



Paired ¹⁴C-¹⁰Be exposure ages from Mount Murphy, West Antarctica: Implications for accurate and precise deglacial chronologies

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- 10 Abstract. Cosmogenic-nuclide surface exposure ages provide empirical data for testing the accuracy of models simulating the timing and pace of ice sheet response to a warming climate. Increasing emphasis is being placed on obtaining exposure ages that both accurately constrain Holocene deglaciation and are precise enough to capture ice sheet change at the submillennial scale. However, the accuracy of Holocene deglacial chronologies can be compromised by nuclide inheritance when measuring longer-lived nuclides, such as ¹⁰Be. Short-lived in situ-produced ¹⁴C is unique because it is largely insensitive to
- 15 nuclide inheritance pre-dating the last glacial maximum (LGM), and when combined with longer-lived nuclides can be used to constrain complex ice sheet histories over Holocene timescales. Here, we present new in situ ¹⁴C exposure ages from Mt Murphy, West Antarctica. Many of the new in situ ¹⁴C ages are inconsistent with published ¹⁰Be ages, suggesting samples collected from the same elevation above the modern ice were exposed at different times. We investigate potential explanations for such conflicting exposure histories by analysing paired ¹⁴C-¹⁰Be data of Holocene age presently archived in the informal cosmogenic-
- 20 nuclide exposure-age database (ICE-D, <u>https://version2.ice-d.org/</u>). Our analysis reveal that neither geologic sources of uncertainty due to variations in geologic setting nor modelled scenarios of subsurface nuclide production explain conflicting paired ¹⁴C-¹⁰Be exposure ages observed at Mt Murphy. Furthermore, we observe that repeat in situ ¹⁴C concentrations measured in 15 of 31 samples do not replicate within their nominal 6 % (2σ) analytical uncertainty and identify ~ 2 kyr of excess unquantified scatter from Mt Murphy in situ ¹⁴C exposure ages. Taken together, these results suggest analytical uncertainty for in situ ¹⁴C
- 25 measurements may currently be underestimated. We provide recommendations for improving measurement precision that will benefit future Holocene deglaciation studies including analysis and publication of more replicate measurements, and the continuation of efforts to quantify and minimise sources of scatter in blank measurements.





1. Introduction

- Increasing emphasis is being placed on glacial chronologies that both constrain the timing of ice surface change during the
 Holocene epoch and provide validation for model simulations at sub-millennial scale resolution (Hippe, 2017; Nichols et al., 2019; Jones et al., 2022; Johnson et al., 2022). To provide Holocene deglacial chronologies for ice sheet models at sub-millennial scale resolution, cosmogenic radionuclide (e.g., in situ ¹⁴C and ¹⁰Be) exposure ages must both be accurate and precise. Accurately determining a Holocene exposure age relies on the assumption that the sample being dated is free from nuclides accumulated during periods of surface exposure that pre-date the LGM (Balco, 2011). The prevalence of cold-based ice and subsequent lack of basal
 erosion, however, often leads to longer-lived nuclides such as ¹⁰Be (half-life; 1.387 Myr) persisting over multiple glacial cycles impacting the accuracy of deglacial chronologies (Balco, 2011; Hein et al., 2014). The short half-life of in situ ¹⁴C of (5700 ± 30)
- years), results in the total inventory of in situ ¹⁴C in a sample decaying to below detectable levels in ~30 kyr, making in situ ¹⁴C largely (and uniquely) insensitive to pre-LGM exposure. Following efforts to develop and improve in situ ¹⁴C extraction (Lifton, 1997; Lifton et al., 2001, 2015b; Hippe et al., 2009, 2013; Fülöp et al., 2010, 2015; Goehring et al., 2014, 2019a; Lamp et al., 2019)
- 40 in situ ¹⁴C, has been increasingly applied to accurately determine Holocene exposure ages where ¹⁰Be inheritance is known or suspected (White et al., 2011; Briner et al., 2014; Nichols et al., 2019). Combining analyses of the short-lived in situ ¹⁴C nuclide with longer-lived ¹⁰Be (or ²⁶Al) presents a valuable approach to reveal and quantify complex exposure histories (Hippe, 2017). However, there is added value in this approach if measurement precision of both nuclides is sufficient to resolve past ice sheet behaviour at the sub-millennial timescale, which is necessary to distinguish early-mid Holocene retreat of the West Antarctic Ice
- 45 Sheet from possible late Holocene ice sheet readvance for which there is emerging evidence (Kingslake et al., 2018; Venturelli et al., 2020; Balco et al., 2023; Venturelli et al., 2023).

In this study, we measured in situ ¹⁴C in a selection of samples from Mt Murphy, a volcanic edifice adjacent to Thwaites Glacier in the Amundsen Sea Embayment (Fig. 1a) to investigate if published ¹⁰Be ages (Johnson et al., 2020; Adams et al., 2022) contained small amounts of nuclide inheritance. However, new in situ ¹⁴C ages suggest two conflicting exposure histories at Mount

- 50 Murphy. Some paired in situ ¹⁴C-¹⁰Be ages from the same sample are concordant (paired ¹⁴C-¹⁰Be ages agree within uncertainty), indicating the sample experienced a simple post-LGM exposure history. Other samples returned discordant exposure ages (paired ¹⁴C-¹⁰Be ages did not overlap within analytical uncertainty) indicating that, since post-LGM exposure, a sample experienced burial or there were changes in the nuclide production rate (Balco et al., 2019). Discordant in situ ¹⁴C-¹⁰Be exposure ages have previously permitted detection of considerable inheritance in ¹⁰Be nuclide concentrations ranging from 10s–100s kyr (e.g., Nichols et al., 2019).
- 55 However, the paired ¹⁴C-¹⁰Be data from Mt Murphy presented here are distinct because i) both new in situ ¹⁴C and ¹⁰Be exposure ages are younger than the LGM and ii) replicate in situ ¹⁴C ages presented in this study do not reproduce within currently stated uncertainties to such an extent they suggest conflicting mid and late-Holocene exposure histories, which is problematic given the need for exposure age chronologies capable of reconstructing post-LGM ice sheet change on sub-millennial timescales.

Here, we describe an investigation into potential explanations for co-existing concordant and discordant paired ¹⁴C-¹⁰Be 60 Holocene exposure ages observed at Mt Murphy. We do this by revisiting the data of Johnson et al., 2020 and Adams et al., (2022),





and performing a more in-depth examination of sources of uncertainty associated with both in situ ¹⁴C and ¹⁰Be exposure ages. First, we present a new in situ ¹⁴C dataset from Mt Murphy (paired with previously published ¹⁰Be measurements) (Fig. 1a) and assess the accuracy and reproducibility of this new dataset. We then contextualise the new Mt Murphy dataset by analysing available ¹⁴C-¹⁰Be paired exposure age data that is of Holocene age (< 11.7 ka) from Antarctica (Fig. 1b) and globally (Fig. 1c). These paired ¹⁴C-¹⁰Be data are primarily sourced from the Informal Cosmogenic-nuclide Exposure age Database (ICE-D; Balco, 2020b) (https://version2.ice-d.org/). Through this work we aim to use the new and existing paired ¹⁴C-¹⁰Be exposure age data to assess, progress and identify steps the community could take to consistently produce robust Holocene glacial chronologies.

1.1 Sources of uncertainty that impact in situ ¹⁴C and ¹⁰Be exposure ages

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To provide additional context for our results and discussion, we first outline sources of uncertainty that need to be accounted for when calculating in situ ¹⁴C and ¹⁰Be exposure ages. The source of uncertainty over which cosmogenic nuclide practitioners have the least control is geologic uncertainty, inherent in a sample from its time of collection in the field and rooted in the limited knowledge we have of a samples true exposure history and any processes that may have modified production of nuclides following exposure (Dunai, 2010). The two main sources of geologic uncertainty are nuclide inheritance (described above) and post 75 depositional disturbance caused by shielding, erosion, and/or rolling of a sample (Gosse and Phillips, 2001; Balco, 2011). Steps taken to reduce geologic uncertainty include a robust and detailed geologic interpretation of deposits or depositional features being dated (Balco, 2011) and statistical techniques (e.g., Johnson et al., 2014; Heyman et al., 2016). Comprehensive summaries of geologic uncertainty and previous and ongoing efforts to quantify it can be found in Balco et al., (2011, 2020b).

The second major source of uncertainty comes from our ability to measure the nuclide concentration accurately and 80 precisely within a sample. A crucial distinction, and source of potential confusion, is that we commonly refer to the accuracy and precision associated with determining the concentration of a sample (Jull et al., 2015), which itself forms a component of the total uncertainty of an exposure age (Balco, 2020a). Data producers make efforts to minimise contributions of measurement uncertainty from two main sources: uncertainties introduced during sample preparation and sample measurement by accelerator mass spectrometry (AMS). Measurement of the cosmogenic nuclide ¹⁰Be is now relatively well-established and routine following

incremental efforts to reduce sources of laboratory sample preparation uncertainty (Kohl and Nishiizumi, 1992; Corbett et al., 2016, 85 2022) and improve AMS performance (Rood et al., 2010, 2013; Merchel et al., 2012; Wilcken et al., 2022). These efforts have resulted in ¹⁰Be measurement precision on typical quartz interlaboratory comparison materials (e.g., CRONUS-A, CoQtz-N) of between ~2-4 % (Jull et al., 2015; Phillips et al., 2016a; Binnie et al., 2019).

Measurement of the isotope 14C (radiocarbon) by AMS is also routine with precision consistently reported in the range of 90 2-3% (Roberts et al., 2010; Scott et al., 2017; Aerts-Bijma et al., 2021). In situ ¹⁴C measurement from quartz is, however, less mature, in part due to the significant challenges associated with its sample preparation and extraction (Lifton et al., 2001; Hippe et al., 2009; Fülöp et al., 2010; Goehring et al., 2014; Lifton, 1997). Laboratory intercomparison studies of CRONUS-A indicate the coefficient of variation (CoV) of in situ 14 C is currently in the range of 6–8 %, double the values reported for 10 Be (Phillips et al., 2016a). Recent improvements to the in situ ¹⁴C extraction process include identification of potential contaminant "dead" carbon





- 95 sources during quartz purification (Nichols and Goehring, 2019), and automation of ¹⁴C extraction lines to reduce risk of atmospheric ¹⁴C contamination and potential for human error (e.g., Lifton et al., 2015, 2023; Goehring et al., 2019b; Lupker et al., 2019). Potential refinement of the in situ ¹⁴C combustion step is also being explored (Lifton et al., 2023) as well as omitting graphitisation in favour of directly analysing in situ ¹⁴C using gas source AMS (e.g., Lamp et al., 2019).
- The final major source of uncertainty comes from transforming a measured nuclide concentration into an exposure age, which requires estimating the production rate due to secondary spallation reactions, which accounts for the majority of surface production (Dunai, 2010), and by muons (Balco, 2017). Production rate uncertainties have been incrementally reduced by improvements in scaling models, especially using more recent models based on particle-physics simulations (Lifton et al., 2014; Argento et al., 2015a, b). Estimates of the ¹⁰Be production rate uncertainty from spallation are currently in the range of 6 % (Borchers et al., 2016; Marrero et al., 2016). However, in the case of in situ ¹⁴C a spallogenic production rate uncertainty could not be fitted to the data because of the large scatter in measured in situ ¹⁴C concentrations (in excess of an assumed 7.3 % measurement uncertainty) observed at in situ ¹⁴C calibration sites (Borchers et al., 2016).

Muons account for a smaller proportion of total cosmogenic nuclide production at the surface, but this quantity differs for ¹⁰Be and in situ ¹⁴C. For ¹⁰Be the proportion of cosmogenic nuclide production by muons at the surface is between 1.5–2 %. In the case of in situ ¹⁴C ~ 20 % of surface production is estimated to be from muons (Lupker et al., 2015). The total uncertainty on

- 110 computing a production rate by muons at an arbitrary location has been estimated between 10–25 % (Balco, 2017). This results in a total maximum scaling uncertainty of only 0.5 % for estimating ¹⁰Be production by muons. However, for in situ ¹⁴C, the same 10–25 % uncertainty on calculating production by muons equates to a 5 % uncertainty on the total surface production rate estimate (Balco, 2017). Because the in situ ¹⁴C production rate by muons as a proportion of total surface production is an order of magnitude higher than for ¹⁰Be, it results in the ¹⁴C-¹⁰Be production ratio increasing with depth below the surface (Hippe, 2017). A sample that
- 115 is buried under a thin layer of rock, ice, till or other material, and then rapidly exhumed by plucking can, therefore, exhibit seemingly "impermissible" paired ¹⁴C-¹⁰Be concentrations due to differences in the ¹⁴C-¹⁰Be total production ratio at the surface versus at depth (Hippe, 2017; Rand and Goehring, 2019).

In summary, sources of geologic, sample preparation and exposure age calculation uncertainty impact the accuracy and precision of Holocene deglaciation chronologies. An increase in paired ${}^{14}C{}^{-10}Be$ measurements in the recent ~ 5 years, driven by

120 greater ¹⁴C extraction throughput (Lifton et al., 2015b; Goehring et al., 2019a) provide many new data to make an assessment of the application of both nuclides and investigate sources of uncertainty, particularly of in situ ¹⁴C. In the following sections, we investigate the cause of concordant and discordant paired in situ ¹⁴C-¹⁰Be exposure ages at Mt Murphy and potential causes for the large amounts of scatter in reported in situ ¹⁴C measurements using both the new in situ ¹⁴C data from Mt Murphy and existing paired in situ ¹⁴C-¹⁰Be data extracted from ICE-D.













Figure 1 (overleaf): Panel (a) a Landsat-9 satellite image of the Turtle Rock, scoria cone and Notebook Cliffs sites at Mt Murphy showing locations of samples with new in situ ¹⁴C exposure ages and previously published ¹⁰Be exposure ages. Grounding line position uses data from (Milillo et al., 2022) and Antarctic Coastline is from version 7.7 of the Antarctic Digital Database. Panel (b) shows Antarctic and Panel (c) global site locations of paired ${}^{14}C{}^{-10}Be$ ages (sites 1-29) where both: i) apparent ${}^{10}Be$ exposure ages are < 4x older than apparent ¹⁴C exposure ages ii) ¹⁰Be exposure ages are of Holocene age (< 11.7 ka). Site numbering uses the 130 order of the specific site ID (lowest to highest) that locations have been assigned in ICE-D (Balco, 2020b). Panel (b) Antarctic paired ¹⁴C-¹⁰Be site locations (1-16) are specified in an inset figure key. Panel (b) abbreviations indicate the Antarctic Peninsula (AP), Amundsen Sea Embayment (ASE), Ross Sea embayment (RSE) and Weddell Sea embayment (WSE). Details of global site locations (17-29) displayed in **Panel (c)** are specified in Results. Table 2. Green squares in Panel c) indicate locations where multiple in situ ¹⁴C measurements have been made on the same sample including Lake Bonneville, Utah, Northwest Highlands, Scotland and 135 Leymon High, Northwest Spain (see Fig. 9, Table S3). Note in Panel (a) the corresponding site number from the global site index (1-29) is specified in **bold** italics along with the name of the sample site, e.g., Turtle Rock (2). Note in **panel** (b) paired in situ ¹⁴C-¹⁰Be sites 2, 7, 11 and 13 also contain replicate in situ ¹⁴C measurements but only the paired ¹⁴C-¹⁰Be symbol (orange circle) is displayed.

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2 Methods

2.1 In situ ¹⁴C analysis of Mt Murphy samples

We selected nine samples from Mt Murphy (Table 1) for in situ ¹⁴C analyses that had previously been measured for ¹⁰Be (Johnson et al., 2020; Adams et al., 2022). The vertical thinning history derived from these ¹⁰Be analyses implies the latest period of exposure of those samples occurred during the Holocene. We ensured that paired ¹⁰Be and ¹⁴C exposure ages cover a wide elevation range by selecting samples from three different locations around the Mt Murphy massif (Notebook Cliffs, samples collected from 893–834 m a. s. l., Turtle Rock, 700–441 m a. s. l., and a scoria cone adjacent to Kay Peak, 240–180 m a. s. l.). A general geological description of these sites (see Fig. 1a) is provided in Johnson et al (2020) and Adams et al., 2022. Geomorphic descriptions and supporting information of the nine samples with paired ¹⁴C-¹⁰Be measurements are provided in Supplement S1,and

150 Table S2.

We selected five of the nine samples: CIN-108, CIN-112, TUR-117, TUR-132 and NOT-103, for repeat in situ ¹⁴C measurements. The in situ ¹⁴C extraction of NOT-103 failed, therefore, only four in situ ¹⁴C measurements were repeated. We performed quartz mineral separation for in situ ¹⁴C samples at Imperial College London largely following Corbett et al., (2016) We omitted the froth flotation step (used to separate feldspars and quartz) following recommendations made by Nichols and Goehring

- (2019), and instead performed 3 x 1 % HF/HNO₃ etches to isolate the quartz. Quartz purity was determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES), after which ~10 g of purified quartz from each sample was sent to Tulane University (New Orleans, USA) for in situ ¹⁴C extraction. Extraction of in-situ ¹⁴C (n = 9) was performed using the fully automated Carbon Extraction and Graphitisation System (CEGS) at Tulane University following the methods of Goehring et al., (2019a). The quartz sample was fused with lithium metaborate (LiBO₂) flux to ensure total sample melt <1300 °C and complete</p>
- 160 liberation of in-situ ¹⁴C (Lifton et al., 2001). First quartz was step heated at 500°C for 1 hour to remove atmospheric ¹⁴C before being combusted at 1100°C for 3 hours to liberate in situ ¹⁴C (in the form of CO₂). Liberated CO₂ was cryogenically purified before being collected in a measurement chamber, quantified monometrically and diluted with ¹⁴C-free CO₂ to ensure a measurable sample size (Goehring et al., 2019a). CO₂ was graphitized using standard H₂ reduction methods over an Fe catalyst (Slota et al., 1987).





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Several changes were made to the configuration of the extraction line prior to the replicate measurements (n = 4). Alterations to the line included a new coil trap, which changes how gas is extracted and trapped following combustion (Lifton et al., 2023) and the introduction of a new mullite tube for ¹⁴C extraction due to failure of the previous tube. Mullite tubes often undergo a "break in" period, during which initial ¹⁴C blanks are higher but often fall with continued use (Goehring et al., 2014, 2019a; Pigati et al., 2010).

¹⁴C/¹²C isotope ratios were measured by accelerator mass spectrometry at the National Ocean Sciences Accelerator Mass Spectrometry Facility (NOSAMS) (Woods Hole, USA) using the methods described in Longworth et al., (2015) Stable isotope $(\delta^{13}C)$ analysis was undertaken at the University of California, Davis Stable Isotope Facility. Data reduction (to convert ${}^{14}C/{}^{12}C$ 170 ratios to ¹⁴C/C_{total}) followed methods outlined in Hippe and Lifton (2014) which account for the specific differences of ¹⁴C production within a mineral lattice compared to ¹⁴C incorporation into organic material. The long-term average ¹⁴C blank used to correct the initial measurements of in situ ¹⁴C (n = 9) is $4.53 \pm 0.24 \times 10^4$ atoms g⁻¹. For replicate measurements (n = 4), a higher short-term average ¹⁴C blank of $7.14 \pm 0.30 \times 10^{4}$ ¹⁴C atoms g⁻¹ is used (Table S1).

We assigned a 6 % (1 σ) uncertainty to each in situ ¹⁴C measurement concentration reported by AMS before calculating 175 exposure ages. The 6 % uncertainty follows recommendations of Hippe (2017) and others that reporting of in situ ¹⁴C data should fully propagate uncertainty relating to intra-laboratory scatter as well as AMS measurement errors and errors relating to blank correction. The 6 % uncertainty exceeds the reported analytical uncertainty for all in situ ¹⁴C measurements made for this study and reflects the repeatability of measurements of CRONUS-A extracted at Tulane. This 6% uncertainty has been adopted by studies 180 where in situ ¹⁴C extraction was carried out at Tulane University, e.g., Nichols et al., (2019). The in situ ¹⁴C concentration of CRONUS-A extracted at Tulane has a long-term value of $6.12 \pm 0.32 \times 10^5$ (n = 10) (Goehring et al., 2019a).

We calculated exposure ages for the new in situ ¹⁴C measurements, as well as for the published ¹⁰Be measurements using version 3 of the online calculators (https://hess.ess.washington.edu/math/v3/v3 age in.html) with the "LSDn" production rate scaling method for neutrons, protons, and muons (following Lifton et al. (2014) and summarised in Balco (2017)) and the primary production rate calibration data set of Borchers et al. (2016). When comparing in situ ¹⁴C and ¹⁰Be exposure ages at Mt Murphy, we 185 used the external uncertainty, which includes the 6 % 10 measurement uncertainty propagated in quadrature with the production rate and scaling scheme uncertainties. We report all exposure ages assuming no erosion or snow cover (making them "apparent" exposure ages) and a sample density of 2.7g cm⁻³ to maintain consistency with Johnson et al. (2020) and Adams et al., (2022). In situ ¹⁴C AMS data and corresponding calculated exposure ages are available from the NERC UK Polar Data Centre, 190 https://doi.org/10.5285/dbb30962-bbf3-434a-9f27-6de2f61a86e2 (Adams et al., 2024).

2.2 Extraction of paired ¹⁴C-¹⁰Be analyses from ICE-D and paired nuclide diagrams

To compare paired ¹⁴C-¹⁰Be exposure ages from Mt Murphy with other ¹⁴C-¹⁰Be datasets, we used a SQL search filter implemented in MATLAB to extract ¹⁴C-¹⁰Be pairs from ICE-D (Balco, 2020b), <u>https://version2.ice-d.org/antarctica/</u> [last accessed 29.03.2024] which meet the following criteria: 1) the ratio of the ¹⁰Be exposure age to the ¹⁴C exposure age is < 4:1, and 2) the ¹⁰Be 195 apparent exposure age is < 11.7 ka (indicating the sample was exposed during the Holocene). We applied these filters to remove





¹⁰Be apparent exposure ages older than the Holocene because ¹⁰Be inheritance is known to impact measurement accuracy. Sites where paired ¹⁰Be and ¹⁴C measurements satisfy the filter criteria are listed in Table 3 and numbered in Fig. 1c.

- Paired nuclide diagrams (Fig. 2) represent a useful method of presenting exposure/burial histories and can potentially indicate uncertainty or help to explain scatter in a dataset (see Granger, 2006, for a detailed description of paired nuclide diagrams). We classified paired nuclide plots generated from ¹⁴C-¹⁰Be pairs extracted from ICE-D into three distinct types: Type 1 represents a sample with simple exposure history (only one period of exposure), Type 2 represents a sample with a complex exposure history (multiple periods of exposure and burial), and Type 3 represents a sample with an impermissible concentration ratio (where an ellipse plots above the line of constant exposure in the "forbidden zone"). The Type 3 scenario can indicate analytical inconsistencies, for example, ¹⁴C contamination increasing in situ ¹⁴C concentrations (Nichols and Goehring, 2019). In certain cases,
- a "Type 3" nuclide ratio may be explained geologically because the constant exposure line assumes a surface production rate rather than subsurface production.



Figure 2: Paired nuclide diagram with key features labelled. Note that the X axis includes the concentration of the longer-lived nuclide, in this case ¹⁰Be, and the Y axis is the ratio of the concentration of the shorter- to longer-lived nuclide, in this case ¹⁴C-¹⁰Be. Both axes are normalised to the local nuclide production rate at each sample location using the LSDn scaling model. Uncertainty ellipses (68 % confidence) are plotted using the ellipse.m code from the online calculators formerly known as the CRONUS-Earth online calculators (Balco et al., 2008). Constant exposure line (upper black), steady erosion line (lower blue), and steady-state erosion island (yellow shaded) are labelled on the figure. Paired nuclide diagram terminology from Granger (2006).

2.3 Investigating sources of geological uncertainty

Previous studies exhibiting concordant and discordant ¹⁰Be and ¹⁴C exposure ages (Balco et al., 2019) or seemingly impermissible ¹⁴C-¹⁰Be concentration ratios (Rand and Goehring, 2019) have been explained by invoking geological processes. We,





therefore, investigated sources of geological uncertainty in the Mt Murphy dataset in two ways. Firstly, we compared Mt Murphy paired ¹⁴C-¹⁰Be exposure ages and concentrations to similar datasets identified from the filter analysis, including a discussion of the differing geological settings of Mt Murphy and other sites. Secondly, we used the search filter to identify sites with impermissible ¹⁴C-¹⁰Be concentration ratios (Type-3 datasets) and then, using MATLAB, modelled scenarios to reproduce the higher ¹⁴C-¹⁰Be concentration ratios observed in these Type-3 datasets by 1) increasing erosion rate estimates and 2) simulating a prolonged duration of burial of a sample under a thin layer of ice at shallow to moderate depths (100–2000 g⁻¹ cm²) in the subsurface.

2.4 Investigating in situ ¹⁴C reproducibility

To investigate possible sources of sample preparation uncertainty, we focus on situ ¹⁴C reproducibility through evaluation of both new exposure age data from Mt Murphy and analysis of existing exposure age datasets in ICE-D. In situ ¹⁴C measurement reproducibility of the new Mt Murphy analyses was assessed with the repeat measurements of four different samples. To expand the scope of the reproducibility analysis, we performed a search of the ICE-D exposure age database for all existing in situ ¹⁴C exposure ages for which two or more measurements have been made of the same sample excluding measurements of laboratory intercomparison materials, such as CRONUS-A. After identifying samples with repeat in situ ¹⁴C measurements from ICE-D, we determined how many repeat measurements reproduced within measurement uncertainty. To assess reproducibility, we applied a blanket measurement uncertainty of 6 % (based on the reproducibility of CRONUS-A reported from the extraction laboratory at Tulane) of the total reported in situ ¹⁴C concentration reported from AMS measurements for each replicate. In cases where a study had reported a nuclide concentration with an associated measurement uncertainty exceeding 6 %, we used the larger value reported

3. Results

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240 3.1 In situ ¹⁴C exposure ages from Mt Murphy

by the study for the reproducibility assessment.

Thirteen in situ ¹⁴C measurements (including four replicate measurements) were performed on nine erratic samples recovered from Notebook Cliffs, Turtle Rock, and the un-named scoria cone from surfaces situated between 893 and 179 m a. s. l. When examining in situ ¹⁴C reproducibility exposure ages calculated from nuclide concentrations are reported with 1 σ internal uncertainties. In situ ¹⁴C exposure ages range from 9.0 ± 1.0 ka to 3.1 ± 0.2 ka (Table 1, Figs. 3, 4a and 4b), with an average exposure age of 6.0 ± 2.1 ka (mean and standard deviation). At scoria cone, in situ ¹⁴C exposure ages exhibit considerable scatter over a small

elevation range (180 - 240 m a. s. l.) with ages ranging from 9.0 ± 1.0 ka to 3.4 ± 0.3 ka. The spread of situ ¹⁴C exposure ages derived from initial measured in situ ¹⁴C concentrations is ~ 6 kyr, and some samples at higher elevations yield younger exposure ages than samples from lower elevations, which is the inverse of the expected age-elevation pattern associated with ice thinning through time. Apparent exposure ages from Notebook Cliffs (850 – 900 m a. s. l.) are 2 kyr younger than those from the scoria cone 250 (180 – 240 m a. s. l.).

Repeat in situ ¹⁴C measurements (n = 4) were made on samples TUR-117, TUR-132, CIN-108, and CIN-112 from the (low scoria cone 180–240 m a. s. l. and intermediate elevation (Turtle Rock, 450–650 m a. s. l.) sites. In situ ¹⁴C exposure ages calculated





from repeat measurements range from 8.2 ± 0.8 ka to 7.2 ± 0.7 ka (Table 1, Fig. 3). Some exposure ages derived from replicate measurements reproduce within internal measurement uncertainties (at 1 σ, Fig. 3), whilst others do not. For example, in situ ¹⁴C
exposure ages from TUR-117 and CIN-112 do not reproduce within range of their internal uncertainties at 1 σ (Table 1) or indeed 2 σ, with initial ages 3–5 kyr younger than those of replicate measurements. In other words, TUR-117-R, CIN-112-R are respectively 165 % and 112 % older than initial ages from the same samples, exhibiting significant scatter in excess of their internal uncertainties. CIN-108-R (7.8 ± 0.8 ka) is 24 % older than the exposure age calculated for CIN-108 (6.3 ± 0.6 ka) and does not reproduce within 1 σ internal uncertainty. TUR-132-R does, however, reproduce within 1 σ internal uncertainty. Neither the Turtle Rock nor scoria
cone site show a systematic bias in terms of reproducibility, with each site yielding one unreproducible in situ ¹⁴C exposure age.

260 cone site show a systematic bias in terms of reproducibility, with each site yielding one unreproducible in situ ¹⁴C exposure age. There is also no correlation between sample lithology and in situ ¹⁴C reproducibility, as ages derived from both granite and gneiss samples do not reproduce within internal uncertainties. Notably, initial analyses of samples from Notebook Cliff (n = 3) and TUR-123 from > 600 m a. s. l. are systematically younger than all repeat ¹⁴C ages from samples below 500 m a. s. l. (including the reproducibility analyses, Fig. 3), which is inconsistent with the expected age-elevation pattern associated with ice thinning.

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Table 1: New in situ ¹⁴ C exposure ages from sites at Mt Murphy: Notebook Cliffs (NOT), Turtle Rock (TUR) and scoria con	ne
(CIN). Exposure ages are reported propagating 6 % measurement uncertainties. Exposure ages were calculated using the LSE	Эn
scaling scheme. Sample IDs appended with R denote repeat measurements. See Table S1 for full in situ ¹⁴ C AMS dataset.	

Sample ID	Location	Latitude	Longitude	Elevation	Lithology	Exp. Age	Inter. Err.	Exter. Err.
		DD	DD	(m a.s.l.)		(ka)	1 σ (ka)	1 σ (ka)
TUR-123	Turtle Rock	-75.370	-111.292	639	granite	3.8	0.3	0.4
TUR-117	Turtle Rock	-75.381	-111.306	451	granite	3.1	0.2	0.3
TUR-132	Turtle Rock	-75.383	-111.309	446	granite	7.9	0.8	1.0
NOT-103	Notebook	-75.391	-111.139	852	granite	3.8	0.3	0.4
NOT-104	Notebook	-75.388	-111.117	893	granite	4.1	0.3	0.4
NOT-107	Notebook	-75.388	-111.090	885	granite	5.2	0.4	0.6
CIN-102	scoria cone	-75.219	-111.023	239	gneiss	9.0	1.0	1.3
CIN-108	scoria cone	-75.216	-111.019	181	granite	6.3	0.6	0.7
CIN-112	scoria cone	-75.216	-111.017	179	aplite	3.4	0.3	0.3
TUR-117-R	Turtle Rock	-75.381	-111.306	451	granite	8.2	0.8	1.1
TUR-132-R	Turtle Rock	-75.383	-111.309	446	granite	7.4	0.7	0.9
CIN-108-R	scoria cone	-75.216	-111.019	181	granite	7.8	0.8	1.0
CIN-112-R	scoria cone	-75.216	-111.017	179	aplite	7.2	0.7	0.9

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Published ¹⁰Be exposure ages and initial in situ ¹⁴C exposure ages measured from samples TUR-132, CIN-102 and CIN-108 overlap within their respective 1 σ external uncertainties making them concordant (Fig. 4a). However, most of the paired ¹⁴C-¹⁰Be ages (n = 6, including all NOT samples, TUR-117, TUR-123, and CIN-112) are discordant and have apparent exposure ages that are mid-late Holocene (5–3 ka). Where in situ ¹⁴C and ¹⁰Be ages are concordant, the in situ ¹⁴C age is systematically older and





early- to mid- Holocene (9–6 ka). Ages calculated from three of four replicate measurements overlap at 1 σ external uncertainty with the corresponding ¹⁰Be exposure ages (Fig. 4b). However, the in situ ¹⁴C exposure age calculated from the repeat measurement of sample CIN-108 is discordant with the ¹⁰Be exposure age from the same sample. We note the initial in situ ¹⁴C measurement of CIN-108 resulted in a concordant ¹⁴C-¹⁰Be exposure age pair.

We now compare paired nuclide diagrams of ¹⁴C-¹⁰Be concentration data from Mt Murphy. Based on initial in situ ¹⁴C concentrations, samples from Notebook Cliffs, Turtle Rock, and scoria cone (Fig. 5a) can be classified as Type 1, Type-2 and to an extent Type-3 nuclide ratios (see section 2.3). Samples TUR-132, CIN-102 and CIN-108 plot within the steady-state erosion island (Type-1) and display concordant in situ ¹⁴C and ¹⁰Be exposure ages (Fig. 5a). The remaining samples (NOT-103, NOT-104, TUR-123, TUR-117, and CIN-112) yield paired ¹⁴C and ¹⁰Be nuclide concentrations that plot below the steady state erosion line and thus indicate complex exposure histories (Type-2). Samples plotting below the steady erosion line (n = 6) include all the young in situ ¹⁴C ages which are discordant with respect to the ¹⁰Be exposure age from the same sample. TUR-138, for which both ¹⁰Be and in

situ ¹⁴C were measured for an earlier study of Mt Murphy (Johnson et al., 2020), displays an impermissible ¹⁴C-¹⁰Be ratio (Type-3).



Figure 3: Plot of in situ ¹⁴C ages vs elevation comparing in situ ¹⁴C exposure ages calculated from both initial and replicate in situ ¹⁴C concentrations. Ages derived from initial in situ ¹⁴C concentrations are depicted by red circles and ages derived from replicate concentrations by yellow triangles. In situ ¹⁴C exposure ages are plotted with 1 σ internal uncertainties calculated from the 6 % measurement uncertainty applied to in situ ¹⁴C concentrations. Replicate in situ ¹⁴C uncertainty bars displayed with thicker lines.







Figure 4: Mt Murphy paired ¹⁴C-¹⁰Be exposure age versus elevation plots (a) calculated using initial in situ ¹⁴C concentrations and (b) using both initial (greyed-out) and replicate in situ ¹⁴C concentrations. In situ ¹⁴C exposure ages plotted with 1σ external uncertainties following propagation of nominal 6 % in situ ¹⁴C measurement uncertainties. We report exposure ages with 1 σ external uncertainties when comparing exposure ages calculated from in situ ¹⁴C and ¹⁰Be concentrations measured in the same sample (see Methods section 2.1).





When comparing the replicate in situ ¹⁴C measurements three paired ¹⁴C-¹⁰Be concentration ratios plot within the steady
state erosion island (Fig. 5b), indicating these samples experienced a simple exposure history (Type 1). The position of sample TUR-132-R is comparable to its position when plotted using the initial measured in situ ¹⁴C concentration. In contrast, using initial ¹⁴C concentrations, TUR-117 and CIN-112 plot below the steady erosion line, suggesting complex exposure histories (Type-2). The positions of TUR-117 and CIN-112 on the paired isotope plot (Type-2) are consistent with in situ ¹⁴C exposure ages that exhibit discordance with ¹⁰Be ages from the same sample (Fig. 4a). Older in situ ¹⁴C exposure ages calculated from repeat measurements
(TUR-117-R, CIN-112-R; Fig. 4b) are, however, consistent with Type-1 ¹⁴C-¹⁰Be concentration ratios (suggestive of simple exposure histories).



Figure 5: Paired ¹⁴C-¹⁰Be nuclide diagrams using new in situ ¹⁴C concentrations from Mt Murphy samples. Panel (**a**) shows ¹⁴C-¹⁰Be nuclide ratios using initial in situ ¹⁴C concentrations and panel (**b**) shows ¹⁴C-¹⁰Be ratios using in situ ¹⁴C concentrations from repeat measurements. The *x*-axis represents the ¹⁰Be concentration normalised to its production rate (atoms g⁻¹ yr⁻¹) and the *y*-axis represents the ratio of the concentration of ¹⁴C, the shorter half-life nuclide, normalised by its production rate to ¹⁰Be – the longerlived nuclide. Ellipses are plotted using the LSDn scaling scheme (68 % confidence). In situ ¹⁴C extraction for TUR-138 (panel **a**) was performed at LDEO.

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3.2 Analysis of paired ¹⁴C-¹⁰Be measurements in ICE-D.

Following examination of ¹⁰Be and in situ ¹⁴C exposure ages from Mt Murphy we now investigate other locations with paired ¹⁴C-¹⁰Be measurements. Specifically, we look at sites within (Fig. 1b) and outside (Fig. 1c) Antarctica in which i) the ratio of the ¹⁰Be exposure age to the ¹⁴C exposure age is < 4:1, and ii) the ¹⁰Be exposure age is Holocene (< 11.7 ka). A summary of all sites with paired ¹⁴C-¹⁰Be exposure ages returned from ICE-D that satisfy our search criteria (n = 29, Fig. 1) is provided in Table 2, and age vs elevation plots and paired nuclide diagrams can be viewed in Supplement S4. The total number of paired in situ ¹⁴C-¹⁰Be measurements of Holocene age (n = 255) at the time of extraction from ICE-D [last accessed - 29.03.2024] is low compared to the over 28,000 total paired ²⁶Al-¹⁰Be measurements in ICE-D (although many predate the Holocene). We identify three endmember





datasets with paired nuclide diagrams that exhibit a predominant Type 1, 2, or 3 ¹⁴C-¹⁰Be concentration ratio (Fig. 6). We observe
Type-1 paired ¹⁴C-¹⁰Be concentrations (indicative of simple exposure) at Kangiata Nunata Sermia (KNS), Greenland (Young et al., 2021; Fig. 6b). Type-2 ¹⁴C-¹⁰Be concentrations (indicative of complex exposure) are observed in the forefield of the Rhône Glacier (Goehring et al., 2011). Finally, Type-3 ¹⁴C-¹⁰Be concentrations (indicative of "forbidden" exposure histories) are observed on the Antarctic Peninsula, proximal to the Sjögren Glacier (Balco and Schaefer, 2013). The Mt Murphy ¹⁴C-¹⁰Be dataset displays elements of all three types, including those observed at KNS (Type-1), Rhône Glacier, Switzerland (Type-2) and to a limited extent Sjögren Glacier. Type-1 exposure ages from this present study (e.g., CIN-108, TUR-132, CIN-102) are most similar to paired ¹⁴C-¹⁰Be measurements from KNS, Greenland (Young et al., 2021, Fig. 6b). Type-2 concentrations in this present study (e.g. NOT-104, CIN-112, TUR-117) are comparable to paired ¹⁴C-¹⁰Be concentrations from the Rhône Glacier, Switzerland (Goehring et al., 2011, Fig. 6c). Finally, ¹⁴C-¹⁰Be concentration ratios observed at Sjögren Glacier (Balco and Schaefer, 2013, Fig. 6d) mostly

plot above the simple exposure line (Type-3). In addition, some paired ¹⁴C-¹⁰Be concentrations from Conness Glacier forefield in
 the Sierra Nevada Range, California (Jones et al., 2023) plot as non-uniform ellipses. These are all, however, associated with very young exposure ages (last few hundred years), and so are not discussed further here. Focusing on the conditions surrounding each of the three endmember ¹⁴C-¹⁰Be datasets may help explain our observations from the Mt Murphy paired ¹⁴C-¹⁰Be dataset.



Figure 6: Paired in situ ¹⁴C-¹⁰Be nuclide concentrations from (a) Mount Murphy, (b) Kangiata Nunata Sermia (KNS), Greenland
 (Type-1, dominated by concordant ages), (c) Rhône Glacier, Switzerland, (Type 2, complex exposure – burial history) and (d)
 Sjögren Glacier, Antarctic Peninsula (Type 3 – "impermissible" exposure history-dominated dataset). NB: Other paired ¹⁴C-¹⁰Be datasets classified using the same system are displayed in Table 2. All paired ¹⁴C-¹⁰Be concentration ratios are normalised to the sample-specific production rate using the LSDn scaling scheme (Lifton et al., 2014). For further information on paired nuclide diagrams and "Type" classification scheme, see section 2.3.





345 3.3 Geological uncertainty and modelled subsurface production scenarios

3.3.1 Tucker Glacier and Mt Murphy age vs elevation datasets

Our first assessment of geological uncertainty compares paired ¹⁴C-¹⁰Be ages from Mt Murphy and Tucker Glacier in the Ross Sea Embayment of Antarctica. Tucker Glacier does not meet the second search criteria (¹⁰Be ages < 11.7 ka) but shares many characteristics with the Mt Murphy dataset. These characteristics include many samples with paired ¹⁴C-¹⁰Be measurements, a relatively large degree of scatter in nuclide concentrations from samples at the same elevations, and a mixture of concordant and discordant paired in situ ¹⁴C-¹⁰Be exposure ages (Fig. 7a). The youngest in situ ¹⁴C ages at this site are also discordant with respect to corresponding ¹⁰Be ages. The distribution of exposure ages from Shark Fin is similar to the Mt Murphy dataset (Fig. 4) when plotted with elevation. Paired ¹⁴C-¹⁰Be concentration ratios are, however, largely consistent with simple exposure histories (Fig. 7b).



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Figure 7: Paired ¹⁴C-¹⁰Be exposure ages (a) and paired nuclide diagram (b) from Shark Fin nunatak, Antarctica. Error bars in panel a represent 1σ external uncertainties as in situ ¹⁴C and ¹⁰Be production rates in the study were derived from different calibration datasets, one in situ ¹⁴C sample was saturated so is not displayed. In situ ¹⁴C exposure age uncertainties are plotted following propagation of a nominal 6 % internal uncertainty following the original study of Balco et al. (2019).

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3.3.2 Increased ¹⁴C-¹⁰Be ratio by higher in situ ¹⁴C subsurface production by muons relative to ¹⁰Be

Our second assessment of geologic uncertainty models scenarios where samples are subject to either rapid exhumation from ice, or prolonged burial under ice, leading to higher in situ ¹⁴C production relative to ¹⁰Be production in the subsurface (see section 1.1).





In both scenarios the in situ ¹⁴C nuclide concentration is expected to increase relative to ¹⁰Be and may explain impermissible Type-3 ¹⁴C-¹⁰Be concentration ratios observed at Sjögren Glacier (see Sect. 2.4). 365



Figure 8: Plots showing modelled subsurface production scenarios that lead to a higher in situ ¹⁴C relative to ¹⁰Be ratio than typical for the surface. Panel (a) shows ^{14}C - ^{10}Be nuclide ratios as a function of erosion rate integrated over a time t, assuming both ^{10}Be and in situ 14 C nuclide concentrations are zero at the LGM (t = 20000 years). The black line represents the constant exposure line and blue line the steady erosion line including muon production. Grey dots indicate modelled ¹⁴C-¹⁰Be nuclide concentration ratios 370 for an erosion rate which is specified above each dot (mm kyr⁻¹). Panel (b) shows modelled ¹⁴C-¹⁰Be nuclide concentrations as a function of burial under different ice thicknesses over Holocene timescales (plotted as isolines). The black line represents the constant exposure line, but we omit the steady erosion line to improve legibility. On both plots red ellipses indicate ¹⁴C-¹⁰Be concentration ratios measured in samples from Sjögren Glacier, Site C (68 % confidence). Plots are generated using the surface and subsurface production rate estimating code from Balco et al., (2023).





We first model rapid exhumation of a sample from overlying ice, which can be thought of as increasing the erosion rate, to investigate if this scenario would yield ¹⁴C-¹⁰Be ratios comparable to ratios observed in Sjogren Glacier samples (Fig. 8a). We observe that an increase in erosion rate to between 1000–2000 mm kyr⁻¹ results in high modelled ¹⁴C-¹⁰Be ratios (> 1.5), comparable to ratios observed in the Sjögren Glacier samples. Faster erosion rates, however, also result in modelled ¹⁰Be concentrations of < 2000 atoms g⁻¹, considerably lower than ¹⁰Be concentrations measured in the Sjögren Glacier samples. At slower erosion rates of 300–400 mm kyr⁻¹, the modelled ¹⁰Be concentration is comparable to concentrations at Sjögren Glacier, but the modelled ¹⁴C-¹⁰Be ratio is < 1, which is much lower than the high ¹⁴C-¹⁰Be ratio of most Sjögren Glacier samples.

We model for a second scenario whereby a sample is buried at specific depths of 1, 2 and 5 metres under ice, over Holocene 385 timescales (Fig. 8b). For this scenario, we observe that significant burial at depths below 2–3 metres are required generate a high modelled ¹⁴C-¹⁰Be ratio which is comparable with ratios observed in Sjogren Glacier samples. Burial under 5 metres of ice increases the ¹⁴C-¹⁰Be ratios to match the highest ratios observed at Sjögren Glacier. However, the ¹⁰Be nuclide concentration is extremely low at these ratios (< 1000 at g⁻¹).

390 **3.4 Sample preparation uncertainty - Mt Murphy and ICE-D in situ**¹⁴C reproducibility

We now describe results of new in situ ¹⁴C repeat measurements from Mt Murphy and repeat measurements extracted from ICE-D (Fig. 9). Although the Mt Murphy sample size is small (n = 4) it is notable that 3 out of 4 in situ ¹⁴C concentrations do not replicate within the 1 σ 6% measurement uncertainty (Fig. 4), and 2 of 4 in situ ¹⁴C concentrations do not replicate at 2 σ . In addition to the Mt Murphy results, a further 25 samples for with repeat in situ ¹⁴C measurements are also in ICE-D, and a further two samples with replicate in situ ¹⁴C measurements from the Leymon High Core (Lupker et al., 2015) bringing the total number of samples for

- 395 with replicate in situ ¹⁴C measurements from the Leymon High Core (Lupker et al., 2015) bringing the total number of samples for which multiple in situ ¹⁴C measurements exist to 31. The majority of replicates are from samples sourced from the Antarctic Peninsula (n = 5; Balco and Schaefer, 2013), Weddell Sea Embayment (n = 5; Nichols et al., 2019), Promontory Point (Pleistocene Lake Bonneville), Utah (n = 5; Lifton et al., 2015a) and the Northwest Highlands, Scotland (n = 5; Borchers et al., 2016). From these 31 samples with replicate in situ ¹⁴C measurements, we observe 18 of the 31 samples display one or more in situ ¹⁴C
- 400 measurements that do not replicate within a 6 % 1 σ measurement uncertainty (Fig. 9). There is a slight improvement in reproducibility at 2 σ , however, 15 of 31 samples still exhibit one or more in situ ¹⁴C measurements that are not reproducible (see Fig. S9).









scoria cone and Sjögren Glacier are indicated by shading. See Table S3 for full list of Sample ID's and corresponding sites.

Table 2 (Overleaf): Full list of paired ¹⁴C-¹⁰Be surface exposure ages extracted from ICE-D using the following filters: i) apparent
 ¹⁰Be exposure age is < 4x than apparent ¹⁴C exposure age ii) ¹⁰Be exposure ages are of Holocene age (< 11.7 ka). Paired nuclide ratio type refers to the dominant position of paired ¹⁴C-¹⁰Be ratio ellipses on the paired nuclide diagram. Paired nuclide diagrams from each site feature one Type, e.g., 1, 2 or 3 or a mixture of types. Instances of the secondary less dominant type are denoted in brackets. Abbreviations for AMS and in situ ¹⁴C extraction laboratories are as follows; CEREGE (Centre Européen de Recherche et d'Enseignement des Géosciences de l'Environnement), ETH Zurich (Swiss Federal institute of Technology in Zurich), (KIST
 (Korean Institute of Science and Technology), LDEO (Lamont Doherty Earth Observatory), LLNL (Lawrence Livermore National

- Laboratory), NOSAMS (National Ocean Sciences Accelerator Mass Spectrometer Laboratory at the Woods Hole Oceanographic Institute) and SUERC (Scottish Universities Environmental Research Centre). Shark Fin nunatak ¹⁰Be ages are > 11.7 ka, so do not meet one of our search criteria but are included here due to the similarities in the age vs elevation profile of Shark Fin nunatak and the Mt Murphy data. [#]Engabreen glacier paired ¹⁴C-¹⁰Be data are not in ICE-D but are included to demonstrate a geological solution
- 425 to a paired ¹⁴C-¹⁰Be "Type 3" dataset. *Unpublished in situ ¹⁴C data obtained from the informal cosmogenic-nuclide exposure age database (ICE-D). These data are, however, freely available in ICE-D under the public release requirements of the National Science Foundation (NSF) U.S. Antarctic Program which requires data be made publicly available 2 years after collection.





Location ID	Short Name (ICE-D)	Site Name, Location	¹⁴ C- ¹⁰ Be ratio	In situ ¹⁴ C extraction	AMS lab ¹⁴ C	Reference ¹⁰ Be dataset	Reference in situ ¹⁴ C dataset
1	REA	Mt.Rea, Sarnoff Mts.	3	Tulane	NOSAMS	Stone et al., (2003)	ICE-D*
2	TR	Turtle Rock, Mt Murphy	12(3)	Tulane	NOSAMS	Johnson et al., (2020)	This paper(Johnson et al., 2020)
3	NMAS	North Masson Range, Frannes Mts.	3 (1)	SUERC	SUERC	Mackintosh et al., (2007)	White et al., (2011)
4	SKF	Sharkfin nunatak, Tucker Gl.	1 (2)	Tulane	TLNL	Balco et al., (2019)	Balco et al., (2019)
5	MZS	Mario Zuchelli Station, Terra Nova Bay	1	Tulane	TLNL	Goehring et al., (2019b)	Goehring et al., (2019b)
9	WHITS	Mt. Whitmore, Whitmore Mts.	N/A	Tulane	NOSAMS	Spector et al., (2019)	Spector et al., (2019)
7	KAYCONE	scoria cone, Mt Murphy	1 2	Tulane	NOSAMS	Adams et al., (2022)	This paper
8	NOTE	Notebook Cliffs, Mt Murphy	2	Tulane	NOSAMS	Johnson et al., (2020)	This paper
6	HOPE	Mt. Hope, Beardmore Glacier	2 (1)	Tulane	NOSAMS	Spector et al., (2017)	ICE-D*
10	CMARSH	Cape Marsh, Robertson Island	1	KIST	KIST	Jeong et al., (2018)	Jeong et al., (2018)
11	FRAMNES	Cape Framnes, Jason Peninsula	1 (2)	KIST	KIST	Jeong et al., (2018)	Jeong et al., (2018)
12	SJOC	Site C, Sjogren-Boydell Fjord	3	Tulane	NOSAMS	Balco and Schaefer, (2013)	ICE-D*
13	DRYE	Site E, Drygalski Glacier	1 (2)	Tulane	NOSAMS	Balco and Schaefer, (2013)	ICE-D*
14	KRING	Mt. Kring, David Glacier	3	Tulane	NOSAMS	Stutz et al., (2021)	ICE-D*
15	DIAMOND	Diamond Hill, Darwin-Hatherton Glaciers	3	Tulane	NOSAMS	Hillebrand et al., (2021)	Hillebrand et al., (2021)
16	DANPB1	Danum Platform, Darwin-Hatherton Glaciers	1 (3)	Tulane	NOSAMS	Hillebrand et al., (2021)	Hillebrand et al., (2021)
17	Goeh2011-A	Rhône Glacier, Swiss Alps	2	LDEO	Uni of Arizona	Goehring et al., (2011)	Goehring et al., (2011)
18	Hipp2014-A	Gotthard Pass, Swiss Alps	1	ETH Zurich	ETH Zurich	Hippe et al., (2014)	Hippe et al., (2014)
19	KBR1	KNS bedrock 1, Kangiata Nunata Sermia	1	LDEO	CEREGE	Young et al., (2021)	Young et al., (2021)
20	KBR2	KNS bedrock 2, Kangiata Nunata Sermia	1	LDEO	CEREGE	Young et al., (2021)	Young et al., (2021)
21	INGLE1	Outboard ice margin, Inglefield Land	1	ETH Zurich	ETH Zurich	Søndergaard et al., (2020)	Søndergaard et al., (2020)
22	MACAULAY	Macaulay Boulder Field, Southern Alps	1 (3)	LDEO	LLNL-CAMS	Putnam et al., (2010)	(Schimmelpfennig et al., 2012)
23	MARRAIT	Marrait Moraine, Jakobshavn Isfjord	1	LDEO	LLNL-CAMS	Young et al., (2013)	Young et al., (2014)
24	GHm-Out5	Grey Hunter massif, MacArthur Mts., Yukon	1	Tulane	NOSAMS	Goehring et al., (2022)	Goehring et al., (2022)
25	SISPEN-W	Sisimiut Peninsula, Kangerlassuaq	1	LDEO	CEREGE	Sbarra et al., (2022)	Sbarra et al., (2022)
26	Mammoth-Forefield	Mammoth Gl., Wind River Range, WY	1	Tulane	NOSAMS	Jones et al., (2023)	Jones et al., (2023)
27	Conness-Forefield	Conness Gl., Sierra Nevada, CA	123	Tulane	NOSAMS	Jones et al., (2023)	Jones et al., (2023)
28	JIF-Forefield	Juneau Ice Field, Coast Mts., AK	123	Tulane	NOSAMS	Jones et al., (2023)	Jones et al., (2023)
29	N/A	Engabreen Glacier, Norway#	3	Tulane	NOSAMS	Rand and Goehring, (2019)	Rand and Goehring, (2019)





430 4. Discussion

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This study compares new in situ ¹⁴C exposure ages from Mt Murphy with published ¹⁰Be ages from the same samples. A total of 3 of 4 new in situ ¹⁴C repeat measurements do not replicate within their 1 σ internal uncertainties (Fig. 4), and samples with concordant and discordant ¹⁴C-¹⁰Be exposure ages suggest both simple and complex exposure histories at the same elevation (Fig. 4 and Fig. 5). Results from the filter analysis of Holocene ¹⁴C-¹⁰Be exposure ages in ICE-D (Table 2) indicate some ¹⁴C and ¹⁰Be nuclide pairs exhibit Type-3 (impermissible) ¹⁴C-¹⁰Be ratios (Fig. 6). We also identify a total of 31 samples from Mt Murphy, ICE-D [last accessed - 29.03.2024] and literature sources for which multiple in situ ¹⁴C measurements have been made. Of these, 18 samples exhibit at least one repeat in situ ¹⁴C measurement that does not reproduce within a 6 % 1 σ internal measurement uncertainty (Fig. 9).

440 4.1 Key observations from the Mt Murphy paired ¹⁴C-¹⁰Be exposure ages

- We begin the discussion focusing on key results from the Mt Murphy paired ${}^{14}C{}^{-10}Be$ exposure ages, and corresponding ${}^{14}C{}^{-10}Be$ ratios. Three of the four replicate in situ ${}^{14}C$ measurements, yield exposure ages that do not overlap within internal uncertainty (1 σ) with initial ${}^{14}C$ exposure ages from the same sample (Fig. 3). TUR-117-R and CIN-112-R also do not replicate at 2 σ internal uncertainty with initial ${}^{14}C$ exposure ages from the same sample. These initial in situ ${}^{14}C$ exposure ages suggest deglaciation from 5–3 ka and are discordant with ${}^{10}Be$ exposure ages from TUR-117 and CIN-112. TUR-117 and CIN-112 yielded two of the six exposure ages calculated from initial in situ ${}^{14}C$ measurements of samples ranging from 150–900 m a. s. l. which were systematically young (5–3 ka, Fig. 4a). These systematically young in situ ${}^{14}C$ exposure ages appear to contradict the currently interpreted deglacial history that the ice surface at Mt Murphy lowered to an elevation of ~ 150 m a. s. l. by 6 ka (Johnson et al., 2020; Adams et al., 2022; Balco et al., 2023). In addition, the young discordant in situ ${}^{14}C$ exposure ages from higher elevations
- 450 (Notebook Cliffs and TUR-123) and older reproducible in situ ¹⁴C ages from lower elevations are inverted with respect to and contradict the expected age-elevation pattern associated with ice thinning. The two samples measured for in situ ¹⁴C that did reproduce within their uncertainties at 2 σ (TUR-132 and CIN-108) were also concordant in respect to existing ¹⁰Be exposure ages from the same sample (Fig. 4b). From the Mt Murphy replicate measurements (n = 4), the two young in situ ¹⁴C ages are not reproducible at 2 σ internal uncertainty, but both older exposure ages are reproducible at 2 σ internal uncertainty. In summary, Mt
- 455 Murphy paired ¹⁴C-¹⁰Be exposure ages display both concordance and discordance across multiple sites. Concordant exposure ages are consistent with Type-1 ¹⁴C-¹⁰Be ratios and discordant exposure ages consistent with Type-2 (and Type-3) ¹⁴C-¹⁰Be ratios. Concordant ¹⁴C-¹⁰Be exposure ages exhibit in situ ¹⁴C ages which are reproducible at 2 σ internal uncertainty whereas discordant ¹⁴C-¹⁰Be exposure ages do not.

A bootstrap linear regression analysis (see Supplement 2) of in situ ¹⁴C and ¹⁰Be exposure age datasets from Kay Peak and a scoria cone adjacent to Kay Peak indicate in situ ¹⁴C and ¹⁰Be chronologies are broadly similar with respect to the timing of deglaciation, implying they are equally accurate (see Fig. S10). There is, however, significant excess scatter of ~1849 years in the in situ ¹⁴C ages (Supplement S2, Table S4) that cannot be accounted for by the nominal 6 % 1σ internal measurement uncertainty





for in situ ¹⁴C adopted in many studies (e.g., Balco et al., 2019; Nichols et al., 2019). The new in situ ¹⁴C exposure ages and existing ¹⁰Be exposure data and ¹⁰Be-¹⁴C ratios from Mt Murphy (Fig. 4, Fig. 5) raise two important questions: 1) Is there a geological explanation for co-existence of concordant and discordant paired in situ ¹⁴C and ¹⁰Be exposure ages, often at the same elevation, as well as a mix of Type 1, Type 2 (and Type-3) ¹⁴C-¹⁰Be ratios? and 2) Is there a way to explain why 3 of 4 replicate in situ ¹⁴C analyses which do not produce within their 6 % 1σ internal measurement uncertainties? The second question is especially pertinent because there cannot be a geological explanation for large variations in concentrations of the same nuclide from the same sample.

470 4.2 A Geological explanation for paired ¹⁴C-¹⁰Be sample concordance and discordance

First, we examine the Notebook Cliffs, Turtle Rock, and scoria cone sites to determine if localised geological changes at Mt Murphy permit the existence of paired ¹⁴C-¹⁰Be discordant exposure ages at the same elevation as paired ¹⁴C-¹⁰Be concordant exposure ages. A trimetrogon aerial flightline photo shows that in 1966, in contrast to today, the lower scoria cone outcrop was almost completely buried by ice. This finding indicates that samples CIN-112 and CIN-108 were shielded by ice for a non-zero time

- 475 between 6.4 ka and present (Adams et al., 2022; Balco et al., 2023). A discordant initial in situ ¹⁴C age for CIN-112 (3.4 ± 0.3 ka, 179 m a. s. l.) younger than the ¹⁰Be exposure age (6.6 ± 0.4) from the same sample and other higher elevation in situ ¹⁴C ages from scoria cone supports such burial occurring during the late Holocene. The in situ ¹⁴C replicate measurement, CIN-112-R, however, yields an exposure age of 7.2 ± 0.9 ka, in agreement with the existing ¹⁰Be age. Both in situ ¹⁴C exposure ages determined from measurements of sample CIN-108 (collected from the same outcrop and elevation as CIN-112) are early-mid Holocene (CIN-108 –
- 480 6.3 ± 0.7 ka; CIN-108-R 7.8 ± 1.0 ka). With the exception of the initial ¹⁴C exposure age from sample CIN-112, all exposure ages from the lower scoria cone outcrop (Adams et al., 2022) suggest that ice cover during the late Holocene was short-lived.

At both Turtle Rock and Notebook Cliffs, there is little evidence to suggest prolonged cover or burial of samples. At Turtle Rock, discordant ¹⁴C-¹⁰Be exposure ages of TUR-117 and TUR-123 could be due to individual samples being partially shielded by till or ice debris cover during the Holocene, but the preferential sampling of topographic highs makes this less likely (Johnson et al.,

485 2020). Furthermore, the in situ ¹⁴C exposure age of TUR-117-R (8.2 ± 1.1 ka) agrees with the existing ¹⁰Be exposure age at the site. There is no geological explanation for the same nuclide (in situ ¹⁴C) measured on the same sample (TUR-117) yielding two different in situ ¹⁴C exposure ages.

At Notebook Cliffs all ¹⁴C exposure ages (n = 3) are late Holocene and discordant with existing ¹⁰Be ages, implying inheritance in ¹⁰Be and prolonged burial of all three samples. The three Notebook Cliffs in situ ¹⁴C exposure ages contradict evidence from lower elevations of Mt Murphy that indicate early to mid-Holocene deglaciation from 9–6 ka (Johnson et al., 2020; Adams et al., 2022; Balco et al., 2023). In situ ¹⁴C ages from Notebook Cliffs could be reconciled with the currently accepted Mt Murphy

- deglaciation history if a localised ice dome had persisted atop Notebook Cliffs, shielding samples until the late Holocene. The flat top of the Notebook Cliffs site would favour persistence of a post-glacial ice dome; however, there is no physical evidence for this having occurred (Johnson et al., 2020). The difference in discordant in situ ¹⁴C concentrations (CIN-112, TUR-117) and replicate
- 495 measurements from Turtle Rock and scoria cone (CIN-112-R, TUR-117-R) imply, however, that repeat in situ ¹⁴C measurement from Notebook Cliffs may have been in closer agreement with the Notebook Cliff ¹⁰Be exposure ages. In summary, evidence for





localised geological and topographical drivers of repeated burial and exposure of samples at Mt Murphy are currently lacking, with the exception of late Holocene ice cover of samples CIN-108 and CIN-112 at the lower scoria cone outcrop.

500 4.2.1 Comparisons of the Mt Murphy and Shark Fin nunatak paired in situ ¹⁴C-¹⁰Be datasets

The ¹⁴C-¹⁰Be paired exposure age dataset from Shark Fin Nunatak adjacent to Tucker Glacier in the Ross Sea Embayment (Fig. 7) shares many similarities with the new Mt Murphy dataset. At Shark Fin Nunatak, Balco et al. (2019) explain the range of concordant and discordant paired in situ ¹⁴C-¹⁰Be ages by samples of the same lithology having been initially exposed at higher elevations upstream for varying durations of time, subjecting them to a higher nuclide production rate. They postulate that exposed boulders then fell from cliffs and were transported supraglacially to their present position at Shark Fin nunatak. The suggestion of prior exposure followed by supraglacial transport is supported by evidence of extensive weathering of many of the Shark Fin samples. An upstream, higher elevation origin is also highly plausible due to the alpine setting of both Tucker Glacier and its tributary, Whitehall Glacier (Fig. 10b).

- The overall conclusion at Shark Fin nunatak was that the youngest and discordant in situ ¹⁴C ages reflect the most straightforward and probable exposure history, while concordant in situ ¹⁴C-¹⁰Be samples must have been subject to varying amounts of inheritance (Balco et al., 2019). The majority of ¹⁰Be exposure ages, and a few paired ¹⁴C ages range between 20–30 ka, precede the Holocene, which suggests that those samples have varying amounts of ¹⁰Be and in situ ¹⁴C inheritance. It should be noted that, at Shark Fin nunatak, both the in situ ¹⁴C ages and ¹⁰Be ages are older than Holocene age, whereas at Mt Murphy all paired ¹⁴C-¹⁰Be exposure ages being examined are Holocene.
- 515 There are very few areas of exposed bedrock immediately upstream of Mt Murphy (Fig. 10a), making a similar geological explanation for the distribution of paired ¹⁴C-¹⁰Be exposure ages at Mt Murphy as at Shark Fin Nunatak problematic and unlikely. The only site with rock outcrop immediately upstream of Mt Murphy is Mt Takahe, but Mt Takahe does not possess the same lithology as erratic boulders observed at Mt Murphy e.g., aplite, granite, and gneiss (Johnson et al., 2020; Adams et al., 2022), and is instead composed of extrusive igneous rock (see online database for Ohio State Polar Rock Repository; https://prr.osu.edu/collection/ [web accessed 15.02.2024]). The only area of exposed rock nearby where granite has been observed is the Kohler Range (Fig. 11, Fig. S6). However, the Kohler Range does not lie upstream of Mt Murphy in the present ice flow configuration (Fig. 11a). A radical past re-organisation of ice flow would be required for erratics to be transported to Mt Murphy
 - from the Kohler Range, for which there is no post-LGM evidence. In addition, erratics of exotic lithology from Notebook Cliffs, Turtle Rock and scoria cone measured for both ¹⁴C and ¹⁰Be are much less heavily weathered than samples at Shark fin nunatak,
- 525 and so could not be considered to have arrived at Mt Murphy via supraglacial transport (for further geological information on Mt Murphy samples see Supplement S1). In summary, Mt Murphy is a remote site, and erratics sampled here show little evidence of having been transported supraglacially from a higher elevation site upstream. Therefore, the existence of discordant and concordant ¹⁴C-¹⁰Be ages cannot be explained geologically in the same way as they can at Shark Fin nunatak. Furthermore, at Mt Murphy, with the exception of the scoria cone, there is little evidence from local topography to explain concordant-discordant paired ¹⁴C-¹⁰Be
- 530 exposure ages, although there is room for speculation at higher elevation sites. Still, any geological justification no matter how





complex cannot explain irreproducible in situ ¹⁴C exposure ages at multiple sites. Paired ¹⁴C-¹⁰Be exposure ages at Shark Fin nunatak (Fig. 8a) reflect the same patterns of concordance and discordance observed at Mt Murphy (Fig. 4). However, ¹⁴C-¹⁰Be ratios at Shark Fin nunatak (Fig. 8b) to a varying extent all suggest a simple exposure history whereas at Mt Murphy, samples exhibit simple, complex, and seemingly impermissible exposure histories (Fig. 5).



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Figure 10: Regional comparison of Mt Murphy (a) and Tucker Glacier (b) demonstrating the comparative topographic isolation of Mt Murphy compared to Tucker Glacier. Orange circles show paired ¹⁴C-¹⁰Be sample sites mentioned in the text. Black lines in both images show locations of grounding lines determined by differential satellite radar interferometry (Rignot et al., 2011a). Ice flow speeds are displayed to improve visualisation of ice streams and were downloaded from MEaSUREs InSAR-Based Antarctica
540 Ice Velocity dataset Version 2 (Mouginot et al., 2012, 2017; Rignot et al., 2011b) [last accessed 25.05.2023]. To improve visualisation exposed rock outcrops in Panel (a) are displayed red using the Antarctic rock outcrop dataset (Burton-Johnson et al., 2016). Both satellite images are sourced from Landsat Image Mosaic of Antarctica (LIMA; lima.usgs.gov).



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4.3 Discussion of ¹⁴C-¹⁰Be endmember datasets from ICE-D analysis

In this section, we further contextualise Mt Murphy paired ¹⁴C-¹⁰Be concentration ratios by discussing the relevant background of existing datasets from Kangiata Nunata Sermia, Rhône Glacier and Sjögren Glacier, which display dominant Type-1, Type-2, and Type-3 nuclide concentration ratios, respectively.

The Type-1 endmember ¹⁴C-¹⁰Be dataset is from KNS, Greenland. The data exhibit concordant ¹⁴C-¹⁰Be exposure ages and ¹⁴C-¹⁰Be ratios consistent with a simple exposure history (Young et al., 2021). Observations from the KNS study site are well documented, both historically back to the 1850's (Young et al., 2021 cf. Fig. 10) and presently (Young et al., 2021 cf. Fig. 2). As a result, observational uncertainties are minimal, which permits the maximum permissible duration of Holocene exposure to be constrained, in this case to ¹⁰Be of 10.20 ± 0.23 ka (Young et al., 2021). The study in which this dataset was generated benefitted

- from local production rate calibration datasets from West Greenland for ¹⁰Be and in situ ¹⁴C (Young et al., 2014), ensuring that production rates and the ¹⁴C-¹⁰Be production ratio were regionally constrained (Young et al., 2021). Finally, sources of analytical uncertainty of in situ ¹⁴C were propagated for each sample based on long-term CRONUS-A measurements from the Lamont Doherty
- 555 Earth Observatory where extraction took place (Lamp et al., 2019). All six samples without ¹⁰Be inheritance possess ¹⁴C-¹⁰Be ratios that are indistinguishable and therefore imply a simple exposure history. In terms of accuracy, the ¹⁰Be ages obtained from KNS are confirmed by concordant ¹⁴C ages. The Type-1 dataset from KNS is associated with well constrained concordant paired ¹⁴C-¹⁰Be exposure ages that permit the Holocene deglaciation history of the site to be reconstructed with high confidence. From the filter analysis of ICE-D, eight other sites display ¹⁴C-¹⁰Be concentration ratios that are exclusively Type-1: Mario Zuchelli Station and
- 560 Cape Marsh-Robertson Island from Antarctica (Jeong et al., 2018; Goehring et al., 2019b) and six sites from elsewhere around the globe (see Table 2). However, five of these eight sites all contain only one ¹⁴C-¹⁰Be pair each, which were extracted based on the Holocene search filter.

The Type-2 endmember dataset is taken from the Rhône Glacier exposure age dataset from the Swiss Alps (Goehring et al., 2011). Notebook Cliffs appears to be the only other dataset that exclusively exhibits Type-2 ¹⁴C-¹⁰Be concentration ratios (Table

- 3). Much like paired ¹⁴C-¹⁰Be ratios from Notebook Cliffs and paired ¹⁴C-¹⁰Be ratios based on the initial in situ ¹⁴C measurements of TUR-117 and CIN-112 (Fig. 5a), the ¹⁴C-¹⁰Be ratios from Rhône Glacier plot within the area of complex exposure. The Rhône Glacier study effectively utilised the complex exposure and burial history of the samples uncovered from the forefield of the glacier to determine when it likely expanded and contracted during the Holocene (Goehring et al., 2011). Historical records aid with the interpretation of Rhône Glacier exposure ages (much like at KNS) as the samples are known to have been covered in the recent past
- 570 when the glacier expanded during the Little Ice Age (Goehring et al., 2011). The Rhône Glacier dataset is more straightforward to interpret than the Mt Murphy dataset because all ¹⁴C-¹⁰Be exposure age pairs (except one outlier) plot below the steady erosion line. At Mt Murphy, apparent discordant and concordant ¹⁴C-¹⁰Be paired exposure ages from the same elevation suggest that some, but not all, samples were shielded during the Holocene, requiring a complex geologic model. The model used to explain exposure ages at Shark Fin Nunatak, however, cannot be applied to Mt Murphy (see Sect. 4.2), and so the cause of Type-2 ¹⁴C-¹⁰Be ratios at Mt
- 575 Murphy currently lacks a coherent geological explanation.





The Type-3 endmember dataset from Sjögren Glacier - Site C (Balco and Schaefer, 2013) displays impermissible ¹⁴C-¹⁰Be concentration ratios (when assuming a constant surface production rate). At Mt Murphy, no samples have impermissible ¹⁴C-¹⁰Be ratios, but TUR-138 almost plots above the constant exposure line, and CIN-102 and TUR-132 also display high ¹⁴C-¹⁰Be ratios (Fig. 4). Exposure ages from Sjögren Glacier samples are bimodal. One group of mid-Holocene exposure ages (including replicates) decreases in age with elevation, from 4.8 ka at 120 m a. s. l. to 3.4 ka at 40 m a. s. l., whilst some samples suggest extremely recent exposure between just 200–500 years ago (Balco and Schaefer, 2013). From analysis of ¹⁰Be ages, it was suggested that the site was

- fully deglaciated at 3.5–4.5 ka, partially covered again by thickening of the adjacent Boydell Glacier at or after ca 1.4 ka, and then deglaciated again between 1969 and the present (Balco and Schaefer, 2013). A complex exposure scenario at Sjögren Glacier may have, therefore, created specific conditions in which seemingly implausible ¹⁴C-¹⁰Be ratios can be explained geologically if samples
- 585 at Sjögren Glacier-Site C were buried under a thin layer of cold-based ice during the Holocene, prior to more recent exposure. An impermissible ¹⁴C-¹⁰Be concentration ratio (when assuming surface production) could therefore become plausible if samples at Sjögren Glacier were subject to long periods of subsurface production during the Holocene or recent rapid exhumation from moderate depth (Rand and Goehring, 2019).

590 4.4 Investigating the effect of subsurface production by muons on ¹⁴C-¹⁰Be ratios

We now evaluate results of modelling high in situ ¹⁴C relative to ¹⁰Be production in the subsurface and explore if this can lead to a plausible geological explanation of the Sjögren Glacier Type-3 dataset. Two scenarios were modelled where an offset in situ ¹⁴C relative to ¹⁰Be production by muons could lead to high impermissible ¹⁴C-¹⁰Be concentration ratios at Sjögren Glacier: 1) An increase in erosion rate and 2) a duration of burial of a sample at shallow to moderate depth (1–5 metres) in the subsurface.

595 Type-3 ¹⁴C-¹⁰Be ratios observed at Sjögren Glacier, however, could not be reconciled by modelling faster erosion rates (a faster rate of ice surface thinning) or prolonged burial under a thin layer of ice.

Increasing ice surface thinning (erosion rates) above 300–400 mm kyr⁻¹ leads to resulting modelled in situ ¹⁴C-¹⁰Be nuclide concentration ratios plotting above the constant exposure line, but only when ¹⁰Be concentrations are low (see Fig. 9a). None of the Sjögren Glacier samples, however, exhibit low ¹⁰Be nuclide concentrations (range; 18070 ± 591 to 27670 ± 686 at g⁻¹). The surface nuclide concentration is inversely proportional to the erosion rate, meaning there is an intersection between maximising the duration of greater ¹⁴C production over an integrated depth and minimising the impact of the faster rate of decay of ¹⁴C relative to ¹⁰Be. Consequently, high ¹⁴C-¹⁰Be ratios are only permitted when ¹⁰Be concentration in a sample is low, due to the increasingly fast removal of accumulated nuclides at higher erosion rates.

Modelling prolonged burial of a sample at depths greater than ~ 2.5-3 metres under ice leads to high ${}^{14}C{}^{-10}Be$ ratios comparable to ${}^{14}C{}^{-10}Be$ ratios of Sjögren Glacier samples (Fig. 9b). The high ${}^{14}C{}^{-10}Be$ production ratio at depth is due to the almost complete attenuation of the high energy neutron flux (Dunai, 2010), and dominance of nuclide production due to negative muon capture (Heisinger et al., 2002; Balco, 2017). However, high ${}^{14}C{}^{-10}Be$ ratios are again only observed when the ${}^{10}Be$ concentration is low (< 1000 at g⁻¹). Over longer burial durations where greater concentrations of ${}^{10}Be$ accumulate, the high in situ ${}^{14}C$ to ${}^{10}Be$ production ratio is offset by the much faster decay rate of in situ ${}^{14}C$ relative to ${}^{10}Be$. In addition, the likely exposure history at





- 610 Sjögren Glacier, Site C indicated from ¹⁰Be ages is 2-3 kyr of initial surface exposure followed by shallow burial under a thin ice layer at 1.4 ka (Balco and Schaefer, 2013). In this case, the significant duration of surface exposure precludes the high ¹⁴C-¹⁰Be concentration ratios observed in the Sjögren Glacier samples, due to the significant quantities of in situ ¹⁴C and ¹⁰Be atoms already accumulated in the sample at the surface production ratio.
- In summary, observed ratios of ¹⁴C-¹⁰Be relative to ¹⁰Be concentrations at Sjögren Glacier cannot be reconciled by either faster erosion (ice surface thinning) rates or prolonged burial at shallow to moderate ice thicknesses (1–5 metres). In both scenarios, if the concentration of ¹⁰Be atoms is low, high modelled ¹⁴C-¹⁰Be ratios comparable to the ¹⁴C-¹⁰Be concentration ratios in Sjogren Glacier samples are observed. However, as soon as ¹⁰Be nuclide concentrations exceed 1000–2000 atoms g⁻¹, high ¹⁴C-¹⁰Be ratios are no longer observed due to 1) high erosion rates rapidly removing accumulated nuclides and or 2) the more rapid decay of in situ ¹⁴C relative to ¹⁰Be offsetting the higher ¹⁴C-¹⁰Be subsurface production ratio. All measured in situ ¹⁴C nuclide concentrations from
- 620 Sjögren Glacier Site C also appear to be systematically offset by approximately 5000 extra ¹⁴C atoms, suggesting a potential contaminant source of in situ ¹⁴C to these samples. A high proportion of samples from Sjögren Glacier Site C are vein quartz (see Table S3), which has previously been speculated to impact in situ ¹⁴C extraction due to the frequent presence of fluid inclusions (Nichols et al., 2019). Therefore, it is possible Type-3 ratios observed at Sjögren Glacier are due to an additional carbon source present in the quartz or incorporated during in situ ¹⁴C extraction.
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4.5 Assessing the reproducibility of in situ ¹⁴C

We now evaluate in situ ¹⁴C reproducibility determined from the results of Mt Murphy replicate sample measurements and the wider ICE-D database. In this study only 1 of 4 of the Mt Murphy replicate in situ ¹⁴C measurements reproduced using a 6% 1 σ measurement uncertainty. The Mt Murphy replicates contribute to a total of 18 of the 31 samples extracted from ICE-D where one

- or more replicates measurements do not reproduce within the nominal 1 σ 6 % analytical uncertainty based on repeat measurements of CRONUS-A at Tulane Laboratory (Goehring et al., 2019a). Many replicate measurements included in the ICE-D Holocene filter analysis are from Sjögren and Drygalski Glaciers (n = 5), sites which also yielded many impermissible paired ¹⁴C-¹⁰Be ratios (Fig. 7). Results from the ICE-D filter of reproducibility (Fig. 10), therefore, suggest a possible link between in situ ¹⁴C reproducibility and Type-3 ¹⁴C-¹⁰Be concentration ratios. The lack of an apparent geologic explanation for repeat in situ ¹⁴C measurements from
- 635 field samples not reproducing within 1 σ 6 % analytical uncertainties suggests that sample preparation uncertainty estimation may be too low. We therefore investigate in situ ¹⁴C reproducibility by examining CRONUS-A reproducibility and in situ ¹⁴C process blank values (Table 3).

4.5.1 Sources of uncertainty affecting in situ ¹⁴C reproducibility – CRONUS-A

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In situ ¹⁴C concentrations for CRONUS-A reported from extraction facilities range from $6.12 \pm 0.32 \times 10^5$ at g⁻¹ (Goehring et al., 2019a) to $7.28 \pm 0.03 \times 10^5$ at g⁻¹ (Lupker et al., 2019). The CRONUS-A value reported from Tulane is 5–10 % lower than other in situ ¹⁴C extraction laboratories and below the "consensus" interlaboratory value (n = 23) of 6.97 x 10⁵ atoms g⁻¹ of Jull et al. (2015). Interlaboratory comparison of CRONUS-A in situ ¹⁴C values therefore suggest a 6 % measurement uncertainty is too



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low (Jull et al., 2015). LDEO report a higher than average in situ ¹⁴C concentration for CRONUS-A of 7.18 ± 0.15 x 10⁵ atoms g⁻¹
(Lamp et al., 2019), making the CRONUS-A value reported from Tulane 14.8 % lower than the value reported from LDEO. The difference between CRONUS-A values reported by Tulane and LDEO, however, explains why the ¹⁴C-¹⁰Be ratio from sample TUR-138, (extracted at LDEO) plotted above the constant exposure line (Fig. 5a, Fig. 7a). Reducing the in situ ¹⁴C concentration of TUR-138 by 14.8 % from 2.03 x 10⁵ at g⁻¹ to 1.73 x 10⁵ at g⁻¹ results in TUR-138 plotting within the steady-state erosion island.

650 Table 3: Summary table of CRONUS-A intercomparison material and long-term blank values reported from different in situ ¹⁴C extraction facilities. Note – Tulane and LDEO are examined more closely over several measurement cycles as in situ ¹⁴C measured from Mt Murphy samples was extracted at both facilities. The latest AixMICADAS gas ion source AMS measurements reported by LDEO are also included to highlight improved ¹⁴C background levels using this extraction technique. *In Balco et al., (2022) long-term blank values for Tulane surface sample measurements presented in this study are not reported but blank variability at Tulane 655 over this time period is discussed at length.

Extraction Laboratory	CRONUS A (at g ⁻¹)	No. CRONUS-A	Long-term blank (at g ⁻¹)	Associated Publication
Tulane - pre-2019	$6.12\pm 0.32\ x\ 10^{5}$	10	$0.98\pm 0.32\;x\;10^{5}$	Goehring et al., 2019
Tulane - Mt Murphy (initial)	$6.12\pm 0.32\ x\ 10^5$		$4.53 \pm 0.24 \; x \; 10^4$	*Balco et al., 2022
Tulane - Mt Murphy (replicates)	$6.12\pm 0.32\;x\;10^{5}$		$7.14 \pm 0.30 \; x \; 10^4$	*Balco et al., 2022
LDEO - Graphitised pre-2014	$6.74\pm 0.10\;x\;10^{5}$	5	$1.19\pm 0.37\;x\;10^{5}$	Lamp et al., 2019
LDEO - Graphitised post-2014	$7.18\pm 0.15\ x\ 10^{5}$	7	$1.19\pm 0.37\;x\;10^{5}$	Lamp et al., 2019
LDEO - AixMICADAS	$6.62\pm 0.09\;x\;10^{5}$	5	$0.75\pm 0.04\;x\;10^{5}$	Young et al., 2021
ETH Zurich (2011 - 2013)	$7.09\pm 0.39 \; x \; 10^5$	13	$3.48 \pm 2.04 \; x \; 10^4$	Lupker et al., 2015
ETH Zurich 2018	$7.28\pm 0.03\ x\ 10^{5}$	7	$2.63 \pm 1.05 \; x \; 10^5$	Lupker et al., 2019
Cologne	$6.72\pm0.71\ x\ 10^{5}$	6	$1.00\pm 0.68\ x\ 10^4$	Fulop et al., 2015
ANSTO	$6.93\pm 0.44\ x\ 10^{5}$	14	$0.98\pm 0.68\ x\ 10^4$	Fulop et al., 2019
PRIME Lab (Purdue)	$6.89\pm 0.04\;x\;10^{5}$	6	$1.84\pm 0.38\;x\;10^{5}$	Lifton et al., 2015
University of Arizona	$7.08\pm0.17\;x\;10^{5}$	12	$3.40\pm 0.90\;x\;10^4$	Lifton et al., 2023
Working interlaboratory - Mean	$6.93 \pm 0.44 \ x \ 10^5$	23		Jull et al., 2015
Working interlaboratory - Median	6.97 x 10 ⁵	23		Jull et al., 2015

Inconsistencies in the interlaboratory reproducibility of CRONUS-A and corresponding underestimation of in situ ¹⁴C measurement uncertainty has been documented in previous studies highlighting that laboratories uniformly underestimated the magnitude by which empirical coefficients of variation exceeded average reported analytical uncertainties for all nuclides (Phillips et al., 2016a; Jull et al., 2015). However, the underestimation in the reported analytical uncertainty exceeds 300 % for ¹⁴C on the CRONUS-A material (Phillips et al., 2016b). Moreover, in an effort to calibrate in situ ¹⁴C spallogenic production. Borchers et al. (2016) found

material (Phillips et al., 2016b). Moreover, in an effort to calibrate in situ ¹⁴C spallogenic production, Borchers et al. (2016) found that scatter of in situ ¹⁴C measurements in excess of an assumed uncertainty of 7.3 % was more significant than variability between production rate calibration sites (Borchers et al., 2016). Impurities in quartz mineral separates have previously been evidenced to





negatively impact the reproducibility of ¹⁰Be (Corbett et al., 2022), and it is possible quartz impurities are also contributing to the lack of in situ ¹⁴C reproducibility in our Mt Murphy dataset. However, a preliminary investigation based on ICP-EOS elemental data of Mt Murphy samples measured for in situ ¹⁴C in this study found no significant link between low abundances of elemental impurities in quartz mineral separates and in situ ¹⁴C reproducibility (see Supplement S3, Table S4, Table S5).

The CRONUS-A intercomparison material is derived from a high elevation site (1612 m) in Antarctica with millions of years of constant exposure, making it saturated with respect to ¹⁴C (mean value = $6.93 \pm 0.44 \times 10^5$, Jull et al., 2015). Reproducibility estimates from CRONUS-A are, therefore, only representative for high concentration samples, for which AMS counting errors and blank contributions are typically low (Hippe, 2017). Achieving the same level of measurement precision in a sample with a lower concentration of in situ ¹⁴C in a sample is more challenging, and a typical sample exposed during the Holocene will yield an in situ ¹⁴C concentration lower than CRONUS-A. For instance, sample 10-MPS-022-CSP from the Schmidt Hills ($8.3 \pm 0.8 \text{ ka}$, 352 m a. s. 1.) has an in situ ¹⁴C concentration of $1.28 \pm 0.20 \times 10^5$ at g⁻¹ (Nichols et al., 2019). Samples exposed during the Holocene, and

675 particularly those at low elevations such as the scoria cone and Kay Peak, are therefore more sensitive to blank correction than CRONUS-A.

4.5.2 Assessing uncertainty affecting in situ ¹⁴C reproducibility – in situ ¹⁴C blank measurements

- For our samples, the blank correction applied to in situ ¹⁴C repeat measurements was higher than that of the initial ¹⁴C measurements (see Table 3). We also assigned a nominal 6 % 1σ measurement uncertainty to in situ ¹⁴C concentrations in this study which assumes scatter in process blank concentrations was normally distributed (Goehring et al., 2019a). The distribution of the long-term ¹⁴C measurement background of samples from the Tulane University extraction facility over which our initial and replicate samples were measured in this study is, however, long-tailed and non-time dependent (Balco et al., 2022).
- Blank correction of in situ ¹⁴C AMS measurements using a long-tailed distribution is appropriate for low concentration samples such as subglacial bedrock cores (Balco et al., 2023) for which blank variability constitutes the dominant source of uncertainty in the in situ ¹⁴C measurements (Balco et al., 2022). Applying this same blank correction (assuming a long-tailed distribution) to in situ ¹⁴C concentrations measured in our Mt Murphy surface samples results in 2 of 4 in situ ¹⁴C replicate samples reproducing at 68 % confidence. In addition, the exposure age calculated from the in situ ¹⁴C concentration CIN-108-R becomes concordant with the CIN-108 ¹⁰Be exposure age (68 % confidence). However, uncertainty ranges in Mt Murphy surface sample concentrations are very large when using the long tailed blank correction (see Fig. S8), especially for the replicates reported with a higher blank (7.14 ± 0.30 x 10^{5 14}C atoms g⁻¹). For instance, the 68 % confidence uncertainty estimate for the in situ ¹⁴C concentration

of CIN-112-R is +24.1 %, -11.2%, which when propagated results in an uncertainty on the exposure age of CIN-112-R of > 4 kyr. The differences in in situ concentrations and replicate in situ ¹⁴C concentrations may be explained, in part, by several

changes made to the in situ ¹⁴C extraction line at Tulane between the extraction of in situ ¹⁴C for the initial and replicate measurements, including the addition of a new coil trap (Lifton et al., 2023) and a new mullite tube which has previously been observed to increase background ¹⁴C (see methods section 2.1). It is not clear how significant the high blank may have been to the final measurements, or if it represents a systematic addition of in situ ¹⁴C to the replicate in situ ¹⁴C exposure ages. It does, however,



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reiterate the importance of minimising blank levels, and potentially suggests that the long-tailed blank correction discussed at length in Balco et al., (2022) should be applied to in situ ¹⁴C measurements, especially for samples such as those in bedrock cores and our Mt Murphy surface samples.

4.5.3 Reducing in situ ¹⁴C background – gas-ion source AMS of non-graphitised in situ ¹⁴C samples

Directly measuring CO₂ has the potential to lower the blank value as it removes a source of in situ ¹⁴C background contribution from graphitisation (Hippe et al., 2013; Bard et al., 2015). The LDEO extraction facility noted improved procedural blanks by measuring situ ¹⁴C using the AixMICADAS gas-ion source AMS instrument, reporting a 35 % reduction in the average in situ ¹⁴C blank concentration from 1.19 ± 0.37 x 10⁵ atoms g^{-1 14}C to 0.75 ± 0.04 x 10⁵ atoms g^{-1 14}C (Lamp et al., 2019). AMS measurements also yielded a lower CRONUS-A concentration of 6.59 ± 0.09 x 10⁵ atoms g^{-1 14}C (Lamp et al., 2019), much closer to CRONUS-A measurements reported from Tulane (difference < 6000 atoms g⁻¹). The lower CRONUS-A in situ ¹⁴C concentration and reduced background using AixMICADAS may suggest a source of contaminant ¹⁴C impacted earlier CRONUS-A extraction at LDEO (Lamp et al., 2019; Young et al., 2021). Notably, in situ ¹⁴C CRONUS-A samples extracted at LDEO and measured using the AixMICADAS gas ion source AMS are now much closer to long-term CRONUS-A values reported from Tulane. Blanks also seem to be an order of magnitude lower (1-4 x 10⁴ at g⁻¹) for extraction lines where sample combustion is performed without LiBO₂ flux (Hippe et al., 2013; Lupker et al., 2015; Fülöp et al., 2019), as opposed to flux-based systems (1–4 x 10⁵ at g⁻¹) (Pigati et al.,

2010; Goehring et al., 2014; Lifton et al., 2015b). Mean blank values from systems not using LiBO₂ flux are though subject to larger 715 uncertainties ranging from 60-160 % (1 σ), reflecting the large scatter in the blank data (Hippe, 2017).

More recent in situ ¹⁴C measurements performed on the AixMICADAS AMS yield a mean CRONUS-A value of $6.62 \pm 0.09 \times 10^5$ atoms g⁻¹ (n = 5) (Young et al., 2021). Notably, 10 of 12 in situ ¹⁴C exposure ages reported in Young et al. (2021) which make up the ¹⁴C-¹⁰Be paired exposure ages from Kangiata Nunaata Sermia (KNS), were measured using the AixMICADAS gas ion source AMS in concert with the LDEO CRONUS-A samples reported above. Concordant ¹⁴C-¹⁰Be exposure ages and Type-1 ¹⁴C-

- ¹⁰Be ratios from KNS all suggest a simple exposure history (Young et al., 2021). In this case, the removal of potentially contaminant sources of in situ ¹⁴C from the extraction process (Young et al., 2021) has coincided with the generation of ¹⁴C exposure ages, which are both accurate and consistent with paired ¹⁰Be measurements from the same sample (Fig. 7b). The two graphitised samples measured at LLNL-CAMS have uncertainties that are 7.7 % and 10.4 %, in contrast to samples measured using a gas ion source at CEREGE, where total ¹⁴C concentration uncertainties ranged from 4.3 % to 5.2 % (Young et al., 2021). We do note, however, that
- 725 in situ ¹⁴C measurements of all five graphitised samples from Promontory Point, Lake Bonneville (Lifton et al., 2015a) largely agree within 6 % 1σ analytical uncertainties (Fig. 10, Table S3). The CoV for Promontory Point samples is also 4.7 % (Lifton et al., 2015a), which is better than the CRONUS-A intercomparison CoV of 6.3 % reported by Jull et al. (2015). Therefore, we acknowledge that graphitisation of samples, can produce results comparable in quality to non-graphitised samples, but that graphitisation itself presents a potential source of uncertainty that can be avoided.





4.6 Summary and suggestions for future work

The findings presented in this paper suggest that stated laboratory uncertainties underestimate the true measurement uncertainty of in situ ¹⁴C. This is consistent with previous findings from the CRONUS-Earth Project (Phillips et al., 2016b; Borchers et al., 2016). The current interlaboratory variation in reported CRONUS-A in situ ¹⁴C concentrations of ~ 15 % and in situ ¹⁴C blank variability are highlighted as issues impacting the accuracy and precision of in situ ¹⁴C measurements, as they have been previously (Hippe, 2017; Jull et al., 2015; Phillips et al., 2016a). We suggest that a new blank correction approach using a long-tailed blank distribution (Balco et al., 2022, 2023) may better account for the true analytical uncertainty in ¹⁴C measurements, especially at low concentrations. The blank correction results in larger uncertainties on in situ ¹⁴C concentrations, allowing for greater measurement scatter, and thereby improving measurement reproducibility. Ongoing progress including automation of in situ ¹⁴C extraction (Goehring et al., 2019a; Lupker et al., 2019; Lifton et al., 2023) will help facilitate analysis of additional replicates and process blanks, which is needed to improve the precision of in situ ¹⁴C measurements. With a focus on improving ¹⁴C analytical reproducibility and precision, we therefore make the following suggestions which will ultimately contribute to the provision of robust combined ¹⁴C-¹⁰Be chronologies:

- Routinely undertaking and reporting more in situ ¹⁴C replicate measurements. This will provide a check on quality control.
- Adopting the blank correction procedure outlined in Balco et al. (2023) for low concentration samples where blank scatter is dominant and blanks do not exhibit a normal distribution. This will provide a better appreciation of the true analytical long-tailed uncertainty distribution present in in situ ¹⁴C measurements.
 - Undertaking a comparison study of the reproducibility of graphitised versus non graphitised in situ ¹⁴C measurements of the same sample(s) and associated blank performance.
 - Quantifying if quartz impurities are contributing to poor reproducibility, in a more in-depth study similar to Corbett et al., (2022).

5 Conclusion

- In this study, we have assessed new ¹⁴C-¹⁰Be exposure ages from Mt Murphy, West Antarctica, in the context of a wider repository of global Holocene age ¹⁴C-¹⁰Be paired measurements extracted from the informal cosmogenic-nuclide exposure age database (ICE-D, <u>https://version2.ice-d.org/</u>). New paired ¹⁴C-¹⁰Be exposure ages from several sites at Mt Murphy display conflicting exposure histories. Young in situ ¹⁴C ages from high elevations that are discordant with ¹⁰Be measured in the same sample appear to have deglaciated after concordant paired ¹⁴C-¹⁰Be exposure ages from lower elevations with simple exposure histories. Statistical analysis of a large in situ ¹⁴C exposure age dataset (n = 20) from Mt Murphy also indicates scatter in excess of
- 760 analytical uncertainty of almost 2 kyr. There is no apparent geological explanation for divergent concordant-discordant exposure histories nor excess scatter observed within the in situ ¹⁴C dataset. Instead, we find that 3 of 4 replicate in situ ¹⁴C measurements performed on samples from Mt Murphy do not reproduce within a 6 % 1σ measurement uncertainty, with 2 of 4 measurements still





not reproducing at 2σ. Furthermore, concordant ¹⁴C-¹⁰Be pairs at Mt Murphy with simple exposure histories exhibit reproducible in situ ¹⁴C concentrations, but discordant in situ ¹⁴C exposure ages suggestive of complex exposure are not reproducible. Our observations from Mt Murphy are reflected in reported in situ ¹⁴C concentrations from ICE-D, where replicate in situ ¹⁴C concentrations measured in 18 of 31 samples fail to reproduce within the 6 % 1σ measurement uncertainty (15 of 31 at 2σ) one or more times. In addition, Sjögren Glacier (a site where 2 of 3 replicate in situ ¹⁴C concentrations are irreproducible) exhibits paired ¹⁴C-¹⁰Be production ratios which exceed theoretical limits and lack geological explanation.

- In summary, the results of our analysis of ¹⁴C-¹⁰Be exposure ages from ICE-D are consistent with the interpretation that discordant ¹⁴C-¹⁰Be exposure ages from Mt Murphy are a result of isolated issues of in situ ¹⁴C reproducibility, while concordant ¹⁴C-¹⁰Be pairs are consistent with a deglaciation history from 9–6 ka identified in previous studies. Currently, in situ ¹⁴C measurement uncertainty may therefore be underestimated due to additional, as yet unquantified, sources of scatter. Several factors may contribute to the low in situ ¹⁴C reproducibility observed in this study and require further investigation. These include long term blank variability within in situ ¹⁴C extraction facilities, differences in CRONUS-A measurements between in situ ¹⁴C extraction laboratories, and the influence of changes to the Tulane extraction line between in situ ¹⁴C initial and replicate measurements. Quartz impurity may also impact sample reproducibility, although our preliminary examination of ICP-OES data found no evidence to
- impurity may also impact sample reproducibility, although our preliminary examination of ICP-OES data found no evidence to suggest that quartz sample purity contributed to lack of in situ ¹⁴C reproducibility in our samples. Quantifying the excess scatter observed in this study is important because, if used in isolation, in situ ¹⁴C exposure ages appear to currently lack the precision needed to reconstruct Holocene deglacial histories at sub-millennial resolution.

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Data and code availability: Some of the in situ ¹⁴C data examined in this study were obtained from the informal cosmogenicnuclide exposure age database (ICE-D) and remain unpublished. These data are, however, freely available in ICE-D under the public release requirements of the National Science Foundation (NSF) U.S. Antarctic Program which requires data be made publicly available 2 years after collection. In situ ¹⁴C AMS and exposure age data shown in Table 1 will be publicly accessible in the UK Polar Data Centre https://doi.org/10.5285/dbb30962-bbf3-434a-9f27-6de2f61a86e2

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