed, so LOC was difficult to quantify and no LOC  $\tau$  values could be calculated for those stations. Mincks et al. (2005) and Sañé et al. (2013) reported that EHAA measurements in Antarctic Peninsula sediments could have blank problems at some stations (i.e., detectable EHAA well below the zone of bioavailable organic matter).

Measuring porewater profiles of oxidants/metabolites (such as  $\text{O}_2$  or  $\text{NO}_3^-$ ) probably has been the most successful of all of the non- $^{14}\text{C}$  based techniques for calculating LOC abundances and LOC  $\tau$  values in marine sediments (Hammond et al., 1996; Sayles et al., 2001). The porewater approach was used to substantiate LOC characteristics determined using the  $^{14}\text{C}$ -based technique, as mentioned earlier in

the discussion. Employing the porewater approach often requires extensive seagoing equipment (such as benthic landers, whole core squeezers, glove bags, gimbaled centrifuges, or porewater harpoons) as well as analytical instrumentation (e.g., oxygen microelectrodes). In contrast, the  $^{14}\text{C}$ -based technique, only requires megacore sampling at sea and frozen sample storage. In order to calculate organic carbon consumption rates using the porewater oxidant/metabolite approach, the stoichiometric ratio between organic carbon oxidized ( $\text{C}_{\text{ox}}$ ) and oxidant consumed (or metabolite produced) must be measured or assumed. Hammond et al. (1996) reported that the  $\text{O}_2:\text{C}_{\text{ox}}$  ratio varied from 1.2 to 3.2 for equatorial Pacific stations between  $2^\circ\text{S}$  and  $5^\circ\text{N}$ . Consequently, assessing the abundance of reactive organic matter (LOC) in marine sediments, based on the  $^{14}\text{C}_{\text{org}}$  gradient in the surface mixed layer, appears to be one of the most viable approaches for measuring LOC abundances and turnover times. This approach has proven effective in all of the sedimentary environments in which it has been employed (i.e., West and East Antarctic Peninsula shelves (Isla & DeMaster, 2018; Taylor, 2018; Taylor et al., 2020; this study) and California Borderland (this study)).

#### 4.5. Sensitivity Analysis and Potential Limitations of the Approach

Error propagation was conducted throughout the  $^{14}\text{C}$  data work up (see Table S2). The range in the relative uncertainties for the LOC values was typically about 50% (ranging from 8% to 140%). The relatively large error in the calculated LOC values originates from the error associated with subtracting somewhat similar  $\text{Fm}$  values for  $\text{Fm}_{\text{PLANKTON}}$ ,  $\text{Fm}_{\text{SAMPLE}}$ , and  $\text{Fm}_{\text{ASYMPTOTE}}$  (see Equation 2). Most of the error comes from the numerator in this equation, in which the  $\text{Fm}_{\text{ASYMPTOTE}}$  is subtracted from the  $\text{Fm}_{\text{SAMPLE}}$  (with the difference decreasing to zero with depth). The relative error in the LOC inventories (i.e., integration of LOC profile with depth) is considerably less, averaging 23% (range from 8% to 35%) in the four cores examined as part of this study.

LOC  $\tau$  values typically had a relative uncertainty of about 50–65% (range of 40–104%). Given the paucity of LOC data in the literature, these are significant contributions, despite the relatively large error bars, to our understanding of LOC turnover times, which rarely are determined in most diagenetic studies. The large relative error in the LOC  $\tau$  values is understandable because the error in the LOC  $\tau$  value comes from the error in the bioturbation coefficient ( $D_b$ , typically about 25%) plus twice the relative error in the slope of the  $\text{Ln}(\text{LOC})$  versus depth profile (typically 20%) (because “ $k$ ” =  $D_b$  (slope) $^2$ ).

High-precision Accelerator Mass Spectrometer analyses have a relative uncertainty of approximately 0.4%. Despite these precise measurements, the calculation of LOC abundances has a fairly high uncertainty associated with it, because of the reasons (i.e., error propagation) mentioned above. The greater the difference between  $\text{Fm}_{\text{PLANKTON}}$  and  $\text{Fm}_{\text{ASYMPTOTE}}$ , the smaller the relative error in the LOC abundance value. For example, in the collapsed ice shelf studies on the East Antarctic Peninsula shelf (Taylor, 2018), the difference between  $\text{Fm}_{\text{PLANKTON}}$  and  $\text{Fm}_{\text{ASYMPTOTE}}$  was large because the organic carbon underneath the ice shelf was quite old ( $>10$  kyr), which led to reduced relative error in the LOC abundances. Similarly, the difference between  $\text{Fm}_{\text{PLANKTON}}$  and  $\text{Fm}_{\text{ASYMPTOTE}}$  was relatively large in the California Borderland sediments (this study) because the local plankton have a bomb  $^{14}\text{C}$  signal ( $\text{Fm} = 1.0737$ ) and the  $^{14}\text{C}_{\text{org}}$  asymptotic value is 0.7552 (equivalent to an age of 2,200 years). In this study, the linearity in the  $\text{Ln}(\text{LOC})$  versus depth plots is quite good, with  $R^2$  values ranging from 0.69 to 0.99 and  $p$  values ranging from 0.00001 to 0.079 (see Figure 4). These trends provide confidence in the overall approach of using  $^{14}\text{C}_{\text{org}}$  data to assess the abundances and turnover times of Labile Organic Carbon (LOC) in marine sediments.

The  $^{14}\text{C}$ -based approach to assessing LOC abundances and LOC  $\tau$  values is limited to some extent by the accuracy of assessing the depth of the asymptote in the  $^{14}\text{C}_{\text{org}}$  profile (usually presumed to be the base of the bioturbated sediment layer; see Section 4.2). The LOC inventory is quite sensitive to the depth of the  $^{14}\text{C}$  asymptote, as evidenced by differences in LOC abundances and inventories in West Antarctic Peninsula shelf sediments as measured in this study ( $^{14}\text{C}$  asymptotes equaled 4–10 cm depth versus the  $^{14}\text{C}$  asymptotes measured in Taylor et al. (2020) in which the  $^{14}\text{C}$  asymptote equaled 25–30 cm depth). Both depictions of LOC characteristics can be correct; they simply characterize LOC of different reactivity and different timescale (i.e., 100-day timescale when using  $^{234}\text{Th}$  as the bioturbation tracer in near-surface sediments, which tracks more labile organic carbon, and 100-year timescale when using  $^{210}\text{Pb}$  as the deep bioturbation tracer of slower sediment mixing of less reactive LOC) (Smith et al., 1993; Fornes et al., 2001). A more robust defi-

dition of LOC (e.g., based on organic carbon molecular structure) would be optimal for studies such as this one, however, no such analysis exists currently. Until a molecular-based assay is developed for determining LOC, this radiocarbon-based approach is useful in revealing the distribution, turnover time, and diagenetic fate of LOC in marine sediments.

#### 4.6. Additional Implications and Insights From the $^{14}\text{C}$ -Based LOC Approach

Having corroborated the utility of the  $^{14}\text{C}$ -based approach for determining LOC abundances and LOC turnover times, this radiochemical technique can be used to explore other aspects of LOC characteristics. Relatively little data exist on the distribution of Labile Organic Carbon (LOC) in marine sediments, and even less information is available concerning LOC  $\tau$  values (or reactivity) in these deposits. Several studies have indicated how important mineralogical substrates can be in preserving reactive organic matter in marine sediments (Blattmann et al., 2019; Hemingway et al., 2019; Keil et al., 1994). Such studies would benefit significantly by knowing the abundance and turnover time of LOC in surface sediments (as provided by the  $^{14}\text{C}$ -based LOC approach).

There are only a few studies (e.g., Hammond et al., 1996; Sayles et al., 2001) in which there is any inference on the trend of LOC turnover time with depth in the sediment column. As mentioned in Section 4.2, Taylor et al. (2020) measured LOC abundance in the upper 22 cm of West Antarctic Peninsula sediments using this  $^{14}\text{C}$  approach. Bioturbation effects were measured in the upper 2–5 cm using  $^{234}\text{Th}$ , whereas the deeper bioturbation intensity (3–22 cm) was assessed using  $^{210}\text{Pb}$ . These data, along with the  $^{14}\text{C}_{\text{org}}$  profile, enabled assessment of LOC  $\tau$  values in the rapidly mixed surface layer (LOC  $\tau$  = 0.8–10 years) as well as in the more slowly mixed subsurface layer (LOC  $\tau$  = 32 to >600 years). Clearly, the LOC is “aging” and becoming less reactive over time as a result of diagenetic reactions, such as processing by benthic fauna as well as microbial degradation.

The  $^{14}\text{C}$ -based LOC approach also can be used to examine the fate of sedimentary LOC during diagenesis following sudden changes in the nature of organic carbon deposition, such as that following ice shelf collapse. Based on the  $^{14}\text{C}$ -gradient technique, Isla and DeMaster (2018) were able to examine the fate of LOC in the seabed (distribution and turnover) following the 2002 ice-shelf collapse of the Larsen B (East Antarctic Shelf). Multicores were collected from the ice-shelf-free Larsen B area during 2007, with reoccupation of four sites in 2011. This sampling approach enabled “snapshots” of LOC characteristics at reoccupied stations 5 and 9 years after ice-shelf collapse. Consequently, the  $^{14}\text{C}$  approach enabled assessment of non-steady-state LOC characteristics (such as LOC inventory and LOC  $\tau$ ) as the seabed became progressively enriched in plankton (and LOC), following ice-shelf collapse. As new  $^{14}\text{C}$  data bases become established in the literature, e.g., van der Voort et al. (2020), it would be useful to examine sedimentary  $^{14}\text{C}$  profiles near the sediment-water interface from other locations to assess the ubiquity of the  $^{14}\text{C}$  decreasing profiles observed in these Antarctic Peninsula and California Borderland studies.

### 5. Conclusions

Measurement of  $^{14}\text{C}_{\text{org}}$  profiles in the upper 5–10 cm of the seabed has been shown to be a useful technique for quantifying labile organic carbon (LOC) abundances in marine sediments. A steady-state diagenetic model for labile organic matter, which incorporates sediment bioturbation coefficients, was used to determine LOC turnover times, LOC e-folding depths, and LOC inventories. These LOC data were compared to LOC literature values (primarily based on porewater profiles) to evaluate the efficacy of the overall  $^{14}\text{C}_{\text{org}}$ -based approach. Consistent agreement between the values from this study and those reported in the literature provides confidence that the relatively new  $^{14}\text{C}_{\text{org}}$  analytical technique is scientifically sound and useful as a tool for exploring additional LOC processes occurring in the seabed (such as aging) as well as characterizing the nonsteady state evolution of LOC characteristics in marine sediments, following ice-shelf collapse. The  $^{14}\text{C}_{\text{org}}$  approach for measuring sedimentary LOC is consistent with the observation that benthic fauna and microbes receive nearly all of their nutrition/energy by assimilating labile organic carbon (primarily recently deposited planktonic material) and not bulk organic carbon from the seabed.



## Data Availability Statement

Original analytical data from this study have been submitted to the following FAIR-aligned data repository (US Antarctic Program Data Center; Dataset ID: 601319; DOI: 10.15784/601319). However, all of this information is provided in the Supplemental Data section of this paper as well (Tables S1 and S2 and Figure S1).

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