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Data report: composite section and chronology of IODP Sites 339-U1385 and 397-U1385 based on postcruise XRF analysis[1]

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Contents

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Abstract

Following International Ocean Discovery Program (IODP) Expedition 397, split archive halves from Site 397-U1385 were analyzed at 1 cm resolution using an Avaatech X-ray fluorescence (XRF) core scanner at the University of Cambridge (United Kingdom). The resulting Ca/Ti profiles were used to revise the composite depth scales of Integrated Ocean Drilling Program Sites 339-U1385 and 397-U1385.

Expedition 339 recovered a nearly continuous sediment sequence to 151.5 meters below seafloor (mbsf) at Site U1385, except for a gap spanning Marine Isotope Stage (MIS) 11/Termination V at 53.06 mbsf. This interval was successfully retrieved during Expedition 397, completing the 1.45 My section. The existing depth scale for Site 339-U1385 was revised to integrate this missing sequence. Using dynamic programming, the Ca/Ti signal from Site 397-U1385 was stretched and compressed to align with the revised depth scale of Site 339-U1385. This alignment enables direct transfer of age models and isotopic records between the two.

Below 1.45 Ma (148.83 m core depth below seafloor, Method A [CSF-A], or 176.64 m core composite depth below seafloor [CCSF]), the chronology at Site 397-U1385 is established through benthic oxygen isotope stratigraphy and orbital tuning. The isotope record aligns with Site 339-U1385 for MISs 41–47 and extends to MIS 61, where a hiatus (~1.72–1.87 Ma) removed MISs 62–70. Below the hiatus, pronounced precession-driven variations in Ca/Ti serve as the basis for astronomical tuning, extending the chronology to 5.3 Ma. The Site 397-U1385 record provides a near-continuous record of changes in sediment composition at millennial resolution to the base of the Pliocene.

1. Introduction

International Ocean Discovery Program (IODP) Site U1385 (37°34.285′N, 10°7.562′W; 2578 m below sea level), referred to as "the Shackleton site," has become a benchmark record for millennial climate variability (MCV) for the last 1.45 My (Figure **F1**) (Hodell et al., 2023). The site was first drilled in November 2011 during Integrated Ocean Drilling Program Expedition 339 when cores from Holes U1385A–U1385E were recovered (Expedition 339 Scientists, 2013; Hodell et al., 2013b), reaching a maximum depth of 151.5 meters below seafloor (mbsf) (Figure **F2**). In November 2022, the site was reoccupied during IODP Expedition 397 when Holes U1385F–U1385J were drilled, extending the sedimentary section to 400 mbsf (**Hodell et al.**, 2024b). Most recently, IODP

Expedition 401 returned to the site and deepened it to 552.5 mbsf (Flecker et al., 2024). Reoccupations of Site U1385 are designated by prefixing the site name by its expedition numbers: 339-U1385, 397-U1385, and 401-U1385.

Here, we report the X-ray fluorescence (XRF) data collected postcruise for Site 397-U1385, which is used to revise the shipboard composite depth scale and integrate the stratigraphy with Site 339-U1385. Integration of Site 401-U1385 by Expedition 401 scientists will soon follow (Flecker et al., 2024).

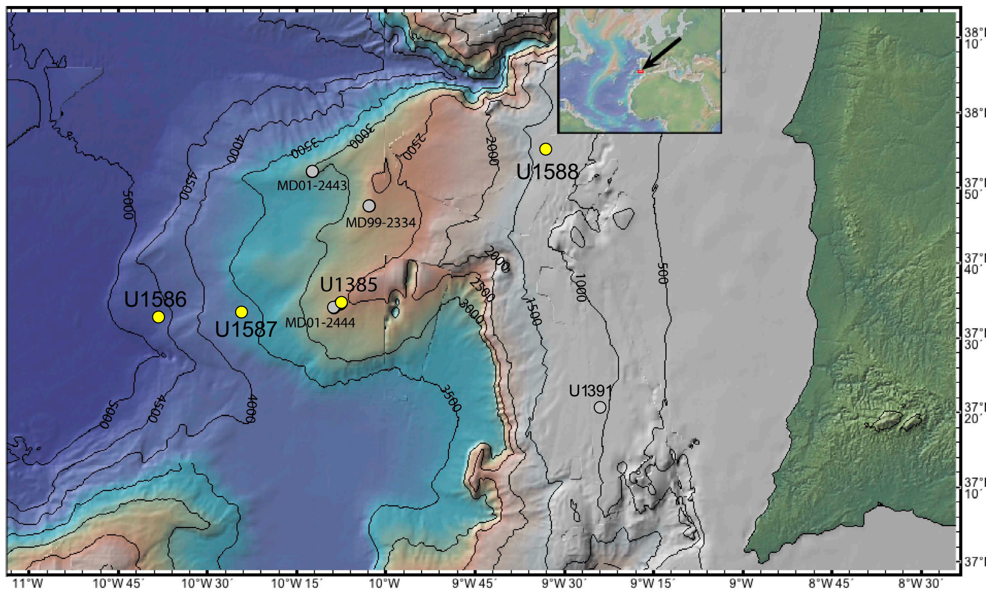

Figure F1. Location of Expedition 397 sites on Promontório dos Príncipes de Avis (PPA) off Portugal. Also shown are Marion Dufrense (MD) piston cores and Integrated Ocean Drilling Program Expedition 339 Site U1391. Map is modified from Hodell et al. (2015) and was made with GeoMapApp (**http://www.geomapapp.org**) using the bathymetry of Zitellini et al. (2009).

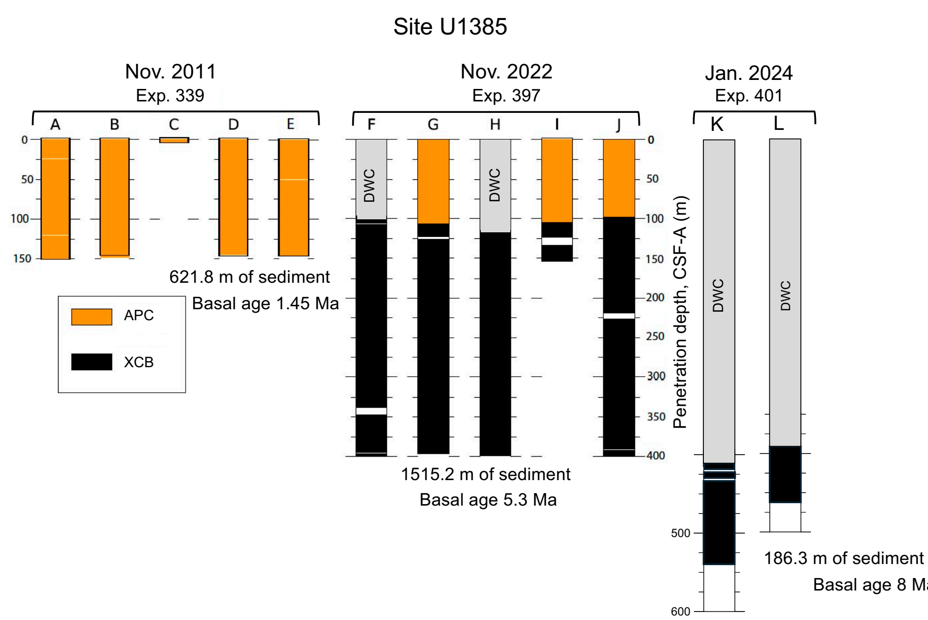

Figure F2. Core recovery from each Site U1385 hole, Expeditions 339 (Holes U1385A–U1385E), 397 (Holes U1385F–U1385J), and 401 (Holes U1385K and U1385L). Holes U1385F, U1385H, U1385K, and U1385L were drilled ahead before core recovery began at depth. Gray = drilled without coring (DWC), white = no recovery. APC = advanced piston corer, XCB = extended core barrel.

A composite section for Site 339-U1385 was originally constructed by correlating Ca/Ti ratios across Holes U1385A–U1385E (Hodell et al., 2015). This record extends to Marine Isotope Stage (MIS) 47, with a basal age of ~1.45 Ma based on correlation to the global oxygen isotope stack (Lisiecki and Raymo, 2005; Ahn et al., 2017), referred to hereafter as LR04. The sequence is nearly continuous, except for a short (~30,000 y) hiatus spanning late MIS 12 and early MIS 11. It was hoped that Expedition 397 would recover this missing interval by targeting a nearby location.

The primary drilling objectives of Site 397-U1385 were to (1) recover the MIS 11/Termination V interval and (2) extend the sedimentary section below the base of Site 339-U1385 to the base of the Pliocene. Both objectives were successfully achieved (**Hodell et al.**, 2024b). Because of heavy sampling of the working halves of cores from Expedition 339 Holes U1385A–U1385E, the upper ~150 m were redrilled in Holes U1385G, U1385I, and U1385J during Expedition 397 (Figure **F2**), providing additional core material for sampling and future research.

The stratigraphy of Site 339-U1385 is well established, with several age models available based on orbital tuning, oxygen isotopes, and correlation to ice cores (Hodell et al., 2015, 2023). Integration of Sites 339-U1385 and 397-U1385 into a unified depth scale will permit transfer of these established chronologies and isotope stratigraphy from Site 339-U1385 to the new cores recovered at Site 397-U1385.

The objectives of this data report are to (1) present the high-resolution XRF core scanning data from Site 397-U1385, (2) construct a revised composite section for Site 339-U1385 by filling the MIS 11/Termination V gap with Site 397-U1385 data, (3) optimally align core depths at Site 397-U1385 with those from Site 339-U1385 for the upper ~150 mbsf, (4) refine the shipboard affine and splice tables for Site 397-U1385, and (5) develop a robust preliminary chronology for Site 397-U1385 to the base of the Pliocene.

2. Methods

2.1. XRF core scanning

The split archive halves from Site 397-U1385 were analyzed using an Avaatech XRF core scanner in the Godwin Laboratory at the University of Cambridge (United Kingdom) to obtain semiquantitative elemental data at a 1 cm resolution across all holes. Scanning the full 1515.2 m of recovered core was a monumental effort that took over a year of continuous analysis, but the resulting high-quality records justified the undertaking. A total of 147,379 measurements were acquired for each of the 10 and 30 kV energy settings.

Before XRF scanning, each core was allowed to equilibrate to room temperature, and each section was imaged using the Avaatech linescan camera system prior to XRF analysis. The surface of each section was then carefully scraped clean, surface irregularities were flattened, and sulfide nodules were removed and labeled as samples. The core surface was covered with a 4 µm thin SPEXCertiPrep Ultralene foil to avoid contamination and reduce desiccation (Richter et al., 2006), which was gently smoothed with a roller to remove air under the surface. The surface area irradiated by X-radiation was 120 mm^2, defined by a slit size of 10 mm along core and 12 mm cross core. Cores were scanned at (1) 10 kV and 750 µA with a 20 s count time and no filter and (2) 30 kV and 500 µA with a 20 s count time using a Pd thin filter.

Elemental intensities were obtained through postprocessing of the XRF spectra using Canberra WinAxil software with standard settings and spectral fitting models. Although intensities are proportional to elemental concentrations, they are also influenced by matrix effects, physical properties, sample geometry, and scanner settings (Tjallingii et al., 2007). To minimize geometric and matrix-related variability, log-ratios of elemental intensities were calculated, which provide a more robust proxy for relative concentration (Weltje and Tjallingii, 2008; Dunlea et al., 2020).

Although Ca and Ti are measured simultaneously at both 10 and 30 kV, the 10 kV counts are significantly greater than those at 30 kV and are considered to have a greater signal-to-noise ratio. For Zr/Sr, we ratio to Sr instead of Ca because Zr and Sr are measured in the same 30 kV scan and have similar fluorescence and counts. The log(Ca/Ti) is highly correlated with measurements of weight percent $CaCO_3$ made by coulometric titration during Expedition 397 (**Abrantes et al.**, 2024a), with an r^2 of 0.967 (Figure **F3**). The following regression equation allows estimation of $CaCO_3$ content from the Site U1385 XRF data:

$$CaCO_3\ (wt\%) = 38.664 \times \log(Ca/Ti) - 24.145.$$

Previous regression analyses of log(Ca/Ti) and $CaCO_3$ content from Piston Cores MD01-2444 and MD01-2443 also revealed strong correlations (Hodell et al., 2013a). The slopes of the regression equations are consistent within analytical error, with differences in intercepts attributed to variations in sediment water content. Notably, Site 397-U1385 cores were measured shortly after recovery, whereas Piston Cores MD01-2443 and MD01-2444 were analyzed over a decade later, allowing for postrecovery water loss.

2.2. Oxygen isotope stratigraphy

Oxygen and carbon isotope measurements were performed on the planktonic foraminifer *Globigerina bulloides* from the 250–300 µm size fraction. For benthic foraminifers, primarily *Cibicidoides wuellerstorfi* and occasionally *Cibicidoides mundulus* were used for analysis from the >150 µm size fraction. Analytical procedures followed the methods described in Hodell et al. (2015).

2.3. Stratigraphic correlation

The methodology for composite section and splice construction followed standard IODP protocols and mirrored the procedures used to generate the shipboard splice during Expedition 397 (**Abrantes et al.**, 2024a). Depths on the core depth below seafloor, Method A (CSF-A), scale (equivalent to mbsf) were adjusted using bespoke IODP Correlator software to align correlative features in the Ca/Ti records, resulting in a revised depth framework: core composite depth below seafloor (CCSF). The calculated depth offsets for each core are provided in the affine table (see TABLES in **Supplementary material**). Upon completion of the affine table, a spliced composite section was constructed by selecting continuous, overlapping intervals from multiple holes to produce a stratigraphically complete record for Site U1385 (see TABLES in **Supplementary material**). The updated affine (CCSF-397-U1385-FGHIJ-20250507-Hodell) and splice (SPLICE-397-U1385-FGHIJ-20250507-Hodell) tables are also available from the IODP Library Information Management System (LIMS) database at **https://web.iodp.tamu.edu/LORE**.

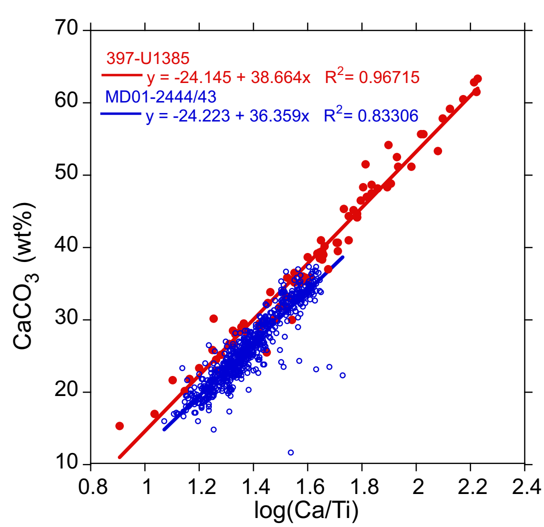

Figure F3. Comparison of $CaCO_3$ weight percentage (measured by coulometric titration on discrete samples) and log(Ca/Ti) values at corresponding core depths, Site 397-U1385 (Hodell et al., 2024b) and Piston Cores MD01-2443 and MD01-2444 (Hodell et al., 2013a). Strong correlation supports use of log(Ca/Ti) as a proxy for $CaCO_3$ content.

3. Results

3.1. Filling the gap at Site 339-U1385

Site 339-U1385 contains a stratigraphic hiatus that removed part of Termination V and MIS 11. Recovery of MIS 11 has proven difficult during the two expeditions; however, Core 397-U1385I-6H captured the high-carbonate, sticky mud deposited during this interval (Figures **F4**, **F5**). The Ca/Ti record clearly shows two peaks related to the two precession minima that occurred during MIS 11e. Oxygen isotope analysis of foraminifers further confirms the presence of Termination V and MIS 11 in Core 6H (Figure **F6**).

To splice the missing sequence into the Site 339-U1385 composite, a 4.46 m interval from Core 397-U1385I-6H (Sections 6H-4, 70 cm, to 6H-7, 75 cm) was inserted (Figure **F7**). This interval was placed at 53.858 corrected revised meters composite depth (crmcd) in the Site 339-U1385 composite depth scale, as defined by Hodell et al. (2015). Consequently, the remaining portion of the 339-U1385 composite section was shifted downward by 2.192 m, beginning at 56.128 crmcd, which is redefined as 58.320 crmcd*. New age-depth tie points were added to the existing age model to assign ages to the MIS 11/Termination V interval inserted from Site 397-U1385.

3.2. Revised affine and splice tables for Site 397-U1385

During Expedition 397, a shipboard splice was constructed to 452.7 m CCSF by correlating physical properties across five holes (U1385F–U1385J) (**Hodell et al.**, 2024a). The original affine table and splice table were subsequently revised ahead of the Expedition 397 sampling party at the Bremen Core Repository (BCR) using high-resolution Ca/Ti data obtained from XRF core scanning. This revised sampling splice (SPLICE-397-U1385-FGHIJ-20230426) served as the primary reference for guiding sample selection.

Minor revisions to the sampling splice were necessary following the sampling party when comparison with IODP Sites U1586 and U1587 revealed a duplicated cycle in the Pliocene section of Site U1385. Every effort was made to adhere as closely as possible to the original sampling splice to

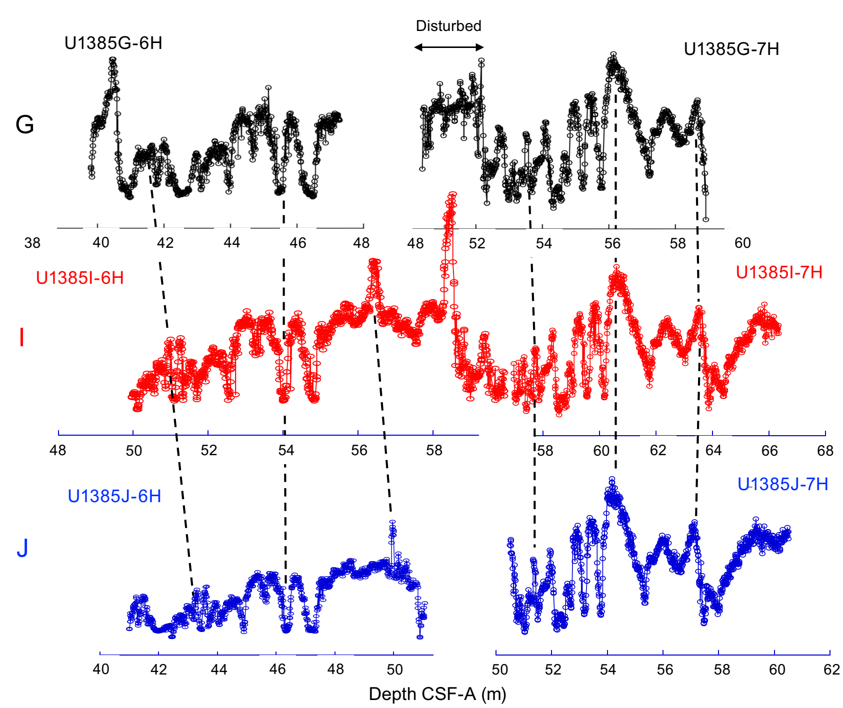

Figure F4. Ca/Ti signals for Cores 6H and 7H in Holes U1385G, U1385I, and U1385J showing tie lines connecting correlative features across cores. Only Core 397-U1385I-6H recovered a complete section across MIS 11/Termination V. The two highest peaks in Ca/Ti in Core 397-U1385I-6H are assumed to correlate with two precession minima during MIS 11e.

ensure that discrete samples will fall on the revised splice, minimizing potential gaps. The updated affine and splice tables for Site 397-U1385 are presented in TABLES in **Supplementary material** and are available from the LIMS database at **https://web.iodp.tamu.edu/LORE**.

3.3. Correlating Site 397-U1385 to Site 339-U1385

Dynamic time warping is a family of algorithms that optimally aligns two time series by stretching and squeezing one series to match the other. We used the Match algorithm (Lisiecki and Lisiecki, 2002) to optimally align the spliced Ca/Ti signal of Site 397-U1385 with that of Site 339-U1385

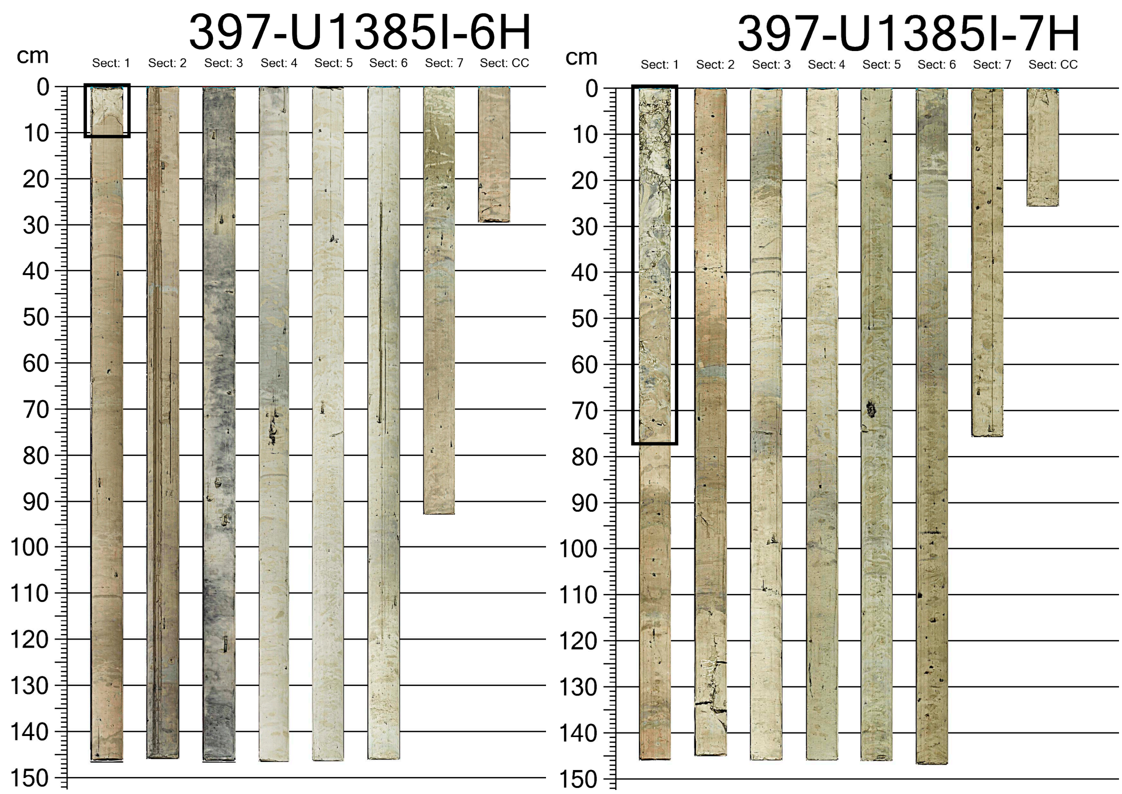

Figure F5. Core photographs taken during Expedition 397 shortly after core splitting (U1385I-6H and 7H). Images have been enhanced by adjusting brightness and contrast to emphasize core disturbance and color changes. Dark patches in Section 6H-3 indicate reducing core conditions that rapidly disappear upon exposure to atmospheric oxygen. Black boxes = disturbed intervals in tops of each core.

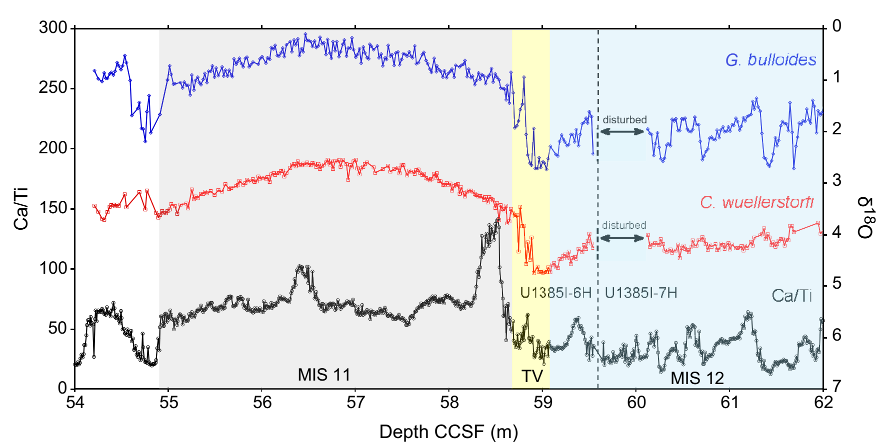

Figure F6. Oxygen isotopes of *G. bulloides* and *Cibicidoides* and Ca/Ti (397-U1385I-6H and 7H). Gray shading = MIS 11, blue = MIS 12, yellow = Termination V.

(Figure **F7**). The Ca/Ti signals were first smoothed with a Gaussian process (GP) filter from the GPy Python library (**https://gpy.readthedocs.io/en/deploy/GPy.core.html** with a Matern32 kernel). This treatment allowed for alignment of the records to within a few centimeters accuracy. Match provides default penalty settings for the length of the equivalent sections (nomatch), average scaling of the mapping (speedpenalty), deviation from constant scaling of the alignment (speedchange), and adherence to the inserted tie point (tiepenalty). The speedpenalty and speedchange were relaxed slightly to improve the total penalty score by ~10% compared to the default settings. The warping function consists of 1198 match pairs of CCSF depths at Site 397-U1385 to equivalent crmcd* depths at Site 339-U1385. Linear interpolation between match pair points is used to calculate the crmcd* depth for any CCSF depth value. The base of the Site 339-U1385 record (168.702 crmcd*) corresponds to 178.4 m CCSF. Below this level, 9.698 is subtracted from the Site 397-U1385 CCSF depth to calculate crmcd*.

3.4. Chronology

For the past 1.45 My, the age model for Site 397-U1385 was obtained by precisely correlating its Ca/Ti record with that of Site 339-U1385 (Figure **F8**). This high-resolution correlation enables the direct transfer of any existing age model from Site 339-U1385 to Site 397-U1385 (Figure **F9**).

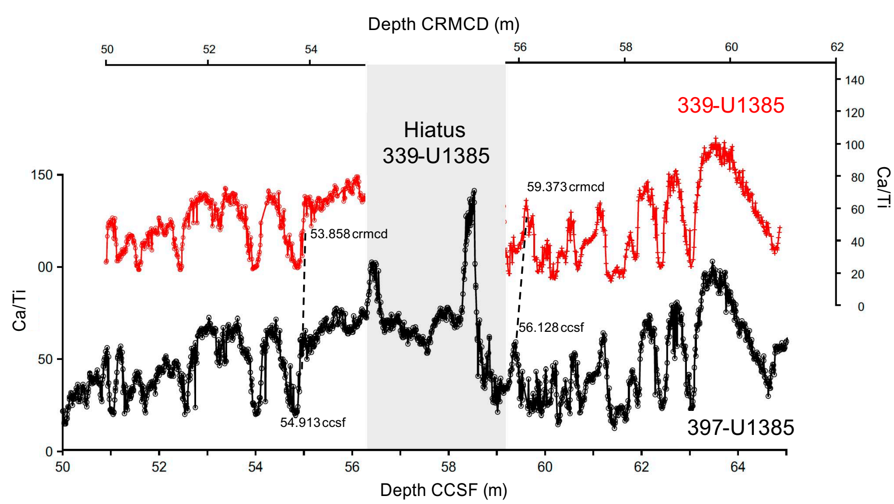

Figure F7. Tie points used to splice 4.46 m section from Site 397-U1385 to fill gap over MIS 11/Termination V at Site 339-U1385.

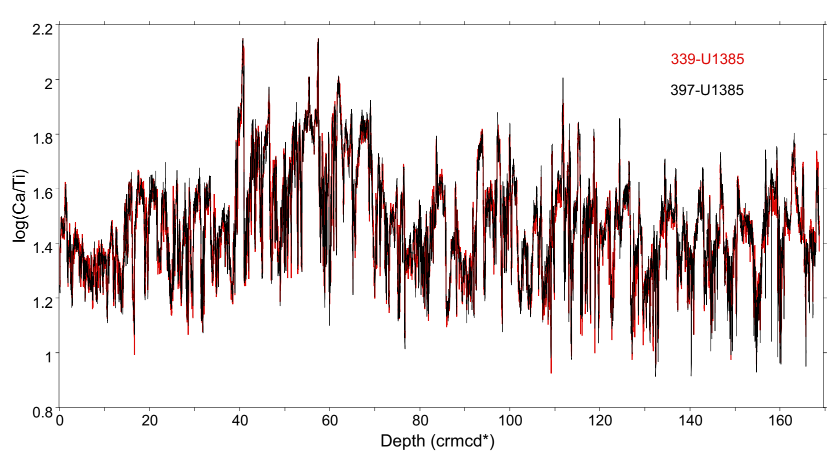

Figure F8. Comparison of correlated Ca/Ti signals using Match (Lisiecki and Lisiecki, 2002), Sites 397-U1385 and 339-U1385. Depth scale (crmcd) of Hodell et al. (2015) was revised to crmcd* by inserting missing MIS 11/Termination V interval recovered from Site 397-U1385.

To assess the stratigraphy below 1.45 Ma, oxygen isotopes were measured on benthic foraminifers and compared with the LR04 ProbStack (Ahn et al., 2017; Lisiecki and Raymo, 2005). Because some regional differences exist between Atlantic $\delta^{18}O$ and the globally stacked record (Zhou et al., 2024), we also compared the Site U1385 benthic $\delta^{18}O$ record to Integrated Ocean Drilling Program North Atlantic Site U1308 (Hodell and Channell, 2016).

The Site 397-U1385 isotope record overlaps with Site 339-U1385 for MISs 41–47, where the two records are in good agreement (Figure **F10**). Oxygen isotope stages are recognized to MIS 61, where a hiatus occurs that removed the interval between ~1.72 and 1.87 Ma, corresponding approximately to MISs 62–70. The oxygen isotope record continues with MIS 71 below the hiatus. The hiatus occurs somewhere between Samples 397-U1385G-20X-5, 125 cm, and 20X-6, 85 cm, although there are no obvious signs of erosion or slumping (e.g., convoluted bedding). Further study of this hiatus interval is ongoing at Site U1587 with the hope this gap at Site U1385 can be partly or wholly filled with material from another site.

Beyond 2 Ma, strong precession-driven cycles in physical properties (e.g., color reflectance and magnetic susceptibility) and XRF-derived elemental ratios (e.g., Ca/Ti) provided the basis for constructing an astronomically tuned age model for Site 397-U1385. Peaks in Ca/Ti were aligned with precession minima, corresponding to maxima in Northern Hemisphere summer insolation with no lag. The amplitude modulation of precession by eccentricity provides a robust framework for unambiguous tuning between ~2 and 5.3 Ma (Figure **F11**). The reproducibility of the Ca/Ti precession cycles at Sites U1385, U1586 (**Abrantes et al.**, 2024b), and U1587 supports the continuity of the spliced sections. A low-resolution benthic oxygen isotope record permits identification of MISs to 3.4 Ma (Figure **F10**). The generally good agreement between Site U1385 benthic $\delta^{18}O$ on the precession-tuned timescale and LR04 stack provides support for the astrochronology.

Sedimentation rates average 5.4 cm/ky from 5.3 to 3 Ma when they begin to increase until leveling off at ~2.5 Ma (Figure **F12**). This increase reflects enhanced clay delivery to the site during times of lowered sea level associated with stronger glaciations following the intensification of Northern

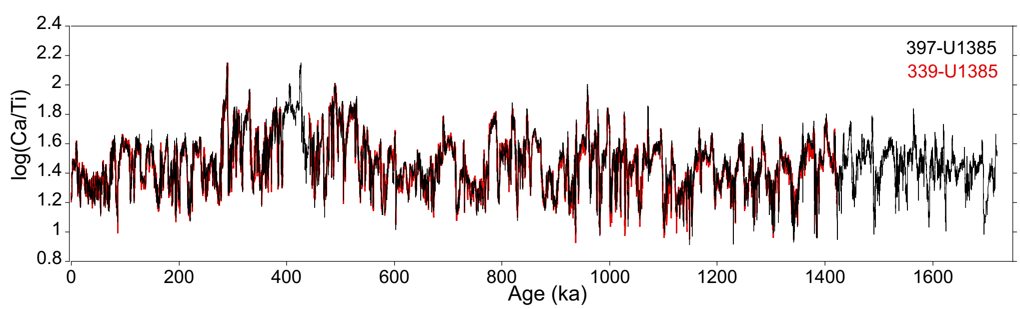

Figure F9. Log(Ca/Ti) records with overlap for last 1450 ky, Sites 339-U1385 and 397-U1385.

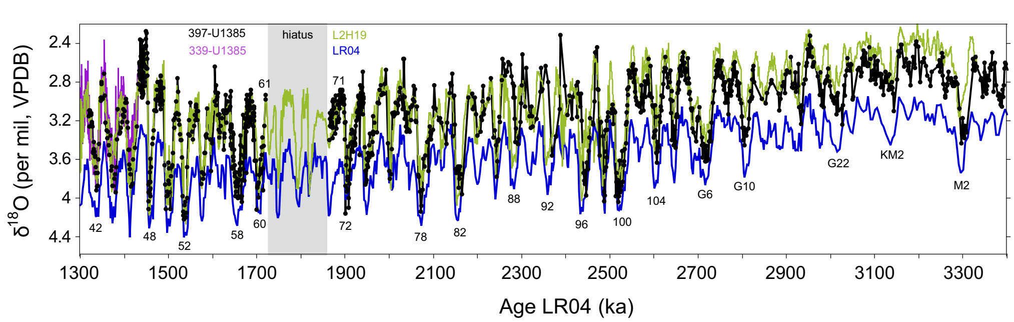

Figure F10. Benthic oxygen isotope record of Site 397-U1385 compared to Sites 339-U1385 and U1308 and LR04 ProbStack. Gray = hiatus at Site 397-U1385 between 1720 and 1870 ka, corresponding to approximately MISs 62–70. Site U1385 record is on LR04 timescale to 2 Ma and precession-tuned timescale thereafter. Records are offset for clarity. LR04 record is adjusted to $\delta^{18}O$ values of *Uvigerina*, but Site U1385 and U1308 records are not.

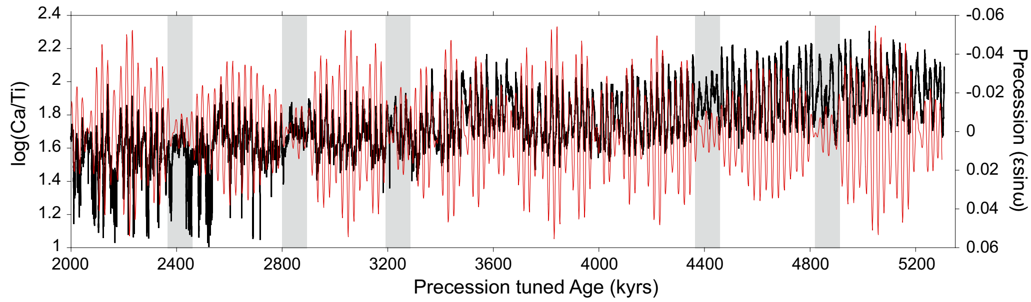

Figure F11. Tuning of peaks in log(Ca/Ti) at Site 397-U1385 (black) with precession minima (red). Gray = times of low eccentricity, which modulate the amplitude of precession and are evident in Site U1385 Ca/Ti record.

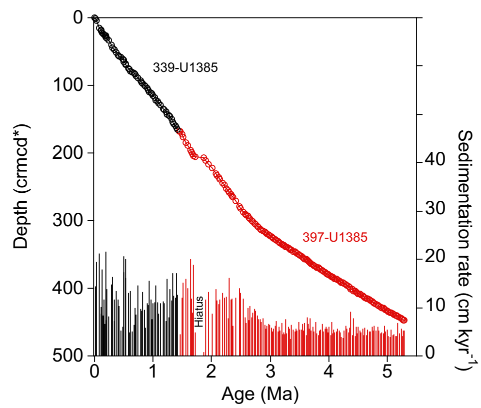

Figure F12. Age-depth relationship and interval sedimentation rates for proposed chronology, Sites 339-U1385 and 397-U1385. Note increase in sedimentation rates between ~3 and 2.5 Ma and hiatus between 1.72 and 1.87 Ma.

Hemisphere Glaciation (Hodell et al., submitted). Sedimentation rates average 11 cm/ky during the Quaternary. The sampling frequency of the XRF data is 1 cm, which translates into an average sampling rate in time of about 200 y for the Pliocene older than 3 Ma and 100 y thereafter in the latest Pliocene and Quaternary. Although the actual resolution is likely to be greater because of bioturbation and drilling disturbance (e.g., biscuiting in extended core barrel [XCB] cores), it is nevertheless high enough to detect millennial events.

4. Summary

Site U1385 serves as a stratigraphic reference section for the other Expedition 397 sites because of its well-established stratigraphy and chronology. Using high-resolution postcruise XRF core scanning data, we constructed a revised composite depth scale and splice for Site 397-U1385 to 448.34 m CCSF. We also filled the stratigraphic gap at Site 339-U1385 caused by the MIS 11/Termination V hiatus using the sediment sequence recovered in Hole U1385I (see FIGURE in **Supplementary material** for an interactive graph of planktonic and benthic oxygen isotopes and Ca/Ti for the last 1500 ky on the LR04 timescale with approximate marine isotope stage boundaries of Lisiecki and Raymo, 2005).

The Ca/Ti records of Sites 339-U1385 and 397-U1385 were correlated using dynamic programming, enabling the transfer of the isotope stratigraphy and chronology from Site 339-U1385 to Site 397-U1385 for the past 1.45 My. A preliminary benthic oxygen isotope record indicates a hiatus between ~1.72 and 1.87 Ma, corresponding to MISs 62–70. Beyond this interval, prominent precession cycles in physical properties and elemental ratios (e.g., Ca/Ti) allowed the construction of an astronomically tuned age model that extends to the base of the Pliocene.

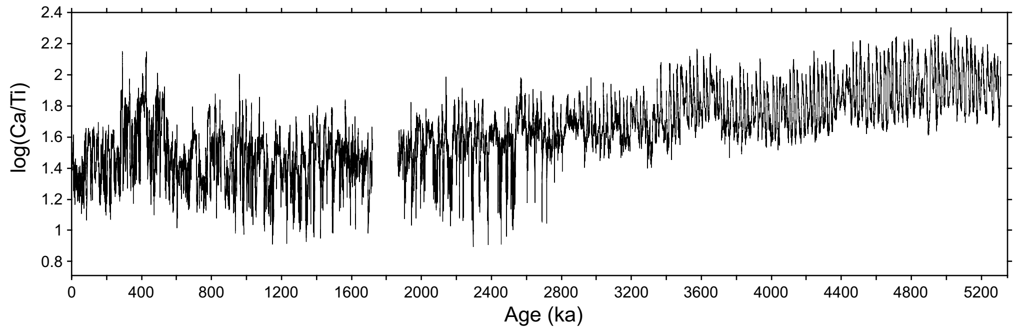

Figure F13. Log(Ca/Ti) for last 5300 ky, Site 339-U1385. Record is continuous except for hiatus between 1720 and 1870 ka.

The Site 397-U1385 record provides a near-continuous 5.3 My long record of changes in sediment composition at millennial resolution to the base of the Pliocene (Figure **F13**). The interpretation and paleoclimatic significance of the XRF data are discussed in Hodell et al. (submitted).

5. Data availability

Ca/Ti for all measured sections are included in TABLES in **Supplementary material** and permanently archived at Zenodo (**https://zenodo.org/communities/iodp**). Ca/Ti and Zr/Sr are also provided for the spliced stratigraphic section of Sites 339-U1385 and 397-U1385. Complementary data sets have been developed for the other Expedition 397 sites, including Sites U1586 (**Abrantes et al.**, 2025), U1587 (Alvarez Zarikian et al., in press), and U1588 (S. Kaboth-Bahr, unpubl. data).

6. Acknowledgments

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