

## **IODP Expedition 401: Mediterranean–Atlantic Gateway Exchange**

### **Week 4 Report (31 December 2023–6 January 2024)**

During Week 4 we began coring Hole U1610A (proposed Site GUB-02A) from the base of casing at 501.9 meters below seafloor (mbsf). Coring was interrupted at 827.8 mbsf by a brake failure in the top drive; while it was being repaired we switched coring systems from the extended core barrel (XCB) to rotary core barrel (RCB). Coring reached 953.9 mbsf by the end of the week, toward the target depth of 1464 mbsf for this site.

### **Operations**

Week 4 of the expedition began with the rig floor team continuing to raise the mud motor bottom-hole assembly (BHA) that had been used to install 10¾ inch casing to 501.9 mbsf in Hole U1610A. The mud motor and underreamer were flushed with 25 bbl of drill water and the BHA was disassembled. The bit cleared the rotary table at 0302 h on 31 December 2023, ending casing operations.

The upper guide horn was moved back into place, and the XCB BHA was assembled with a polycrystalline diamond compact (PDC) bit. We elected to start coring in Hole U1610A with the XCB coring system because it had recovered good quality cores at equivalent depths in Hole U1609A. We anticipated that we would have to change to the RCB coring system at some depth in Hole U1610A, but the additional time to exchange BHAs would be outweighed by the good quality of the XCB cores. The nonmagnetic drill collar (NMDC) was left out of the assembly to improve the robustness of the BHA, and a lockable float valve (LFV) was included for potential downhole logging. The bit was lowered close to the seafloor and the subsea camera was deployed to guide reentry.

Hole U1610A was reentered at 0940 h, and during this process the depth to seafloor was found to be 556.3 meters below sea level (mbsl), identical to the precision depth recorder (PDR) reading, but shallower than the previous estimate of 561.7 mbsl. The camera was raised back to the ship by 1030 h, and the bit was lowered to the base of the hole, 505.2 mbsf. The hole was swept with 30 bbl of sepiolite mud, and at 1315 h the first science activity at the site was to run the Sediment Temperature 2 (SET2) tool. The SET2 was held 5 min at the mudline, followed by the main 10 min hold in the formation at 505.2 mbsf. At 1430 h we started coring. At midnight, scientists, technical staff, and crew members met on the bow to ring in the new year.

XCB coring continued until 0700 h on 4 January 2024. At Core 27X, we started a more rigorous headspace gas sampling protocol for the depth interval 754 to 960 mbsf at this site, following the recommendation by the IODP Environmental Protection and Safety Panel (EPSP) and Texas A&M University Safety Panel. The headspace gas results from each core were analyzed before advancing the bit to collect the next core. This protocol was in effect because a detailed preexpedition analysis of 3-D seismic data showed that there is very low risk of finding gas at the Site U1610 location. Headspace gas results from all XCB cores were found to be in the normal (safe) range of methane/ethane ratios and absolute methane values. Cores U1610A-2X to 36X penetrated from 505.2 to 827.8 mbsf and recovered 322.6 m (93%).

At 0700 h on 4 January there was a failure in the top drive brake system, causing the brake to engage and overheat. We stopped coring and pulled up Core 36X, which had advanced 3 m. The bit was raised to 793.5 mbsf, the top drive was racked to allow repair, and the bit was raised to 495.2 mbsf just inside the casing. The first interval of top drive inspections and repair ran from 0945 to 1330 h. The cause for the failure appeared to be the exhaust valve on the brake. A portion of the diaphragm in the exhaust valve had broken off, lodged in the valve, and kept air pressure to the energizing bladder behind the brake bands. This kept the brake engaged. There was significant damage to the brake and bladder assembly from the resultant overheating. The entire brake assembly needed to be replaced with the spare unit from the warehouse.

Meanwhile, we decided to change the coring system from XCB to RCB for the remainder of the hole. We raised the bit to the ship, clearing the seafloor at 1423 h and the rig floor at 1630 h. Repairs to the top drive continued and were complete by 0215 h on 5 January. The replacement and start-up testing took 13.25 h, overall. The rig floor team then assembled the RCB BHA with a new 9 $\frac{7}{8}$  inch PDC bit and lowered it to 533 mbsl, 23 m above seafloor. The subsea camera was deployed and the ship was positioned for reentry. The view of the cone was partly obscured by a school of fish, which had been attracted by the camera lights. After switching off the lights for a short time, the fish dispersed and Hole U1610A was reentered at 0548 h. The bit was lowered to 810 mbsf. The top drive was picked up and the bit washed down to 827.8 mbsf to start RCB coring. Cores U1610A-37R to 49R penetrated from 827.8 to 953.9 mbsf and recovered 126.1 m (70%). Sepiolite mud sweeps were pumped at 899.0, 915.0, and 944.0 mbsf. Headspace gas results from these cores are within the normal range. Core 50R marked the end of the special headspace gas protocol, with no resultant issues. The procedure resulted in a delay of ~45 min per core, across 23 cores. The week ended at midnight on 6 January with Core 50R in progress at 960.9 mbsf.

## Science Results

### *Lithostratigraphy*

The following lithological units are tentatively defined from macroscopic and smear slide analysis of Cores U1610A-2X to 45R (505.1–915.1 mbsf).

Unit I extends from 505.1 to 699.1 mbsf (Sections U1610A-2X-1 to 22X-CC). This unit is composed of calcareous mud and calcareous silty mud, with minor coarser sandy horizons. Calcareous mud is predominantly grayish olive (10Y 5/2), and calcareous silty mud is grayish green (5GY 5/2). Coarser horizons are mostly greenish gray (GLEY1 5/10Y), but occasionally dark greenish gray (GLEY1 4/10Y). Contacts between lithologies are gradational and are marked by subtle color changes. Calcareous nanofossils are abundant and there are occasional shell fragments and pyrite nodules. Bioturbation is sparse to moderate, occasionally abundant, or absent. Trace fossils include *Chondrites*, *Planolites*, *Thalassinoides*, *Palaeophycos*, and *Zoophycos*, and rare *Asterosoma*, *Arenicolites*, *Spirophyton*, *Schaubcylindrichnus*, and *Phycosiphon*. Sometimes burrows are filled with silt and very fine sand. Due to the fine-grained nature of the sediments, primary structures are difficult to observe, but occasionally fine horizontal, subparallel, and cross-laminations are present in the calcareous silty mud sediments. Although it is not common in the coarser sandy intervals, occasionally the silt and sand deposits contain normal grading. Sediments in Unit I are initially interpreted as hemipelagic, very fine-grained contourites, and fine-grained turbidites.

Unit II extends from 699.1 to 735.6 mbsf (Sections U1610A-23X-1 to 26X-5). This unit is very homogenous but primarily consists of calcareous mud with some calcareous clay. Calcareous muds are dark greenish gray (GLEY1 4/10Y), and calcareous clays are dark grayish olive (10Y 4/2). Contacts between lithologies are gradual with subtle color changes. There are foraminifera and pyrite with occasional shell fragments disseminated throughout. Bioturbation is sparse and occasionally moderate. Trace fossils include *Chondrites*, *Planolites*, *Thalassinoides*, *Zoophycos*, and rare *Palaeophycos*. No primary structures are observed in Unit II. Sediments in Unit II are initially interpreted as dominantly hemipelagic deposits.

Unit III extends from 735.6 to 796.4 mbsf (Sections U1610A-26X-6 to 32X-CC). Unit III is composed of calcareous muds and calcareous clays, but the vertical variability between these two lithologies is more evident in comparison to Unit II. Calcareous muds are dark greenish gray (GLEY1 4/10Y), and calcareous clays are dark grayish olive (10Y 4/2). Contacts between lithologies are gradual. Shell fragments, pyrite, and foraminifera are disseminated throughout Unit III. Bioturbation is predominantly sparse, sometimes moderate, and occasionally abundant or absent. Trace fossils include *Chondrites*, *Planolites*, *Thalassinoides*, *Palaeophycos*, *Schaubcylindrichnus*,

*Zoophycos*, and rare *Arenicolites*. Although primary structures are difficult to observe in fine-grained sediments, black organic matter laminae were observed in one core. Unit III sediments are interpreted as dominantly hemipelagic deposits.

Unit IV extends from 798.0 to 837.5 mbsf (Cores U1610A-33X to 37R). Unit IV is more homogeneous with respect Unit III, but primarily consists of calcareous mud with minor calcareous clay. Calcareous muds and clays are homogeneous and predominantly dark greenish gray (GLE Y1 4/10Y) and occasionally dark grayish olive (10Y 4/2). Contacts between lithologies are gradual. Shell fragments and pyrite are disseminated throughout. Bioturbation is sparse to moderate, and occasionally abundant. Trace fossils include *Condrites*, *Planolites*, *Thalassinoides*, *Zoophycos*, and rare *Palaeophycos* and *Schaubcylindrichnus*. No primary structures were observed in these sediments. Unit IV sediments are initially interpreted as dominant hemipelagic deposits.

Unit V extends from 837.5 to 915.1 mbsf (Cores U1610A-38R to 45R). Unit V consists of calcareous muds and clayey calcareous ooze, with minor sandy silt deposits. Calcareous muds are predominantly dark greenish gray (GLE Y1 4/10Y), and clayey calcareous oozes are a slightly lighter greenish gray (GLE Y1 5/10Y); however, occasionally the color of sediments in this unit becomes gradually lighter or darker without changing lithology. Contacts between lithologies are gradual and usually correspond to a change in color, but sometimes the boundaries are sharp. There are shell fragments, pyrite, and foraminifera disseminated throughout the unit. Bioturbation is sparse to abundant, with the clayey calcareous ooze lithology exhibiting a noticeable increase in bioturbation compared to the calcareous mud intervals. Trace fossils include *Chondrites*, *Planolites*, *Thalassinoides*, *Zoophycos*, and *Palaeophycos*. Although primary structures are difficult to observe in fine-grained sediments, there are some laminations. Deposits from this unit are not horizontal, but are tilted with a variable low angle. Unit V sediments are interpreted as dominantly hemipelagic deposits.

### *Biostratigraphy*

Micropaleontologists sampled, processed, and observed 50 core catcher (CC) samples from Hole U1610A. All calcareous microfossils are typically abundant, with moderate to good preservation. Foraminifera and calcareous nannofossil assemblages in the first core, Core U1609A-2X, at 514.9 mbsf, indicate an age of around 3.50 Ma. The disappearance of *Globorotalia puncticulata* is recorded between Sections 3X-CC and 4X-CC, giving an age of about 3.57 Ma. Between Sections 10X-CC and 11X-CC, the highest occurrence (HO) of *Discoaster tamalis* is recorded, giving an age around 3.80 Ma. Between Sections 11X-CC and 12X-CC, the highest common occurrence of *Globorotalia margaritae* provides an age of 3.98 Ma. Two other events are recorded: the lowest occurrence (LO) of *G. puncticulata* between Sections 18X-CC and 19X-CC, and the HO of *Ceratolithus acutus* between Sections 23X-CC and 24X-CC (giving ages of

~4.52 and 5.04 Ma, respectively). Finally, the last datum recorded is the HO of *Orthorabdus rugosus*, at 5.23 Ma between Sections 30X-CC and 31X-CC. From there to the last sample of the week to be analyzed (from Section 49R-CC), no further clear age-diagnostic datums are observed.

Thirty-two core catcher samples were analyzed for benthic foraminifera assemblages. The variation of assemblages with lithology was notable: benthic foraminifers, especially the uvigerinids, are more abundant in the calcareous mud intervals (that are richer in organic matter). An influx of shallow water assemblages and the presence of many detrital fragments is noted in Section 39R-CC and may be attributed to a gravity flow. Pyritized microfossils, pyrite, and sand grains are common in most CC samples. Other than foraminifers, the presence of ostracods, echinoderm plates, spines, and fish teeth were also noted in the samples. The fish teeth were collected for postexpedition neodymium isotope analysis. Five to 10 specimens of *Planulina wuellerstorfi* and/or *Cibicidoides pachyderma* species were also picked from each sample for postexpedition oxygen isotope analyses.

### *Paleomagnetism*

We collected and measured 164 discrete oriented cube samples from the working half core sections of Hole U1610A, three to five from each core. First, we measured the anisotropy of magnetic susceptibility (AMS) and bulk magnetic susceptibility (MS) using the MFK2 KappaBridge instrument. Results show an overall vertical direction of the  $k_{\min}$  axis, in agreement with a sedimentary fabric. After the AMS measurements, the natural remanent magnetization (NRM) of 85 cube samples was measured on the JR-6A AGICO spinner magnetometer, then the samples were alternating field (AF) demagnetized. Initially, steps of 5, 10, 15, and 20 mT were applied. Later, steps of 30, 40, 50, and 60 mT, and in some cases up to 100 mT were added to fully demagnetize the characteristic magnetic components. The NRM is significantly stronger than in Site U1609, with an average of  $10 \times 10^{-4}$  A/m. Detailed analyses showed that a low coercivity overprint component was generally removed below 10 mT, after which a second component remained that mostly had reversed directions. This component could not be properly demagnetized in the applied magnetic fields, and in some cases the samples acquired a gyroremanent magnetization (probably indicating the presence of greigite) that obscures the primary signal. The inclination directions from the discrete samples were mostly reversed between 505 and 800 mbsf, although five intervals were observed with normal polarity inclinations.

In addition, we measured the NRM of all the archive half-core sections from Hole U1610A on the superconducting rock magnetometer (SRM). An AF demagnetization was performed at 5, 10, 15, and 20 mT with measurement of the remaining NRM being taken at 2 cm resolution after each step. NRM intensity in Hole U1610A ranges from

$1.5 \times 10^{-4}$  to  $5 \times 10^{-3}$  A/m, and NRM remaining below 10 mT is likely related to overprint caused by core drilling. To suppress the influence of scattered directions, we ran a smoothing window of 1 m for the inclination values. Based on the smoothed inclinations, normal polarity intervals were still hard to recognize, probably because of the presence of a pervasive reversed signal or overprint.

Finally, we compared the inclination results of the discrete samples with the SRM data. This confirms that normal directions are highly suppressed by a dominantly reversed component, and at this moment we refrain from presenting a detailed magnetostratigraphy for this hole. More advanced measurements on how to best separate the different magnetic components are required to provide more robust age constraints for this hole.

### *Geochemistry*

At Hole U1610A the safety protocol for drilling between 740 to 960 mbsf required the methane concentration in headspace gases and methane/ethane ratios to be reported to the drilling team prior to advancing to drill the next core. Headspace gas is routinely measured for safety monitoring by the organic geochemistry team from a discrete sample taken at the top of a core section and promptly processed and measured. In all headspace samples, the methane concentrations remain <13,000 ppmv (well below threshold), ethane concentrations are <12 ppmv, and the minimum ratio of these two gases ( $C_1/C_2$ ) is 406. These low concentrations of methane and ethane and the dominance of methane (low  $C_1/C_2$  ratio) present no concerns for drilling safely as they indicate a microbial rather than petrogenic source. Methane and ethane were commonly detected, while ethene, propane, isobutane, and isopropane were detected in trace amounts.

Void space was also sampled directly when gas pockets appeared in the core, yielding concentration and composition results within 16 min, which is helpful contextual information as these data are available sooner than the 46 min required for standard headspace gas protocols. In void spaces, the absolute concentrations are not meaningful as they are essentially methane pockets (measured methane is close to 100%), but we found similar trace constituent composition to that measured with the standard interstitial headspace gas protocol. In void spaces, the  $C_1/C_2$  ratio minimum is 1930, indicative of microbial rather than petrogenic sourcing. For safety monitoring we rely upon the headspace gas standard protocol data, and drilling continued as soon as the headspace data were available and reported.

The inorganic geochemistry team sampled 42 interstitial water (IW) samples and two bottom water samples from Hole U1610A and measured salinity, pH, and alkalinity. One IW sample was collected from every XCB core (Cores 2X to 34X) and RCB core (Cores

35R to 49R), except where recovery was short (<3 m core) or poor quality (abundant fractures), in which case the IW sample was not taken. Approximately 10 mL of IW was extracted initially, which decreased to ~6 mL of water during RCB coring. Alkalinity ranges from 1.4 to 4.8 mM, pH varies between 7.5 and 8.0, and salinity is 32 to 35 within IW samples. The bottom water samples each have an elevated salinity of 36, indicating we were sampling the Mediterranean Overflow Water.

Sediment samples were obtained from the squeeze cake residues from the IW sampling at Hole U1610A. Additionally, several samples were selected to understand lithological variations described by the sedimentology group. Sediment samples were dried, ground, and weighed to measure various chemical properties. Calcium carbonate content ( $\text{CaCO}_3$  wt%) is relatively invariant at this site compared to the prior site, with a mean of 33.9 wt% and standard deviation of 2.9 wt% on 40 samples of Pliocene age. This batch included samples selected for color variations, implying that color variations are driven by changes in components other than carbonates, likely the siliciclastic fraction. We also explored relationships between carbonates and physical properties data. We found that the relationships differed from those at Hole U1609A, where carbonate dominated the variance in natural gamma radiation (NGR). Here, we found that the minimal variations in carbonate concentration did not drive NGR variance, again implying that other components, presumably siliciclastic debris, are important at this site in the Gulf of Cádiz.

### *Physical Properties and Downhole Measurements*

Physical properties measurements in Hole U1610A generally show short-scale (2–4 m) repetitive variation on a slowly increasing compaction trend with depth. The trends are partly obscured by the effects of XCB biscuiting and undergauge RCB cores.

The interval cored this week can be divided into three units: 505.2–700 mbsf, 700–800 mbsf, and 800–827.8 mbsf. The first unit (505.2–700 mbsf) shows subtle long-term trends in NGR and MS in addition to clear ~2 m scale cycles. The second unit (700–800 mbsf) generally has slightly higher NGR, MS, and gamma ray attenuation (GRA) bulk density than the first unit, but with a lower amplitude and frequency of cycles. The third unit (800–827.8 mbsf) has generally higher NGR, MS, and GRA density values, and longer wavelength cycles than the overlying units.

The SET2 probe results gave a temperature of 24.8°C at 505.2 mbsf. Using this result and assuming a seafloor temperature of 12°C, the geothermal gradient at the site is 25.3°C/km.

## Outreach

The new year kicked off with seven ship-to-shore tours, reaching hundreds of students ranging in age from 11 years old to graduate students in university. Tours were conducted for organizations in the United States, China, and Japan. One piece of feedback received from a high school teacher said, “The kids really enjoyed it! ... While my new department members were dubious at first about the relevance, they really see the importance of this type of interaction now!”

Videos published on social media performed well this week, reaching more than 50,000 views. Outreach Officers collaborated with multiple institutions to amplify social media content, including Louisiana State University and Princeton University. The best performing posts on each platform were the Science in 60 Seconds [video featuring Wout Krijgsman](#) on X, the video featuring [how tiny cups are created](#) by water pressure on Instagram and Threads, and the [New Year’s traditions video](#) on Facebook.

## Technical Support and HSE Activities

### *Laboratory Activities*

- Staff processed cores and samples from Hole U1610A. Total expedition core recovery is 1485.83 m so far.
- The EPO-TEK<sup>®</sup> epoxy, used for making thin sections, has passed its expiration date. Other epoxies on the ship were tested but were found to be unreliable. New epoxy needs to be purchased for Expeditions 402 and 403.
- We serviced two pneumatic staplers, used for the core boxes, as one was leaking and the other one did not close the staple properly.
- Section Half Imaging Logger (SHIL): Some of the cropped images were not saved. The problem was that the “crop image” button could be clicked twice before the “save” occurred. The second click reset the crop, resulting in the noncropped image being “saved.” The image file was then rejected by the software, as it includes all borders and the label, and placed in the “error” folder. This problem went back to the beginning of the expedition and has now been fixed by the programmers.
- Paleomagnetism: Chill water remains relatively “clean” because of the combination of a bag filter installed by a ship engineer and the sediment trap canister installed by JRSO staff. The Haskris chiller continues to keep temperature at 69°F consistently.
- The NGR has been producing values that are too high at the top of core sections and too low at the bottom. One explanation for this observation is that the core

top extends too far past the center of the starboard detector in the instrument. Introduction of a 1 cm spacer at the top of the NGR tray has improved the NGR output so that there is now better continuity in NGR values between sections. We will investigate further after coring concludes at Site U1610.

#### *IT Activities*

- Troubleshooting the vibration isolated television (VIT) survey channel encoder after it failed when the VIT was brought back to the ship following the release of the reentry system. We are looking for a suitable replacement for the encoder.

#### *Developer Activities*

- Deployed an update to LIMS-to-Excel (L2E) with a new function that downloads images with top and bottom depths embedded in the filename to make them easier to import into TechLog software.
- Worked with a staff scientist onshore to make changes to the LORE Sample Report to address issues with offset in section vs. depth in sample values.
- Worked on the iRIS project to fix the broken wires that turn on/off the tracer pump.

#### *HSE Activities*

- Emergency shower and eye wash stations were tested.