UT-GOM2-2 Prospectus: Science and Sample Distribution Plan- version 2.3

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1. Executive Summary

The University of Texas (UT), Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope Project (GOM2), will perform the UT-GOM2-2 drilling and coring expedition in the Terrebonne Basin, Gulf of Mexico outer continental shelf. This is the scientific plan for the acquisition, storage, analysis, and distribution of core and other collected samples for UT-GOM2-2.

The UT-GOM2-2 expedition will sample and analyze the physical, chemical, and biological properties of the hydrate-bearing layers, thereby illuminating the origin, dynamic behavior, and response of this system to perturbation (whether by climate or energy production). A systematic measurement program will sample the hydrate-bearing layer from the seafloor downward. Novel technology, developed during the project, will be used to extract cores from a mile beneath the ocean and study those cores in laboratories worldwide, while keeping the samples under their original pressure. Samples taken near the seafloor will constrain the flux of carbon from the sediment into the ocean and determine whether there have been recent temperature perturbations at the ocean floor. Through sampling of the microbes, and analysis of the surrounding pore fluid, we will illuminate the depths and rates at which microbes are generating methane beneath the seafloor. Our analyses will inform biological, geochemical, and geomechanical models to constrain the role of gas hydrates in the carbon cycle and the potential for gas hydrates as an energy resource.

We will drill and core up to two vertical wells, one up to 9470 ft below sea level in the offshore Gulf of Mexico in Terrebonne Basin, Walker Ridge Block 313. We will spend up to 30 days at sea mobilizing, executing, and demobilizing. We will spend an additional days performing ‘dockside’ operations at Geotek Coring, Salt Lake City, UT (GCI and SLC) to complete the primary analysis of the recovered core. Finally, samples will be shipped to various institutions for secondary analysis.

We will characterize the Orange sand and a portion of the Upper Blue sand hydrate reservoirs and their bounding units, characterize dissolved methane concentration and gas molecular composition with depth, measure the in-situ temperature and pressure profile, and determine the high resolution geochemical, geobiological, and sedimentary profiles in the shallow muds below the sea floor (fbsf).

Pressure core and conventional core will be collected in up to two holes adjacent to one previously drilled well. We will use the DOE Pressure Coring Tool with Ball Valve (PCTB) to obtain pressure core. In the first hole, conventional core will be collected using Geotek’s APC or XCB coring tool and pressure

core will be collected using the PCTB in the cutting shoe configuration (PCTB-CS). In the first hole, temperature and pressure measurements will also be made. In the second hole, only pressure core will be acquired using the PCTB in the face bit configuration (PCTB-FB).

We will use Geotek's Pressure Core Analysis and Transfer System (PCATS) to log and X-ray the pressure cores. PCATS will also be used to subsample the recovered pressure cores at hydrate-stable conditions, and to transfer samples to pressurized storage chambers. On-board and dockside, subsamples will undergo quantitative degassing to determine dissolved methane concentration and hydrate saturation. Pressure core in storage chambers will be transported to UT and stored at the UT Pressure Core Center (PCC) and other institutions.

Conventional cores will be processed on board and dockside. On-board, conventional core will be run through a Geotek IR Scanner to create a thermal image to identify where hydrate dissociation had just occurred. Sections of conventional and depressurized core will be cut for microbiology and pore water analysis. Sections for pore water analysis will be squeezed and ephemeral pore water measurements will be completed on board. Preserved pore water samples will be shipped to the University of Washington (UW) for analysis. Microbiology samples will be sent to Oregon State University for analysis. Void and headspace gas samples will be collected, and strength measurements made on-board.

At SLC, conventional and prematurely depressurized core will be run through CT 3D scanning, Geotek MSCL whole core logging, core splitting, split core scanning, primary litho- and biostratigraphy. Moisture and density (MAD) whole round samples will be cut. Additional whole core samples will also be cut for geomechanical testing. Thermal conductivity, fall cone, and vane shear measurements will be made. Cores will be split, scanned, and described. MAD and geomechanical whole round samples will be sent to Tufts University for analysis. Subsamples of split core will be shipped to the University of New Hampshire (UNH), USGS, UT, and others for secondary litho- and biostratigraphy. Split core archival and working halves will be placed in long term cold storage at UT.

The Preliminary Expedition Report will be issued 2 months post-expedition. The Expedition Report will be published 1-year post-expedition. We are eager to support hydrate science by the broader community and requests for data and/or samples can be made to UT.

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2. Motivation

2.1. Gas Hydrates in the Global Carbon Cycle

About 10,000 billion tons of organic carbon (in land plants, peat, soil, organic matter dissolved in the ocean, and fossil fuels) constantly cycle through the solid Earth, the ocean, and the atmosphere. The atmosphere currently contains only ~800 billion tons of carbon, and carbon cycle changes significantly affect greenhouse gases and global climate. 5-22% of this global organic carbon is trapped in gas hydrate, an ice-like substance composed of methane and water. Most of this massive carbon reservoir lies in continental margin sediments within a layer that extends downward from the seafloor and can reach thicknesses of ~1,000 m (3,280 ft.). This layer interacts with the Earth's Ocean and, occasionally, the atmosphere. This dynamic carbon reservoir is a potential energy resource, a potential source of geohazards, and a potential driver for climate change. For all these reasons, we need to have a better process understanding of how these hydrate reservoirs form and how these reservoirs respond to perturbation.

2.2. Gas Hydrates and Energy

The natural gas stored in gas hydrates makes hydrate reservoirs one of the most abundant unconventional energy resources on Earth. For coastal nations with limited energy resources, this is a potential domestic energy source to provide energy security today. Japan, South Korea, India, and China have active programs trying to understand this resource. The global energy system is undergoing a major transition toward sources with low or no greenhouse gas emissions. In the U.S., the role of natural gas in replacing coal-based energy generation and reducing CO2 emissions is well documented. Methane hydrate is found in existing hydrocarbon production areas (deepwater Gulf of Mexico and Alaska). Methane is the cleanest hydrocarbon fuel and methane from hydrates may be an important future U.S. energy source.

2.3. Hydrates and Climate

Methane is a greenhouse gas 84 times more potent than carbon dioxide (CO_2) over a 20-year timeframe. Its atmospheric concentration is hundreds of times less than that of CO_2, yet methane drives ~25% of the radiative forcing of anthropogenic CO_2. Meanwhile, atmospheric methane concentrations have increased three times as rapidly as those of CO_2 since the preindustrial age. This has spurred research on methane in the carbon cycle, and the role of the hydrate system in this cycle is a pressing frontier research problem.

Methane in oceanic hydrates is largely generated by microbes. Archaea (single celled microorganisms) consume organic matter in sediments buried beneath the ocean floor and generate methane that is frozen as hydrate. The system is complex and dynamic: methane gas flows, hydrates form, and hydrates dissociate in response to pressure and temperature perturbations. In many locations above concentrated methane hydrate deposits, methane vents into the overlying ocean where it is either oxidized, resulting in potential for ocean acidification, or occasionally (typically in quite shallow water) reaches the atmosphere where it could contribute directly to global warming. Large-scale hydrate dissociation events and the consequent methane emissions have been proposed to cause large climate perturbation in the geologic past. Methane flowing upward is also oxidized within near-seafloor sediments, leading to a flux of dissolved carbon into the ocean. The workings of this sedimentary carbon recycling factory and the role played by gas hydrates are not yet completely understood.

2.4. Hydrate and CO_2 Sequestration

In deep concentrated hydrate reservoirs (at depths greater than those affected by changes in deep ocean temperatures), technologies are being developed to store CO_2 while producing methane. This approach is nearly carbon neutral: similar amounts of carbon are stored by CO_2 injection as are produced as natural gas. In so doing, CO_2 is stored as an immobile, solid, CO_2-hydrate, the reservoir's geomechanical stability is maintained (reducing environmental impact), and temperatures are maintained allowing gas production.

3. Scientific Objectives

Five key scientific objectives are detailed below. The combined impact of these objectives is to obtain a systems understanding of gas hydrate formation and dissociation in coarse-grained sediments at WR 313. The objectives inform two models or scales of investigation: the reservoir-scale (Figure 3-1) and the basin-scale (Figure 3-2).

We will inform reservoir-scale production models by obtaining and maintaining sediment at in situ conditions (pressure core), determining hydrate concentration, gas composition, age, sediment texture, pore water chemistry, permeability, compression, capillary behavior, and strength for several different hydrate reservoirs and their bounding units at WR 313. Characterizing the reservoir and seal properties will lead to better prediction of reservoir perturbation behavior and help us test wither hydrates formed from long-range or short-range transport.

We will inform basin-scale models by collecting sediment (some at in situ conditions), gas, and pore water samples, in situ pressure and temperature with depth; determining gas and hydrate concentration, gas composition, age, sediment texture, pore water chemistry, sedimentology, variations in organic carbon, permeability, compression, capillary behavior, and strength for the basin system at WR 313. Characterization of these properties with depth will help us understand the origin and evolution of the hydrate system in response to organic matter deposition, microbial methane formation, and fluid migration. This will inform how hydrate is generated and what role the hydrate system plays in the carbon cycle.

Each one of the following objectives test these reservoir scale and basin scale models in some way.

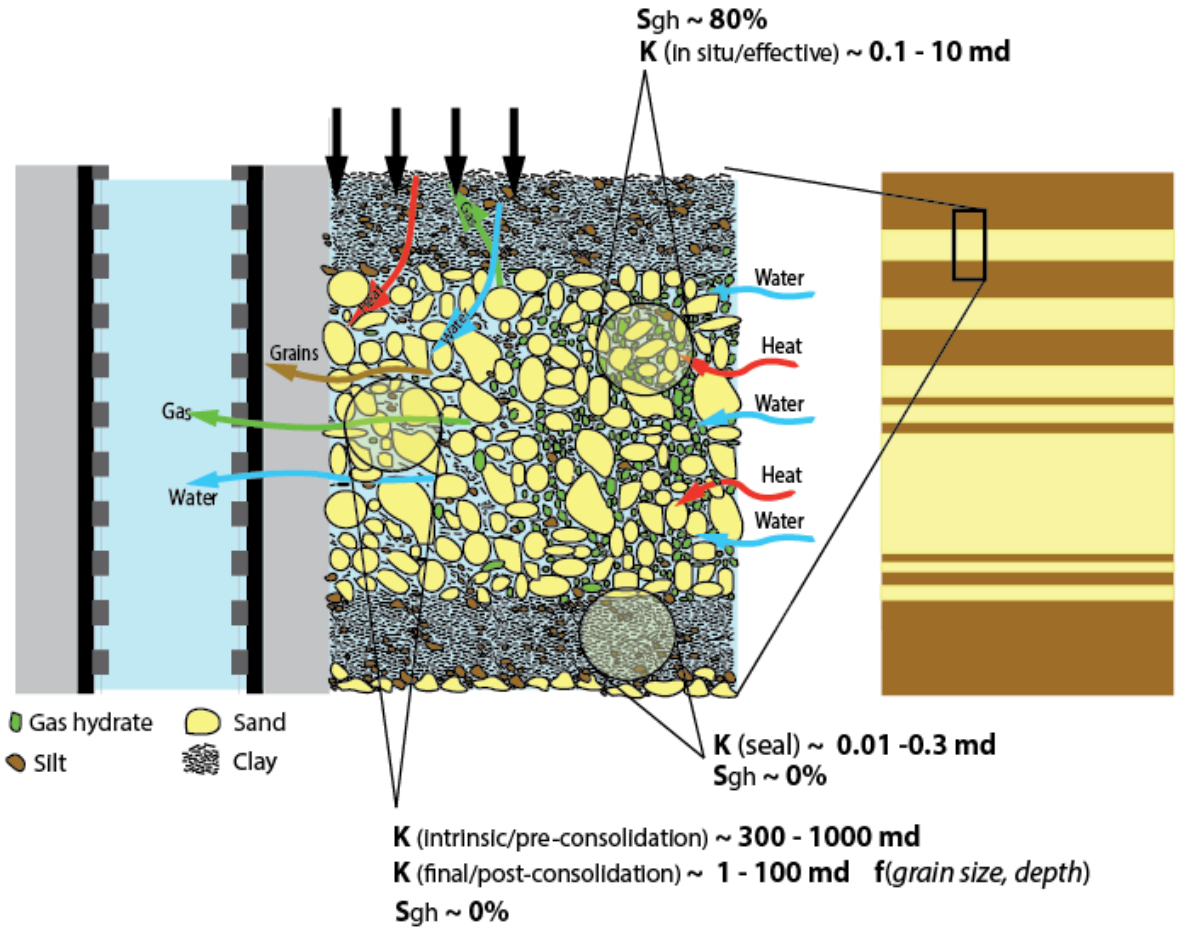

Figure 3-1. Conceptual view of hydrate response to perturbation. In this example, pore pressure is reduced in the wellbore. As a result, hydrate trapped within the porous medium dissociates into water and methane and both travel towards the well bore. Heat (red arrows) is drawn into the reservoir due to endothermic cooling when the hydrate dissociates. The reservoir is capped and underlain by fine grained sediments. Observations and experiments will determine the concentration, and petrophysical properties of this hydrate system and inform models of hydrate perturbation. Modified from Bowell and Collet 2016

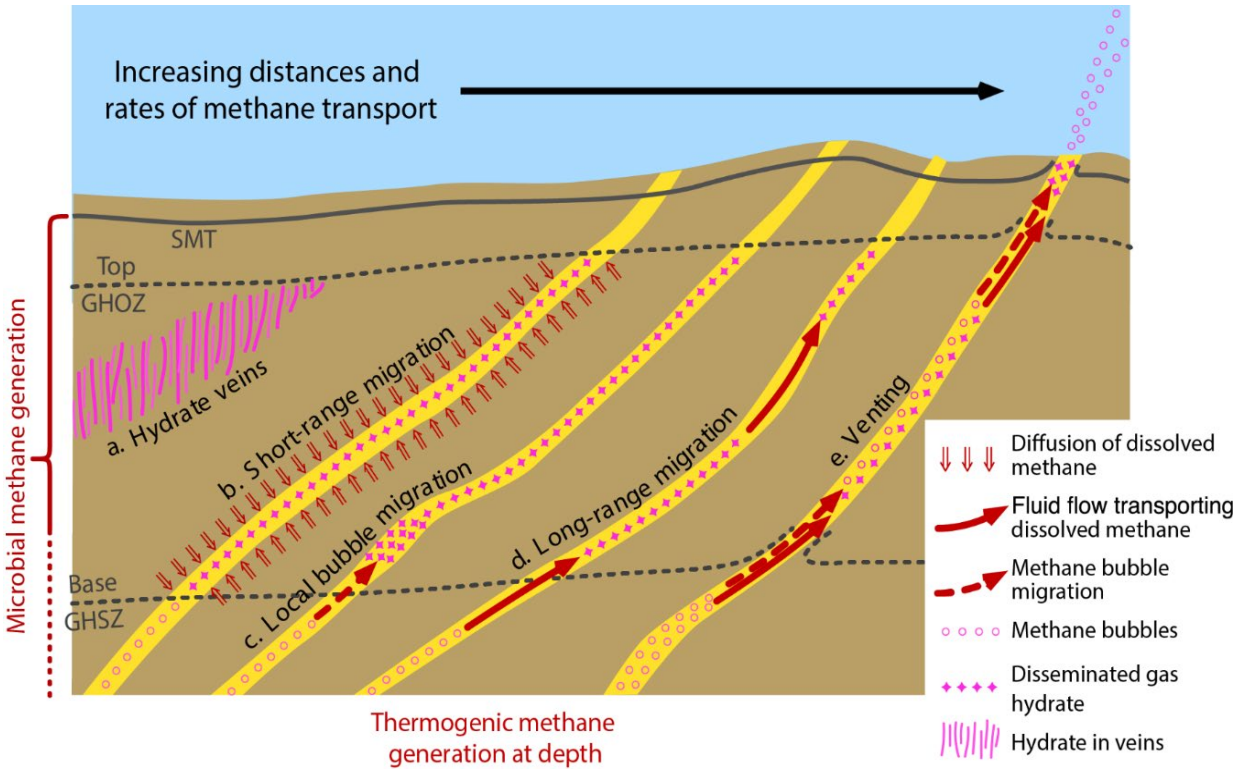

Figure 3-2. Methane migration mechanisms and gas hydrate formation in fine-grained marine sediments (darker shade) containing coarse-grained layers (lighter shade). Typically, gas hydrates occur in a gas hydrate occurrence zone (GHOZ) whose top is below the seafloor and is controlled by the depth where methane concentration in pore water reaches solubility. The settings marked A through E illustrate processes that take place as the overall rate of methane transport increases. In intervals dominated by fine-grained sediments, gas hydrate sourced from in situ microbial methane forms in veins and fractures (A). In coarse-grained layers, gas hydrate is found disseminated in the pore space. Hydrate can form from microbial methane that diffuses from adjacent fine-grained sediments as in short-range migration (B). When sedimentation buries layers below the GHSZ, methane in hydrate will turn into gas bubbles, which can migrate upward to enrich hydrate deposits just above the GHSZ (C). Fluid flow can transport dissolved methane that originates below the GHSZ as in long-range migration (D). Where large amounts of gas are present below the GHSZ and/or fluid flow is intense, free gas can migrate through the GHSZ forming a vent (E). After Malinverno and Goldberg 2015

3.1. Objective 1: Characterize the Orange sand and Upper Blue sand hydrate reservoirs and their bounding units

Characterization will include the following reservoir and bounding mud properties: 1) hydrate concentration, dissolved methane concentration, and gas composition, 2) pore water solute concentration and composition, 3) lithofacies identification, grain size, and sorting, 4) permeability, 5) compressibility, 6) strength behavior, 7) sediment composition and age, 8) microbial communities and activity, 8) and physical properties such as mineral and clay composition, porosity, and liquid limit. Characterizing these properties will allow us to better understand transport processes in and

around the reservoir and seal, providing insight in terms of gas migration and hydrate formation, and hydrate production behaviors.

3.1.1. Objective 1 Rationale

The Orange sand is the thickest and cleanest (consistently low gamma-ray) reservoir penetrated in the Terrebonne basin based on logging-while-drilling (LWD) logs (Figure 3-3). The Orange sand may represent a levee deposit on the flank of a submarine channel or it represents a regional sheet sand that was subsequently incised by the channel (Figure 3-3 A). The GC-955 reservoir is a levee deposit adjacent to a submarine channels (Figure 3-3 A). We interpret that the turbidite flows that formed the Orange sand were less mud prone, likely higher-energy, that they are coarser grain-size and that they have greater bed-thickness than the sandy silt levee deposits cored in GC 955 area.

The structure of the hydrate reservoir is different from that at GC-955 (Figure 3-3 B). At GC-955, the levee channel system is highly faulted and draped across the crest of an anticline. Its hydrate accumulation is controlled by 4-way closure over the anticline (Figure 3-3 B). At Terrebonne, the Orange sand does not have significant faulting and crosses the hydrate stability boundary (Figure 3-3 B). A distinct hydrate zone, gas-leg, and underlying water bearing zone in the Orange sand are identifiable in seismic reflection data in both cross sections and maps.

The effective stress at the Orange sand is roughly twice that of the GC-955 reservoir. Thus, both the seal and perhaps the reservoir will be more compacted than at the GC-955 location. While the effective stresses are different, the in-situ temperatures at the reservoir are likely similar based on estimates of the geothermal gradient (Figure 3-3 B).

Characterization of pressure cores from the Orange sand hydrate-bearing reservoir will provide critical information for understanding methane sourcing, hydrate formation, methane migration mechanisms, provide critical inputs for reservoir and basin models for predicting hydrate stability and in situ production. Methane and other light hydrocarbon concentrations paired with carbon and hydrogen isotopic ratios can provide insight into whether the methane is thermogenic or microbial in origin. In addition, chemical concentration gradients of the pore water within the confining sediments above and below the reservoir can provide both an indication that there is recent flow into or out of the permeable reservoirs and can be used to estimate the composition of the fluid in the reservoir. These can provide information about the rate and direction of solute diffusion, which in turn provide insights on fluid flow.

Specifically, characterizing pressure cores from within the Orange sand and its bounding units will characterize a marine hydrate reservoir that is distinct from the only other pressure-cored reservoir in the Gulf of Mexico (GC-955) (Figure 3-3 A, B, C). The Pressure Core Analysis and Transfer System (PCATS) will provide velocity and density logging, and X-ray scanning, which will allow for characterization of bed thickness, sedimentary structures, qualitative estimates of hydrate concentration, and lithofacies distribution. Lithofacies-specific cuts for quantitative degassing, permeability analysis, and grain size analysis will allow us to better understand the lithologic control on hydrate saturation and fluid flow.

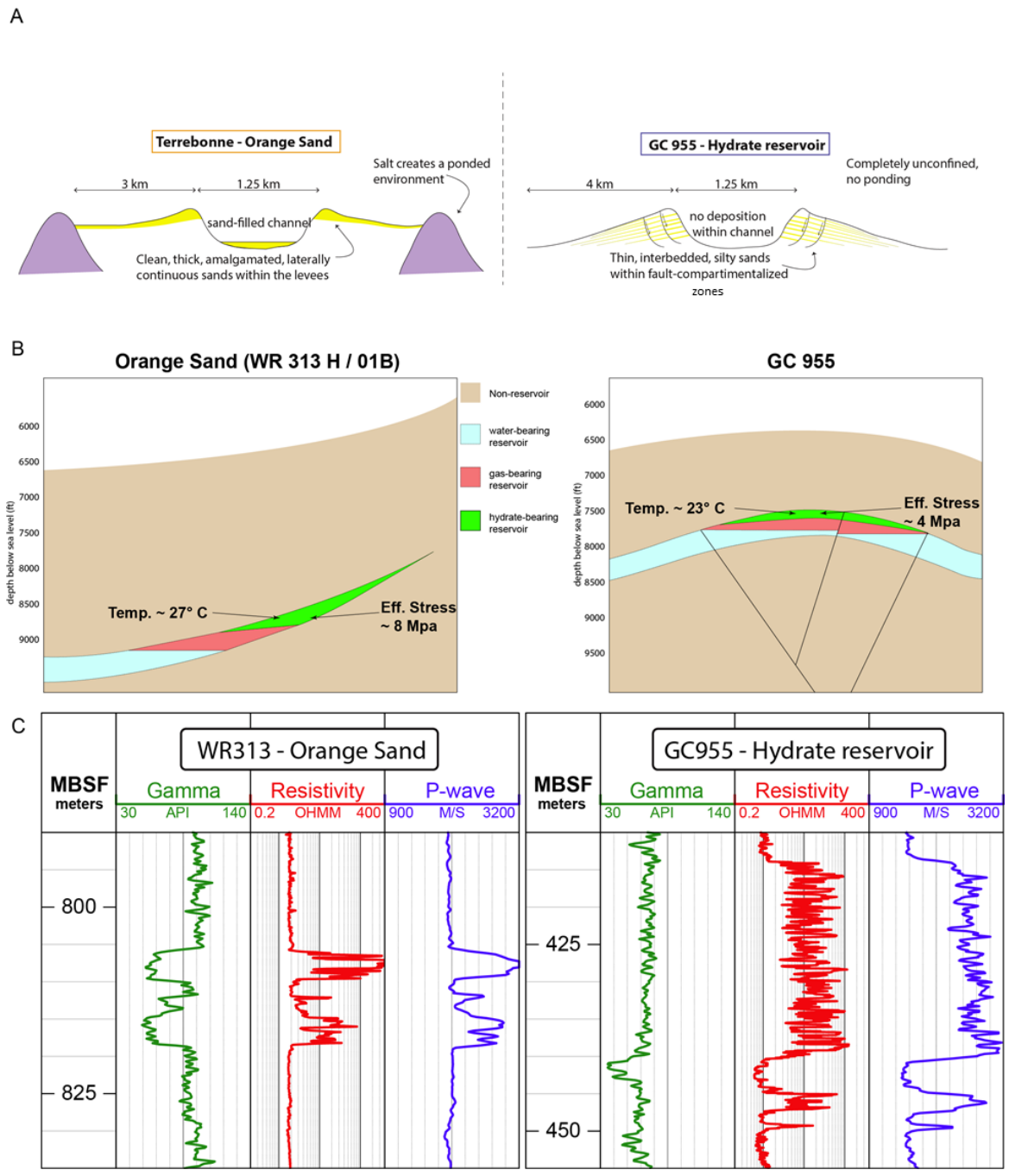

Figure 3-3. Comparison of WR 313 Orange sand and GC 955. (A) Schematic depositional environment for the primary drilling objective – Orange sand (left); sandy levee deposits are laterally extensive, steeply dipping (~15°), and pinch-out in the up-dip direction; schematic depositional environment for interbedded sandy-silt and muddy-silt reservoir in GC 955 (right) – levee associated with non-depositional channel ; (B) simplified cross-sections showing Orange sand in Block WR 313 (left), and tested hydrate reservoir in Block GC 955 (right); estimated temperature and effective stress are shown in each location; (C) Comparison of log signature between Orange sand in WR313-H001 (left), and tested hydrate reservoir in GC955-H001 (right). Logs from the Gulf of Mexico Gas Hydrates Joint Industry Project Expedition II (JIP II).

3.1.2. Plan to meet Objective 1

We will meet Objective 1 by pressure coring through the Orange sand, Orange sand bounding mud, and a portion of the Blue sand in the in the first hole, H003. Pressure core analysis will be done on-board and dockside. Conventional core analysis will be done on depressurized pressure cores. We will characterize the 1) hydrate concentration, dissolved methane concentration, and produced gas composition, 2) pore water dissolved solute concentration and composition, 3) lithofacies identification, grain size, and sorting, 3) permeability, 4) compressibility, 5) strength behavior, 6) sediment composition and age, 7) microbial communities and activity. We will illuminate the diffusion rate and direction of methane and other solutes diffusion by taking background cores 16.4, 49.2, and 148 ft (5, 15 and 45 m) above and 49.2 ft (15 m) below the orange sand.

3.2. Objective 2: High resolution geochemical and sedimentary profiles: understanding the Hydrate System.

3.2.1. Objective 2 Rationale

High resolution lithostratigraphic, chronostratigraphic, geochemical, geomechanical, and microbial profiles are essential for understanding fluid sources, microbial methane generation, and the geological evolution of the hydrate system in the Terrebonne Basin. These data will contribute to developing a model for the origin and evolution of the hydrate system. This model will describe the biogeochemical cycling and will constrain the role of the methane hydrate in the carbon cycle.

A high-resolution profile is especially important in sedimentary sequences containing frequent lithologic transitions (as seen in GC 955 and in the Terrebonne basin). A continuous coring approach has been successful in scientific drilling programs to reveal key insights into the geologic evolution and biogeochemical cycling in a range of continental margin environments. Figure 3-4shows examples of the geochemical, lithostratigraphic, and biostratigraphic data discussed above from International Ocean Discovery Program (IODP) 353, which encountered methane hydrates in thinly-bedded turbidites in cores from a borehole (IODP U1445) on the Indian margin in the Bay of Bengal.

To understand the source and origin of methane in hydrate-bearing sediments of the Gulf of Mexico it is essential to determine the nature of the microbial communities that exist in the sediments. We will test three hypotheses for the origin of the methane in the hydrates: 1) methane is produced by microbes buried in the sediments proximal to the hydrates, 2) methane is transported or migrated within fluids that are coming from much deeper in the section, or 3) methane is present by some combination of these two mechanisms.

We will look at rates of silicate weathering, an important buffer on pore water pH (Solomon et al., 2014; Wallmann et al., 2008). Weathering reactions are not only important for pH, but may also be required to keep methanogenesis as a thermodynamically feasible catabolic pathway (i.e. by scrubbing out metabolic products (Solomon et al., 2014)). To better understand this reaction network requires continuous cores for pore water inorganic and organic chemistry, as well as sediment TOC, total inorganic carbon (TIC), carbonate composition, etc.

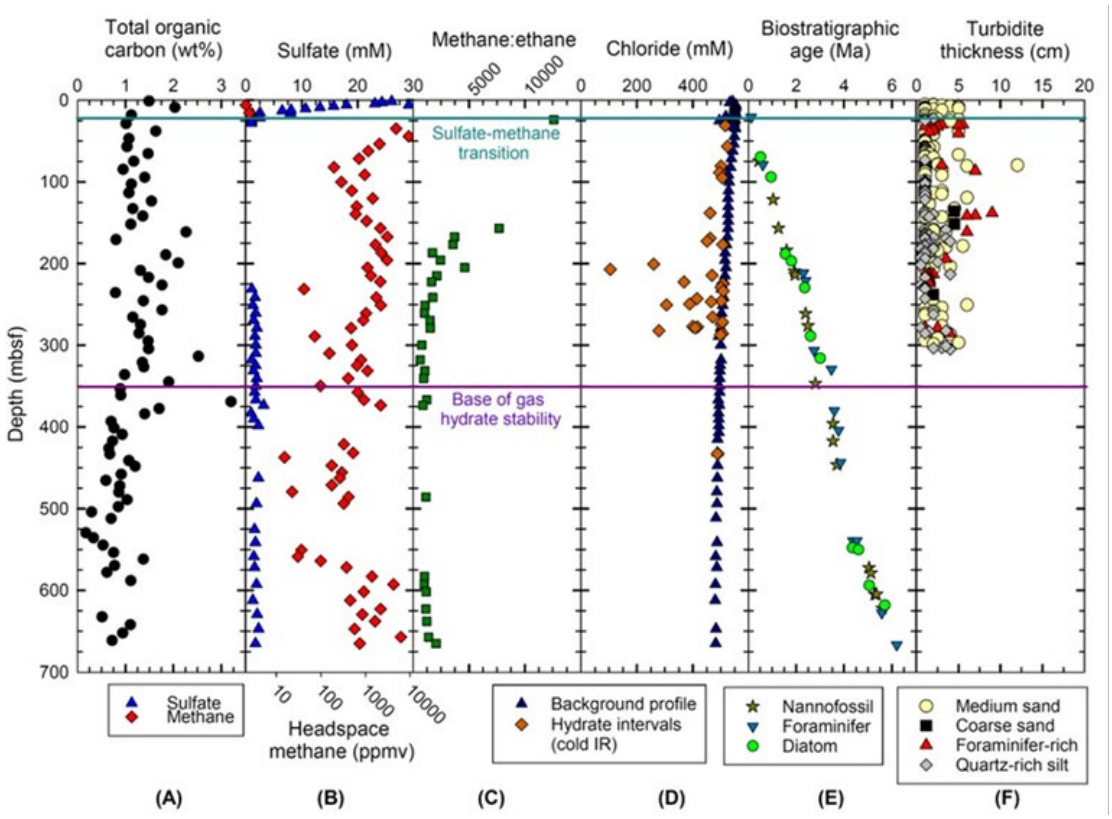

Figure 3-4. Example geochemical data from IODP Site U1445 in the Mahanadi Basin, northern Bay of Bengal that encountered a hydrate system in thinly bedded turbidites. This example shows the type of higher-resolution geochemical, lithostratigraphic, and age data that can be acquired with continuous coring. A) Total organic content is relatively high and decreases with depth. B) The sulfate methane transition (SMT) is noted as the depth of sulfate disappearance and onset of methane production. C) The methane/ethane ratio (C1/C2) decreases with depth D) Hydrate-bearing layers (identified as cold spots with an infrared camera). E) Biostratigraphic age from nannofossils, foraminifers and diatoms. F) Turbidite thickness classified by four main type of occurrences. All data are from (Clemens et al., 2016).

3.2.2. Plan to Meet Objective 2

A sedimentary profile with high resolution pore water, sedimentology, physical properties, microbiological, and mechanical properties sampling will be acquired at hole WR313 H003. We will continuously core to at least 500 fbsf, spot conventional and pressure core to the Red sand, and pressure core to total depth.

We will derive the following data:

a. Measure organic matter content and source indicators (total organic carbon, bulk organic $\delta^{13}C$, C/N ratios) with depth to constrain the amount of organic carbon available for microbial fermentation and methanogenesis, and determine if this organic carbon can drive sufficient in situ microbial methane production to form high saturation hydrate in the Orange sand and Upper Blue sand. We will measure the conversion of organic carbon to inorganic carbon and burial.

b. Observe abrupt transitions and general behavior of the pore water composition to infer fluid flow, hydrate formation/dissociation, and diagenesis. To observe a comprehensive record of fluid sources, the sulfate-methane transition (SMT), and minor hydrate occurrences, continuous conventional coring is required to as great a depth as possible. Pore-water and microbiological samples selected at a high resolution (every 3 m average, 0.5 m in key transitions) will reveal abrupt transitions in microbial processes and methane production. A high-resolution profile will reveal potential anomalous fluid flow intervals within the basin that can transport deeper methane or substrates for methanogenesis (thermogenic components, CO_2). For example, a local increase in C2+ hydrocarbons in headspace gas or anomalies in pore water boron or "enriched $\delta^{18}O$" or Li isotopic composition may indicate advection of deeper fluids. Infrared scanning of conventional cores and measurement of pore water Cl and $\delta^{18}O$ will be used to characterize the distribution of hydrate in fractures and thin sands. Authigenic minerals such as carbonates and iron sulfides that record past pore water conditions and microbial processes (methanogenesis, anaerobic oxidation of methane) will be sampled and analyzed from the continuous core.

c. Determine the age of the strata through nannofossil biostratigraphy in both holes. In the continuous cored section, in the upper 250 fbsf, we will use benthic foraminfer $\delta^{18}O$ chemostratigraphy to create an age model on glacial-interglacial timescales. Through this, we will characterize variation in sedimentation rates and organic carbon accumulation rates. Sedimentation rate influences the hydrate system because it impacts the deposition and preservation of organic carbon, methane oxidation, and the burial of pore waters.

d. Characterize the continuous record of lithologic properties including the reservoir seals. The bounding seal is a key component of the hydrate petroleum system that allows for gas hydrate accumulation. The effectiveness of the seal is affected by the permeability and presence of fractures, which are influenced by its composition. A profile of continuous core will provide an opportunity to characterize the physical and geomechanical properties of the seal, including grain size, sediment composition, and permeability. This allows for an integrated approach to understand how depositional processes influence the seal component of the hydrate petroleum system. Cores from the Krishna-Godavari Basin, offshore India indicate that the presence of diatoms in hemipelagic clays increase the porosity/permeability of seal material overlying the hydrate system (Jang et al., 2019).

e. Determine presence, numbers, and activities of key microbial communities responsible for methane generation and link these observations to pore-water, lithologic, and formation properties. Sediment samples will be analyzed for microbial community characteristics (including functional genes that methanogenic microbes use to make methane), methanogen biomass or cell numbers, and methanogen activity. These data will be used in combination with other data collected on the expedition to refine reactive transport models that estimate rates of methane production at different depths in the reservoir.

3.3. Objective 3: Measure the in-situ temperature and pressure profile

3.3.1. Objective 3 Rationale

We commonly predict the temperature at the bottom-simulating reflector (BSR) by assuming that this reflector records the phase boundary where vapor, water, and hydrate coexist: hydrate and water overlie this boundary and methane vapor and water underlie it. For this calculation,

we assume that pore pressure is hydrostatic, pore water is of seawater salinity, and the hydrocarbon is composed of only methane. We then solve for the temperature at three phase equilibrium (e.g. Figure 3-5purple, red and blue dots). In fact, multiple observations have shown that this phase boundary does not always lie at its predicted temperature. A different hydrocarbon composition, salinity, in situ pressure, or capillary effects could all cause this discrepancy. Direct measurement of temperature, pressure, and pore water composition may resolve these discrepancies and allow us to better understand the conditions of hydrate formation.

By measuring the temperature profile and calculating the geothermal gradient, we will quantify the thermodynamic state of all hydrate reservoirs penetrated in the borehole. Locations near the phase boundary will be most susceptible to natural and human induced perturbations. For example, in Figure 3-5, the hydrates within the Orange sand will begin to dissociate when pressure decreases by 2.7 MPa. In addition, the higher the in-situ temperature, the greater the sensible heat present and the more production that will be possible before the reservoir freezes due to temperature decrease during production. Temperature must also be determined to calculate the in-situ methane solubility, measure dissolved methane concentration, determine the onset of dissolved methane saturation, and measure hydrate saturation. By measuring the in-situ pressure, we will determine whether the pore pressure is indeed hydrostatic and what the thermodynamic conditions are.

In addition, it is necessary to measure the temperature profile in order to calculate the in-situ methane solubility, measure dissolved methane concentration, determine the onset of dissolved methane saturation, and measure hydrate saturation.

3.3.2. Plan to meet Objective 3

Formation temperature will be measured in two manners. We will measure pressure and temperature with a penetrometer to a depth of ~975 feet below seafloor (fbsf) in hole WR313 H003. We will use the 'Temperature 2 Pressure' (T2P) probe. The tool is only compatible with PCTB-CS BHA.

In addition, we will measure temperature while piston-coring in H003 using the IODP APC temperature sensor (APCT-3, APCT Tool Sheet (tamu.edu)). In this approach, two sensors embedded in the cutting shoe of the piston corer record the cutting shoe temperature while the piston-core is advanced, held in the formation for 10 minutes, and the inner core barrel is extracted. The in-situ temperature is then inferred from the acquired temperature history. APCT temperature measurements will be made from the seafloor to the depth of our final APC core whereupon we will switch to XCB coring. This depth is currently unknown.

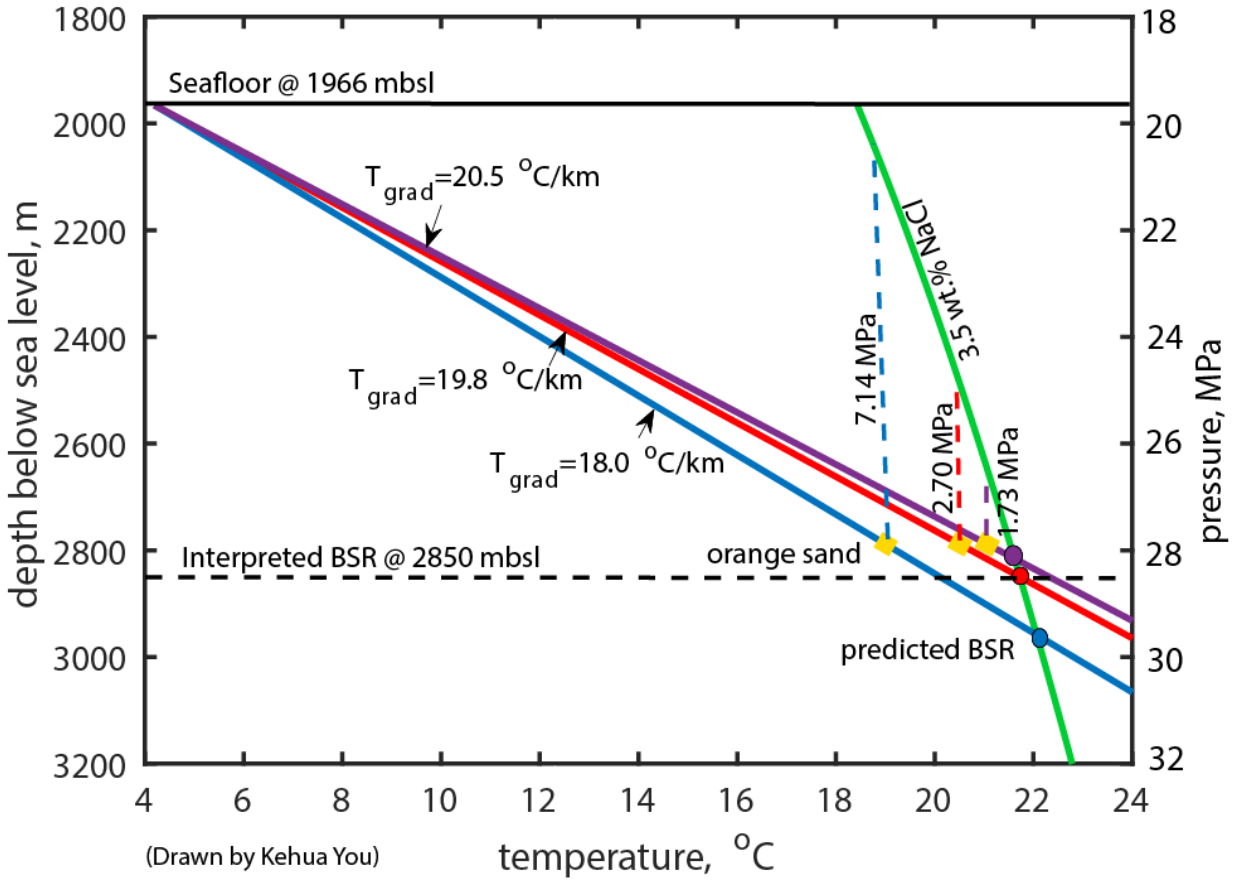

Figure 3-5. Inferred temperature and pressure at the H001 hole. A temperature gradient of 19.8 °C/km (red line) and a seafloor temperature of 4.2 °C results in a temperature at the depth of the bottom-simulating reflector (black dashed line) that is at the phase boundary (green line). This is our inferred in situ temperature. A higher temperature gradient (purple line) intersects the phase boundary above the BSR (purple dot), whereas a lower temperature gradient intersects phase boundary beneath the BSR (blue dot). The pressure and temperature inferred for the Orange sand for the three different temperature gradients are shown by the yellow dots. To calculate the phase boundary (green line), pore pressure is assumed to be hydrostatic, pore water is assumed to be of seawater salinity, and only methane is assumed to be present.

3.4. Objective 4: Characterize dissolved methane concentration and gas molecular composition with depth

3.4.1. Objective 4 Rationale

A downhole profile of dissolved methane concentration can be used to infer where hydrate is either present or currently forming. If the moles of methane produced from a depressurized sample exceed the maximum moles of methane that can be dissolved in the pore water at in situ temperature, pressure, and salinity (the methane solubility), then it is interpreted that hydrate has formed (Figure 3-6). The amount of methane present in a pressure core in excess of

solubility is used to calculate hydrate saturation. Alternatively, if the concentration is less than the solubility, it is interpreted that hydrate is not present.

Modeling studies have suggested that microbial methane generated in fine-grained muds can migrate over short distances into thin sand layers to form high-saturation methane hydrate (Cook and Malinverno, 2013). This process results in a distinct dissolved methane concentration gradient in the muds bounding these sand layers (Figure 3-6). We may be able to observe this variation with our methane concentration methods. In addition, changes in microbial communities, gas composition, and organic matter composition in the transitions to these thin sands may reveal if short-range migration of microbial gas is driving hydrate formation.

Modeling of dipping sands from Terrebonne Basin suggest that this short-range migration is sufficient to form high-saturation hydrates in thin sands such as the Red sand (Cook and Malinverno, 2013), but insufficient for forming high-saturation hydrates in thick sands (You and Flemings, 2018). Other processes such as long-range gas transport, overpressure-driven flow, or gas recycling at the base of gas hydrate stability are required to form high saturations in thick sands (Nole et al., 2016; Nole et al., 2018; You and Flemings, 2018). Free gas flow in a sand from below the base of hydrate stability will result in elevated dissolved methane and a diffusional gradient at the top of the reservoir but not below the reservoir (You and Flemings, 2018). Dissolved methane in transitions to thick sands along with the hydrate saturation distribution within them will determine if long-range transport is driving hydrate formation. We will test this model by collecting closely spaced (20 m) pressure cores between the Orange sand and Blue sand.

The gas composition will illuminate the genetic source of the gas. The molecular composition of the hydrocarbons (C1-C5), with noble gas concentrations, and C and H isotopes of methane will be used to determine the relative contribution of microbial or thermogenic hydrocarbon sources. The C and H isotopes of methane will also illuminate the pathways of methanogenesis (Figure 3-6). These pathways can be microbial, such as 1) hydrogenotrophic methanogenesis (i.e., carbon dioxide (CO_2) reduction); 2) acetoclastic methanogenesis (i.e., acetate (CH_3COOH) fermentation); or 3) methylotrophic methanogenesis (i.e., methanol (CH_3OH reduction) (Whiticar, 1999); or thermogenic. Natural gas formed by thermogenic processes, for example, is distinguished from microbial sources by higher levels of longer-chained aliphatic hydrocarbons (C2+) and an increase in the stable isotopic composition of C and H in methane.

Finally, we plan to analyze clumped isotopologues of methane to further constrain microbial and thermogenic pathways, and possibly the temperature, and hence, the depth at which the methane formed.

It is increasingly recognized that the microbial factory is responsible for huge deposits of natural gas (Katz, 2011; Rice and Claypool, 1981). Surprisingly, results from GC-955 suggest that this deposit is sourced by microbial methane, even though it overlies a thermogenic hydrocarbon source and there are indications of upward gas transport. The processes of microbial methane biogenesis and, in particular, the influence of physical and biogeochemical factors on methanogenesis rates occur are poorly known. Downcore profiles of methane concentration and molecular and isotopic composition will first determine how much of the methane is microbial, and then inform and constrain current biogeochemical models of methane production.

As organic carbon is buried below the sulfate-methane transition, communities of methanogens and fermenting bacteria will consume organic matter and produce methane. The influence of

organic matter content/quality, lithology, and substrate availability on the rate of methanogenesis are poorly constrained. By determining systematically at what depth and age microbes are currently generating methane, along with quantifying the increase in dissolved methane with depth, we will better constrain these biogenic models.

Modeling of dipping sands from Terrebonne Basin suggest that this short-range migration is sufficient to form high-saturation hydrates in thin sands such as the Red sand (Cook and Malinverno, 2013), but insufficient for forming high-saturation hydrates in thick sands (You and Flemings, 2018). Other processes such as long-range gas transport, overpressure-driven flow, or gas recycling at the base of gas hydrate stability are required to form high saturations in thick sands (Nole et al., 2016; Nole et al., 2018; You and Flemings, 2018). Free gas flow in a sand from below the base of hydrate stability is expected to result in elevated dissolved methane and a diffusional gradient at the top of the reservoir but not below the reservoir (You and Flemings, 2018) which can be tested by pressure coring. Dissolved methane in transitions to thick sands along with the hydrate saturation distribution within will determine if long-range transport is driving hydrate formation. We will test this model by collecting closely spaced (20 m) pressure cores between the Orange sand and Blue sand.

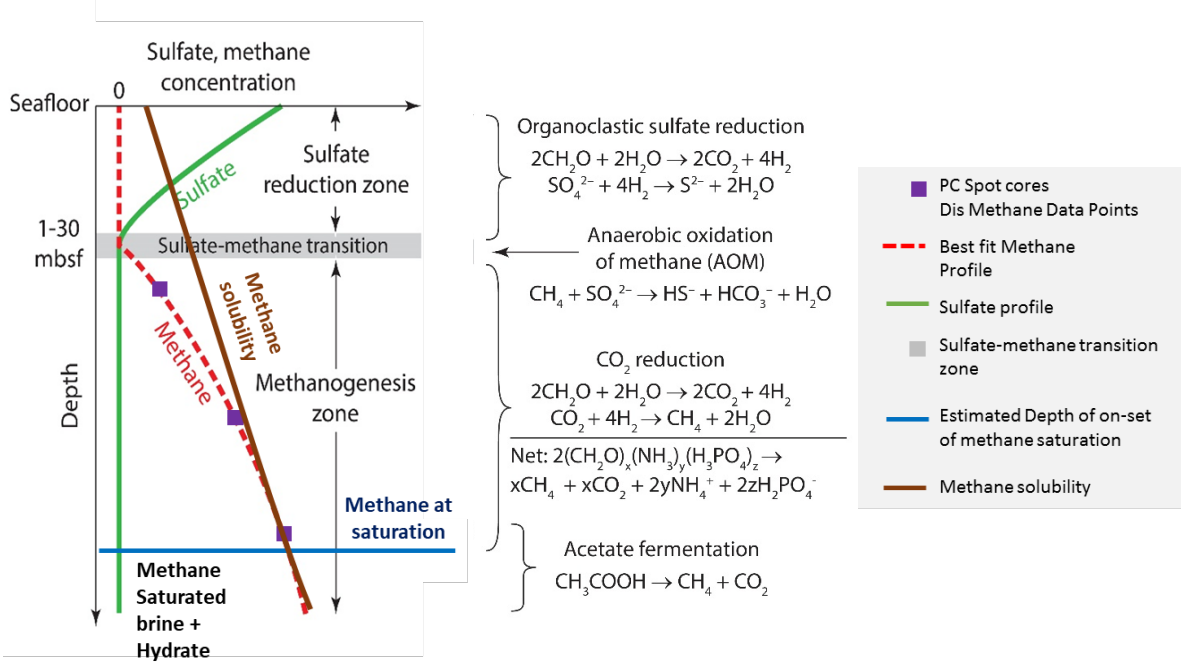

Figure 3-6. Schematic diagram of the microbial methane production and oxidation processes typical of continental margin sediments. In these equations CH_2O represents organic matter.

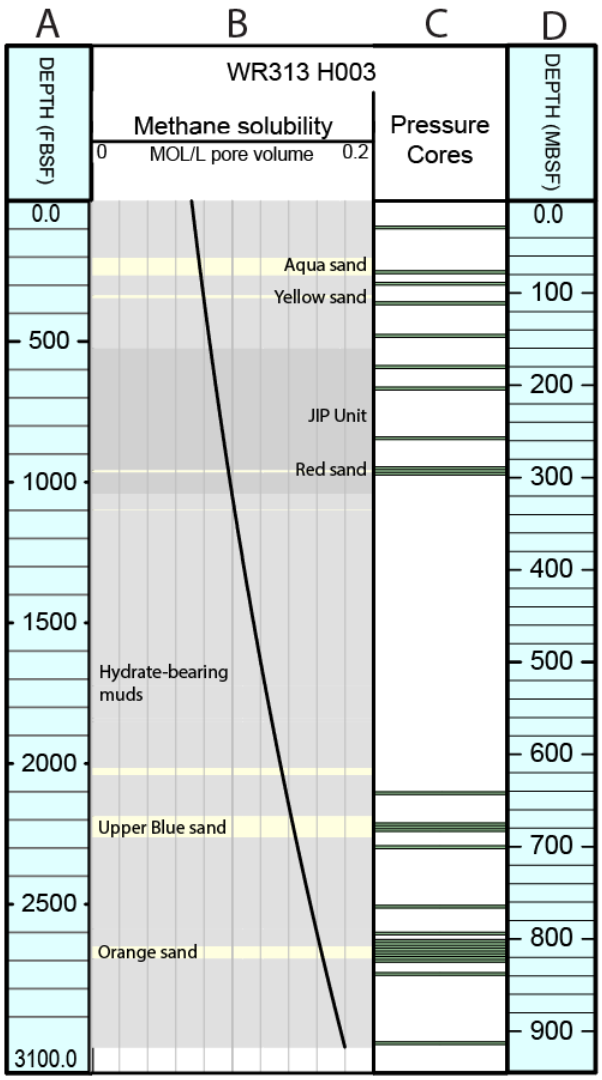

Figure 3-7. H calculated maximum concentration of methane in the pore water. A) Depth in feet below the seafloor (FBSF). B) Maximum concentration of methane (methane solubility) in H003 at in situ temperature, pressure and salinity in moles per liter of pore water volume (mol/L pore volume) is shown with solid black line. Hydrostatic pressure, a constant temperature gradient (see next section), and seawater salinity are assumed. The JIP mud unit is highlighted in darker grey and sand units in light yellow. C) Location of H003 pressure cores where the dissolved methane concentration and possible hydrate saturation will be calculated. D. Depth in meters below the seafloor.

3.4.2. Plan to meet Objective 4

We will meet Objective 3 by pressure coring over a range of depths in the muds surrounding the coarse-grained hydrate intervals to obtain a profile of dissolved methane and gas composition. The location of these pressure cores will be coordinated between the two holes. Initial dissolved methane concentrations from H003 will be used to predict concentrations in H002 and adjust coring points. Deeper pressure cores in WR313 H003 will focus on the interval between the Orange and Blue Sand to test the long-range transport model. The dissolved methane

concentrations for WR313 H003, together with analyses from conventional coring, will focus on characterizing the microbial methane 'factory' and target an expected increase in dissolved methane from below the sulfate-methane transition (SMT) to the depth at which methane reaches maximum solubility. The depth of the SMT is commonly within the upper 20 m in methane-bearing continental margin sediments.

We will acquire a depth profile of dissolved gas concentration and the gas molecular/isotopic composition to characterize the gas source and the microbial methane production. Degassing experiments will be performed on longer intervals of high-quality core to be able to resolve changes in dissolved methane. Quantitative degassing of pressurized core sections will directly measure the volume of gas and methane produced, and will use this methane volume with core volume and porosity to calculate the dissolved methane concentration. The molecular (C1-C5) hydrocarbon composition of the hydrocarbons (C1-C5) of the produced gas will be measured. The isotopic composition of methane ($\delta^{13}C$ and $\delta^{2}H$) and CO_2 ($\delta^{13}C$) will also be measured. We will also measure any atmospheric N_2 or O_2 contamination.

3.5. Objective 5: Reservoir characterization—other targets of interest

WR313 H002 and H003 contain another sand of interest, the Red sand, that will be characterized.

3.5.1. Objective 5 Rationale

By coring the Red sand, we will characterize another hydrate reservoir at a different thermodynamic state. This shallower sand is further from the hydrate stability boundary and at lower effective stresses. We will have the opportunity to examine whether there are fundamental differences in hydrate reservoirs the system is further from the hydrate stability boundary. Coring the Red sand will provide insight on a variety of questions including: 1) does hydrate form in thin sands via methane diffusion? 2) What is the form and concentration of fracture-filling hydrate in clay surrounding the thin sand? 3) What is the variation in sediment grain size and composition between reservoirs?

3.5.2. Plan to meet objective 5

We will meet Objective 6 by pressure coring the Red sand. Pressure core analysis will be done on-board and dockside. Conventional core analysis will be done on depressurized pressure cores, as possible. We will characterize the 1) hydrate concentration, dissolved methane concentration, and produced gas composition, 2) pore water dissolved solute concentration and composition, 3) lithofacies identification, grain size, and sorting, 3) permeability, 4) compressibility, 5) strength behavior, 6) sediment composition and age, 7) microbial communities and activity.

Table 3-1. Interpreted effective stresses in the middle depth of each sand penetrated by H001.

Location	Sand	Depth in the center (mbsl)	Interpreted effective stress (MPa)
H001	Aqua sand	2040	0.49
	Yellow sand	2073	0.75
	Red sand	2262	2.57
	Upper blue sand	2649	6.54
	Orange sand	2783	7.95

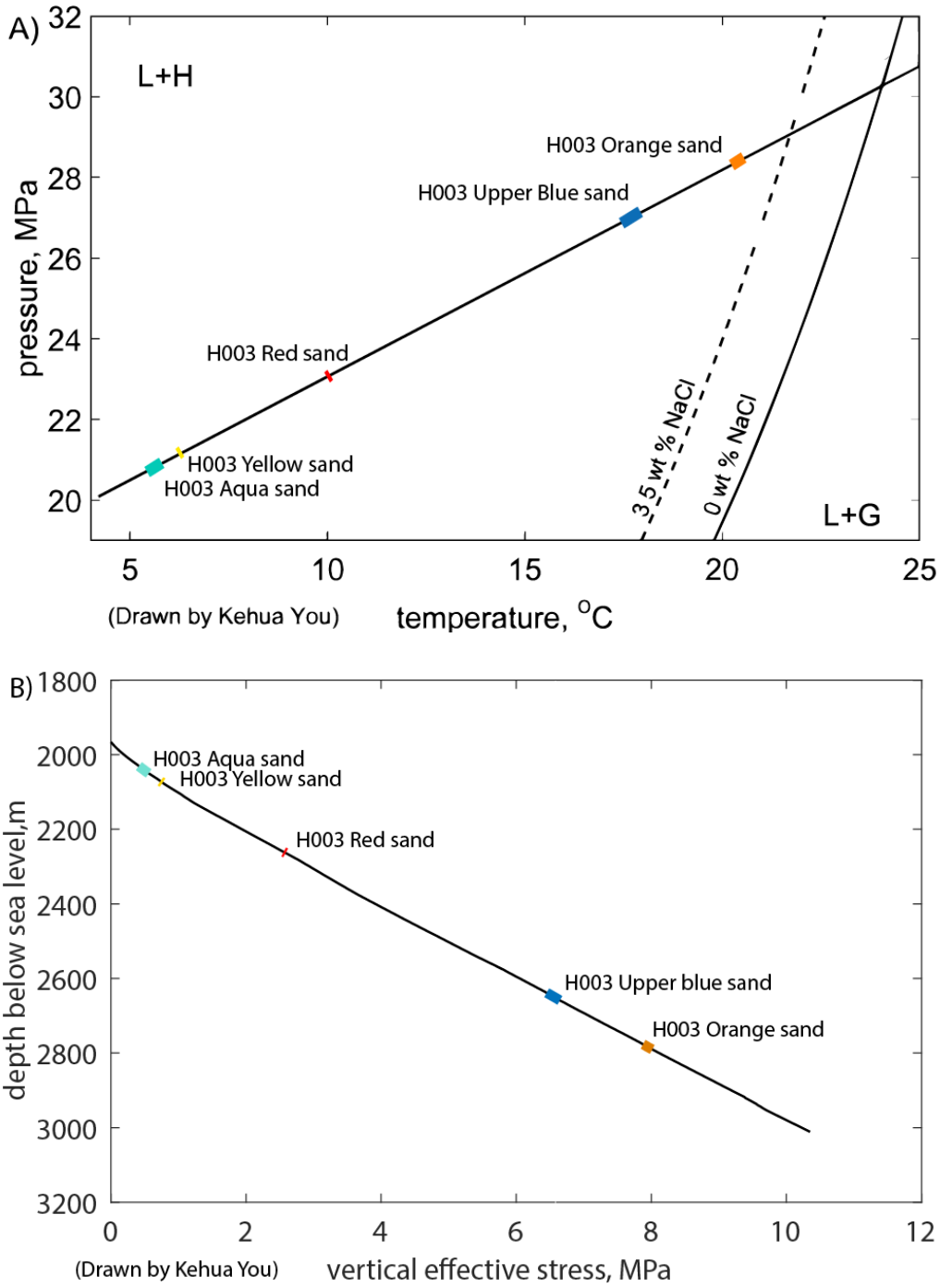

Figure 3-8. In-situ pressure, temperature, depth, and stress of each sand A) Position of each sand (as described in Section 5.1.1 H002 and H003 units and tops) for water- and hydrate-bearing sand reservoirs within the methane hydrate phase diagram. The black line tracks the P-T path for Location H from the seafloor (left) to total depth (right). The phase boundary where vapor, hydrate, and water can coexist is marked for seawater salinity (dashed line) and fresh water (solid line). B) Vertical effective stress for H (black line).

4. Measurements to Inform Scientific Objectives

The following section discusses the planned scientific measurements and the information they provide. These measurements will enable us to successfully meet the six science objectives (see Section 3. Scientific Objectives).

4.1. Pressure Core Analysis

Pressure cores will be acquired in the key hydrate-bearing sands, bounding muds, and background muds to meet Objectives 1, 4, and 5 as discussed in Sections 3.1, 3.4, and 3.5, respectively. Depressurized cores of background muds will be processed as conventional cores to help meet Objective 2 (Section 3.2).

For more information on the movement and processing of pressure cores and produced gases from pressure cores, see the following sections: Sections 7.1.1 On-Board Pressure Core, 7.2.1 Dockside Pressure Core, 7.3.1 UT Pressure Core, 7.1.4 On-Board Gas Analysis, 7.2.4 Dockside Gas Analysis, 7.3.1 Ohio State Gas Analysis, and 7.3.2 TBD Clumped Isotopes.

4.1.1. Pressure core logging and imaging

Pressure core logging and imaging of pressure cores will be performed to determine the amount and quality of pressure core recovered, the amount of fall-in material, the lithofacies present, to discriminate hydrate-bearing sediment, and to determine core cutting locations. Logging and imaging will include p-wave velocity, gamma density, 2D X-ray imaging, and 3D X-ray tomography. These scans are used to determine the specific analysis plan for each core.

4.1.2. Pressure core sampling

Whole round samples of lithofacies-specific pressure cores will be cut for quantitative degassing and gas analysis, geomechanical testing, liquid nitrogen (LN2) depressurization for microbiology, rapid depressurization for pore water analyses, and distribution to other institutions.

Quantitative degassing of pressure cores will be performed. Quantitative degassing of hydrate bearing sands will be used to measure dissolved methane concentration, hydrate saturation, and assess the composition and source of the dissociated gas. Slower depressurization experiments with small pressure decrements are used to estimate the in-situ salinity based on the pressure and temperature condition of the sample at the onset of dissociation.

Quantitative degassing of clean background mud (no hydrate-filled fractures or coarse-grained beds/laminations) will be performed to measure the dissolved methane profile and gas composition with depth. Quantitative degassing of background mud with hydrate fractures will determine the hydrate saturation and gas composition within fracture-filled muds.

Gas produced from quantitative degassing will be used to measure hydrocarbon, carbon dioxide, and noble gas content and hydrocarbon isotope and isotopologue ratios. Clumped methane isotopologues will be analyzed. Special handling, such as LN2 depressurization, will be available to preserve high-hydrate sections as intact cores for microbiological or sedimentological analysis.

Geomechanical testing of pressure cores at in situ conditions will be conducted. Strength and transport properties will be determined at all levels of the hydrate system.

All depressurized pressure core section (mainly from quantitative degassing) that remain intact will be moved to the conventional core processing flow for conventional core analysis. Depressurizing pressure cores for conventional core analysis is particularly important for intervals where conventional coring is not planned so that cores can be fully characterized for pore water chemistry, solid-phase geochemistry, physical properties, geomechanical properties, and microbial community composition over profiles to total depth. A section from at least one PCTB core collected within the interval of continuous conventional core will be depressurized specifically to compare microbial communities identified from pressure and conventional cores.

Unconsolidated sediment from degassed core sections will be bagged and stored in Core Storage (~4-6 C). Unconsolidated sediment will be used for mechanical studies (as a reconstituted core), physical properties, biostratigraphy. Biostratigraphy of the depressurized core is also important for understanding sediment accumulation rates and sediment dating of bounding clays around the hydrate-bearing sands.

4.2. Conventional Core Analysis

Conventional cores will be acquired in H003 to meet Objective 2 (see Section3.2). Intact, lithofacies-specific, depressurized (conventionalized) cores acquired in H002 and H003 will be moving into the conventional core flow to meet Objects 1, 2, 3, 5, and 6 (see Section 3).

For more information on conventional core and pressure core produced gas processing, see the following sections: Sections 7.1.2 On-Board Conventional Core, 7.2.2 Dockside Conventional Core, 7.3.4 Tufts University Geomechanics, 7.1.4 On-Board Gas Analysis, 7.2.5 Dockside Gas Analysis, 7.3.1 Ohio State Gas Analysis, 7.3.2 TBD Clumped Isotopes, and other sections in 7.3 Post-expedition.

4.2.1. Conventional core logging and imaging

Conventional cores will be imaged twice with a thermal (IR) track scanner, once immediately when the core is recovered, and then again after cutting and removing whole round samples. IR imaging is valuable for identifying background sediments versus hydrate-bearing anomalies and providing an initial assessment of core quality. Thermal imaging of the remaining conventional core after whole round sampling is useful for monitoring the core thermal evolution after recovery in order to identify additional developing thermal anomalies. The additional thermal scan also provides a backup record of where whole round samples have been removed from the core since the initial IR scan conducted upon recovery and before sampling and cutting into archived sections.

Conventional and depressurized whole round cores will be logged and imaged. Gamma ray attenuation density, P-wave velocity, magnetic susceptibility, resistivity, and natural gamma radiation will be logged using whole round scanning. X-ray CT imaging of the whole core will also be performed. Logging is valuable for understanding stratigraphic context, tracking variation in sediment composition, and determining core sampling locations.

Sediment strength will be measured on whole round cores by hand vane, pocket penetrometer, and Peak and residual strength will be measured using fall cone and a miniature vane strength device. Sediment strength is important for characterizing the mechanical state of the sediments as a function of depth. Hand held vane and penetrometer measurements may provide an initial measurement of strength and will be used to help determine the proper depth to switch from APC to XCB coring (see -UT-GOM2-2 APC to XCB). Thermal conductivity will also be measured.

Split cores will also be logged and imaged. Split core scans will include high resolution magnetic susceptibility, line-scan camera photography, X-ray fluorescence, and color spectrophotometer. Scanning split core and obtaining discrete samples of sediment is important for characterizing the lithostratigraphy, sediment properties with depth, and for interpreting depositional environments and geological history.

Variations in magnetic susceptibility of core samples often represent stratigraphic variation and thus, magnetic susceptibility records are an excellent core to core correlation tool. In addition, the tool can be used to determine the lithological properties, including any changes in sediment provenance and/or diagenetic environment. Magnetic susceptibility in marine sediment records can represent a mixed depositional and diagenetic signal. The primary magnetic susceptibility pattern can be affected by dissolution and precipitation of magnetic mineral phases due to hydrogen sulfide produced during organoclastic sulfate reduction (OSR) and/or anaerobic oxidation of methane (AOM) at the sulfate methane transition zone (SMTZ) (Johnson et al., 2021). This diagenetic alteration can result in decreased magnetic susceptibility at the SMTZ due to (titano) magnetite dissolution and pyrite precipitation. Additionally, AOM-related diagenesis can increase the magnetic susceptibility by producing magnetic iron sulfides, such as greigite or pyrrhotite that can increase magnetic susceptibility (Larrasoaña et al., 2007). Thus, the magnetic susceptibility record can be used to identify potential intervals of diagenetic alteration related to methane cycling.

High-resolution digital imagery of sediment cores will improve our ability to properly identify and interpret sedimentary structures and subsample primary sedimentary materials and diagenetic precipitates. X-ray fluorescence scans can be used to determine the elemental composition of sediment. The color of sediment, measured with the spectrophotometer, reflects important aspects of depositional environments including redox conditions and rates of deposition of organic matter and calcium carbonate.

4.2.2. Conventional core sampling

Gas and a ‘composite’ of whole round samples will be taken from background sediment in the conventional and depressurized cores. Gas samples will include void and headspace gases. The whole round ‘composite’ or ‘set’ will include sub-samples for pore water geochemistry, sediment moisture and density, and microbial communities. Additional whole round samples may be collected to characterize thin-bedded IR anomalies which may indicate cm-scale silt and sand layers hosting gas hydrate. Alternatively, these intervals may be marked for sampling after core splitting. If encountered during core cutting, large pieces of hydrate may be captured using a syringe and placed in a gas bag. Whole round samples will be collected separately for geomechanical properties.

Analysis of the hydrocarbon content of void and headspace gas samples will be primarily used to create a profile of hydrocarbon fluids and gases encountered with depth. Headspace gas samples usually provide a more complete profile and also tend to be enriched in the higher hydrocarbon homologs. Void and headspace gas samples will also be used to monitor the occurrence of potential gas related drilling hazards with a focus on analyzing (C1/C2+C3) gas ratios, where decreasing values (<100) may be indicative of thermogenic derived gas and oil.

Characterization of pore water chemistry can determine the role of biogeochemical and alteration processes within sediments, and the contribution of advected deep fluids into the shallower hydrate stability zone. Samples from the whole round composite will provide background profiles in pore water constituents.

Moisture and density analysis (MAD) will be taken to determine bulk density, dry bulk density, moisture content, grain density, and porosity. These are important physical property measurements used in hydrate saturation and mass accumulation rate calculations. These properties are also important for the interpretation of permeability and geomechanical experiments.

Sediment from MAD whole rounds will be subdivided for measurements of grain size distribution, magnetic properties, mineral composition, and bulk elemental composition (CHNS), including TOC. Correlation of these physical properties, especially grain size distribution, is important for proper interpretation of the geochemistry and geomechanical results.

Grain size distribution will be assessed using laser particle analyzer and hydrometer (settling) methods. Quantitative grain size measurements will allow us to determine grain size effects on the gas hydrate distribution in these records. Supplementing grain size measurements using a laser particle analyzer with measurements using a hydrometer is required to provide a better assessment of clay-sized particles underestimated by the laser partial analyzer method (Germaine and Germaine, 2009).

The composition and activity of the microbial community with depth is important for understanding reservoir microbial methane sourcing and basin evolution. Selected samples will be collected for microbial community characterization (by extraction, amplification and sequencing of DNA), total cell counts, single-cell genome studies, and culture-based characterization of microbial communities. Drilling fluid and PCATS confining fluids (PCATS water) samples will be collected to assess the extent of microbial contamination of the cores. Lab air will be collected and assessed for possible microbial contaminants such that these taxa can be identified as suspect contaminants and eliminated from subsequent analyses.

Geomechanical properties including permeability, lateral stress coefficient, shear strength, and friction angle will be determined from geomechanical analysis of whole cores.

See more conventional core analysis under Section 4.4 Sedimentology below.

4.2.3. Conventional core split core description

After split core scanning, the archive half of cores will be described visually. This macroscopic analysis will document bedding, sedimentary structures, major lithology, relative grain sizes, Munsell color, presence of diagenetic nodules, bioturbation, and drilling/coring disturbance. This description will also involve microscopic sediment analysis via smear slide and coarse fraction sampling and description. These macro- and micro-descriptions will be integrated to create the lithostratigraphic core description and log, which, along with core logging and imaging, will guide additional sampling of the working half.

Lithology from smear slides and coarse fraction sediment descriptions provide the basis for identification of changes in bulk composition. Smear slides allow for a semi-quantitative estimate of major and minor lithologies as well as identification of diagenetic and trace minerals. Smear slide analysis will also allow for an estimation of grain size ranges.

Biostratigraphic sampling and observation of key nannofossil markers species will also be performed from smear slide and coarse fraction analysis and is key to determining sediment age and sedimentation rates.

4.3. Pore Water Analysis

Pore water geochemical analysis is critical for Objectives 1, 4, and 5. Pore water solute, gas, and isotope ratio profiles are used to track *in situ* biogeochemical reactions such as SO_4-reduction, Mn- and Fe-reduction, the anaerobic oxidation of methane, and methanogenesis, as well as to characterize diagenetic reactions such as silicate weathering, authigenic aluminosilicate precipitation, authigenic carbonate precipitation, carbonate recrystallization, and ion exchange. Furthermore, we can use pore water chemical profiles to identify fluids migrating from deeper sources along permeable stratigraphic horizons. The shape of the pore water profiles can be used to quantify reaction rates (e.g. the rate of AOM) and the rates of fluid flow.

Pore water samples will be extracted from whole round core samples on-board. Drilling fluid and PCATS confining fluids (PCATS water) samples will be collected to assess the extent of pore water contamination. The pore water salinity, pH, and alkalinity will be measured on-board as soon as possible, and pore water samples for additional analysis will be preserved on-board. Residual sediment after squeezing the pore water will also be preserved.

For more information on pore water processing, see the following sections: Sections 7.1.5 On-Board Pore Water, 7.2.6 Dockside Pore Water, and 7.3.3 University of Washington Pore Water.

4.3.1. Geochemical tracers that will be measured on-board

The following geochemical tracers will be measured on-board.

Salinity is a routine measurement of dissolved salt content. It is used as an initial assessment of gas hydrate distribution and concentration. It governs the physical properties of the pore water (e.g. density), and is important for determining the limits of the gas hydrate stability field.

Alkalinity is a critical parameter in constraining shallow and deeper carbon cycling, and along with DIC concentrations can be used to calculate the pore water pH and speciation between the weak acids and bases.

4.3.2. Geochemical tracers of biogeochemical reactions measured post-expedition

The following tracers will be measured post-expedition.

Dissolved Inorganic Carbon (DIC) is defined as $[CO_2] + [HCO^{3-}] + [CO_3^{2-}]$. Critical measurement for understanding carbon cycling (including methanogenesis and methane oxidation) and pH in gas hydrate systems. δ^{13}C-DIC is critical for quantitative models of sulfate reduction, anaerobic oxidation of methane, and methanogenesis. It places strong constraints on the source of DIC in pore water systems.

Dissolved Organic Carbon (DOC) is produced during the decomposition of sediment organic matter and is the substrate utilized by methanogens.

Volatile fatty acids (VFAs) are a reactive component of the DOC pool that may be directly converted to methane. Profiles of their carbon isotopic composition provide information on their production and utilization.

Sulfate is consumed during organic matter degradation and the anaerobic oxidation of methane. Below the sulfate-methane transition zone, SO_4 is a valuable, quantitative tracer for drill water contamination.

Dissolved sulfide is an important product of both organocalstic sulfate reduction and anaerobic oxidation of methane, it is important for constraining the sulfur cycle in marine sediments.

Bromide is a product of the decomposition of organic matter that is used to track microbial metabolic reactions in marine sediments. Once released from organic matter, it behaves conservatively within the temperature and pressure conditions anticipated at these sites. Ammonium is also a product of the decomposition of organic matter that is used to track microbial metabolic reactions both within the sulfate reduction zone and within the methanogenic zone. Neither Br nor NH_4 are produced through AOM.

Trace metals (e.g. Fe, Mn, Ni, Co) are important products in the redox sequence of marine pore waters during early diagenesis of organic matter and in the marine sulfur cycle. Many of the trace metals are important nutrients for methanogens and methanotrophs and as such are critical for both methane production and consumption. The isotopes of several of the trace metals are useful for tracking the competition between metal release from sediments, utilization by the microbial community, and sequestration in authigenic minerals.

4.3.3. Geochemical tracers of diagenetic reactions and deeper-sourced fluids measured post-expedition

The following tracers will be measured post-expedition.

Chloride concentrations are affected by evaporite dissolution, and also track the addition or uptake of H_2O. Background Cl profiles provide information on authigenic clay formation and clay dehydration (e.g. the smectite-illite transition) at depth. Negative Cl anomalies are used to estimate in situ gas hydrate concentrations. The stable isotopes of chlorine are helpful for identifying fluids sourced from higher temperatures. Hydrous silicate mineral formation at higher temperatures partitions Cl and ^{37}Cl into the mineral, leaving the residual fluid depleted in Cl and ^{37}Cl.

Calcium, Magnesium, Sodium, and Potassium are the major cations in seawater. They are involved in a wide-range of in situ and deeper fluid-rock reactions. They are used to constrain carbon sinks, diagenetic reactions, deeper-sourced fluids, and fluid flow pathways.

Lithium, Boron, Strontium, Barium, and Si each track fluid-sediment interactions over a wide range of temperatures and depths. The alkali metals, and Li and B in particular, are useful tracers of deeper-sourced fluids, and when combined with lithium and boron isotope ratios, are useful for constraining the temperature at fluid sources. Dissolved Si concentrations provide information on fluid-rock equilibria and fluid sources, and, in some lithologies, is a well-established geothermometer. Pore water strontium isotope ratios are an essential tracer of fluid sources and fluid/rock reactions and are used to distinguish between ash, terrigeneous sediment, biogenic carbonates, and evaporites as sources of strontium to the pore water.

When coupled with dissolved Cl profiles, $\delta^{18}O$ and δD of the pore water are important tracers of the presence of gas hydrates and for estimating *in situ* gas hydrate concentrations. Background profiles provide information on fluid/rock reactions and water sources (i.e. clay dehydration at depth, meteoric water), and are also commonly used in chemical geothermometry.

Cs, Cesium will be used as a contamination tracer in PCATS system. Like SO4 for drilling fluid contamination, Cs will provide quantitative information on the amount of core contamination during cutting and depressurization.

4.4. Sedimentology

Select intervals/plugs of sediment will be taken from the working half and preserved for post-expedition measurement. Samples will be collected for grain size distribution, CHNS, total organic carbon (TOC), rock magnetic properties, XRPD, MAD, etc. These samples along with sediment from whole rounds, pore water residue, and collected from core-catcher and other coring tool parts will be used to construct comprehensive core descriptions containing the compositional, structural, stratigraphic, and diagenetic fabric and facies variations throughout the cores.

For more information on split core processing, see the following sections: Sections 7.2.2.3 Split Core Lab, 7.3.7 UNH Sedimentology, 7.3.8 USGS Rock Magnetics, 7.3.9 UT Biostratigraphy, and 7.3.10 UT Split Core.

Synthesis of the grainsize, CHNS, and sediment composition data specifically can be used to document sediment transport regimes throughout the reservoir and subsequent early diagenesis of hydrate-bearing sediments. Additionally, increased sorting of all samples after organic carbon removal, reflects the variable size of organic carbon deposited during and between possible turbidity current events and documents whether or not both the turbidites and intervening clays contain measurable organic carbon.

Bulk sediment CHNS elemental analysis allows us to sample and measure at a high down core resolution Total Carbon (C), Total Nitrogen (N), Total Sulfur (S), Total Organic Carbon (TOC) and derived Calcium Carbonate (CaCO3), of select samples throughout the records. These measurements will serve to quantify the bulk compositional trends for import gas and gas hydrate related sediment components. (TOC and the C to N ratio (C/N) equates to the organic matter quantity and type, $CaCO_3$ tracks authigenic and biogenic carbonate variations, Total S tracks variations in pyrite and other iron sulfides, produced during sulfate reduction and AOM, as well as organic S.

Higher frequency sampling for grain size and TOC in and around the Red sand will test the viability of methane diffusion as the methane migration mechanism for hydrate accumulations in centimeter-meter thick sands.

Bulk sediment TOC, N, and S isotopes to allow us to look at the sources of organic carbon and evidence for AOM in the records. Coupled with the C/N measurement, the isotopic character of the organic carbon will define relative variations in the relative source (marine or terrestrial) of the carbon.

Changes in magnetic mineralogy may be utilized to track the migration of the SMTZ or gas hydrate stability zone. Specific rock magnetic properties (e.g. isothermal remnant magnetization, hysteresis parameters, low/high temperature susceptibility) will be measured. The characterization of magnetic mineral assemblages will be used to identify zones where AOM-related diagenesis has overprinted the primary detrital magnetic susceptibility signal, and changes in the primary detrital magnetic mineral assemblage.

Authigenic carbonate nodule composition and isotopic measurements yield information about the origins and methane related diagenetic history preserved in the cores. Authigenic carbonates can form both from methane oxidation coupled to sulfate reduction and from methanogenesis coupled to silicate weathering. Sulfide (Pyrite) nodules if present and sampled directly provide a better record of the origins of pyrite and evidence for AOM or OSR in the records.

X-ray powder diffraction (XRPD) reveals the minerology/crystallography of diagenetic nodules and the bulk sediment. The mineralogy, along with isotopic and elemental composition, of the nodules can provide insights into the biogeochemical processes that drove the formation of the nodules.

Nannofossil biostratigraphy samples will used to observe marker species and making age assignments based on established datums. These will then be used to create age-depth plots and calculate sedimentation rates. Additional sampling for coarse fraction foraminifers in the interval of continuous conventional coring can be used to measure benthic foraminifer $\delta^{18}O$ that can be used with global stack records to provide an age model on glacial-interglacial timescales.

4.5. Temperature and Pressure

Formation temperature and pressure measurements will be taken in H003. Temperature will be measured both during piston coring with the APCT and will be measured with a penetrometer. Pressure will be measured with a penetrometer. For more information on how temperature and pressure measurements will be taken, see Section 7.1.3 On-Board Temperature and Pressure.

5. Operational Plan Summary

The following section outlines the key components of the maximum and most-likely operational plans, based on the proposed and most-likely level of funding, respectively. The sections for each plan include high level summary, drilling location, projected tops, high level drilling and coring outline, and expedition schedule.

5.1. Location of holes and Projected Tops including sand targets of interest

The Geologic Program including hole locations, stratigraphy, top hole prognosis, predicted hydrate stability field and pore pressure is detailed in the Operational Plan and summarized here. Figure 5-1 shows the location of the H002 and H003 Holes with in the southern region of the Terrebonne Basin, off the coast of Port Fourchon, LA, Gulf of Mexico. The maximum operational plan includes coring at H002 and H003, while the most-likely plan currently only includes coring at H002.

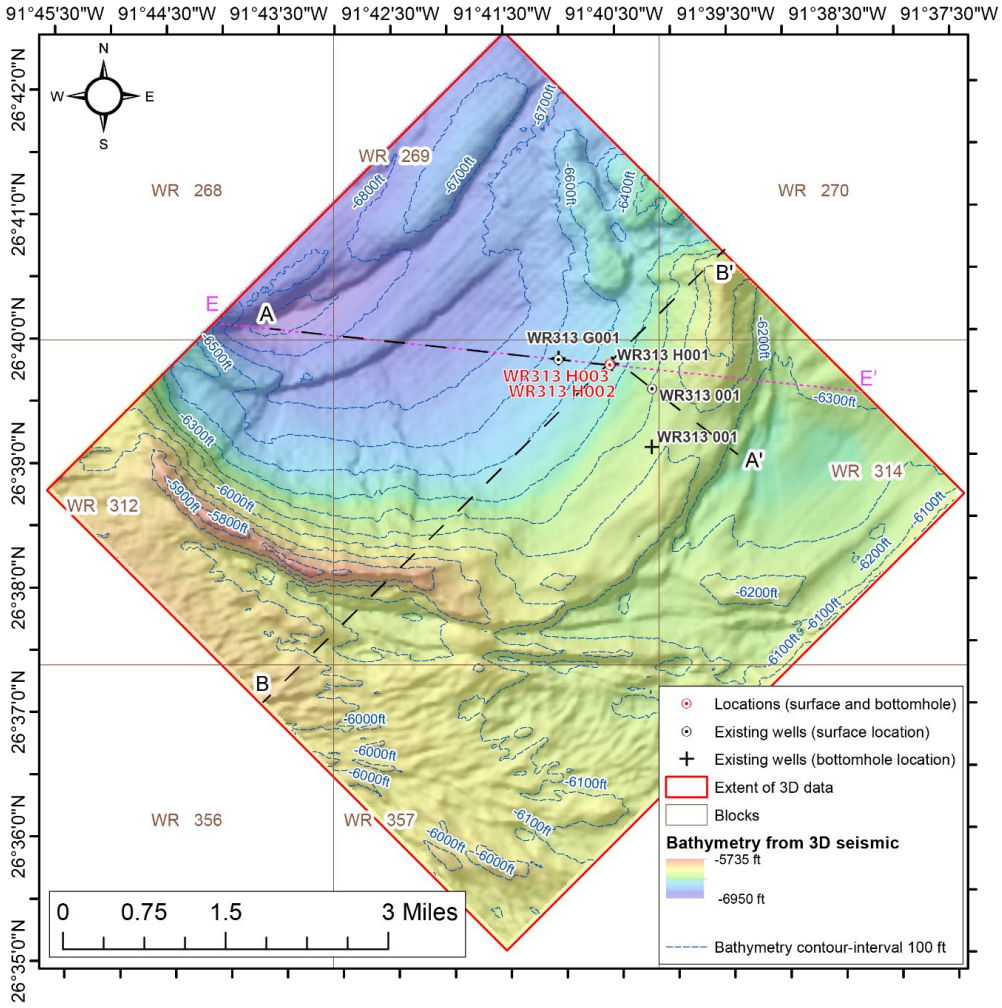

Figure 5-1. Bathymetry map of the area studied in southern Terrebonne Basin. Based on 3D seismic data, showing existing wells and proposed locations in Walker Ridge Block 313 (WR313). 3D seismic data were used with permission of WesternGeco.

5.1.1. H002 and H003 units and tops

The seafloor at WR313 H002 and H003 is projected to be at 6460 feet (1969 m) below sea level (fbsl). H002 and H003 tops were interpreted from H001 using seismic data from WesternGeco. The proposed wells are planned along-strike from WR313 H001, which means the tops depths in the proposed wells are identical to H001. H002 and H003 tops are shown in Table 5-1. Key sands and other targets of interest are:

- Aqua sand (201.5-264.0 fbsf, 62.5 ft thick with 12 ft of dispersed sand)
  - water-saturated coarse-grained sediment
  - within Unit 1 (0-520.0 fbsf)
  - may contain a low concentration of gas hydrate in a ~1.5 ft thick interval
- Yellow sand (333.0-344.0 fbsf, 11.0 ft thick)
  - water-saturated coarse-grained sediment
  - within Unit 1 (0-520.0 fbsf)
- JIP mud unit, Unit 2 (520.0-1038.0 fbsf)
  - composed of mud with hydrate in near-vertical fractures
  - interpreted as a mass transport deposit and is more compacted or de-watered than the overlying mud.
- Red sand (958.0-966.0 fbsf, 8 ft thick)
  - high hydrate saturation coarse-grained sediment
  - within the JIP unit, Unit 2
  - does not appear to be connected between H001 and G001
- Upper Blue sand interval (2180.0-2256.0 fbsf, 76 ft thick with 13 ft of dispersed sand)
  - hydrate-bearing, thinly bedded course-grained sediment
  - within the lower interval of Unit 4 (2000-2285.0 fbsf)
- Orange sand (2642.0-2686.0, 44 ft thick with 39 ft of dispersed sand)
  - thick hydrate-bearing reservoir and the primary coring target in H002
  - course-grained sediment
  - within Unit 5 (beginning at 2285.0 fbsf)
- Base of hydrate stability (BHSZ), approximately 2935 fbsf
  - there is no indication of this event on the well logs or seismic
- Total depth is 3010 fbsf.

Table 5-1. H002 and H003 Projected topsInterpretation and unit descriptions are detailed in the Operational Plan.

			Water depth (ft)	Total depth (fbsf)	Total depth (fbsl)
WR313 H002 and H003			6460	3010	9470
Events, Sands & Units			**WR313 H001**	**WR313 H002 and H003**	
			depth (fbsf)	projected depth (fbsf)	projected depth (fbsl)
Seafloor			0.0	0.0	6460.0
water bearing Aqua sand	Top	**Unit 1**	201.5	201.5	6661.5
	Base		264.0	264.0	6724.0
water bearing Yellow sand	Top		333.0	333.0	6,793.0
	Base		344.0	344.0	6,804.0
Horizon 1000			520.0	520.0	6,980.0
JIP mud unit with low concentration hydrate	Top	**Unit 2**	520.0	520.0	6,980.0
hydrate bearing Red sand	Top		958.0	958.0	7,418.0
	Base		966.0	966.0	7,426.0
JIP mud unit with low concentration hydrate	Base		1038.0	1038.0	7,498.0
Horizon 0800			1038.0	1038.0	7,498.0
water bearing coarse-grained interval	Top	**Unit 3**	1096.0	1096.0	7,556.0
	Base		1100.0	1100.0	7,560.0
hydrate bearing marine mud	Top		1716.0	1716.0	8,176.0
	Base		1722.0	1722.0	8,182.0
hydrate bearing marine mud	Top		1832.0	1832.0	8,292.0
	Base		1846.0	1846.0	8,306.0
Horizon 0500			2000.0	2000.0	8,460.0
hydrate bearing coarse-grained interval	Top	**Unit 4**	2017.0	2017.0	8,477.0
	Base		2042.0	2042.0	8,502.0
hydrate bearing Upper Blue sand	Top		2180.0	2180.0	8,640.0
	Base		2256.0	2256.0	8,716.0
Horizon 400			2285.0	2285.0	8,745.0
hydrate bearing marine mud	Top	**Unit 5**	2578.0	2578.0	9,038.0
	Base		2580.0	2580.0	9,040.0
hydrate bearing Orange sand	Top		2642.0	2642.0	9,102.0
	Base		2686.0	2686.0	9,146.0
WR313 H002 TD				3010.0	9470.0

5.2. Operational Plan

5.2.1. Summary

1. Drill and core two vertical wells, one to 9470 fbsf and the other to 975 fbsf in the offshore Gulf of Mexico in Terrebonne Basin, Walker Ridge Block 313.
2. Spend ~ 28 days for mobilization, drilling, coring, plug and abandonment, demobilization, and contingency.

3. The first and second holes, WR313-H003 (H003) and WR313-H002 (H002) are within 62’ (19 m) of the previously drilled Walker Ridge Block 313 H well WR313-H (H001).
4. Pressure cores, 10’ (~3.0 m) long, will be attempted in each hole (up to 34 total deployments) using the Pressure Coring Tool with Ball Valve (PCTB).
5. The main coring reservoir target is the hydrate-bearing Orange sand [Horizon 0300]. This and other hydrate-bearing and non-hydrate bearing sands will be pressure cored. Pressure cores will also be taken in background mud samples.
6. Two configurations of the PCTB will be used: 1) the PCTB-FB face-bit configuration and 2) the PCTB-CS cutting-shoe configuration
7. Only pressure coring will be carried out in H002.
8. In H003, continuous conventional coring will be done in the shallow muds to measure the sulphate methane transition (SMT) and other geochemical and sedimentary profiles. Formation temperature and pressure will be measured with depth with a penetrometer.
9. Thermal imaging, whole round core sampling, pore water analysis, gas analysis, and initial measurements of sediment strength will be completed on the vessel.
10. The Pressure Core Analysis and Transfer System (PCATS) from Geotek Limited will be used to characterize pressure cores and transfer the samples to pressurized storage devices while on the drilling vessel.
11. Pressure core sections, 3.3’ to 3.9’ (1.0 to 1.2 m) in length and 2.0 inches (5.08 cm) in diameter, will be transported over land to Salt Lake City and then to the UT Pressure Core Center (PCC) for storage, further analysis, and distribution.
12. Time will be spent at Salt Lake City for continued core analysis, core logging using the Multi-Sensor Core Logger (MSCL-S) from Geotek Limited, core splitting, core description and split core processing.

5.2.2. Drilling and coring program

A graphical representation of the UT-GOM2-2 drilling, coring, and downhole testing program is shown in Figure 5-2. For a detailed description of target intervals and coring plan, refer to Section 6.

Coring Plan.

The first hole, WR313 H003, will be drilled solely with the PCTB-CS BHA (bottom-hole assembly). Pressure cores will be acquired in the Red, Blue, and Orange sands; and at other intermittent locations throughout the hole (Figure 5-2, left hole). We will acquire Advanced Piston Corer (APC) and extended Core Barrel (XCB) conventional cores, PCTB-CS pressure cores, and in situ pressure/temperature measurements. H002 will be drilled with the PCTB-FB BHA and we will acquire PCTB-FB pressure cores. Pressure cores will be acquired in and above the Red sand (Figure 5-2, right hole).

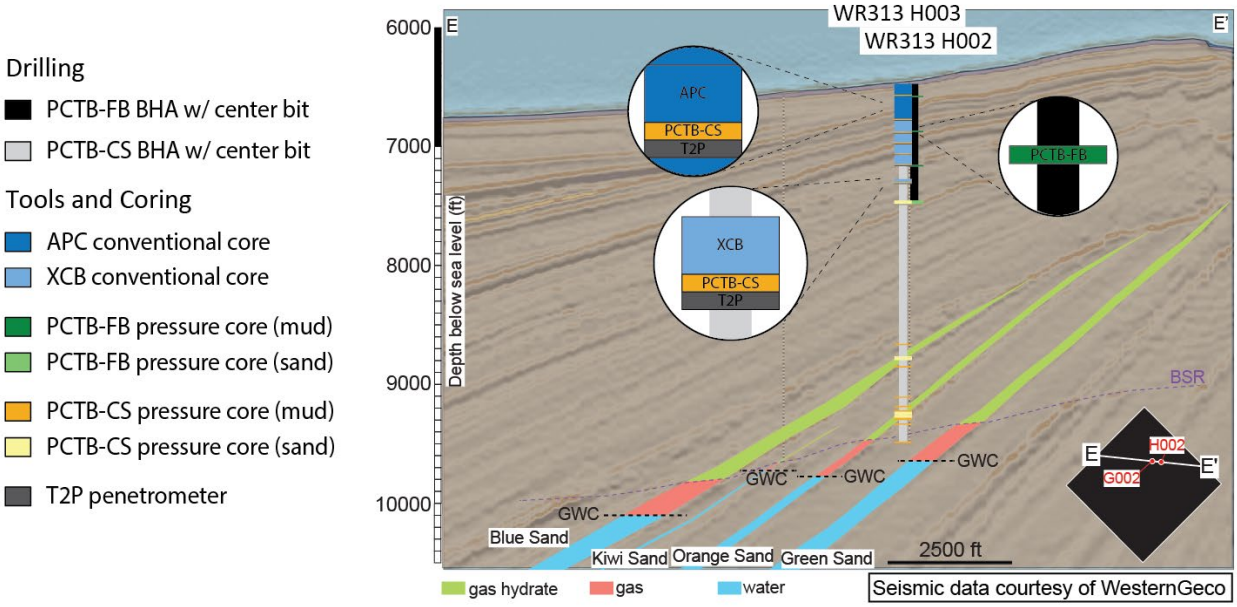

Figure 5-2. Seismic section EE', with graphical representation of UT-GOM2-2 drilling and coring plan for WR313 H002 and WR313 H003. Not to scale.

5.2.3. Schedule

The estimated duration of the UT-GOM2-2 maximum drilling and coring field program is 62.5 days or almost 9 weeks (Table 5-2). Personnel and equipment will be mobilized from the port-of-call to the drilling vessel and preparations will be made for the field science program (3.8 days). Wireline tool full function tests will be completed (0.4 days). Drilling, coring, in-situ measurements, and the on-board science program will be performed at WR313 H003 (16.9 days), and WR313 H002 (3.2 days). Personnel and equipment will be demobilized from the vessel (3.2 days) and remobilized to the dockside core processing location in Salt Lake City, UT (5.0 days). Geotek will image and log the whole round conventional and conventionalized cores (~14 days). The science party will then regroup and conduct the dockside core analysis and science program (~16 days). After the completion of the science party, Geotek will continue to scan split cores.

Table 5-2. UT-GOM2-2 anticipated field program schedule.

No.	TASK	LOCATION	ESTIMATED DURATION (Days)	CUMULATIVE DURATION (Days)
1	Mobilization	Port of Embarkation	3.8	3.8
2	Full Function Tests	Walker Ridge 313	0.4	4.2
2	WR313 H002 Coring Program*	Walker Ridge 313	16.9	21.1
3	WR313 H003 Coring Program*	Walker Ridge 313	3.2	24.3
4	Stage 1 Demobilization	Walker Ridge 313	3.2	27.5
5	Transit and Remobilization	Port of Disembarkation	5.0	32.5
6	Dockside Core Logging	Salt Lake City, UT	14.0	46.5
7	Dockside Science Party	Salt Lake City, UT	16.0	62.5
	* From _WR313-H002 MAXIMUM; includes pre-tour safety meeting and 20% non-productive time			

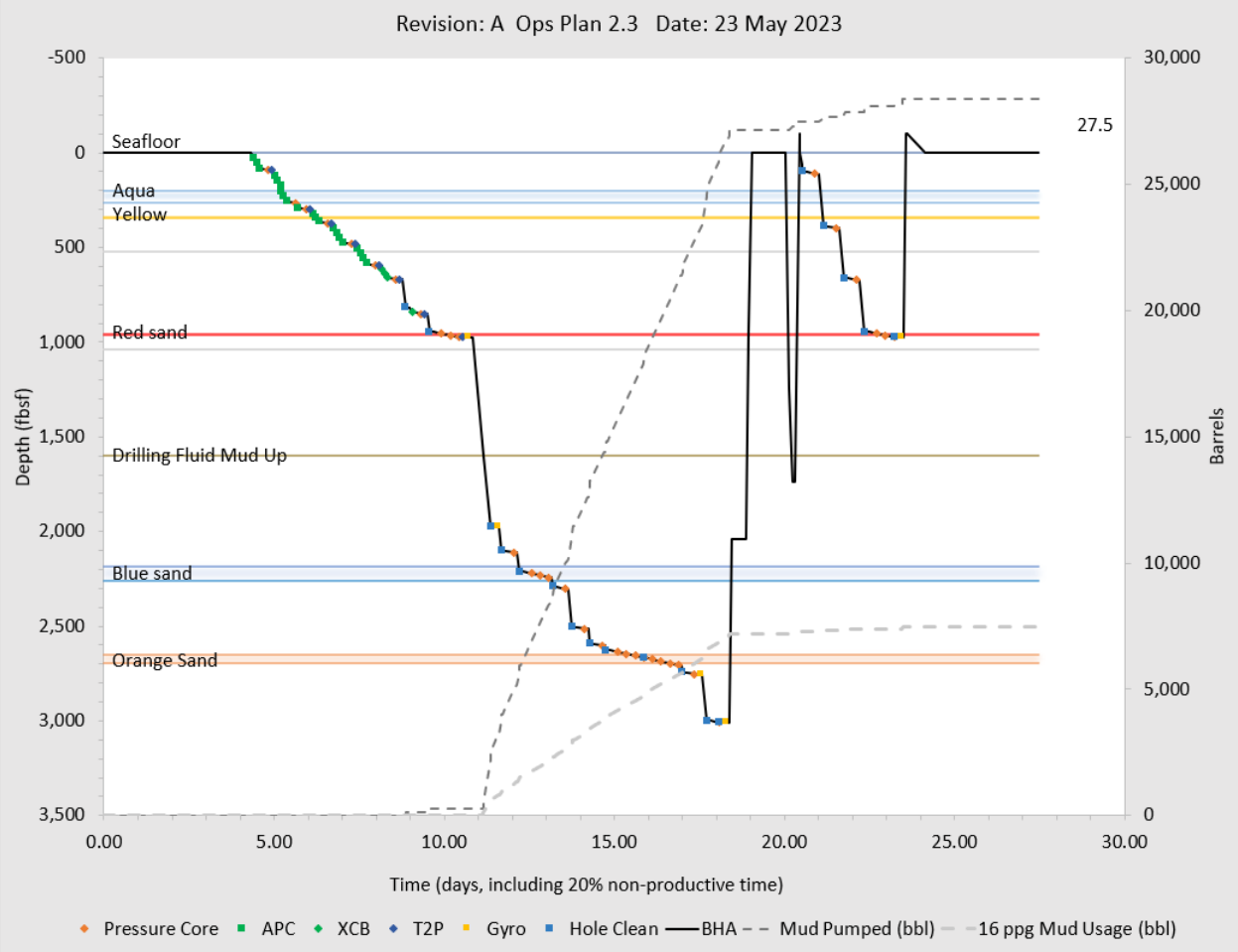

Figure 5-3. UT-GOM2-2 anticipated depth with time. H003 will be drilled with the PCTB-CS BHA. We will acquire Advanced Piston Corer (APC) and extended Core Barrel (XCB) conventional cores, PCTB-CS pressure cores, and in situ pressure/temperature measurements. Pressure cores will be acquired in the Red, Upper Blue, and Orange sands and at other intermittent locations throughout the hole. H002 will be drilled solely with the PCTB-FB BHA (bottom-hole assembly).

5.3. Key Science Equipment, Containers, and Science Providers

The operation plan includes two tables outlining the required containers, container providers, and container activities. These tables are also found in this document in more detail. See Section 8.

Science Containers, Equipment and Personnel.

6. Coring Plans

This section outlines the coring targets and depths required to capture those targets assuming the maximum and most-likely drilling and coring programs. On-Board scientists will correlate acquired cores to logging data so that real time adjustments to coring depths can be made during drilling.

6.1. H003 Planned Core Depths

At H003 25 cores will be taken with the Advanced Piston Corer (APC) or Extended Core Barrel (XCB) conventional coring tool, there will be up to 8 T2P penetrometer deployments, and 11 pressure cores will be taken with pressure coring tool (Table 6-1).

We will take conventional core starting with the Advanced Piston Corer (APC) and transition to taking conventional core with the extended core bit (XCB) (12.1.A.1.3 APC and XCB Coring) with continuous coring down to 670 fbsf. At approximately every 100 ft we will stop conventional coring, take a pressure core, and make a pressure and temperature measurement with the T2P penetrometer (12.1.A.2.1). The initial pressure cores will be used to correlate the depth and interpret the dissolved methane.

We will use these cores to make our first measurements of dissolved methane concentration. We will use this measurement to estimate the depth at which the pore water is saturated with methane, which indicates the depth that hydrate may form.

We will monitor the conventional core for signs of dissolved methane which might indicate that we have crossed the sulfate-methane transition (SMT). We will take an additional pressure core around 165 fbsf. Pressure coring in shallow depths will be done using the PCTB with very low flow rates and minimal to no rotation.

For each T2P deployment we may acquire sediment samples using thin Shelby tubes (See Appendix 12.1.A.2.1 Shelby tubes).

At the Red sand, we will acquire a series of three pressure cores to capture the sand and surrounding mud. At the Blue sand, we will acquire a series of up to 3 pressure cores within the sand.

The target core length for each pressure core is 10 ft, each APC core is XCB core is ~25 ft with the possibility of lengthening it to 31’ on-board. The maximum total core length for WR313-H003 is 280 ft (85.0 m) of pressure core and 625 ft of conventional core assuming 100% successful coring runs and 100% recovery. Table 6-2 shows the Coring Depths, expected lengths, and the expected core sediment type. The table and other details can be found in the source file, Coring Plan v2.3 program working-2023-05-11.xlsx.

Table 6-1. WR313-H003 Coring and T2P Depths, expected lengths . Full details and depths in meters can be found in the source file, Coring Plan v2.3 program working-2023-05-11.xlsx. Blue rows conventional cores. Yellow rows PCTB-CS pressure cores. Orange rows are the locations of temperature and pressure probe deployment. White rows are intervals without coring. Zoom in to read values.

Core Name	Expected Core Type	Comments	Coring Tool	Interval / Core Top	Interval / Core Bottom	Interval / Core Length	Cum PC Recovery	Cum CC Recovery
				fbsf	fbsf	ft	ft	ft
WR313-H003-01H	Background Mud		APC	0	27	27	0	27
WR313-H003-02H	Background Mud		APC	27	55	28	0	55
WR313-H003-03H	Background Mud		APC	55	83	28	0	83
WR313-H003-04CS	Background Mud	Top at 83 FBSF (Target range 63-93), possibly Low Density (not clear), no hydrate-filled fractures	PCTB-CS	83	93	10	10	83
WR313-H003-T2P93	Background Mud	Good, no sand	T2P	93	93	0	10	83
WR313-H003-05H	Background Mud		APC	93	120	27	10	110
WR313-H003-06H	Background Mud		APC	120	147	27	10	137
WR313-H003-07H	Background Mud		APC	147	174	27	10	164
WR313-H003-08H	Background Mud		APC	174	201	27	10	191
WR313-H003-09H	Background Mud	Aqua top 201.5	APC	201	228	27	10	218
WR313-H003-10H	Background Mud	Aqua	APC	228	255	27	10	245
WR313-H003-11CS	Background Mud	Top at 255 FBSF (Target 255 exact as possible), Low/High density transition, no hydrate-filled fractures	PCTB-CS	255	265	10	20	245
WR313-H003-12H	Background Mud	Aqua base 264	APC	265	290	25	20	270
WR313-H003-13CS	Background Mud	Top at 290 FBSF (Target range 270-295), High Density, no hydrate-filled fractures	PCTB-CS	290	300	10	30	270
WR313-H003-T2P300	Background Mud	Good, no sand	T2P	300	300	0	30	270
WR313-H003-14H	Background Mud		APC	300	321	21	30	291
WR313-H003-15H	Background Mud	Yellow top is 333	APC	321	342	21	30	312
WR313-H003-16H	Background Mud	Yellow base is 344	APC	342	364	22	30	334
WR313-H003-17CS	Background Mud	Top at 364 FBSF (Target 364 exact as possible), Low/High Density transition, no hydrate-filled fractures	PCTB-CS	364	374	10	40	334
WR313-H003-T2P374	Background Mud	Good	T2P	374	374	0	40	334
WR313-H003-18H	Background Mud		APC	374	399	25	40	359
WR313-H003-19H	Background Mud		APC	399	424	25	40	384
WR313-H003-20H	Background Mud		APC	424	449	25	40	409
WR313-H003-21H	Background Mud		APC	449	474	25	40	434
WR313-H003-22CS	Background Mud	Top at 475 FBSF (Target 475 exact as possible), Low/High Density transition, no hydrate-filled fractures	PCTB-CS	474	484	10	50	434
WR313-H003-T2P484	Background Mud	Good	T2P	484	484	0	50	434
WR313-H003-23H	Background Mud		APC	484	509	25	50	459
WR313-H003-24H	Background Mud		APC	509	534	25	50	484
WR313-H003-25H	Background Mud		APC	534	559	25	50	509
WR313-H003-26H	Background Mud		APC	559	585	26	50	535

Core Name	Expected Core Type	Comments	Coring Tool	Interval / Core Top	Interval / Core Bottom	Interval / Core Length	Cum PC Recovery	Cum CC Recovery
				fbsf	fbsf	ft	ft	ft
WR313-H003-27CS	Background Mud	Top at 585 FBSF (target range 585-605), High Density, with hydrate-filled fractures	PCTB-CS	585	595	10	60	535
WR313-H003-T2P595	Background Mud	Good, no sand	T2P	595	595	0	60	535
WR313-H003-28X	Background Mud		XCB	595	616	21	60	556
WR313-H003-29X	Background Mud	Good, no sand	XCB	616	638	22	60	578
WR313-H003-30X	Background Mud	Good, no sand	XCB	638	660	22	60	600
WR313-H003-31CS	Background Mud	Top at 660 FBSF (target range 640-680), High Density, with hydrate-filled fractures	PCTB-CS	660	670	10	70	600
WR313-H003-T2P670	Background Mud	Good, no sand	T2P	670	670	0	70	600
WR313-H003-	NA		Drill /wash	670	815	145	70	600
WR313-H003-32X	Background Mud	Good, no sand	XCB	815	840	25	70	625
WR313-H003-33CS	Background Mud	Top at 840 FBSF (target range 820-860), High Density, with hydrate-filled fractures	PCTB-CS	840	850	10	80	625
WR313-H003-T2P850	T2P	Good, no sand	T2P	850	850	0	80	625
WR313-H003-	NA		Drill /wash	850	945	95	80	625
WR313-H003-34CS	Bounding Mud	Red top is 958	PCTB-CS	945	955	10	90	625
WR313-H003-35CS	Sand	Red Sand	PCTB-CS	955	965	10	100	625
WR313-H003-36CS	Bounding Mud	Red bottom is 966	PCTB-CS	965	975	10	110	625
WR313-H003-T2P975	T2P	Check	T2P	975	975	0	110	625
WR313-H003-	NA	Course grain interval 2017-2042	Drill /wash	975	2100	1125	110	625
WR313-H003-37CS	Background Mud		PCTB-CS	2100	2110	10	120	625
WR313-H003-	NA	H001 Upper Blue top is 2180	Drill /wash	2110	2212	102	120	625
WR313-H003-38CS	Sand	Upper Blue	PCTB-CS	2212	2222	10	130	625
WR313-H003-39CS	Sand	Upper Blue	PCTB-CS	2222	2232	10	140	625
WR313-H003-40CS	Sand	Upper Blue	PCTB-CS	2232	2242	10	150	625
WR313-H003-	NA	Upper Blue sand bottom is 2256	Drill /wash	2242	2292	50	150	625
WR313-H003-41CS	Background Mud		PCTB-CS	2292	2302	10	160	625
WR313-H003-	NA		Drill /wash	2302	2504	202	160	625
WR313-H003-42CS	Background Mud		PCTB-CS	2504	2514	10	170	625
WR313-H003-	NA		Drill /wash	2514	2592	78	170	625
WR313-H003-43CS	Background Mud	The 15m diffusion point above Orange is 2593 FBSF	PCTB-CS	2592	2602	10	180	625
WR313-H003-	NA		Drill /wash	2602	2626	24	180	625
WR313-H003-44CS	Bounding Mud	The 5 m diffusion point above Orange is 2625 FBSF	PCTB-CS	2626	2636	10	190	625
WR313-H003-45CS	Sand		PCTB-CS	2636	2646	10	200	625
WR313-H003-46CS	Sand	Orange top is 2642	PCTB-CS	2646	2656	10	210	625
WR313-H003-47CS	Sand	Orange sand	PCTB-CS	2656	2666	10	220	625
WR313-H003-48CS	Sand	Orange sand	PCTB-CS	2666	2676	10	230	625
WR313-H003-49CS	Sand	Orange sand	PCTB-CS	2676	2686	10	240	625
WR313-H003-50CS	Bounding Mud	Orange bottom is 2686	PCTB-CS	2686	2696	10	250	625
WR313-H003-51CS	Bounding Mud	The 5 m diffusion point below the Orange is 2703 FBSF	PCTB-CS	2696	2706	10	260	625
WR313-H003-	NA		Drill /wash	2706	2743	37	260	625
WR313-H003-52CS	Background Mud	The 15 m diffusion point below Orange is 2755 FBSF	PCTB-CS	2743	2753	10	270	625
WR313-H003-	NA		Drill /wash	2753	3000	247	270	625
WR313-H003-53CS	Background Mud	On-board move top to 2950 FBSF to drop gyro at 2753, the diffusion point 45m below Orange is 2854 FBSF, BSR is estimated at 2935 FBSF	PCTB-CS	3000	3010	10	280	625

6.2. H002 Planned Core Depths

A maximum of 6 pressure coring tool deployments are planned (Table 6-2) in H002. Three in the shallow mud interval which will be used to improve our dissolved methane profile. And, three to capture the Red sand and bounding mud.

The target core length for each pressure core is 10 ft. The maximum core length recovered for H002 is 60 ft (18.3 m) assuming 100% successful coring and 100% recovery. Table 6-1 shows the coring depths, expected lengths, and the expected core sediment type. The table and other details can be found in the source file, Coring Plan v2.3 program working-2023-05-11.xlsx.

Table 6-2. H002 Coring Depths and expected lengths . Full details and depths in meters can be found in the source file, Coring Plan v2.3 program working-2023-05-11.xlsx. Green rows are PCTB-FB pressure cores. White rows are intervals without coring. Zoom in to read table values.

Core Name	Expected Core Type	Comments	Coring Tool	Interval / Core Top	Interval / Core Bottom	Interval / Core Length	Cum PC Recovery	Cum CC Recovery
				fbsf	fbsf	ft	ft	ft
WR313-H002-	NA		Drill /wash	0	100	100	0	0
WR313-H002-01FB	Background Mud	High Density, Range 80-120 FBSF, no hydrate-filled fractures	PCTB-FB	100	110	10	10	0
WR313-H002-	NA		Drill /wash	110	389	279	10	0
WR313-H002-02FB	Background Mud	High Density, Range 360-400 FBSF, no hydrate-filled fractures	PCTB-FB	389	399	10	20	0
WR313-H002-	NA		Drill /wash	399	663	264	20	0
WR313-H002-03FB	Background Mud	High Density, Range 643-683 FBSF, with hydrate-filled fractures	PCTB-FB	663	673	10	30	0
WR313-H002-	NA		Drill /wash	673	945	272	30	0
WR313-H002-04FB	Bounding Mud	Red top is 958	PCTB-FB	945	955	10	40	0
WR313-H002-05FB	Sand	Red Sand	PCTB-FB	955	965	10	50	0
WR313-H002-06FB	Bounding Mud	Red bottom is 966	PCTB-FB	965	975	10	60	0

6.3. Quantity of Pressure and Conventional Core

The maximum total length of pressure core that could be recovered in WR313-H002 and WR313-H003 is 340 ft (103.6 m). This calculation assumes 100% successful coring runs, and 100% recovery. This is the maximum amount of core that will need to be logged using the PCATS Quick Scan method (method details are below in Appendix A). The maximum total length of conventional core that will be recovered for WR313-H002 and WR313-H003 is 625 ft (190.5 m). This calculation assumes 100% successful coring runs, and 100% recovery. This is the maximum amount of core that will be logged using the Geotek IR scanners. Table 6-3 outlines the various estimates of pressure and conventional core considering core type, core quality, recovery, PC success.

Table 6-3. Estimated total amount of pressure and conventional core based on core type, pressure coring run success (core is sealed and held at a pressure within the hydrate stability zone), and core

recovery (% of sediment to length of coring interval). The amount of conventional core to process will increase assuming depressurized core are treated as conventional core.

Coring Hole(s)	PC Success	Recovery	Total Pressure Core	Total Conventional Core
	%	%	ft	ft
H002	100	100	60	0
H002	80	100	48	12
H002	70	80	38	10
H003	100	100	280	625
H003	80	100	224	681
H003	70	80	179	545
TOTAL	100	100	340	625
TOTAL	80	100	272	693
TOTAL	70	80	218	554

6.4. Quantity and type of Pressure Core sections to bring back to UT

Sections of 1.0 m each from the recovered pressure core will be brought to UT for geomechanical testing. Enough pressure core sections from each target of interest must be brought back to UT to meet the prioritized science objectives (see Section 3. Scientific Objectives). Table 6-4 presents the planned number of pressure core sections per target of interest. The plan will be adjusted during the expedition based on the success of pressure coring and the quality of pressure core obtained.

Table 6-4. Estimated minimum amount of pressure core to bring back to UT. A. Target of interested as described in Sections 6.1 and 6.2. B. Number and feet (assuming 100% recovery) of planned pressure cores. C. Sand thickness of each target. D. Maximum amount of recovered sand pressure core. F. Maximum amount of recovered mud (either background or reservoir bounding mud). G. Planned number and total feet of sand pressure core sections to bring to UT. H. Planned number and total feet of bounding mud pressure core sections to bring to UT. I. H. Planned number and total feet of background mud pressure core sections to bring to UT. Some trade-offs will be made during the expedition between the different targets and between sand and mud.

A. Target	B. Planned Pressure Cores		C. Sand thickness	D. Sand Recovery	F. Mud recovery	G. Sand Pressure Core to UT		H. Bounding Mud Pressure Core to UT		I. Background Mud Pressure Core to UT	
	# of cores	ft of core	ft	ft	ft	# of sections	ft	# of sections	ft	# of sections	ft
H003 Orange sand and bounding mud	8	80	44	44	36	7	21	2	6	-	-
H003 Blue interbedded sands	3	30	76 with 13 ft of dispersed sand	30, dispersed	-	3	9	-	-	-	-
H002 and H003 Red sand	6	60	8	16	44	2	6	2	6	-	-
H002 and H003 Background Mud	17	170	-	-	-	-	-	-	-	2	6
TOTAL to bring back to UT						12	36	4	12	2	6

7. Core Processing Plan

The following lays out the processing and allocation of core and other samples on-board, dockside, and post-expedition. Additional requests for samples may alter the plan slightly (see Section 10. Requesting Samples and Data).

7.1. On-Board

The following lays out the plan for the processing and allocation of core and other samples on-board. Any steps not completed on-board will be completed dockside. Addition dockside and post-expedition activities are outlined in Section 7.2 Dockside and 7.3 Post-expedition, respectively.

Equipment details and analytical methods can be found in the referenced sections of Appendix A. Expedition sampling handling instructions can be found in the referenced protocol documents.

7.1.1. On-Board Pressure Core

Pressure cores will be acquired, logged, and imaged. Based on these data cores will be cut into sections and those sections will be identified for transport to UT (See Section 7.3.1 UT Pressure Core), quantitative degassing (slow depressurization with the quantification of produced gases), and for rapid depressurization.

7.1.1.1. PCTB Van and laydown area

The PCTB inner core barrel is craned to the PCTB container, where the pressure core chamber is removed from the PCTB inner barrel. The pressure section of the PCTB is checked to ensure that the core has sealed at or above in-situ pressure for background mud cores, and within the hydrate stability zone for hydrate-bearing sand cores. The ball valve is checked.

Poorly sealed cores that are still intact will be treated as conventional cores. Any loose sediment will be bagged for potential grain size analysis, biostratigraphy, CHNS, and other properties.

7.1.1.2. PCATS

The way each pressure core is treated in PCATS, and the resulting core data acquired immediately, will depend on the recovered sediment type and on the resources available (the amount of time between cores, and the number of storage chambers available). The recovery of four types of sediment core are possible with the first being unlikely given our hole cleaning plan: **1) 'fall-in'** cores are any core that may contain detritus accumulated in bottom of hole; **2) 'background mud'** cores are recovered from shales far from reservoir sand; **3)** '**bounding mud**' cores bound sand reservoirs; and **4) 'sand'** cores are from the hydrate reservoirs. The predicted composition of the cores is shown in Table 6-1 and Table 6-2.

More information on the amount of time available can be found in A.3.2.9 PCATS Schedule/Timing. A full analysis of the amount of time available for each pressure core and the planning PCATS processing flow can be found in the expedition Coring plan.

All pressure cores must be cut into sections of 1.2 m or less before demobilization from the vessel.

Step 1. Pressure Core Logging and X-Ray imaging

A. Fall-in pressure cores

In this type of core, a significant amount of the recovered core is composed of detritus that was accumulated in bottom of hole.

We have adjusted the hole cleaning process and are no longer anticipating recovering these types of cores. However, it is still possible that we may encounter a few.

If encountered, Fall-in pressure cores will be quickly scanned (Quick scan, See 12.1.A.3.2.5) and a portion of the core will be fully scanned ('Full scan", See 12.1.A.3.2.6) before cutting. All sections for quantitative degassing (see Step 2) need to be fully scanned. CT imaging is important for quantitative degassing to accurately quantify the volume of the core section being depressurized for accurate calculation of the dissolved methane concentration and/or hydrate-saturation. High-resolution density logs also help improve the accuracy of porosity estimates when MAD is not possible on disaggregated cores post-dissociation.

B. Background Mud pressure cores

Background mud pressure cores are defined as the intermittent pressure cores taken between the reservoir sands. They are selected to represent the background geochemical, geomechanical, and petrophysical properties with depth. They may or may not contain hydrate-bearing fractures and/or thin sands/silts.

Background mud pressure coring is followed by long periods of conventional coring or drilling before another pressure core is attempted (see Section 6. Coring Plan). Thus, there is a lot of time to process these cores before the next core arrives.

Background mud pressure core will be fully scanned ('Full scan", See 12.1.A.3.2.6). After full scans, a plan to section the core will be made (step 2), and the pressure cores will be cut into those sections (Step 3).

C. Sand and Bounding Mud pressure cores

Sand pressure cores are defined as the continuous pressure cores taken from the sand reservoirs as identified in Sections 5.2.1 and 5.2.2. These pressure cores will contain sand and/or interbedded sand and mud. Some, at the top or bottom of the series of pressure cores, may contain sections of the reservoir bounding mud.

There is very little time for on-board processing of many of these cores before the next core arrives.

Sand pressure cores will be quickly scanned (Quick Scan, See Appendix 12.1.A.3.2.5) and some will be cut into sections right away.

Sand pressure core sections will be pushed out of PCATS and into 1.2 m pressure storage chamber (SC_{120}). Uncut sand cores will be pushed into long, 3.5 m temporary pressure storage chamber (SC_{350}, see A.3.1 for more information about these types of core storage chambers). The chamber will be tagged and stored.

The core sections will eventually be brought back from core storage and pulled into PCATS, and fully scanned ('Full scan", see 12.1.A.3.2.6). This may occur on-board or dockside.

If enough time is available, the core will be fully scanned and cut, avoiding the need to be brought back to PCATS later.

Step 2. Core Sectioning and Pressure Core Sampling Plan

The pressure core science team will review each core with Geotek. Geotek will provide an initial recommendation for the section/cut locations based on the data available. UT will make the final decision and the specific plan for the sectioning and allocating of the pressure core will be communicated to Geotek by UT.

A. Identify sections for Transport to UT

1.2 m core storage chambers are the longest chambers that can be transported over land within the US. We will have 1.2 m core storage chambers (SC_{120}) available for transport to UT (see Section 6.4).

A small number of sections (1-3 total) from background and bounding mud pressure cores will be cut and stored for transport to UT for comparing petrophysical and transport properties (See Section 7.3.1 UT Pressure Core for more details). One to three 1.0 m sections of every 10' sand pressure core will be selected for transport to UT (see Section 6.4 for the estimated number for each sand being cored). No fall-in material should be saved for transport to UT.

Sections selected should be from the highest quality core with consistent core diameter, little coring disturbance (long biscuits, no evidence of grooves on core).

B. Identify sections for further PCATS processing

Identify 1.0 m sections of pressure core that still need full scanning and possible additional cutting. These sections will be placed in 1.2 m storage chambers ($SC_{120,}$ See Appendix A.3.1).

C. Identify sections for Quantitative Degassing (6-12 Hour degassing)

10-30 cm sections containing individual lithofacies (as possible) of sand and interbedded mud will be identified for quantitative degassing from the sand pressure core. These sections will be placed in 0.35 m storage chambers ($SC_{035,}$ See Appendix A.3.1).

10-100 cm sections of background and bounding mud will be identified for quantitative degassing. These sections will be placed in 1.2 m storage chambers ($SC_{120,}$ See Appendix A.3.1).

D. Identify sections for very slow degassing (days to weeks)

One or more 15 to 35 cm hydrate-bearing sections will be identified for very slow degassing over several days (<0.5 MPa steps, see 7.2.1.1).

The section(s) selected should be high quality coarse-grained high-hydrate saturation reservoir material where it will be difficult to recover conventional pore water samples. This approach will allow for calculation of the sample salinity based on the pressure and temperature at which hydrate dissociation begins (observed from the onset pressure rebounds during depressurization).

E. Identify sections for liquid nitrogen depressurization

20 cm sections will be identified for LN2 depressurization (or cryo-core, see 7.2.1.2).

One section per core will be frozen before depressurization for microbiology on-board and dockside. One section should be purposefully chosen from a PC in H003 (see 7.2.1 Dockside Pressure Core) for comparing microbiology results from adjacent conventional cores and adjacent sections of quantitatively degassed pressure core. Adjacent sections should be selected for quantitative degassing (Step 2 C.)

F. Identify sections for Rapid Depressurization

20 cm sections of high quality coarse-grained high-hydrate saturation reservoir material will be identified for rapid depressurization for pore water analysis.

Rapid depressurization will occur using one of two rapid degassing methods (See 12.1.A.3.3 Rapid Degassing.

All fall-in material should be rapidly depressurized. Additionally, some sections of pressure core may unexpectedly lose pressure rapidly while in PCATS or storage.

After rapid depressurization, any intact core will be moved to the conventional core flow. Loose sediment will be collected, bagged, labeled and stored for dockside or post-expedition analysis in core storage.

Step 4. Core Section Cutting

The core will be cut into the identified sections. Cut positions may be adjusted after examination of additional X-ray images and p-wave velocity measurements made immediately before cutting if the core material moved inside the liner.

10-30 cm pressurized sections will be placed in 0.35 m storage chambers ($SC_{035,}$ See Appendix A.3.1). Larger sections will be cut and moved SC_{120} storage chambers.

All storage chambers will be pre-fitted with a rabbit with DST, and solid spacers to minimize the total volume of storage fluid to minimize the fluid around the core. The chambers will be tagged with the core name, section, depth reference, DST identifier, and spacer length.

Pressure core sections for quantitative degassing will be brought to the degassing lab (R17). If space is limited they will be temporarily stored in the Core Storage container. Pressure core sections for later LN2 depressurization dockside or transport to UT from SLC will be brought to the Core Storage container.

7.1.1.3. Degassing Lab (R17)

1-3 sections at a time of pressure core will be quantitatively degassed in 6-12 hours on 1 of 3 degassing stations (see Appendix 12.1.A.3.4 Quantitative Degassing for more details).

Most background mud sections will be degassed on-board. Some hydrate-bearing sand sections will be degassed on-board. All remaining sections will be degassed dockside.

Gas will be collected during quantitative degassing (See Gas Collection protocols in UT-GOM2-2 Degassing and Gas Sampling Protocols) and analyzed on-board (See Section 7.1.4 On-Board Gas Analysis) or analyzed post-expedition (see Section 7.3.1 Ohio State Gas and Section 7.3.2 TBD Clumped Isotopes).

As depressurization occurs, high-hydrate saturation and coarse-grained samples will likely not retain their structural integrity, while hemipelagic clay intervals will likely remain intact

and retain reasonable quality. All Intact depressurized cores, no matter how they are depressurized, will be treated as conventional core starting with the on-board conventional core flow Step 2 (see Section 7.1.2 On-Board Conventional Core). All unconsolidated and disturbed sediment will be collected, bagged, labeled, and stored for dockside or post-expedition analysis in the Core Storage container.

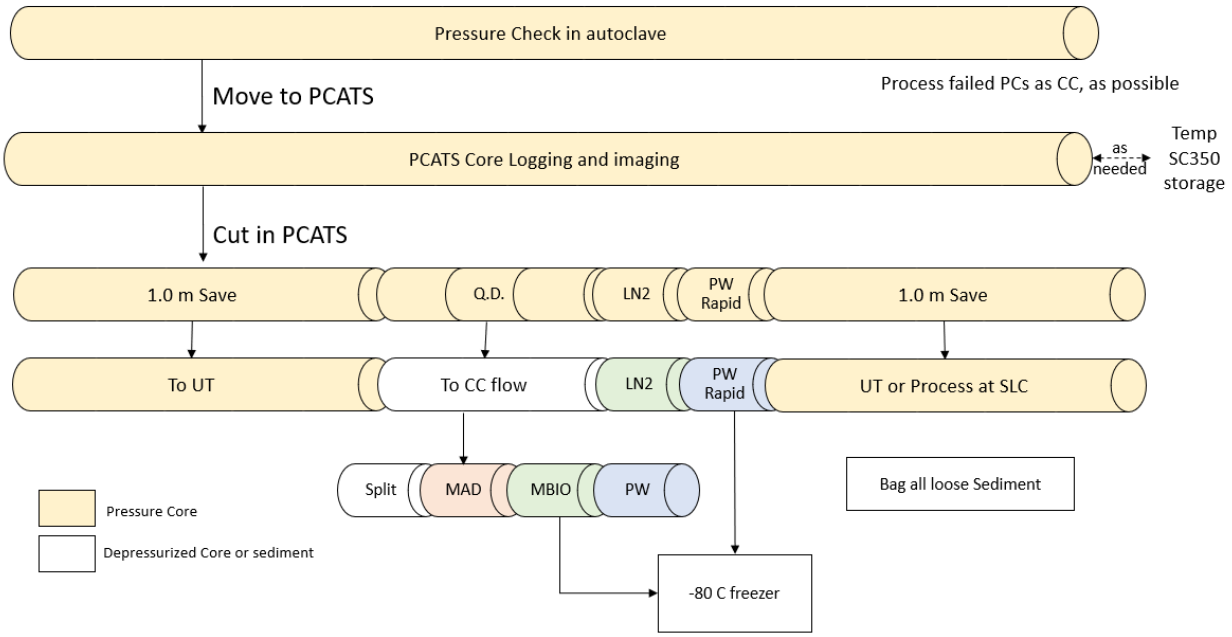

Figure 7-1. Processing of pressure cores. As the core arrives at the Geotek Pressure Core Analysis and Transfer System (PCATs), it will be removed from the pressure core chamber and is imaged and scanned at high resolution (Full scan, see Appendix 12.1.A.3.2.6 PCATS Full Scan Analysis*). If there is not enough time (as with sand pressure cores), an initial core log and image is generated (Quick scan,* see Appendix 12.1.A.3.2.5 PCATS Quick Scan Analysis*), and the full core is transferred to a long (3.5 m) storage chamber or cut and transferred to shorter chambers. As time permits, the core or core sections are brought back to PCATS and the core is fully scanned. From scan data, a core sectioning plan is made. The core is then sectioned and sections are moved into smaller storage and analysis chambers. 1.0 m sections of pressure core will be cut and transferred to UT. 0.1 to 1.0 m sections will be quantitatively degassed (6-12 hour depressurization measuring the amount of gas produced and collecting gas samples for Gas chromatography). Core from slow degassing will be processed as conventional core. Some sections will be very slowly degassed and some depressurized with LN2 (see Section 7.2.1). Core sections for Pore water will be rapidly depressurized. Remaining sections of core including any fall-in will be rapidly depressurized.*

7.1.2. On-Board Conventional Core

APC and XCB conventional cores will be imaged with an IR skate track and sectioned. Conventional and depressurized PCTB (conventionalized) core (7.1.1 On-Board Pressure Core) will be sampled, preserved, and some portion analyzed on-board.

We will do thermal imaging, void gas analysis (C1-C5), core sectioning, whole round core cutting, hand held vane and penetrometer measurements, and headspace gas sampling, pore water squeezing, pore water ephemeral measurements, and pore water sample preservation on all conventional and some depressurized cores on-board.

Conventional cores will not be logged or split on-board (See Section 7.2).

Figure 7-2 shows the core flow for a hypothetical conventional core. Detailed instructions for each step can be found in UT-GOM2-2 DOCKSIDE Science Party Conventional Core Protocols.docx and the Geotek Core Processing protocols.

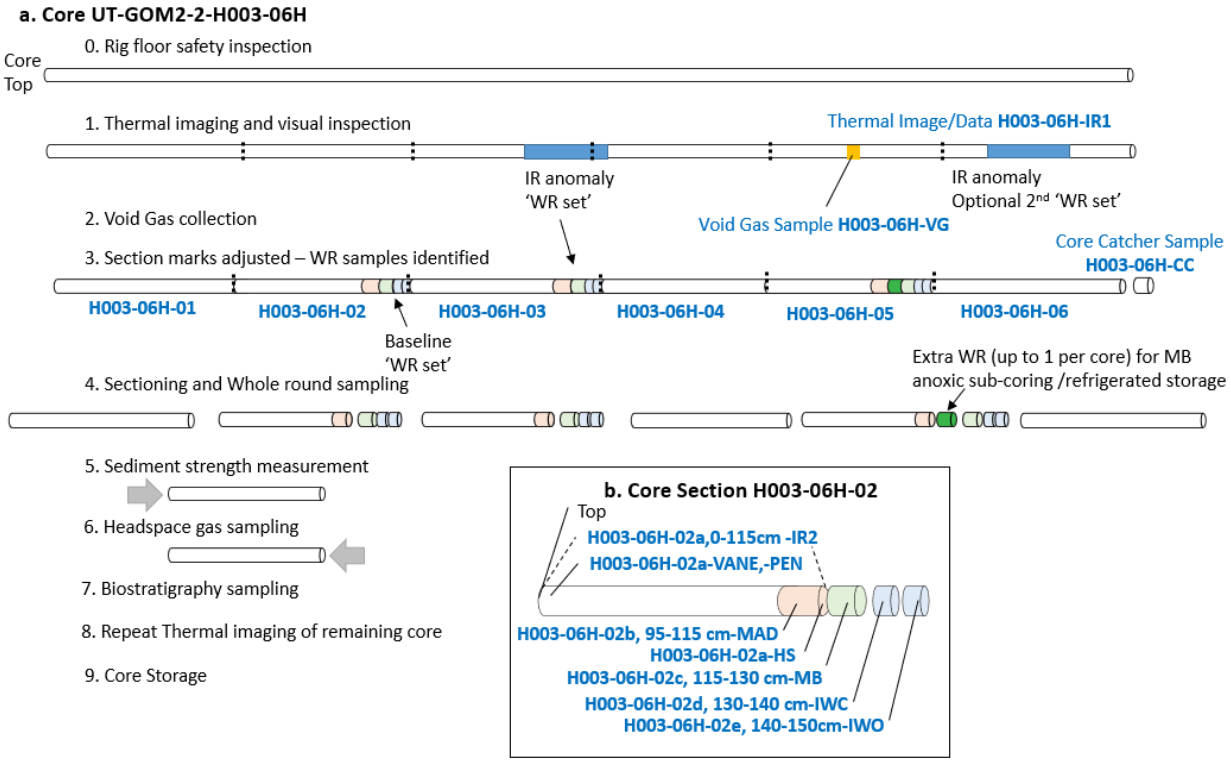

Figure 7-2. Conventional Core Processing. A. ~ 9 m conventional core processing steps for a hypothetical APC/XCB core above the SMT. B. Detailed Section noting whole round set and location of headspace gas sampling, and hand-held vane and penetrometer, and second IR measurement locations.

The processing steps in the core flow are as follows:

7.1.2.1. Conventional Core Laydown area

As APC and XCB conventional cores arrive at the conventional core laydown area, they will be removed from the coring tool, monitored for H2S, and inspected for lengths of expansion/high pressure. Precautions will be taken for the presence of H2S. Precautions will

also be taken as holes are drilled to vent the core liner. The exact protocol for H2S monitoring and core venting will be determined in consultation with the vessel operator.

The core catcher will be curated as a section. If needed, these core catcher samples will be bagged and stored in Core Storage.

7.1.2.2. Core Receiving Lab

As APC, XCB, and conventionalized cores arrive at the Core Receiving Lab they will be laid out and processed according to the following steps.

Step 1. Thermal imaging and visual inspection

APC and XCB Core will be thermally imaged using the Geotek MSCL-IR thermal imaging system with skate track (See Appendix 12.1.A.4.1 Thermal Imaging for details). Initially, 1.5 m long sections (6 sections in every 9 m core) will be marked out but not cut. Science party members will visually inspect the core through the liner for features of interest and any coring damage.

Step 2. Void Gas collection

Free gas trapped within the core liner will be collected. A subset of the gas will be used for an initial analysis of C1-C5 hydrocarbons on-board (See Section 7.1.4 On-Board Gas Analysis). The remainder will be transferred to pre-evacuated copper tube or vials to eventually be shipped to Ohio State for C1-C6 hydrocarbons, CO_2, noble gases, and Isotopic analysis (see 7.3.1 Ohio State Gas and Section 7.3.2 TBD Clumped Isotopes). For planning purposes, we estimate to collect 2 void gas sample sets for every APC core below the SMT and 1 set for every XCB. Samples for Ohio State will be labeled and stored in Core Processing.

Step 3. Core Section and whole round sampling planning

The geochemistry and microbiology team, with Geotek, will finalize a plan for the sectioning and sub-sections of core. Additional sections/cuts may be identified and adjustments made to the original 1.5 m core section size / cutting positions based on thermal images and visual inspection mentioned above. Locations of whole round sample sets including whole rounds for Pore Water, Microbiology, Moisture and Density (MAD), and physical properties will be identified (see Figure 7-2 Step 3).

Conventionalized cores may be very short and these core should be left for analysis dockside.

A. Pore Water

Pore water whole round samples will be cut from the deeper end (bottom) of the core sections (Figure 7-2, B).

Two 10 cm whole round samples for pore water analysis will be cut from the deeper end of each 1.5 m section (~6 sets per every 9 m) to depths just below the estimated SMT as part of the whole round set. The higher sampling rate will capture the expected rapid change in geochemical properties just below the seafloor.

In deeper conventional cores, two 10 cm samples from APC cores and one 15 cm or longer whole round samples from XCB cores for pore water extraction will be cut from the deeper end of approximately every third section (~2 sets/samples per every 9 m). Additional pore water samples will be taken from sections with IR

anomalies. For planning purposes, we assumed to generate five 10 cm samples (two-three sets) for every APC core of this type, and two to three 15+ cm samples from XCB cores.

Two 10 cm samples may be cut from depressurized cores in shallow depths similar to those for APC coring. 15 cm or longer whole round samples may be cut from deeper depressurized core sections.

Pore Water core whole rounds will be capped and the caps will be secured to the liner with electrical tape. Samples will be immediately be labeled, placed in a bucket, and transferred to the Pore Water Lab where they will be trimmed and squeezed on-board.

Ephemeral measurements of pore water alkalinity and pH will be made. Additional pore water will be preserved for various chemical analysis at the University of Washington. See Section 7.1.5 On-Board Pore Water below.

B. Microbiology

Microbiology whole round samples will be cut from the deeper end of the core section directly adjacent to the Pore Water sample as part of the whole round set (Figure 7-2, B).

One 15 cm whole round sample for microbiology will be cut from each 1.5 m section (~6 per every 9 m) from the seafloor to depths just below the estimated SMT.

In deeper conventional cores, one 15 cm whole round sample will be cut from every third section (~2 per every 9 m) directly adjacent to the MAD/physical properties sample for

Additional special microbiology samples may be cut from sections with IR anomalies. For planning purposes, we assumed to generate two and a half 15+ cm samples for every core of this type.

15 cm or longer whole round samples may be cut from depressurized cores for microbiology. Longer cores might be required from deeper depressurized cores as the microbial counts are expected to be low. As possible, one sample of each lithofacies should be cut from each depressurized core section.

A 15 cm section of depressurized sediment from a shallow pressure core in H003 will be cut and preserved in order to make a microbiology comparison between conventional and pressure cores.

Additional whole round samples may be cut based on the needs as specified in possible sample requests.

Microbiology samples will immediately be capped, sealed with electrical tape, labeled, and bagged.

Some microbiology whole rounds may be placed in the refrigerator in the Core Processing lab until they can be sub-cored. Sub-coring will be done in the lab in a flow hood or in the glove box under a sterile, anoxic environment, sediment will be removed from the center of whole rounds. Sub-cored and rind sediment will be separated, sealed, and labeled prior to storage and shipping. Most Microbiology samples will be placed in the -80 C freezer in the Core Processing Lab right away.

Microbiology analysis will be done at Oregon State University and possibly Dauphin Island Sea Lab, Univ of Alabama (See Section 7.3.6 Oregon State Microbiology and Appendix 12.1.A.6 Oregon State Microbiology of Conventional Core).

C. Moisture and Density, Physical Properties (MAD)

20 cm Moisture and Density (MAD) whole round samples will be marked for cutting at SLC from the deeper end of the core section directly adjacent to the microbiology sample (Figure 7-2, B).

Whole round MAD samples will be marked for cutting at SLC from each 1.5 m section (~6 per every 9 m) of APC cores taken at the seafloor to depths just below the estimated SMT. The increased sampling rate will capture the expected rapid decrease in porosity just below the seafloor.

In deeper cores, one 20 cm (MAD) sample will be marked for cutting at SLC from every third section (~2 per every 9 m). Additional MAD samples may be taken from sections with IR anomalies. For planning purposes, we assumed to generate two and a half 20 cm samples for every core of this type.

Step 4. Core Section and Whole Round Cutting

Cores will be cut into sections per the adjusted section marks.

Only selected sections for Pore Water and Microbiology will be cut on-board (Figure 7-2, B). Sections identified for MAD will be cut dockside.

Whole rounds will be removed by the science team and remaining core sections will be used for additional analysis dockside. See Section 7.2.2 Dockside Conventional Core for details.

Step 5. Sediment shear strength

A handheld vane (https://www.humboldtmfg.com/pocket-shear-vane-metal.html) or pocket penetrometer (https://www.humboldtmfg.com/soil-penetrometer-pocket-type.html) measurement will be made in the shallower (top) end of each section (see Figure 7-2, Step 6 and B) with the core oriented horizontally.

Measurements will be made on every core section.

Step 6. Headspace Gas and additional Microbiology discrete sediment samples

Headspace gas and additional microbiology samples will be extracted from the deeper end of the core within the interval marked for MAD whole rounds, adjacent to the microbiology sample (Figure 7-2, B) for every section identified for whole round sampling above (~6 per every 9 m above the SMT and ~2.5 per every 9 m below the SMT, see Figure 7-2, Step 7 and B).

At each spot, four sediment plugs will be collected from the freshly exposed face of the core using 3 mL syringes.

Microbiology samples will be placed in a whirl-pak and stored in the Core Processing Lab refrigerator. Headspace gas samples will be labeled and taken to the Core Processing Lab where two sediment plugs will be extruded into a 30 mL glass vial with 10 mL of 1 M KCl to stop microbial activity. The vial will be purged with nitrogen and sealed. The sample will be labeled and stored upside-down until it can be heated and the headspace

gas extracted for C1-C6, CO_2, and isotopes post-expedition at Ohio State (see 7.3.1 Ohio State Gas). The samples will be labeled and stored upside-down in the Core Processing Lab. The final sediment plug will be treated in the same way, but shipped to USGS Woods Hole.

Plugs will be added to the end of the core where the headspace gas samples were extracted.

Step 7. Biostratigraphy discrete sediment samples

A plug of sediment from the muddiest part of the core catcher section will be extracted for grain size analysis, biostratigraphy, CHNS, and other properties

Step 8. Thermal imaging of remaining Whole Core sections

After all the whole round core samples have been removed, the remaining core will be re-assembled on a half-round core liner with each section in its correct position and with sections of empty whole round liner placed where whole round samples have been cut away. The core will then be run again through the Geotek IR scanner

7.1.2.3. Core Storage

The remaining core is stored in the Core Storage container for later 3D imaging, logging, and split core analysis at SLC.

7.1.3. On-Board Temperature and Pressure

Formation temperature and pressure measurements will be taken with the UT Temperature 2 Pressure (T2P) penetrometer (see Section 6.1and Appendix (12.1.A.2.1) and the IODP temperature sensor within the APC (APCT-3, see Appendix 12.1.A.1.3 APC and XCB Coring) in the first hole, H003.

Formation temperatures will also be taken using the APC temperature sensor from IODP, the APCT-3.

In-situ temperature and pressure will also be compared to pressure and temperature measurements using data storage tags (DSTs). Temperature and pressure will be measured in the borehole using DSTs on the PCTB pulling tool. Temperature and pressure will be measured inside the PCTB inner core barrel at two locations: 1) just above the core liner at the top of the core and 2) at the top of the core barrel (Thomas et al., 2020b).

Temperature and pressure may also be measured in gassy sediments using a DST inserted inside one of two APC cutting shoe chisels (see Appendix 12.1.A.1.3 APC and XCB Coring).

A comparison of measured temperatures from GC 955 and expected WR 313 temperatures are discussed in the UT-GOM2-2 Technical report (Thomas et al., 2020a).

7.1.4. On-Board Gas Analysis

Gas samples collected from pressure core depressurization (see Section 7.1.1 On-Board Pressure Core) and void gas (see Section 7.1.2 On-Board Conventional Core), will be analyzed for C1-C5 hydrocarbons using an Inficon Fusion MicroGC gas chromatograph with molecular sieve and PLOT Q columns and thermal conductivity detectors. Methane (C1), ethane, (C2) propane (C3), n-butane (C4), isobutene (iC4), isopentane (iC5), and n-pentane (C5) will be measured. The detection limit for all gases is 10 ppm; the quantification limit is 30 ppm.

Gases from depressurization, void gas, and headspace gas samples will also be preserved for analysis post-expedition (see 7.3.1 Ohio State Gas and Section 7.3.2 TBD Clumped Isotopes). These samples will be stored in copper tubes, or gas bags. Samples will be labeled and stored in tubs and cases in the Core Processing Lab. All samples will need to be secured for supply boat transfer to the port.

7.1.5. On-Board Pore Water

The following outlines the pore water sample processing steps. See UT-GOM2-2 Pore Water Lab Protocols for detailed sample handling instructions.

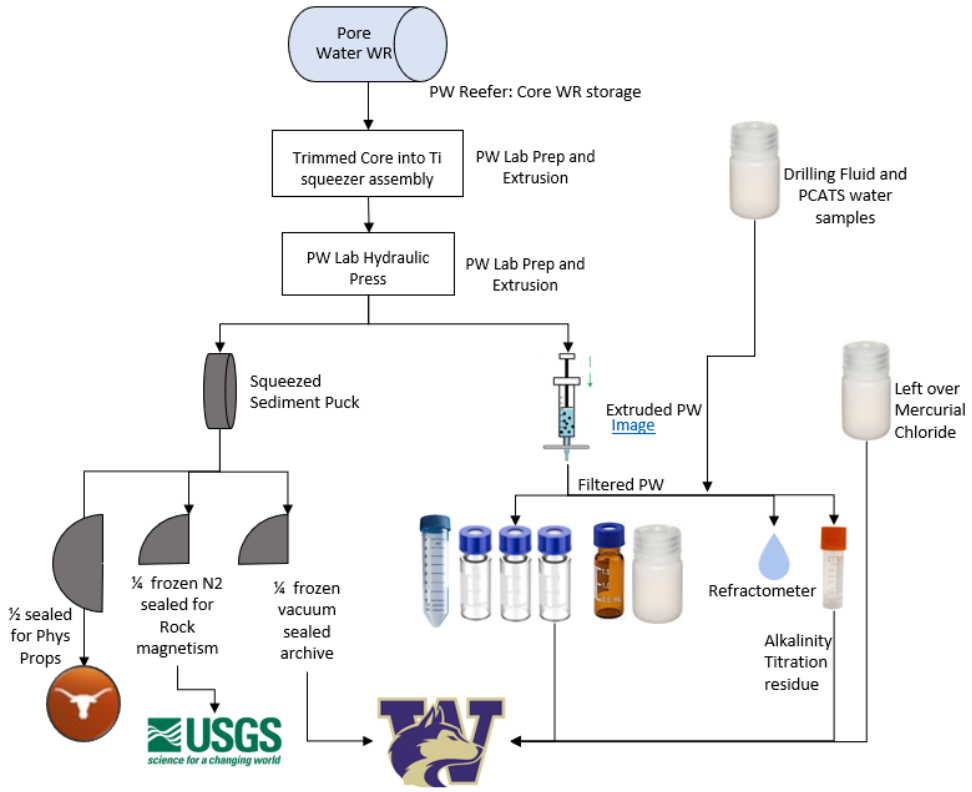

Figure 7-3. Pore Water Processing. Sediment from Pore Water whole rounds will be extracted, trimmed, and squeezed. The remaining sediment puck will be divided for physical properties, rock magnetism, and archiving. The resulting water will be analyzed on-board for salinity and alkalinity, and divided for various analyses at the University of Washington.

7.1.5.1. Pore Water Lab

Step 1. Core Extrusion

Pore water whole rounds will be treated differently depending on the type of coring tool used and the depth they were acquired.

APC mud whole rounds and PCTB whole rounds at APC depths

We anticipate that we will squeeze two separate 10 cm whole round samples to recover a sufficient amount of interstitial water. After squeezing, one of these

whole round samples will be processed for routine geochemical measurements and the other will be processed for organic geochemistry.

APC mud whole rounds will be brought into the refrigerated pore water lab and put into an N_2-filled glove bag to preserve anoxic conditions and limit evaporation. We will first process all the APC samples allocated for routine geochemical analyses before processing the those allocated for organic geochemistry.

Whole round samples will be moved to a second glove bag and extruded from the core liner onto a titanium tray for cleaning. The surface of the extruded core will be carefully scraped with a spatula to remove potential contamination from seawater and drilling fluid. In the second glove bag, trimmed core will then be placed in the pre-flushed Ti squeezer, a piston placed on the top of the sample, and a pre-flushed syringe will be placed into the port. At this point, the Ti squeezer assembly will be removed from the glove bag and taken to the hydraulic press for squeezing.

APC with chisel mud whole rounds

These whole rounds will be treated as APC mud whole rounds above with additional sediment trimmed away from the surface to avoid contamination from any chiseled grooves likely filled with sediment on the exterior of the core.

XCB whole rounds or PCTB mud whole rounds at XCB and deeper depth

XCB mud whole rounds will be brought into the refrigerated lab and placed in the N_2-filled sample storage glove bag to preserve anoxic conditions and limit evaporation.

XCB and PCTB cores do not need to be extruded and cleaned in a glove bag. Whole round cores will be extruded from the core liner onto the titanium cleaning tray on the bench top or using large floor unit. If extruding the core is not possible, the core liner will be cut away.

The surface of the extruded core will be carefully scraped with a spatula to remove potential contamination from seawater and drilling fluid. The trimmed core will then be placed in a Ti squeezer, the sample syringe attached to the squeezer, and the assembly brought to the hydraulic presses in the A/C lab for squeezing.

Sand whole rounds

Pore water whole rounds containing sand bounded by mud may be treated as mud cores per the description above. If the sand is not bounded by mud, special handling may be required to capture pore water that will drain from sand upon extrusion from the core liner. For these WRs, the squeezer assembly with the sampling syringe attached will be placed on the benchtop in the refrigerated lab. The whole round will not be cleaned, but extruded directly into the squeezer. The squeezer assembly will be brought to the hydraulic presses in the A/C lab and processed the same way as the mud cores. Alternatively, the core cap may be punctured and the fluid drained directly into a syringe with a filter attached.

The rind cleaned away from the exterior of each whole round will be bagged, labeled, and kept until processing is complete in the refrigerated section of the Pore Water Lab.

Step 2. Pore Water Squeezing

Ti Squeezers will be carried to one of three manual presses in the A/C section of the lab. The sediments will be squeezed at pressures of up to but not exceeding 30,000 lbs to extract the pore water.

A. Pore Water

During squeezing, pore water is pre-filtered through a prewashed Whatman No. 1 filter placed in the press, above a titanium screen. The extracted pore water will be collected in acid-cleaned plastic syringes. Once the syringe is full or squeezing is complete, a 0.2 μm syringe filter is placed on the syringe and the syringes are stored in a 3rd N_2 filled glove bag in the refrigerated section of the lab until the pore water sample can be subsampled. For the APC whole rounds, the 0.2 μm syringe filter will be flushed with nitrogen before being connected to the syringe.

B. Sediment Squeezed Cake

Sediment squeezed cakes will be removed from the Ti squeezer by removing the base of the squeezer, putting the apparatus on the wood block on the hydraulic press, and pushing the sample out the base with the press. The squeezed cake will then be quartered. Two quarters of the cake will be packed in a heat-sealed bag, labeled, stored in Core Storage, and later shipped to UT. This sediment will be available for physical properties. One quarter will be vacuum sealed, labeled, stored in a -20 C freezer, and later shipped to UW. The final quarter will be heat sealed under nitrogen, labeled, stored in the -20 C freezer in the Core Processing lab, and later shipped in a cooler with freezer packs overnight to USGS Woods Hole.

Step 3. Pore Water allocation plan

The pore water team will assess the volume of pore water extracted from each sample and subsampling for different analyses will be based on the expedition pore water sample allocation plan.

Step 4. Pore Water sampling

Pore water will be divided among the following according to the specific allocation plan for each syringe for the following, as possible. See UT-GOM2-2 Pore Water Lab Protocols for more sample handling details. Unless noted, all power water samples stored in the refrigerator will be stored in the refrigerated pore water lab. Since the refrigerated section of the Pore Water lab will not be powered during supply boat transfer, these samples will need to be secured in Core Storage before transfer to SLC. From SLC, the samples will be shipped in coolers with freezer packs overnight to the University of Washington. All frozen samples will be shipped in LN2 dry shippers overnight.

On-Board and Routine Geochemical Analyses

Table 7-1 outlines the pore water sampling plan for on-board and routine geochemical pore water analysis.

A. Salinity

A drop of pore water will be placed on a temperature-compensated refractometer and the salinity will be measured on-board.

Salinity will be determined with a Reichert temperature-compensated handheld refractometer. The refractometer will be calibrated with IAPSO standard seawater.

B. Alkalinity and pH - IWS

If 15 mL or more of pore water are recovered, a 3.5 mL allocation of pore water will be injected into a 15 mL Falcon centrifuge tube. From the Falcon tube, 3 mL will be pipetted into the titration vessel and analyzed on-board for alkalinity and pH via titration with HCl using the Gran method. Also see Sulphate below.

Total alkalinity is defined as the number of moles of hydrogen ion equivalent to the excess of proton acceptors (bases formed from weak acids with a dissociation constant less than or equal to $10^{-4.5}$ at 25°C) over the proton donors (acids with Ka > $10^{-4.5}$) in one kilogram of sample, such that:

Alkalinity = TA = $[HCO_3^-] + 2[CO_3^{2-}] + [B(OH)_4^-] + [H_3SiO_4^-] + [HPO_4^{2-}] + 2[PO_4^{3-}] + [NH_3] + [OH^-] + [HS^-] + [\text{Org. Acids}] - [H^+] - [HSO_4^-] - [HF] - [H_3PO_4]$

The titrated residue (**IWALK**) will be poured into a 5 mL cryovial. The sample will be labeled and the volume of acid added to the sample during the titration will be noted on the cryovial and in the alkalinity titration notebook. Cryovials will be stored in the refrigerated section of the pore water lab.

Preservation of "routine" geochemical pore water samples

Preserved samples will be analyzed post-expedition at the University of Washington (see 7.3.3 University of Washington Pore Water).

C. δ18O and δD isotopes of pore water - IWOH

1-2 mL allocations of pore water will be preserved in glass vials, labeled, and stored in the refrigerated section of the pore water lab.

D. Halogens and Ammonium - IWHAL

If 10 mL or more of pore water are recovered, 1-2 mL allocations of pore water will be injected into glass vials, labeled, and stored in the refrigerated section of the lab. Samples will be used for post-expedition analysis of

- chlorinity via titration
- ammonium concentrations;
- and as a replicate sample for Br, F, and acetate analyses by ion chromatography.

Note that Cl, Br, and F will be analyzed via IC on the sulfate samples. The precision of Cl determined by titration is better than by IC, which is why we are analyzing by two separate methods.

E. δ^{13}C-DIC – IWDI13C

If 10 mL or more of pore water are recovered, 1-2 mL allocations of pore water will be injected into 2 mL Agilent autosampler vials pre-injected with 10 µL of saturated $HgCl_2$ solution. Samples will be labeled and stored in the refrigerated section of the pore water lab.

F. DIC - IWDIC

If 20 mL or more of pore water are recovered, 1-2 mL allocations of pore water will be preserved in 2 mL Agilent autosampler vials pre-injected with 10 μL of saturated $HgCl_2$ solution. Samples will be labeled and stored in the refrigerated section of the pore water lab.

G. Major/minor elements and isotopes (Na, K, Ca, Mg, Li, B, Si, Ba, Sr, Fe, Mn, δ^7Li, $^{87}Sr/^{86}Sr$, and tracer (Cs)) - IWMAJ

If 3 mL or more of pore water are recovered, 2-15 mL allocations of pore water will be preserved in acid-cleaned HDPE bottles and acidified with Optima grade nitric acid to a pH of 2. Samples will be labeled and stored in the refrigerated section of the pore water lab. Also see sulphate below.

H. Sulfate (SO_4^{-2}) with Cl, Br, and F – IWSO4

If 15 mL or more of pore water are recovered, 0.1 mL of sample will be pipetted from the additional 0.5 mL in the alkalinity and pH Falcon tube housing the alkalinity sample. If pore water recovery is lower, then the 0.1 mL aliquot will be taken from the major/minor element sample above.

The 0.1 mL aliquot will be pipetted into a 15 mL Corning Centristar centrifuge tube containing 10 mL of a 0.5 mM Zn-acetate solution. These samples will be labeled and stored in refrigerated section of the pore water lab.

I. Cl and B isotopes - IWCLISO

If 10 mL or more of pore water are recovered, 2-14 mL Allocations of pore water will be preserved in non-acidified LDPE bottles. The samples will be labeled and stored in the refrigerated section of the pore water lab.

J. DOC, VFAs, and VFA isotopes -IWDOC

2-5 mL allocations of pore water from XCB and PCTB cores will be preserved in pre-combusted glass vials, labeled, and stored in the -20C freezer. Note that for APC cores and depressurized PCTB cores at APC depths, the DOC samples will not be collected from this whole round, but instead be collected with the VFA sample from the organic geochemistry whole round below.

Preservation of organic geochemistry samples from APC Cores

Table 7-2 outlines the pore water sampling plan for organic geochemistry pore water analysis from APC cores.

J. APC DOC, VFAs, and VFA isotopes - IWDOC

2-5 mL allocations of pore water will be preserved in pre-combusted glass vials, labeled, and stored in the -20C freezer.

K. Characterization of DOC pool and organic ligands IWLIG

2-15 mL allocations of pore water will be preserved in acid-cleaned LDPE bottles, labeled, and stored in the -20 C freezer.

L. Trace metals and isotope ratios - IWTRACE

1-20 mL allocations will be preserved in acid-cleaned LDPE bottles and acidified with Optima grade nitric acid to a pH of 2. These samples will be labeled and stored in the refrigerated section of the pore water lab.

Table 7-1. Pore Water sampling plan for on-board and routine geochemical analysis. Zoom in to read table.

Pore Water Allocation - APC/XCB/PCTB Routine Pore Water Geochemistry										
	Personal Samples									
	glass					plastic			shipboard	
	O/H	Halogens	DIC Isotopes	DIC	DOC/VFAs	Majors, Minors, Isotopes	SO4/H2S	Cl+B Isotopes	Alkalinity	Alkalinity residue
code	IWOH	IWHAL	IWDI13C	IWDIC	IWDOC	IWMAJ	IWSO4	IWCLISO	IWS	IWALK
subsample container	2 ml glass vial	2ml glass vial	2 ml agilent vials	2 ml agilent vials	5 ml amber bottles, pre-combusted	4-15 ml Acid-Cleaned Nalgene Bottles	15 ml Corning Centristar Tubes	4-15 ml Acid-Cleaned Nalgene Bottles	14 ml Falcon tubes	5 ml cryovials
treatment	Nothing	Nothing	HgCl2 10 ul	HgCl2 10 ul	Frozen -20C	Acidified to pH2 with Optima HNO3	0.1 ml sample in 10 ml of 0.5 mM Zn-Acetate	Nothing	Nothing	Nothing
45 ml	2.0	2.0	2.0	2.0	5.0	15.0	0.1	14.0	3.0	3.0
40 ml	2.0	2.0	2.0	2.0	5.0	15.0	0.1	8.0	3.0	3.0
35 ml	2.0	2.0	2.0	2.0	5.0	11.0	0.1	8.0	3.0	3.0
30 ml	2.0	2.0	2.0	2.0	5.0	8.0	0.1	6.0	3.0	3.0
25 ml	2.0	2.0	2.0	2.0	2.0	8.0	0.1	4.0	3.0	3.0
20 ml	2.0	2.0	2.0	1.0	2.0	4.0	0.1	4.0	3.0	3.0
15 ml	2.0	1.0	2.0		1.0	4.0	0.1	2.0	3.0	3.0
10 ml	2.0	1.0	1.0			4.0	0.1	2.0		
5 ml	2.0					3.0	0.1			
3 ml	1.0					2.0	0.1			
1 ml	1.0									
Note - APC DOC samples are collected with APC Organic Geochem Whole-Round, Only Collect DOC Samples for XCB and PCTB Cores										

Table 7-2. APC core Pore Water sampling plan for organic geochemical analysis. Zoom in to read table.

Pore Water Allocation - APC Organic Geochemistry				
	glass	plastic		
	DOC/VFAs	Ligands	Trace Metals and Isotopes	SO4/H2S
code	IWDOC	IWLIG	IWTRACE	IWSO4
subsample container	5 ml Amber Glass Bottle (pre-combusted)	4-15 ml Acid-Cleaned LDPE Bottle	4-20 ml Acid-Cleaned LDPE Bottle	15 ml Corning Centristar Tubes
treatment	Frozen -20C	Frozen -20C	Acidified with Optima Nitric to pH 2	0.1 ml sample in 10 ml of 0.5 mM Zn-Acetate
40 ml	5.0	15.0	20.0	0.1
35 ml	5.0	15.0	15.0	0.1
30 ml	5.0	12.0	13.0	0.1
25 ml	5.0	12.0	8.0	0.1
20 ml	5.0	10.0	5.0	0.1
15 ml	2.0	10.0	4.0	0.1
10 ml	2.0	4.0	4.0	0.1
5 ml	2.0	2.0	1.0	0.1

7.1.6. On-Board Drilling Fluid and PCATS Water

Drilling fluid and PCATS confining fluids (PCATS water) samples will be collected and preserved on-board.

We will dope PCATS water with a 10 ppm cesium chloride (CsCl) tracer to allow us to be able to track the total confining fluid contamination during analysis, core cutting, and storage of pressure cores. Drilling Fluid Allocation.

7.1.6.1. On-Board Drilling and PCATS Water Fluid Collection

At least 60 samples of 100 ml of drilling fluid will be collected on-board.

At least 34 samples of PCATS water will be collected from the liquid nitrogen depressurization chamber when PCATS water is replaced with Nitrogen.

Samples will be collected for pore water analysis and microbiology.

A. Pore Water Analysis

Samples will be collected, filtered, and preserved on-board in 50 mL acid-cleaned plastic bottles. Bottles will be labeled and stored in the refrigerated section of the pore water

lab. Samples will be analyzed at the University of Washington to assess the extent of pore water contamination (see Section 7.3.3 University of Washington Pore Water).

B. Microbiology

Samples will be preserved on-board in 50 mL falcon centrifuge tubes unfiltered and immediately labeled and placed in the -80 C Freezer in the Core Processing Lab. Samples will be analyzed for microbial contamination from drilling at Oregon State (see Section 7.3.6 Oregon State Microbiology).

7.2. Dockside

The following lays out the processing and allocation of core and other samples at SLC.

Equipment and analytical method details can be found in the referenced sections of Appendix A. Expedition sampling handling instructions can be found in the referenced protocol documents.

On-board and post-expedition activities are outlined in Section 7.1 On-Board and 7.3 Post-expedition, respectively.

7.2.1. Dockside Pressure Core

All Pressure Core analysis and sampling will be remobilized dockside. Any steps not completed in the on-board pressure core flow as described in Section 7.1.1 On-Board Pressure Core will be completed dockside. The following additional steps will also be completed dockside:

7.2.1.1. Very slow degassing (days to a week)

Hydrate-bearing sections identified to be degassed over several days (<0.5 MPa steps) will be degassed as soon as possible once operations have started. Sample salinity will be calculated based on the pressure and temperature at which hydrate dissociation begins (observed from the onset pressure rebounds during depressurization).

7.2.1.2. Additional Liquid nitrogen depressurization

Pressure core sections identified for LN2 depressurization will be cut, frozen with liquid nitrogen, and depressurized using a special chamber attachment to PCATS as was done on-board. Specific samples may be identified in possible sample requests.

7.2.1.3. Pressure core transport to UT

All pressure core sections for transport to UT will be stored in Geotek overpacks and transported by reefer truck to the UT pressure core center (PCC). See 12.1.A.3.7. Pressure Core Transport over land.

7.2.2. Dockside Conventional Core

MSCL-S (standard MSCL) logging, thermal conductivity, peak and residual strength, X-ray CT imaging, whole round sampling for geomechanical properties, core splitting, and primary split core analysis will be done on conventional core sections previously processed on-board (See Section 7.1.2 On-Board Conventional Core). The dockside core flow is as described below. Additional intact degassed sections (i.e. ‘conventionalized pressure core’) generated dockside will enter the conventional core flow.

Figure 7-3 shows the core flow for a hypothetical 1.5 m core section. The processing steps in the core flow are as follows:

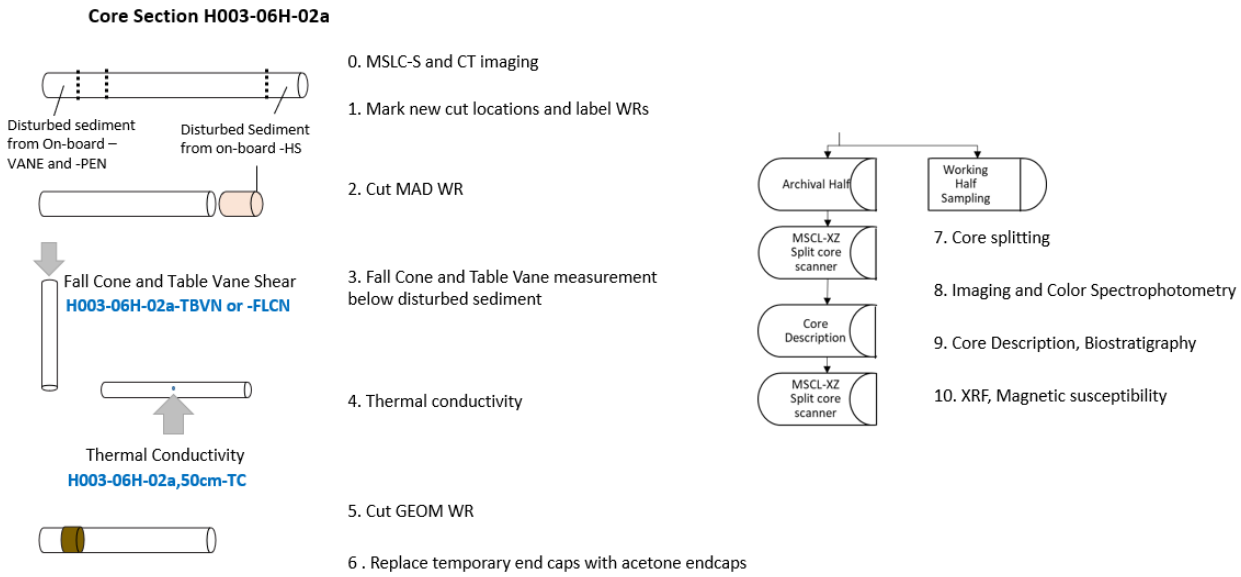

Figure 7-4. Dockside Conventional Core Flow. Conventional and conventionalized core sections will be logged at imaged at SLC. Locations of MAD and Geomechanical whole rounds will be identified from possible sample requests. MAD WRs will be cut and strength and thermal conductivity measurements made before the geomechanical whole round is cut. The section will then be split and described. Discrete sediment samples will be extracted from the working half of the split core section.

7.2.2.1. MSCL Lab

MSCL-S Scanning and CT imaging

Core sections from Core Storage, and conventionalized core sections depressurized and cut dockside will be taken to the MSCL Lab.

In the MSCL Lab, once the core sections are thermally stable, whole round core sections will be logged by Geotek using the Geotek MSLC-S. Logging will include gamma density, P-wave, magnetic susceptibility, resistivity, and natural gamma. See Appendix 12.1.A.4.2 Scanning

Core sections will be imaged with the Geotek CT scanner (see Section 12.1.A.4.3 3D CT).

7.2.2.2. Core Receiving

Step 1. MAD and Geomechanical whole round sample planning

MAD whole rounds sections will be located and geomechanical whole round samples per possible sample request will be identified.

Step 2. MAD whole round cutting

20 cm whole round samples will be cut from conventional and depressurized core sections for MAD as marked on the core liner on-board.

MAD whole round samples will be immediately capped, and the cap secured (core end sealed) with electrical tape to prevent evaporation and retain moisture content. These whole rounds will be labeled, weighed, and packed for shipping(See UT-GOM2-2 Core Receiving Protocols).

All MAD whole round samples will be analyzed for moisture and density and a portion of the sediment will be subsampled for X-ray powdered diffraction (XRPD), CHNS, and grain size distribution by laser particle and hydrometer methods at Tufts University. See Section 7.3.4 Tufts University.

Step 3. Peak and Residual Sediment Strength

Core sections will be oriented vertically with the shallower end up and secured in the miniature vane strength measurement or fall-cone device. Measurements of peak and residual strength will be recorded in the same locations as handheld vane and pocket penetrometer measurements (See Section 7.1.2) from on-board (~ 6 per every 9 m). Measurements will be made using a Fall cone and a Wille Geotechnik fully automated laboratory vane apparatus with a capacity of about 450 kPa. This measurement is based on ASTM D4648. The apparatus is attached to the edge of a lab bench with the vane extending beyond the bench surface. The core section (max length of 1.2 m) is fixed in the vertical orientation with the shallow end on top with a clamp to the side of the bench. The vane is manually inserted several cm into the sediment. A computer controls rotation of the vane and logs force and rotation data. (see UT-GOM2-2 Core Receiving Protocols for more information). Fall-cone measurements will also be made at this same location.

Step 4. Thermal Conductivity

After CT scanning, core sections will be taken to the Core Receiving Lab for measurements of the thermal conductivity. This measurement will be made with a probe mid-section (up to 6 per every 9 m, as time allows without holding up core sections within the flow). The high frequency of the measurements will help identify trends versus scatter in the data from dissolve methane gas expansion. See UT-GOM2-2 Core Receiving Protocols for more information.

Step 5. Geomechanical whole round sampling

MSCL-S and CT images will be inspected and 15 cm whole rounds will be cut portion of the core sections for mechanical testing at Tufts.

Each section will be capped, labeled, and sealed with electrical tape. The sections will be sealed in plastic bags and weighted prior to packing.

MAD and Geomechanical whole rounds will be packed together in coolers with cold freezer packs and shipped to Tufts.

7.2.2.3. Split Core Lab

Step 6. Permanent end caps

Temporary end caps will be removed and replaced with permanent end caps.

Step 7. Core Splitting

Core sections will be split into archival and working halves in a covered area ('tent') just outside the split core lab.

Step 8. Split Core logging

Archival halves will be logged inside using the Geotek MSCL-XZ logger for color spectrophotometry. Images will be captured using the high definition GeoScan Camera. (See 12.1.A.5 Split Core for more details)

Step 9. Core Description, Primary Sedimentology

Archival halves will be placed on a description table next to the sedimentology computer and microscopes. Working halves will be brought inside and placed on a sampling table. Any associated bagged sediment from the core catcher, PCATS, rapid degassing, quantitative degassing, and pore water squeezing will also be brought in from Core Storage.

The archive half of split cores will be used to describe the major and minor lithology, sedimentary structures, bioturbation, colors (Munsell Soil Color Chart), any authigenic nodules, and drilling/coring disturbance. This description will be used to construct lithologic logs that will be used to interpret depositional environment.

Very small sediment samples of major and minor lithology in each core section will be collected using a toothpick from the archive core halves and dispersed on a glass slide and dried. After drying, a cover slip will be adhered to the slide using an optical cement and cured under a UV light. This sample will be described under a petrographic microscope to estimate the abundance of detrital minerals and lithic fragments, microfossils, authigenic minerals, and organic fragments. This petrographic analysis will also estimate the grain size. This semi-quantitative compositional and grain size analysis will be used to classify the sediment type and this information will be integrated with the core descriptions and included in the Lithostratigraphic core descriptions. Slides will be preserved and shipped to UNH. See the UT-GOM2-2 Split Core Sampling Protocols for more details.

Step 10. Discrete samples

Standard and special request discrete samples of sediment from major (fine) and minor (coarse) lithologies will be identified and extracted from the working half of the split core. See the UT-GOM2-2 Split Core Sampling Protocols for more details.

Dockside standard discrete sample set

As the core description and sample identification progresses, flags and other markings will be used to identify sampling locations for one standard set of sediment samples per core section in both the major and minor identified lithologies. The standard discrete set includes laser particle grain size distribution; Course Fraction analysis; CHNS, TOC, and Isotopic analysis; secondary biostratigraphy; rock magnetism; MAD, XRPD, and XRF; additional samples for carbonate nodules (if present), and iron sulfide nodules (if present).

Samples will be removed from the working half and prepared for storage and transport (See Split Core Sampling Protocols) to the University of New Hampshire and other institutions. See 7.3.7 UNH Analysis.

Preservation of

A. Laser particle grain size distribution

2 cm^3 of wet sediment will be identified for laser particle analysis of the grain size distribution at UNH (See 7.3.7.5 UNH Grain Size Distribution by Laser Particle Analysis) of major and minor lithology in each core section and adjacent to other samples, as possible depending on the thickness of the facies. These samples will be collected from representative, lithofacies specific, 1 cm stratigraphic intervals.

B. Coarse Fraction Microscopy

10 cm^3 of wet sediment will be required to sieve a sample for Coarse Fraction Microscopy. Coarse fractions will be flagged. Detrital and authigenic minerals as well as major microfossil groups will be estimated as a percent of this fraction and integrated into the lithostratigraphic core description. These fractions may be used for later post-expedition picking of benthic foraminifers for $\delta^{18}O$ stratigraphy.

C. CHNS, TOC, and isotopic analysis

One 2 cm^3 volume of wet sediment will be identified for CHNS, TOC, and isotopic analysis of C and S (See 7.3.7 UNH Sedimentology), as possible depending on the thickness of the facies.

D. Secondary biostratigraphy

Additional samples will be identified for the observation of marker species and making age assignments. As possible, one 10 cm^3 sample will be collected per core through the continuously cored section, one to two samples will be collected from each background spot core pair in both holes, and multiple samples will be collected from the finer-grained interbeds of each reservoir sand. See 7.3.9 UT Biostratigraphy.

E. Rock magnetism

IODP standard paleomagnetic sample cubes (p-mag samples, 25 x 25 x 19 mm) will be used to sample sediments from the split core surfaces for rock magnetic studies at USGS Woods Hole. These samples will be collected at approximately every 1 m in the same stratigraphic intervals as the UNH CHNS samples. The p-mag samples will be immediately heat sealed with nitrogen to prevent oxidation of the Fe-sulfide fractions. These samples will be labeled, frozen in the -20 C freezer in the Core Processing lab, and later shipped in coolers with freezer packs overnight to USGS Woods Hole.

The results from these samples will be integrated with rock magnetism results from the Pore water residual sediment.

F. MAD, XRPD, and XRD

~8 cm^3 of wet sediment as possible be identified for MAD measurement at Tufts. Results from these samples will be integrated with results from the MAD whole rounds.

After MAD, a few grams of moist material may be identified for X-ray powdered diffraction (XRPD) at James Hutton Institute. The results from these samples would be integrated with XRPD data from the MAD whole rounds.

After MAD, one 2 cm^3 volume of wet sediment will be identified for calibration of the X-ray fluorescence from core scans.

G. Authigenic carbonate and sulfide nodules

Any carbonate or sulfide nodules, if present, will be identified for analysis at UNH (See 7.3.7 UNH Sedimentology).

H. Strength measurements

Shear strength of the sediment will be measured at selected locations on the exposed surface of the split core using either the handheld vane (ASTM D8121) or the pocket penetrometer. These measurements will be compared to the miniature vane strength measurements (see Section 7.1.2 On-Board Conventional Core) and provide information on strength variability within the core.

Special request discrete samples

Additional samples of wet sediment may be extracted from the working half as described in possible sample requests.

A. Additional Grain size and TOC samples

Additional laser grain size and CNHS, TOC, isotope samples will be identified for an Ohio State diffusion study on a specific core section with good recovery where a fine-grained interval either surrounds or bounds a coarse-grained interval. The Red sand and the upper and lower bounding muds are the preferred interval for this study, but another thin sand (1-4 m in thickness) may be identified and used as an alternate. TOC and additional samples will be collected at high frequency (~every 10 cm) in an approximately a 10-meter interval within and surrounding the sand layer.

TOC analysis can be inaccurate due to incomplete removal of the detrital, biogenic, or authigenic carbonate fraction. As such, TOC will be done at UNH where methods have been optimized to remove the carbonate fraction completely (Phillips et al., 2011).

TOC results from these measurements will be integrated with information in the well logs and other core samples.

B. Rock Magnetic analysis of Magnetic susceptibility anomalies

IODP standard paleomagnetic sample cubes (p-mag samples, 25 x 25 x 19 mm) will be used to sample sediments from the split core surfaces for rock magnetic studies at USGS Woods Hole. These samples will be collected from magnetic susceptibility anomalies identified from MSCL-S core logging. The p-mag samples will be immediately heat sealed with nitrogen to prevent oxidation of

the Fe-sulfide fractions. These samples will be labeled, frozen in the -20 C freezer in the Core Processing lab, and later shipped in coolers with freezer packs overnight to USGS Woods Hole.

C. Isotopes of Foraminifers

Two ~10 cm^3 of wet sediment will be identified approximately every 30 cm from 0-68 mbsf to form a foraminifer-based age model (~360,000 years to present).

Additional samples will be collected from the Coarse Fraction. See Standard Discrete sample B.

D. Biogenic Silica

~10 cm^3 of wet sediment will be identified approximately every 30 cm from 0-68 mbsf to investigating the role of meltwater pulses on paleo productivity in the Gulf of Mexico.

Step 11. Magnetic susceptibility, X-ray fluorescence

Archival halves will be logged inside using the Geotek MSCL-XZ logger for magnetic susceptibility and X-ray fluorescence.

Step 12. Split Core Packing

Working and Archival halves will be brought to the core packing table where they will be prepared for transport to UT (See 7.3.8 USGS Rock Magnetics) in the Core Storage container (see UT-GOM2-2 Split Core Sampling Protocols).

7.2.3. Dockside Sediment

Bagged sediment of lithofacies specific depressurized sand cores (see Section 7.1.1 On-Board Pressure Core) will be separated out, sealed, labeled, and shipped to Tufts University to create reconstituted samples for geomechanical testing. See Section 7.3.4.1 Tufts Constant Rate of Strain and Triaxial Testing.

Bagged sediment of lithofacies specific depressurized sand cores will be extracted for all standard split core measurements.

All remaining bagged sediment will be shipped to UT in Core Storage.

7.2.4. Dockside Gas Analysis

Gas samples will be collected and analyzed dockside as was done on-board, as possible. See Section 7.1.4 On-Board Gas Analysis.

Headspace samples for Ohio State will be shipping in robust glass vials. Gas samples from void gas and degassing experiments for Ohio State will be shipped in crimped copped tubes.

7.2.5. Dockside PCATS Water Samples

PCATS Water samples will be collected from pressure core dockside as was done on-board. See Section 7.1.6.

7.3. Post-expedition

The following lays out the processing and allocation of core and other samples post-expedition.

Equipment and analytical method details can be found in the referenced sections of Appendix A.

On-board and Dockside activities are outlined in Section 7.1 On-Board and Section 7.2 Dockside, respectively.

7.3.1. UT Pressure Core

Sections of pressure core will be brought to UT for geomechanical and petrophysical testing. Additional samples will be cut and made available to other institutions.

The follow outlines the flow, allocation, and analysis of pressure core at UT and other institutions.

7.3.1.1. UT Pressure Core Processing

Step 1. Storage

As pressure core arrive from the expedition at UT, pressure cores will be transported to the UT Pressure Core Center (PCC) and placed in storage on the UT Pressure Maintenance and Relief System (PMRS).

Step 2. Allocation Plan

The Expedition Technical Advisory Group (Appendix A) will review the full suite of pressure core data for cores stored at UT. Cores will be compared against approved sample requests and a recommendation for the allocation of pressure core to UT and other institutions will be made to UT. UT will make the final decision and the specific plan for each pressure core will be communicated by UT.

As done on the previous coring expedition, core will be allocated in a manner that maximizes the science that can be achieved at UT and other institutions.

Step 3. High-Resolution Pressure Core Logging and CT imaging

Just prior to sub-sectioning and transfer, pressure cores from storage will be pulled into mini-PCATS, X-ray imaged, logged at high resolution, and CT imaged (3D), (same as the Geotek Full scan (see 12.1.A.3.2.6 PCATS Full Scan Analysis). The full length of core will be imaged.

Logs and images will be compared against previous logs and images.

Step 4. Core Sub-section and Pressure Core Sampling Plan

Pressure core sections will be further divided into sub-sections at UT. Cut positions will be located precisely by comparing the current X-ray image and P-wave velocity measurement with original stored images and data from the expedition. Adjustments may have to be made to the recommended plan from Step 2 if core material is not as expected, has degraded, or has moved inside the liner.

Step 5. Core Sectioning and Sampling Cutting

See Figure 7-4 below. Pressure core in PCATS will be cut into section for permeability and compressibility using Mini-PCATS and subsections moved into the appropriate pressurized analysis chambers.

Additional sections may be cut for microbiology, PCCT analysis, PNATS analysis, Micro CT, ESC, and Micro-Raman, etc. See Appendix 12.1.A.8 through 12.1.A.15.

Mini-PCATS water samples can be collected from Mini-PCATS when Mini-PCATS is emptied after core cutting is complete or from the Mini-PCATS source tank as needed for contamination control.

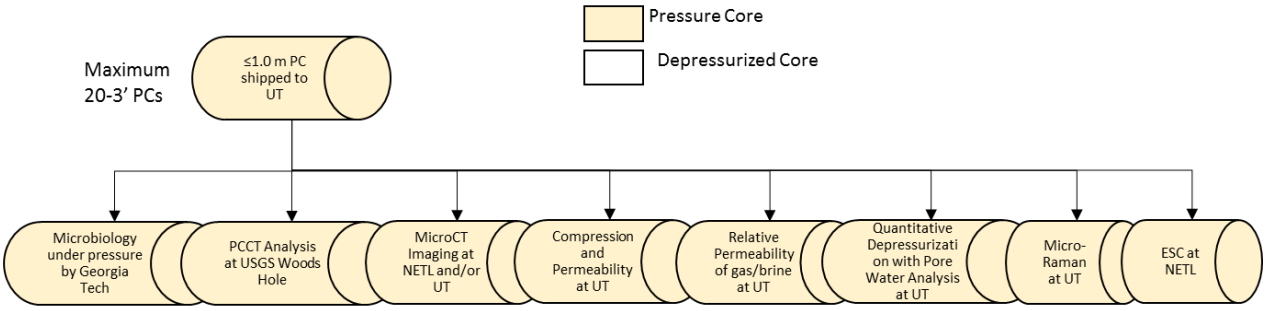

Figure 7-5. Possible Movement and Allocation of Pressure Core Post-expedition from UT. UT Permeability and compression behavior

Pressure core samples will be used by UT to measure compression and permeability using the K0 permeameter. See Appendix 12.1.A.7 UT Compressibility, Permeability of Pressure Core

7.3.1.3. UT Mercury Porosimetry

Pore volume and pore volume distribution with respect to apparent size will be measured using Mercury Intrusion Porosimetry according to ASTM D4404. Samples will be collected from depressurize material or the working half of the cores at UT.

7.3.1. Ohio State Gas Analysis

Void, headspace, and pressure core degassing samples collected on-board and dockside will be analyzed at Ohio State for C1-C6 hydrocarbons, CO_2, and isotopic analysis.

Ohio State will analyze samples from ~30 pressure cores during the same point in the degassing cycle to provide hydrocarbon and noble gas analysis from different reservoirs (including, if pressure core samples are available, the Blue, Orange, Red and Purple sands, as well as the JIP fracture interval).

Ohio State will analyze the composition of background pressure cores to better understand microbial methane production with depth.

7.3.1.1. Hydrocarbons (C1-C6), CO_2, Fixed Gases (N_2, O_2)

Ohio State will analyze hydrocarbon composition (C1 to C6), CO_2, N_2 and O_2 using a combination of: a) SRS Quadrupole MS and b) Trace 1310 Gas Chromatograph equipped with a TCD (Thermal Conductivity Detector) and FID (Flame Ionization Detector) following methods reported previously (Darrah et al., 2014; Jackson et al., 2013; Moore et al., 2018).

7.3.1.2. Noble Gases: ^{4}He, ^{20}Ne, ^{36}Ar, Kr, and Xe

Ohio State will analyze samples collected during a complete degassing of ~2 cores, to look at the variation of gas composition over time. In total, we estimate approximately 45 samples for noble gas geochemistry, 45 samples for hydrocarbon composition and 45 samples of the carbon and hydrogen isotopes of methane and carbon isotopes of CO_2.

Ohio State will purify *in vacuo* (Darrah et al., 2015; Harkness et al., 2017) and analyze noble gases (He, Ne, Ar, Kr, Xe) using a Thermo Fisher Helix SFT mass spectrometer.

7.3.1.3. Isotopes: $\delta^{13}C\text{-}CH_4$, $\delta D\text{-}CH_4$, $\delta^{13}C\text{-}CO_2$

Ohio State will also analyze hydrocarbon composition (C1 to C6) and carbon isotopes from void space and headspace samples. A subset will be measured for H isotopes of methane and C isotopes of CO_2. We estimate we will analyze ~155 void space and headspace gas samples. Pore water and sediment samples will also be analyzed to determine residence time of the fluids, (4 samples of pore water and 4 samples of sediment).

Initial measurements (~20 results) of $\delta^{13}C\text{-}CH_4$ and hydrocarbon gases will be delivered to UT less than 90 days after the samples arrive at Ohio State.

Ohio State will analyze the carbon and hydrogen isotopes of methane ($\delta^{13}C\text{-}CH_4$, $\delta D\text{-}CH_4$) and carbon isotopes of CO_2 ($\delta^{13}C\text{-}CO_2$) of gas samples from pressure cores following methods reported previously (Harkness et al., 2017; Moore et al., 2018) using a Thermo Finnigan Trace Ultra GC, followed by combustion and dual-inlet isotope ratio mass spectrometry using a Thermo Fisher Delta V Plus.

7.3.2. University of Washington Pore Water

Pore water samples will be shipped to the University of Washington.

7.3.2.1. Pore Water $\delta^{18}O$ and δD

The pore water $\delta^{18}O$ and δD isotope ratios will be determined on a Picarro cavity ring-down spectrometer water analyzer at UW.

7.3.2.2. Chlorinity

High precision Cl concentrations will be determined via titration with $AgNO_3$. The average precision of the chloride titrations is typically <0.3%. Note that Cl concentrations determined through this method are actually chlorinity as dissolved Br and I are also precipitated during the titration. Dissolved Br concentrations are analyzed via IC during the sulfate determinations, and can be used to correct the Cl values for AgBr precipitation. In general, precipitation of AgBr contributes about 0.8 to 1.2 mM to the chlorinity value (~0.1-0.2%).

7.3.2.3. Dissolved Inorganic Carbon and Carbon Isotopes

Dissolved Inorganic Carbon will be measured via coulometry at UW. $\delta^{13}C$-DIC will likely be analyzed via isotope ratio mass spectrometry at Oregon State.

7.3.2.4. Sulfate, Chloride, Bromide, and Fluoride Concentrations

Sulfate, Cl, Br, and F will be determined on a Metrohm 882 Compact ion chromatograph at UW.

7.3.2.5. Calcium, Magnesium, Sodium, and Potassium Concentrations

These solutes will be analyzed on a Perkin-Elmer 8300 inductively coupled plasma – optical emission spectrometer (ICP-OES) at UW.

7.3.2.6. Lithium, Boron, Strontium, Barium, Iron, Manganese, and Si Concentrations

These solutes will be analyzed on a Perkin-Elmer 8300 inductively coupled plasma – optical emission spectrometer at UW. Samples will be analyzed for $^{87}Sr/^{86}Sr$ via MC-ICP-MS at Oregon State (see below).

7.3.2.7. Contamination Tracer (Cesium) Concentrations:

Tracer concentrations will be measured on a ThermoFisher Scientific iCAP-RQ inductively-coupled plasma mass spectrometer (ICP-MS) at UW.

7.3.2.8. $^{87}Sr/^{86}Sr$, $\delta^{7}Li$, and $\delta^{11}B$

Samples will be analyzed for $^{87}Sr/^{86}Sr$ via MC-ICP-MS at Oregon State. Samples for $\delta^{7}Li$ and $\delta^{11}B$ will also be analyzed via MC-ICP-MS. Location for these analyses is TBD.

7.3.2.9. $\delta^{37}Cl$

Select samples for $\delta^{37}Cl$ will be analyzed on a ThermoFisher Delta V mass spectrometer after conversion to CH_3Cl at IPGP in Paris, France.

7.3.2.10. Si, NH_4, dissolved sulfide, PO_4

Samples will be analyzed via colorimetry at UW.

7.3.2.11. Dissolved Organic Carbon Concentrations

DOC concentrations will be analyzed with a Shimadzu TOC-Vcsh DOC analyzer at UW.

7.3.2.12. VFAs and Isotopes

Samples will be analyzed via liquid chromatography-isotope ratio mass spectrometry. Location of the analyses is TBD.

7.3.2.13. Trace Metal Concentrations and Isotope Ratios

Trace metal concentrations will be measured on a ThermoFisher Scientific iCAP-RQ inductively-coupled plasma mass spectrometer (ICP-MS) at UW. Isotope ratios will be measured by MC-ICP-MS at UW.

7.3.2.14. Organic Complexes and Ligands

Organic complexes will be measured via by cathodic stripping voltammetry at UW. Organic ligand quantification will be determined by liquid chromatography-electrospray ionization mass spectrometry at UW.

7.3.3. Tufts University Geomechanics

Whole round and bagged sediment samples will be shipped to Tufts University for geomechanical, MAD, and physical properties.

7.3.3.1. Tufts Constant Rate of Strain and Triaxial Testing

Mechanical properties tested at Tufts and will include constant rate of strain testing (ASTM D) to determine compression characteristics, preconsolidation pressure, and permeability as a function of porosity. Triaxial tests will be performed to evaluate the lateral stress ratio, the shear strength, and the friction angle as a function of stress level.

Tests will be run on intact whole round samples and reconstituted samples of sand from depressurized pressure core.

7.3.3.2. Tufts Moisture and Density

Tufts will determine downhole variation in bulk density, dry bulk density, grain density, porosity, and void ratio on material from MAD whole round cores. These analyses will be similar to standard IODP methods and based on ASTM methods D2216 and D854.

In addition, material will be used to measure grain density by water submersion and gas pycnometer at selected locations.

7.3.3.3. Tufts Grain size distribution by Hydrometer

Bulk sediment grain size distribution will be measured at Tufts using the hydrometer method according to ASTM D7928. The sediment will be taken from the MAD whole round samples and plugs removed from the working half of the split core. Additional hydrometer samples may be identified bounding the Red sand. The target sample frequency for this study is every 10 cm. Grain size distribution by hydrometer of samples in the exact stratigraphic intervals as those identified above for the Ohio State diffusion study may be done by Tufts or Ohio State.

Sediment from the MAD samples will also be subsampled for laser particle grain size distribution and CHNS measurements at the University of New Hampshire (See 7.3.7 UNH Sedimentology) and X-ray power diffraction measurements at James Hutton Institute (See 7.3.5 James Hutton X-ray powder diffraction).

7.3.4. James Hutton X-ray powder diffraction

Whole rock and clay fraction mineralogical analysis by X-ray powder diffraction (XRPD) will be performed by the James Hutton Institute (UK) on sediments from the split core working half will be performed at selected locations to provide quantitative information on the overall minerology as well as a detailed distribution of the clay content. Additional samples may be provided by the various PIs to correlate with specific analytical results where XRPD can inform the specific analytical interpretation.

7.3.5. Oregon State Microbiology

Oregon State will broadly investigate microbial properties in samples collected with a focus on characteristics linked to methanogenesis, how this activity may be distributed in the sediments (e.g., coarse- vs. fine-grained sediments), and how active these cells may be. Though challenging because of both the low biomass and the low levels of activity known to occur in deep seafloor systems, Oregon State will select approaches most likely to yield measurable results.

Sediment from the microbiology whole rounds, other samples of interest, and contamination control samples will be used to determine: 1) microbial diversity using DNA sequencing, 2) microbial activity using RNA sequencing, 3) levels of selected functional genes using DNA sequencing, and 4) the level and degree of contamination. See Appendix 12.1.A.6 Oregon State Microbiology of Conventional Core for more details.

7.3.6. UNH Sedimentology

7.3.6.1. **UNH Lithostratigraphic Core Description Summaries**

Lithostratigraphic core description will be based on visual core logging and sediment compositions determined from smear slide and coarse fraction petrography. Smear slide and coarse fraction sediment descriptions provide the basis for identification of changes in bulk composition. Together these data will be used to construct comprehensive core descriptions containing the compositional, structural, stratigraphic, and diagenetic fabric and facies variations throughout the cores.

7.3.6.2. UNH CHNS

UNH will complete CHNS elemental analysis of representative lithofacies specific samples at approximately every 1 meter from the working half of the split core. CHNS samples will also be analyzed from a sub sample of the MAD whole rounds send to Tufts. These samples will be collected from representative lithofacies specific, 1 cm stratigraphic intervals.

Bulk sediment CHNS elemental analysis will be completed at UNH using an Elementar UNICUBE CHNS Elemental Analyzer and yield the following measurements: Total Carbon (TC), Total Nitrogen (N), Total Sulfur (S), Total Organic Carbon (TOC) and derived Calcium Carbonate ($CaCO_3$), of select samples throughout the records. These measurements will serve to quantify the bulk compositional trends for import gas and gas hydrate related sediment components: TOC and the C/N equates to the organic matter quantity and type, $CaCO_3$ tracks authigenic and biogenic carbonate variations, Total Sulfur tracks variations in pyrite and other iron sulfides produced during sulfate reduction and Anaerobic Oxidation of Methane (AOM).

UNH will analyze X-ray fluorescence samples to determine the concentration of individual elements like S or Ca using mass spectrometry. The data will be used for calibration of the X-ray fluorescence core scans.

7.3.6.3. UNH TOC and $CaCO_3$

As part of the bulk sediment CHNS measurements, carbonate free total organic carbon (TOC) and CaCO3 will be determined at UNH at a sample frequency (every~ 1 m) throughout the conventional and pressure cores. Prior to TOC analysis, inorganic carbon (IC) will be dissolved from bulk sediment samples using 6% sulfurous acid applied to weighed samples in amounts and steps optimized for carbonate-rich sediments (Phillips et al., 2011). $CaCO_3$ weight percent will be calculated by multiplying the IC weight percent (IC = TC-TOC) by 8.33 to account for the non-carbon mass fraction. The calculated bulk $CaCO_3$ fraction represents biogenic, authigenic, and any detrital carbonate phases.

7.3.6.4. UNH Isotopes of C and S

Bulk sediment TOC and S isotopes (del ^{13}C and del ^{34}S) will be completed by UNH in collaboration with the University of California Berkeley Center for Stable Isotope Biogeochemistry. These measurements will allow us to look at the sources of organic carbon and evidence for AOM in the records. Coupled with the C/N measurement, the isotopic character of the organic carbon will define relative variations in the source (marine or terrestrial) of the carbon.

7.3.6.5. UNH Grain Size Distribution by Laser Particle Analysis

Grain size analysis will be completed at UNH using a Malvern Mastersizer 2000 Laser Particle Size Analyzer with a Hydro 2000G wet dispersion unit. The Malvern Mastersizer 2000 can measure particles from 0.2 µm to 2,000 µm in diameter. Bulk sediments sampled for grain size will be from 1 cm3 in volume and contained within 1 cm thick stratigraphic intervals, with care taken to not cross major lithologic or grain size bed boundaries, but to capture the range of lithofacies throughout the cores. Most samples collected for grain size will be from split core sampling with additional 1 cm stratigraphic thickness subsamples from the MAD whole round. On a subset of samples with sufficient lithostratigraphic thickness, both Laser Particle Size analyses (UNH) and hydrometer particle size analysis (Tufts) will be completed for comparison.

7.3.6.6. UNH Authigenic Carbonate and Sulfide Nodules

UNH in collaboration with the University of California Berkeley Center for Stable Isotope Biogeochemistry will determine the C and S isotopic signatures for a subset of any authigenic carbonate or Fe-sulfides recovered in the cores.

7.3.7. USGS Rock Magnetics

Paleomagnetic samples will be analyzed at USGS Woods Hole for frequency-dependent magnetic susceptibility.

Specific rock magnetic properties (e.g. isothermal remnant magnetization, hysteresis parameters, low/high temperature susceptibility) from a sub-set of the p-mag samples will be measured at the UNH Paleomagnetism Laboratory and possibly the University of Minnesota Institute for Rock Magnetism.

7.3.8. UT Biostratigraphy

Biostratigraphy samples will be interpreted by a representative of UT. Biostratigraphy smear slides will be used to identify the first and last occurrence of marker species and used to create an age model based on nannofossil biostratigraphic zonation developed for the Gulf of Mexico.

7.3.9. UT Split Core

Working and archival halves of split core will be stored at UT. Cores will be shrink-wrapped and shelved in cold storage.

7.4. Summary of expected core logging and imaging data

Table 7-3 summarizes the core logging and imaging information that will be available as a function of core type and sample type based on the sampling plan above.

Table 7-3. Core Logging and Imaging Summary. Matrix of expected logging and imaging data available and time the data is collected (on-board or dockside) as a function of core type and A. Type of core including expected sediment, and core acquisition method including pressure (PC) and conventional coring and pressure core handling such a liquid nitrogen (LN2) depressurization. B. Core section including pressure core sections going to UT, whole round samples for pore water (PW), microbiology (MBIO), Moisture and Density (MAD), and mechanical testing, and split core working and archival halves. C. Pressure Core Analysis and Transfer System (PCATS) data. Data is collected as part of a Quick or Full Scan. D. Geotek Infra-red Multi-Sensor Core Logger (MSCL-IR) thermal image.

E. Geotek Standard Multi-Sensor Core Logger (MSCL-S) data. Only intact cores will be scanned using the MSCL-S. It is not likely that sand cores will remain intact. F. Split core scanning data. Zoom in to read table.

A. Core Type		B. Core Section	C. PCATS			D. MSCL-IR		E. MSCL-S	F. Split Core Logging
			Quick-scan: 2D X-ray image, P-wave velocity, Bulk Density	Full Scan: High-resolution 2D X-ray images (2 orientations), P-wave velocity, and Bulk density; 3D CT imaging		Thermal imaging	Repeat Thermal imaging	High-resolution 2D X-ray imaging (2 orientations), P-wave velocity, bulk density, natural gamma, and resistivity; 3D CT imaging	Photo scans, Magnetic susceptibility, X-ray Fluorescence, Color reflectance
			ON-BOARD	ON-BOARD	DOCKSIDE	ON-BOARD	ON-BOARD	DOCKSIDE	DOCKSIDE
Sand	PC	to UT	YES		YES				
	Depressurized PC	PW-MBIO-MAD	YES		YES			If possible, MAD only	
		Mechanics & split working half	YES		YES			If possible	
		Split Archival half	YES		YES			If possible	If possible
	LN2 Depressurized PC	MBIO-Sediment fabric	YES		YES				
	Bagged sediment	Reconstituted geomechanics	YES		YES				
Bounding mud	PC	to UT	YES		YES				
	Depressurized PC	PW-MBIO-MAD	YES		YES			YES -MAD only	
		Mechanics & split working half	YES		YES			YES	
		Split Archival half	YES		YES			YES	YES
Background mud	PC	to UT		YES					
	Depressurized PC	PW-MBIO-MAD		YES				YES -MAD only	
		Mechanics & split working half		YES				YES	
		Split Archival half		YES				YES	YES
	Conventional Core	PW-MBIO-MAD				YES		YES -MAD only	
		Mechanics & split working half				YES	YES	YES	
		Split Archival half				YES	YES	YES	YES

7.5. Summary of sampling frequency and estimated total number of samples

7.5.1. Sampling frequency per core

Table 7-4 A summarizes the expected number of samples/measurements for each core type (rows) and sample/measurement type (columns) as described in the Analysis and Sampling Plan above.

7.5.2. Sampling frequency with depth

Geolog well log tables showing the estimated numbers of samples/measurements with depth for each sample type are available.

7.5.3. Estimated total number of samples

Table 7-4 B summarizes the expected number of samples/measurements for each hole and for the total expedition (rows) for each sample/measurement type (columns).

Table 7-4. Estimated maximum number of samples (sample sets, or measurements)

PC represents Pressure Core, APC Advanced Piston Core, and XCB extended core barrel rotary core. A. Approximate section length in cm. B. Estimated number of quantitative degassing samples per core. C. Pressure core sections that will be preserved at pressure and temperature and brought to UT. Note that only a small number of sections from all the background mud PC cores will be brought to UT for K0 testing making the average number of background mud PCs per core, zero. The total estimate for the expedition includes these sections. D. Sections that will go into the conventional core flow. For pressure cores, this is the estimated number of quantitatively degassed sections that will stay intact be able to be moved into the conventional core flow. For conventional cores, the expected number is solely based on the target core length divided by the section length of 150 cm. E. Void gas sample sets to collect per core. Once collected, these samples are split in 2, with half for GC on-board and half for GC at Ohio State. For pressure cores, voids will only be found if the pressure core fails to seal. This estimate assumes 100% pressure coring success. F. Pore water organic whole round samples. G. Conventional pore water whole round samples. Estimate assumes that we can move some PC sand cores to the conventional core flow. Additional pore water samples will be collected from drilling fluid and PCATS as a measure of contamination. H. Pore water whole rounds generated by rapid depressurization of a PC. I. Moisture and Density (MAD) whole round samples. Estimate assumes that we can move some PC sand cores to the conventional core flow. Additional MAD samples will be taken from the split core. MAD samples will be used for x-ray powdered diffraction, and x-ray fluorescence, grain size by hydrometer and other analyses. J. Microbiology whole rounds. Estimate assumes that we can move some PC sand cores to the conventional core flow and that we can collect a whole rounds. Additional pore water samples will be collected from drilling fluid and PCATS as a measure of contamination. K. Microbiology samples generated from cryogenic depressurization of PC sample. A few additional samples may be generated from PC sand cores using the BIO chamber. Estimated number of strength set measurements. This estimate represents the number of hand-held vane, hand penetrometer, table vane, and fall cone measurements. M. Headspace gas sample sets. Each set consists of 4 sediments plugs. These sets are split with half for GC at Ohio State, a quarter for GC at USGS, and a quarter for microbiology. N. Estimated number of thermal conductivity measurements. O. Whole rounds for geomechanical testing. Additional geomechanical testing of sand will be done on pressure core or reconsolidated from bagged sediment. P. Estimated remaining length of whole round core that will be split per core. Q. Split core discrete sample sets including laser grain size, coarse fraction analysis, CHNS, secondary biostratigraphy, MAD/XRF/XRD, and rock magnetism. Other discrete samples will be taken in select intervals.

	Sampling Frequencies per core assuming 100% recovery						
	PC Background mud	PC Bounding mud	PC Sand	APC above SMT	APC below SMT	XCB	Estimated number of samples
A. Section Length (cm)	100	100	30	150	150	150	-
B. Quantitative Degassing	3	3	1.5	-	-	-	84
C. # to UT	0	0	1.75	-	-	-	18
D. sections into CC flow	3	2.5	1.5	5.5	5.5	5.5	218
E. Void Gas	0	0	0	0	2	1	38
F. Pore Water Organics	0	0	0	5.5	5.5	0	65
G. Pore Water	1	2.5	1	5.5	2.5	2.5	118
H. Pore Water by Rapid Degassing	0	0	1	0	0	0	10
I. MAD	1	2.5	1	5.5	2.5	2.5	118
J. Micro	1	2.5	0	5.5	2.5	2.5	108
K. Micro by cryogenic depressurization	1	1	1	0	0	0	33
L. Strength	3	2.5	1	5.5	5.5	5.5	213
M. Head-space Gas	1	1	1	5.5	2.5	2.5	108
N. Thermal conductivity	1	1	1	1	1	1	58
O. Geomech	1	1	0	1	1	1	48
P. Length left after all whole rounds (cm, unless noted)	210	78	5	508	673	673	198 meters
Q. Split Core Discrete Set	3.0	2.5	1.5	11.0	11.0	11.0	355

7.6. Summary of sample storage and movement

Table 7-5 shows a summary of the samples generated, how they will be stored, where they will be stored, how they will be shipped, and where they will be shipped.

Table 7-5. Summary of samples generated, how they will be stored, where they will be stored during different stages of the expedition, where they will be shipped, and how they will be shipped from the SLC. Zoom in to read table.

Sample Generated	Storage method	Storage location on-board	Storage Location during supply boat transfer	Storage location dockside	Ship to (location)	Shipping method
Pressure cores	SC120s	R17 Depressurization Lab and Core Storage			UT	Geotek 40' Reefer Truck
LN2 depressurized core	LN2 Dewars	-80C Freezer in Core Processing			UT and/or Oregon State	LN2 Dewars, requires DOT permit
Gas samples from Depressurization	Copper tubes	Copper tubes in tubs secured on the floor of the Core Processing Lab			Ohio State	FedEx, Requires HAZMAT shipping certification
Void Gas samples	Syringe to glass vials	Secured on shelf in Core Processing Lab				
Headspace gas samples	Glass Vials w/KCl	Secured upside-down on shelf in Core Processing Lab				
Dissociated gas from hydrate-bearing sediment (cc flow)	Glass Vials w/KCl	Secured upside-down on shelf in Core Processing Lab			NA	FedEx, requires HAZMAT shipping certification
Microbiology whole rounds	Capped, sealed with electrical tape, place in Whirl-pak, seal	Refrigerator and -80 C Freezer in Core Processing lab, see protocols			Oregon State	N2 dry shippers overnight
Microbiology - PCATS water and Drilling fluid samples	Falcon tubes	-80 C Freezer in Core Processing Lab				
MAD whole rounds	Capped, sealed with electrical tape	NA	NA	Core Storage, or ship immediately	Tufts	Weigh, ship overnight in coolers with freezer packs
Split Core samples for MAD, XRF, and XRPD	Bagged and sealed	NA	NA	Core Storage, or ship immediately		
Geomechanical Testing whole rounds	Capped, sealed with electrical tape	NA	NA	Core Storage, or ship immediately		
Mechanical Testing-loose sand lithofacies sediment from Pressure Cores	Bagged and sealed	Core Storage (4-6 C)				

Sample Generated	Storage method	Storage location on-board	Storage Location during supply boat transfer	Storage location dockside	Ship to (location)	Shipping method
Pore Water whole rounds	Capped, sealed with electrical tape	Glove bag in refrigerated Pore Water lab	NA	Glove bag in refrigerated Pore Water lab	NA	NA
Frozen Pore water samples	Varied. See Pore Water Protocols	-20 C freezer in the Core Processing Lab			UW	Ship overnight in N2 dry shippers
Pore water residual sediment -UW	Varied. See Pore Water Protocols	Varied. See Pore Water Protocols				
Pore water samples	See Table 5-1 and 5-2	Refrigerated Pore Water Lab	Core Storage (4-6 C)	Refrigerated Pore Water Lab	UW	Ship overnight in coolers with freezer packs
Pore Water - PCATS water and Drilling fluid samples	Acid-cleaned 50-mL HDPEs	Refrigerated Pore Water Lab	Core Storage (4-6 C)	Refrigerated Pore Water Lab		
Pore water residual sediment for Rock magnetism	Bagged and sealed with N2	-20 C freezer in the Core Processing Lab			USGS Woods Hole	Ship overnight in coolers with freezer packs
Split Core samples for rock magnetism	Bagged and sealed with N2	NA	NA	-20 C freezer in the Core Processing Lab		
Split Core samples for XRPD	Bagged and vacuum sealed	NA	NA	Core Storage (4-6 C)	James Hutton Institute	Ship international
Split Core samples of Red sand for Hydrometer	Bagged and sealed	NA	NA	Core Storage (4-6 C)	Tufts or Ohio State	Ship overnight in coolers with freezer packs
Biostratigraphy Smear slides	Boxed	NA	NA	Benchtop in Split Core Lab	UNH	Biostratigrapher will transport
Split Core samples for Biostratigraphy	Bagged and sealed	NA	NA	Core Storage, or ship immediately		Ship overnight in coolers with freezer packs
Sedimentology smear slides	Boxed	NA	NA	Benchtop in Split Core Lab		Ship overnight
Split Core Sulfide samples	Bagged and sealed with N2	NA	NA	Core Storage, or ship immediately		Ship overnight in coolers with freezer packs
Split core samples - all other	Bagged and sealed	NA	NA			
Pore water residual sediment -UT	Bagged and heat sealed	Core storage(4-6 C)			UT	Core Storage (4-6 C)
Working and Archival halves	D-tubes	NA	NA	Core Storage (4-6 C)		
Loose sediment from coring and pressure cores - all other	Bagged and sealed	Core Storage (4-6 C)				

8. Science Containers, Equipment and Personnel

This section provides more detail on the required containers/mobile laboratories, equipment, and staff needed for the science and sampling activities. This section also provides information on who will provide the containers, equipment, and personnel.

8.1. On-board by container

Figure 8-1 shows the containers and laydown areas required on-board and highlights the container provider. Figure 8-1 also identifies the movement of core, gas, and water samples between each container. Table 8-1 lists the on-board container or work area name, type or size, description, major activities requiring processing or testing equipment, when it will be mobilized, and what hook-ups are required.

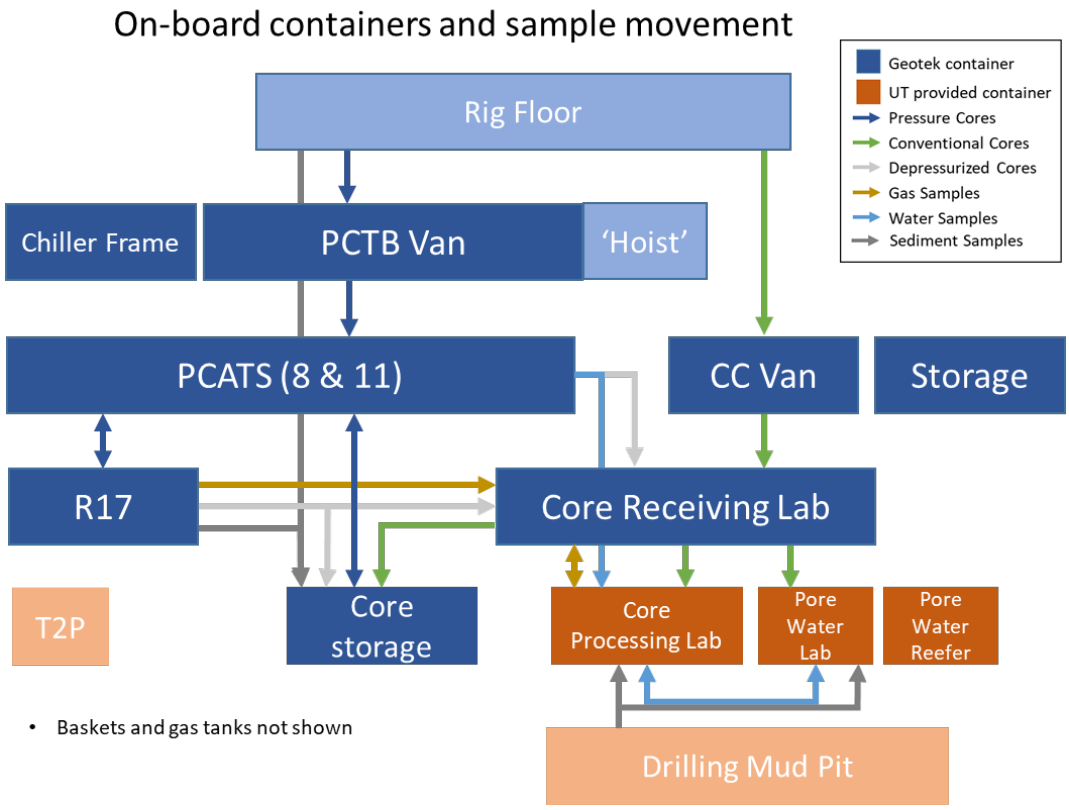

Figure 8-1. On-board containers, providers, and sample movement. Dark blue boxes represent containers that will be supplied by Geotek. Dark Orange boxes represent containers that will be provided by UT. Lightly shaded boxes represent areas on the vessel where the science party will be working. Arrows indicate core and sample movement.

Table 8-1. On-Board container information. Zoom in to read table.

Name	Type	Description	Activities	Reuse or New	Mobilization/ demobilization notes	Required Vessel Hook-up
Rig Floor	Area	Rig Floor	• Wireline tool deployments, Cold Shuck with Chillers	Same as GOM2-1	NA	NA
Utility Frame	20' Frame	Geotek large glycol chillers	• Power, water, air, internet distribution	Same as GOM2-1	-	Power 480 V 3 phase 200 amp water (1", 50 gpm) air (1", 110 psi) LAN jack (CAT6)
Conventional Core Handling	Area	Conventional Coring Laydown	• H2S monitoring, CC core liner venting • Core Catcher sediment collection	NEW	-	NA
PCTB Van	40' container	PCTB coring tools	• PC Pressure checks • Some PCTB assembly, autoclave handling • Collect loose sediment from PCTB	Same as GOM2-1	-	None
CC Van	20' container	Conventional Coring tools	• APX and XCB parts and supplies, handling	NEW	-	None
Storage	20' container	Tools storage		NEW		None
PCATS11	40' container	PCATS Analysis	• PC scanning (X-ray /CT imaging, P-wave, bulk density) • PC cutting and transfer • Rapid degassing	Same as GOM2-1	-	Waste water drain
PCATS8	20' container	PCATS Autoclave and storage vessel handling	• Autoclave and PC storage handling • 15 count PC storage (SC_{350})	Same as GOM2-1	-	None
R17	20' container	Pressure Core storage and degassing	• Quantitative degassing • Gas sampling • 20 count PC storage (SC_{120}, SC_{035})	Same as GOM2-1	Powered on Supply boat during demobilization	None
Core Storage	20' container	Pressure and Conventional Core Storage	• 40 count PC storage (SC_{120}), 10 racks of 4 • 600 m conventional and depressurized core storage, racks • Sediment bag storage • Conventional core and sediment transport to UT	NEW	Powered on Supply boat during demobilization	Power 480 V 3 phase 30 amp
Core Receiving Lab	40' container	Geotek Whole Core Processing Laboratory	• IR scanning • Long core rack (9m) • Void gas sample collection • Sectioning and whole core cutting for microbiology, PW, MAD • Headspace gas sediment sampling • Hand vane and pocket penetrometer • Gas chromatography • CT image processing	NEW	-	Power 480 V 3 phase 60 amp water (0.5", 5-15 gpm) air (0.5", 110 psi) LAN jack (CAT6)
Core Processing Lab	20' container	Microbiology, M&D	• Microbiology sub-coring under N2 • Microbiology contamination control sample preservation • Headspace gas sample processing • -80 C freezer • -20 C freezer	Same as GOM2-1	Powered on Supply boat during demobilization	Power 240/480 V 1 phase 50 amp water (0.5", 5-10 gpm) air (0.5", 110 psi)
Pore Water Lab	10' container	Pore Water Laboratory	• Pore water squeezing • PW ephemeral properties analyses (Salinity, Alkalinity and pH) • PW preservation (Sulfide, DOC, DIC, isotopes, major elements, nutrients, etc.)	NEW	-	Power 240/480 V 1 phase 50 amp water (0.5", 5-10 gpm) air (0.5", 110 psi)
Pore Water Reefer	10' container	Pore Water Refrigerated Lab	• Extruding sediment from liner • PW contamination control sample preservation	NEW	-	Power 480 V 3 phase 30 amp
T2P	Laydown Area	Wireline Pressure and Temperature Probe	• Storage and partial assembly of T2P	NEW	-	None

8.2. Dockside by container

Figure 8-2 and shows the containers and/or permanent labs required dockside and highlights the container provider. Figure 8-2 with the movement of core, gas, and water samples between each container. Table 8-2 lists the on-board container or work area name, type or size, description, major activities requiring processing or testing equipment, when it will be mobilized, and what hook-ups are required.

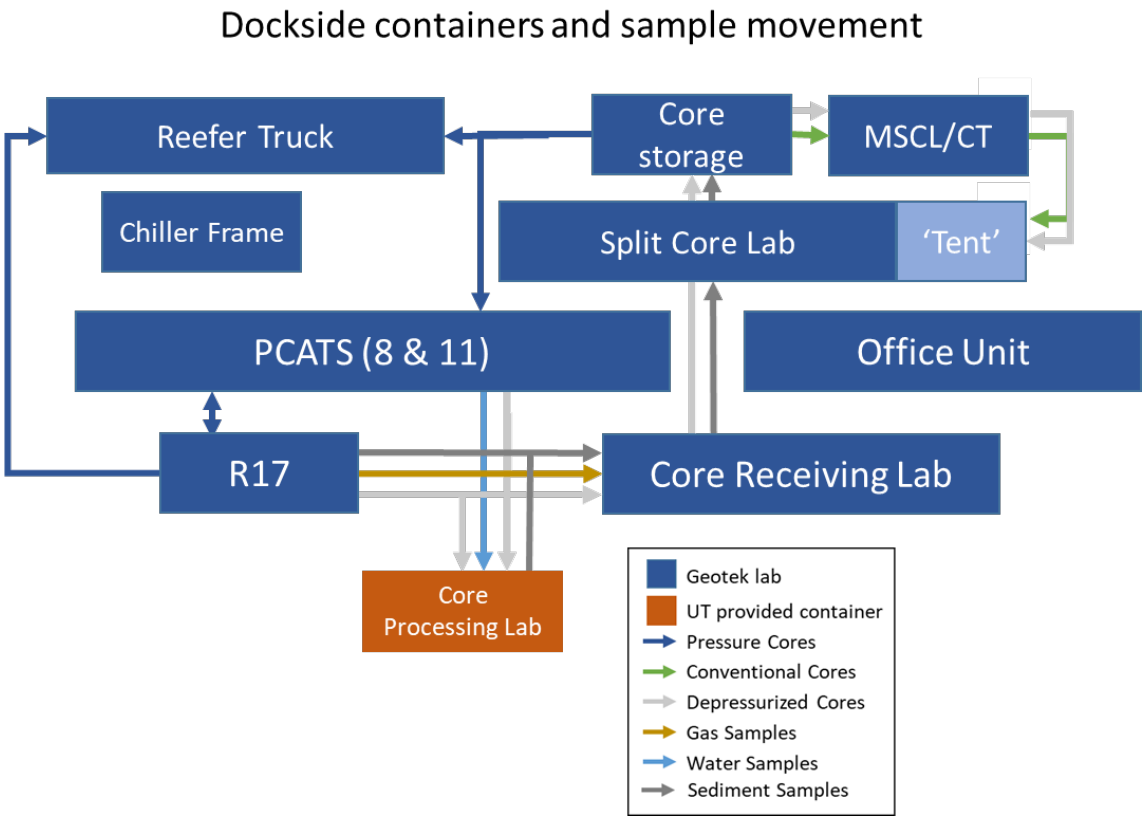

Figure 8-2. Dockside containers, providers, and sample movement. Dark blue boxes represent containers that will be supplied by Geotek. Dark Orange boxes represent containers that will be provided by UT. Lightly shaded boxes represent covered outdoor areas where the science party will be working. Arrows indicate core and sample movement.

Table 8-2. Dockside container information. Zoom in to read table.

Name	Type	Description	Activities	Reuse or New	Mobilization/ demobilization
PCATS11	40’ container	PCATS Analysis	• PC scanning (X-ray /CT imaging, P-wave, bulk density) • PC cutting and transfer • Rapid degassing	Same as GOM2-1	Truck from Harvey Gulf to GCI, hook-up at GCI
PCATS8	20’ container	PCATS Autoclave and storage vessel handling	• PC storage handling • 15 count PC storage (SC_{350})	Same as GOM2-1	Truck from Harvey Gulf to GCI, hook-up at GCI
R17	20’ container	Pressure Core storage and degassing	• Quantitative degassing • Gas sampling • 20 count PC storage (SC_{120}, SC_{035})	Same as GOM2-1	Truck from Harvey Gulf to GCI, hook-up at GCI
Core Storage	20’ container	Pressure and Conventional Core Storage	• PC storage (SC_{120}), 10 racks of 4 • 600 m Conventional and depressurized core storage, racks • Sediment bag storage	NEW	Powered during transport from port to GCI
Core Receiving Lab	40’ container	Geotek Whole Core Processing Laboratory	• Thermal Conductivity and Vane Strength • Whole Core cutting and sectioning for mechanics • Gas chromatography • CT image processing • Sample weighing	NEW DNV	Truck from Harvey Gulf to GCI, hook-up at GCI
Core Processing Lab	20’ container	Microbiology, headspace gas	• Microbiology sub-coring under N2 • Microbiology contamination control sample preservation • Headspace gas sample processing • -80 C freezer • -20 C freezer	Same as GOM2-1	Powered during transport from port to GCI, No power required for transport back to Pro-Log
Geotek Office	40’ container	UT Office Space	• Writing, data Analysis • Workstations, seismic and log correlations	Same as GOM2-1	NA - on site at GCI
MSCL /X-ray	20’ container	Core Scanning, Core imaging	• CT X-ray imaging • MSCL whole core scanning (Gamma density, P-wave, Mag susceptibility, Resistivity; natural gamma)	NEW	NA - on site at GCI
Split Core Tent	Tent	Core Splitting	• Core splitting	NEW	NA - on site at GCI
Split Core Lab	Permanent lab at GCI	Split Core Analysis	• Split core scrapping • Split core scanning (magnetic susceptibility, Photo scanning, X-ray fluorescence, color reflectance) • Core layout, sampling • Smear slide prep and microscopy	NEW	NA - on site at GCI
Overpack - TRANS36	40’ Reefer Truck	Overpack reefer truck	• Pressure Core transport	Same as GOM2-1	Powered during transport from port to GCI and GCI to UT

8.3. Science Party

Table 8-3 shows the required On-board Science Party staff level and possible assignments for each coring hole. Table 8-4 shows the required Dockside Science Party staff level for each coring hole.

Table 8-3. Required On-board science party staff level and possible assignments.

Title	Location	H002	H003
Company Man	Head office	1	1
Chief Scientist	Head Office	1	1
Operations Reporting	Head Office	1	1
Curation Staff Scientist	Connex	1	1
Observer	TBD	1	1
T2P	TBD	-	1
Geotek - Coring – PC Processing, Logging, Gas Analysis, CT processing	Rig Floor, PCTB Van, CC Tools, PCATS, G9	12	12
Geotek CC imaging and Whole Round Processing, Void Gas Collection	Core Receiving, CC Storage	-	-
Drilling Data and Core Log Integration	Connex, Core Receiving	2	2
CC Analyst (Vane and pocket penetrometer, headspace gas and hydrate Samples) *	Core Receiving, CC Storage	-	2
Pore Water Geochemist	Pore Water Lab	2	4
Methane/Hydrate Geochemist (Quantitative Degassing)	R17	4	4
Microbiology, Headspace Gas, Control Sample Curation	Core Processing	2	2
Photographer/Videographer (only at beginning or end)		2	-
TOTAL		**30**	**32**

Table 8-4. Required Dockside science party staff level.

Title	Location	Dockside
Chief Scientist	Connex	1
Curation Staff Scientist	Connex	1
Geotek - Coring – PC Processing, Logging, Gas Analysis, CT processing	PCATS, G9	12
Geotek CC Logging, CT imaging, and whole round processing, Split core logging	Core Receiving, Split Core Lab	-
Methane/Hydrate Geochemist (Quantitative Degassing)	R17	4
Whole Core Geomechanics, Thermal conductivity	Core Receiving, Core Processing	2
Split Core Description	Split Core Lab	2
Split Core Sampling, Curation	Split Core Lab	2
TOTAL		**24**

9. Reporting

Similar to IODP expeditions a Preliminary Expedition Report will be issued 2 months post-expedition. The preliminary report will review the background and expedition objectives and discuss achievement of those objects; describes drilling, coring, and dockside operations; and presents principal standard measurements for each site. Members of the on-board and dockside Science party will contribute to and author the report.

Expedition Reports will be published 1-year post-expedition. Expedition reports will contain the expedition summary, a methods chapter, and site reports that present site operations and on-board and dockside results for geochemistry, microbiology, physical properties, sedimentology, biostratigraphy, downhole measurements, and other results (e.g. UT-GOM2-1 Expedition Reports (Flemings et al., 2018)

Expedition data and samples will be protected by a 1-year post-expedition moratorium, during which time data and samples from the expedition will be available only to the expedition's science party and approved shore-based participants. recipients of samples and data incur obligations to conduct research and report on the derived science outcomes in peer-reviewed scientific literature and/or expedition data reports., and make the data publicly available.

10. Requesting Samples and Data

Requests for Data and/or Samples can be made to UT using a UT-GOM2-2 Sample Request Form. Requests will be review by our Core Analysis and Distribution Technical Advisory Group.

Submit Inquiries to by e-mail to: carla.thomas@utexas.edu, Subject Line: [P.I. Last Name] UT-GOM2-2 Sample and Data Request

11. Acknowledgment

This work is a compilation of writing from many of members of the GOM research team. The compilation led by Carla Thomas and writing including Peter Flemings, Manasij Santra, Jaime Morrison, Jesse Houghton, Kehua You, Kevin Meazell, Alexey Portnov, Carla Thomas, and Aaron Price (University of Texas), Rick Colwell (Oregon State), Evan Solomon (University of Washington), Ann Cook, Derek Sawyer, and Tom Darrah (Ohio State University), David Divins and Joel Johnson (University of New Hampshire), Alberto Malinverno (Lamont-Doherty Earth Observatory), John Germaine (Tufts University), and Steve Phillips (USGS); and others including Peter Schultheiss and Melanie Holland (Geotek); members of the GOM2 Advisory Group including Tim Collett (USGS); and Tom Pettigrew (Pettigrew Engineering, Ltd).

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12. Document Tracking

12.1. Releases and changes to the document

Table 12-1. Science Plan document edits and releases

Revision	Description	Date	Modifications
V1	Initial Release	2020-08-05	• The first release was created from a previously released Operational and Science Plans reviewed by the GOM2 Advisory team. Details were added by the 2018-2020 Core Analysis Team.
V2	Major revision	2021-10-11	• Major rework of Section 5 (now sections 3 and 6) including suggested edits from the GOM2 Technical Advisory Group by the 2020-2021 Science Planning Team • Update to the pressure core flow • Major update to the conventional core flow • Clarified the timing and use of Ohio State's vane shear device. • Added a microbial comparison of conventional and pressure cores. • Updated the moisture and density plan. • Updated the conventional core flow at the dock to add parallel processing of whole round and split cores. • Prioritized the allowance PC pair above the Blue sand (potential blue sand seal) in H002. • Added detailed plans for profiling pore water methane concentration. • Added opportunity to collect anoxic samples from pressure cores using Geotek's LN2 apparatus at the dock • Specified James Hutton Institute as the location for XRPD. • Specified UNH as the primary location for TOC and laser particle grain size analysis. Included duplicates of samples analyzed using the hydrometer method at Tufts. • Reworked and added details to Appendix A. • Updated final destination of split core to UT cold storage. • Added tables estimating the amount of pressure core to bring to UT, summarizing collected logging and imaging data, summarizing the planned sampling frequency and estimating the total number of each sample type, summarizing sampling with depth, and summarizing the storage and movement of samples during the expedition.
V2.2	Minor revision	2022-10-27	• Second hole moved from G002 to H003 to reduce cost, contrasted proposed maximum plan vs, most-likely funded plan • Removed science objective to contrast hydrate reservoir properties at different structural levels • Updates to estimated time and resources, resulting samples and sample types
V2.3	Minor revision	2023-06-05	• H003 with conventional and pressure coring moved to be the first hole. Deep pressure cores moved to H003. H002 is now the second shallow coring hole. • Number of cores set modified to reflect what is possible with the DOE proposed "Add second hole" plan. • MAD samples will now be cut at dockside.

Appendix A. Detailed Descriptions and Analytical Methods

The following appendix contains detailed descriptions of many aspects of the science plan including equipment and analytical methods. Sample handling protocols are published separately. See UT-GOM2-2 Protocols.

A.1. Drilling Fluid

A full discussion of the drilling fluid can be found in the operational plan. We plan to drill/core with seawater to 1600 FBSF. Below 1600 FBSF we will use a water-based mud. Intermittent gel sweeps will be used to clean the borehole.

A.1.1 Drilling Fluid Contamination

Drilling Fluid will potentially contaminate the PCTB, APC, and XCB cores, however we do not plan to implement tracers, such as perfluorocarbon (PFC) compounds, within the drilling mud to quantify trace-level contamination from the coring process (Expedition 337 Scientists, 2013; House et al., 2003; Lever et al., 2006; Smith et al., 2000a; Smith et al., 2000b). Since most cores will be collected below the sulfate-methane transition, which has been observed to occur between 13 and 308 fbsf (4 and 94 mbsf) in the northern Gulf of Mexico (Coffin et al., 2008; Expedition 308 Scientists, 2006; Kastner et al., 2008; Paull et al., 2005; Pohlman et al., 2008; Presley and Stearns, 1986; Smith and Coffin, 2014), the presence of sulfate will be used as an indicator of contamination of pore waters with drilling fluid at the micromolar level (Our current detection limit with a seawater matrix is 0.1 mM). In addition, comparison of microbial communities in drilling fluid and seawater to those in core samples will be used to identify possible microbial contamination from drilling fluids. This approach has been used successfully in other scientific drilling operations (Colwell et al., 2011; Davidson et al., 2011; Inagaki et al., 2015; Pedersen et al., 1997).

A.2. Coring

A.1.2 PCTB Coring

The PCTB (Pressure Coring Tool with Ball) is a coring system designed to recover core samples while keeping the core within the hydrate stability zone by maintain or boosting the pressure at or above the in-situ pressure. Detailed descriptions of the PCTB and PCTB configurations can be found in the Operational Plan and Thomas et al (2020b).

A.1.2.1 PCTB Cores

The PCTB generates cores with the following:

- Core diameter nominally 5.08 cm (2.0 inches)
- Core liner internal diameter 5.36 cm (2.1 inches)
- Core liner outer diameter 6.03 cm (2.4 inches)
- Core liner length 3.05 m (10 ft)

A.1.2.2 PCTB Data Collection

The parameters in Table 0-1 below will be tracked as part of the operation of the PCTB.

We anticipate to have four interchangeable PCTB pressure chambers (autoclaves) on-board. Each autoclave is equipped with a fluid sampling and drain port, and a 5,000 psi (34.5 MPa) burst disk. There is also a pressure transducer for monitoring the autoclave internal pressure once the tool is on deck and an autoclave pressure relief valve which can be operated manually. The autoclave is not designed for core degassing.

The autoclave is deployed with two Star-Oddi Data Storage Tags (DST Centi-TD temperature depth recorder, DST) to measure the chamber internal temperature and pressure. One is located inside at the top of the chamber. The other is inside, in the core follower, or 'rabbit' (DST Rabbit) and measures the chamber temperature and pressure at the top of the core. A third DST is outside the autoclave, within the PCTB pulling tool, and measures the temperature and pressure of the borehole.

Analysis of the DST data is used to determine whether the sample remained within the hydrate stability field (Inada and Yamamoto, 2015) as the PCTB is recovered to the rig floor. Examples pressure data from successful run of the PCTB resulting in a complete tool stroke, ball valve closure, and nitrogen boost occurring at the target depth with recovery of core can be found in Thomas et al. (2020b)

Table 0-1. List of Parameters that will be collected during PCTB operation.

Core Log	Date
	Site
	Core #
Hardware (which tools are running and how they are configured	Autoclave Number (A/C #)
	Pressure Section Configuration (Pres Sect #)
	Upper Section Configuration (Up Sect #)
	Reservoir Pressure (psi)
	Reservoir Pressure (MPa)
	Set/Boost Pressure (psi)
	Set/Boost Pressure (MPa)
	DST Plug ID number
	DST rabbit ID number
Cored Location / Depth	Core Depth (mbsf)
	Water depth (m)
	Depth below rig floor (m)
Pressure and Temperature Data	In situ Pressure (MPa)
	Recovery Pressure (psi)
	Recovery Pressure (MPa)
	Plug Data Storage Tag Minimum Pressure (MPa)
	Rabbit Data Storage Tag Maximum Temperature ^{o}C
	Time out of Hydrate Stability Zone from Rabbit Data Storage Tag (min)
	P/T Notes
	Date into pipe

Coring and Processing Time	Time into pipe
	Date out of pipe
	Time out of pipe
	Total Time in pipe (hr)
	Date in PCATS
	Time in PCATS
Core Recovery	Cored Interval (m)
	Total curated length (m)
	% Recovery of Cored Interval
Drilling Parameters from the rig floor	Drill String Rotation (RPM)
	Pump Rate (L/min)
	Weight on Bit (T)
	Pull-out Force (T)
	Rate of Penetration (m/h)

A.1.3 APC and XCB Coring

Conventional cores will be acquired using the Geotek Advanced Piston corer (G-APC) and their Extended core Barrel (G-XCB). The G-APC is a hydraulically actuated piston corer designed to recover cores from very soft to firm sediments that cannot be recovered with rotary coring. The G-PAC cutting shoe will be modified to accept the IODP temperature sensor and to include the option of coring with or without chisels (Figure 0-1). The chisels may be deployed if the sediment is too gassy, or perceived to be too gassy, to safely handle APC cores on the rig floor and to minimize core loss from expanding gases as the core is raised to the rig floor. One of the chisels will include space for a Data Storage Tag (DST) capable of recording temperature and pressure.

The G-XCB is typically deployed when the formation becomes too hard for piston coring.

A DST may be deployed on the APC and XCB pulling tool.

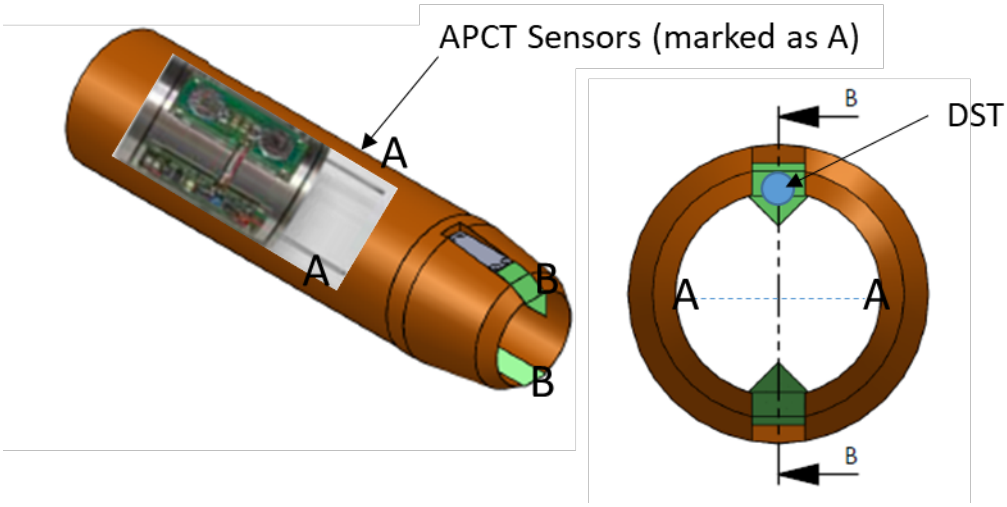

Figure 0-1. Schematic of APC cutting shoe with IODP APCT probe inside. Left) side view of the APCT probe contains two temperature sensors (marked as A) and optional chisels to create flow paths for expanding gases (marked as B). Right) cross sectional view.

A.1.3.1 APC and XCB Cores

The APC and XCB work with the wider inner diameter PCTB-CS BHA and produce conventional core with the following:

	G-APC	G-XCB
Cut core diameter / Cutting shoe inner diameter	6.20 cm (2.4 inches)	5.85 cm (2.3 inches)
Core liner internal diameter	6.63 cm (2.6 inches)	
Core liner outer diameter	7.14 cm (2.8 inches)	
Core Liner length	9.5 m (31.1 ft)	
Core Throw	9.5 m (31 ft	7.6 m (25 ft

A.1.3.2 Conventional Coring Tool Data Collection

The parameters in

Table 02 below will be tracked as part of the operation of the conventional coring tools.

Table 0-2. List of Parameters that will be collected during conventional coring operation.

Core Log	Date
	Site
	Core #
Hardware (which tools are running and how they are configured)	TBD
Cored Location / Depth	Core Depth (mbsf)
	Water depth (m)
	Depth below rig floor (m)
Pressure and Temperature Data	In situ Pressure (MPa)
Coring and Processing Time	Date into pipe
	Time into pipe
	Date out of pipe
	Time out of pipe

	Total Time in pipe (hr)
	Date in PCATS
	Time in PCATS
Core Recovery	Cored Interval (m)
	Total curated length (m)
	% Recovery of Cored Interval
Drilling Parameters from the rig floor	Drill String Rotation (RPM)
	Pump Rate (L/min)
	Weight on Bit (T)
	Pull-out Force (T)
	Rate of Penetration (m/h)

A.2. Penetrometer

A.2.1 Temperature-2-Pressure (T2P) probe

The T2P is a penetrometer tool that logs temperature, pressure, and acceleration when the needle penetrates about 4 ft into the formation. Temperature and pressure are logged at a rate of 1 measurement per second and acceleration at 10 measurements per second. There is one thermistor (temperature sensor) in the tip of the instrument needle, and two pressure transducers (Figure 0-2 B). Some information about formation permeability can be inferred from the difference in pressure response between the two transducers.

A.2.2 Probe Deployment Tool (PDT)

The PDT is a device designed to deploy a penetrometer, such as the T2P or the SET(P), on a wireline using a single mechanical running/pulling tool through the bottom hole assembly (BHA). The outer barrel of the PDT latches into the BHA while the inner barrel, attached to the T2P, helps drive the T2P into the formation. The inner barrel also allows the T2P to remain isolated from the drill string/BHA to eliminate and residual heave movement of the BHA while the T2P is collecting data.

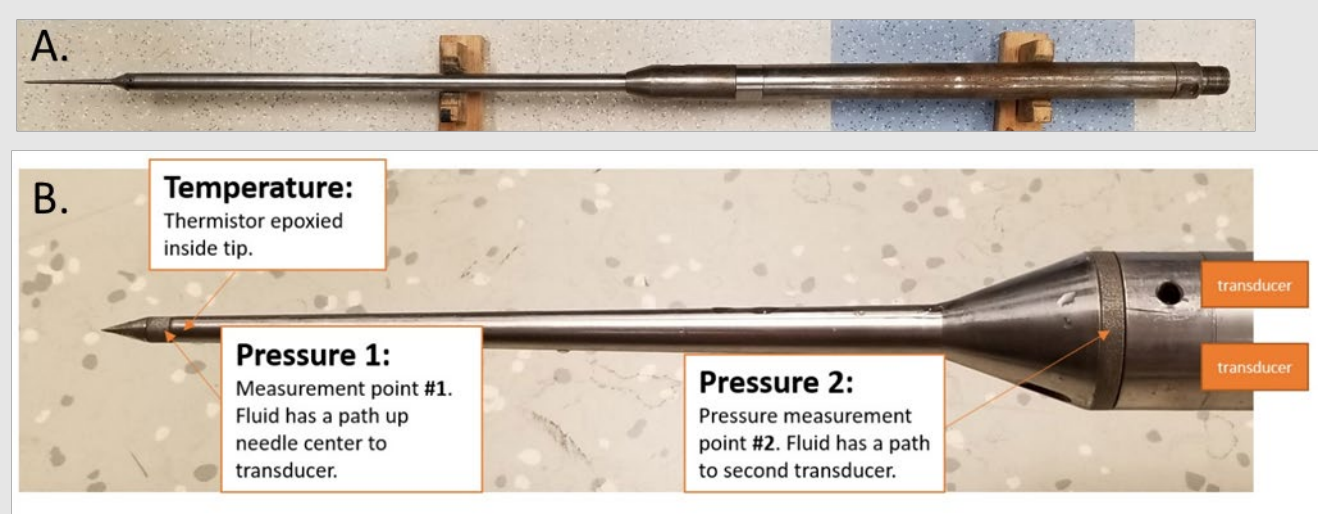

Figure 0-2. Images of the Temperature-2-pressure (T2P) measurement probe A. Image of the full length (8 ft) T2P probe. About half this length penetrates the formation. The point where the T2P screws onto the PDT cab be seen on the right. B. Close-up view of the T2P needle showing the positions of the temperature and pressure sensors in the needle. At each pressure measurement point is a porous metal disc that allows a fluid path to the transducers.

A.2.1 Shelby tubes

From ASTM D1587 / D1587M - 15 Standard Practice for Thin-Walled Tube Sampling of Fine-Grained Soils for Geotechnical Purposes:

Thin-walled tube samples are used for obtaining intact specimens of fine-grained soils for laboratory tests to determine engineering properties of soils (strength, compressibility, permeability, and density).

A.3. Pressure Cores

A.3.1 Pressure Core Storage

The types of storage chambers listed below are considered the standard for all hydrate expeditions around the world. The storage chambers will be stainless steel and compatible with PCATS. Each pressure core chamber has two safety valves at the bottom of the tank: a 35.5 MPa (5150 psi) pressure relief value, to keep the internal pressure close to 35 MPa, and a 43.75 MPa (6345 psi) rupture disk, to prevent explosion of the tank in case the 35.5 MPa relief valve fails. Plastic cylinders ("cones") containing the same types of pressure-temperature loggers used in the PCTB can be placed in the chambers prior to transferring a sample if a pressure-temperature record is desired during storage.

Geotek will provide three types of pressure storage vessels.

A.3.1.1 Geotek SC_{350} chambers: 3.5 meter

Geotek will provide temporary storage chambers on the vessel that will store cores as long as the largest core length that is captured in the PCTB autoclave plus any core expansion, up to 3.5 meters. These chambers are for temporary storage only until sub-sampling and moving the core

sections into 1.2 m or 0.35 m chambers for shipment, quantitative degassing, transport over land, and long-term storage.

A.3.1.2 Geotek SC_{120} chambers: 1.2 meter

U.T. will obtain from Geotek 1.2m storage chambers (Figure 0-3). 1.2 m chambers are the longest core length that can be shipped overland and that can be stored at UT and handled by the UT Mini-PCATS.

The approximate weight of each chamber will depend on the maximum length of core that it can contain. The 1.2 m chambers will be approximately 180 cm in length, 30 cm in width, and weigh approximately 100kg (220 lbs.) when full.

The storage chambers described are not rated for DOT (Department of Transportation) capability to transfer the cores overland. Instead, a DOT rated 'Overpack Technology' is used. See "Overpack Technology" description below. These chambers will ultimately be transferred to shore-based facilities at the University of Texas using this Overpack technology.

A cone containing a DST pressure-temperature recorder can be added to the storage chamber.

A.3.1.3 Geotek SC_{035}: 0.35 Meter, Degassing Chambers

Geotek will provide 0.35 m storage chambers for degassing analysis.

Figure 0-3. Image of 1.2 m and 0.35 m storage chambers on-board in Geotek cold storage

A.3.2 Pressure Core Analysis and Transfer System (PCATS)

PCATS is a specialized system created and operated by Geotek that is designed to characterize and transfer cores at elevated pressures (see Figure 0-4 and Figure 0-5). PCATS has the capability to receive cores from the PCTB autoclave; log cores using 2D X-ray, 3D CT imaging, P-wave velocity and bulk density (Schultheiss et al., 2011); cut cores into smaller sections; and transfer cores into pressure storage or analysis chambers.

Figure 0-4. PCATS in the Geotek Reefer on location (http://www.geotek.co.uk/services/pressure_core_analysis)

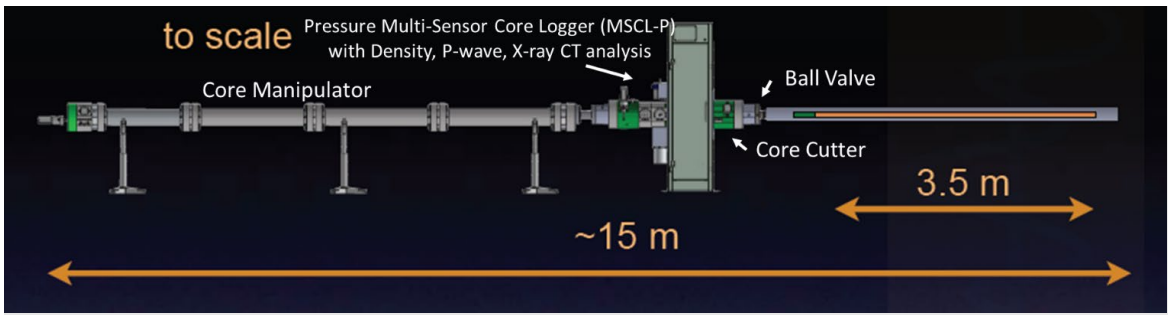

Figure 0-5. PCATS schematic (not to scale)
http://www.geotek.co.uk/services/pressure_core_analysis.

A.3.2.1 PCATS Pressure Core cutting

The core can be cut at a precise location to create subsamples using a combination pipe cutter and guillotine tool. The operation of the core cutter on the core liner is similar to a pipe cutter with a rolling cutting disk. The cutter is pressure-balanced and advanced under manual control

across the core, while the core is rotating, enabling the operator to feel the cutting wheel moving through the core liner. Additional information on the cutting process is provided by live graphs of torque data from the motor. Once the core liner has been parted, the thin stainless steel of the guillotine is used to slice through the sediment. The overall quality of the cuts through the liner are excellent, with little cracking or burring at the edges. However, the quality of cuts through the sediment depend on the sediment properties. Through competent, homogenous softer material the cuts are sharp, clean, and perpendicular to the liner, but in hard sands cleavage along bedding planes may occur.

A.3.2.2 PCATS P-wave Velocity

Ultrasonic P-wave velocity is measured with a pulse transmission technique. The two 500 kHz acoustic transducers are mounted inside the aluminum pressure housing, perpendicular to the core axis. The transducers are also perpendicular to but co-located along the core with the gamma ray beam. The P-wave velocity is calculated from the pulse travel time across the core material and the internal diameter of the core liner ultrasonic velocity with a precision of ±1.5 m/s and an accuracy of approximately ±5 m/s. The pulse travel time across the core material is calculated by subtracting the travel time offset, which is the time required for the pulse to transit the core liner as well as the pressurizing fluid between the transducers and the core liner at a given temperature. Temperature is monitored constantly in PCATS, so the appropriate travel time offset can always be applied.

A.3.2.3 PCATS Gamma Density

Gamma density is calculated from the attenuation of a collimated beam of monochromatic photons from a nominal 10 mCi (370 MBq) 137Cs source. The source is active enough to penetrate both the core and the aluminum pressure housing (wall thickness of 11 mm), and is shielded in lead with a rotating lead shutter (5mm diameter collimator). The gamma source used during this expedition was built by Geotek but was kindly on loan from the core analysis laboratories on *D/V Chikyu*. The Tl-doped NaI detector is calibrated to record only the primary energy emitted by the 137Cs source (662 keV), which ensures that the beam attenuation through scattering is accurately reported. Calibration of gamma attenuation to gamma density, and from there to bulk density, relies on a set of standards of known average bulk density. The standards of choice for calibration of gamma attenuation to gamma density in standard sediments (water-saturated aluminosilicates) are aluminum and water of known thicknesses inside core liner. This results in similar electron density in the calibration pieces and the core, allowing gamma density and bulk density to track each other with high precision, though the resultant data are still technically reported as “gamma density” rather than “bulk density.” The source, detector, and calibration protocols are the same as are used with Multi-Sensor Core Logger (MSCL) systems in laboratories and on research vessels around the world. More Information on MSCL can be found at http://www.geotek.co.uk/products/gammadensity. Quick scan utilizes a 5 second count time while full scan uses a 10 second count.

A.3.2.4 PCATS X-ray Imaging

X-ray images are collected using a variable intensity, microfocal X-ray source and a digital flat-panel detector. The source can provide energies up to 130 keV. The combination of microfocal source and high resolution flat-panel detector enables images to be collected with typical spatial resolutions of 100-150 microns. To collect linear X-rays of core larger than the detector, the core analysis program takes sequential snapshots, moving the core past the detector, and creates a montage of the central section of each snapshot.

Quick scans will use a target core X-ray intensity of 10,000, an X-ray source voltage and current of 120 kV & 400 µA.

To collect data for X-ray CT reconstruction full scan, the core analysis program rotates the core while collecting images. Reconstructions are generated using appropriate algorithms suitable for rotational scans in a cone beam. To help ensure that the core remains aligned with the axis of rotation, mechanical centralizers are used on either side of the X-ray detector when possible. For full scan, images will also be collected for X-ray CT reconstruction using the same X-ray settings as a quick scan. Data includes 400 images at 0.8˚ intervals collected every 6.7 cm down the core in subsections as time permits. Note that in all X-ray images provided by Geotek, dense objects which obscure the X-ray beam are dark.

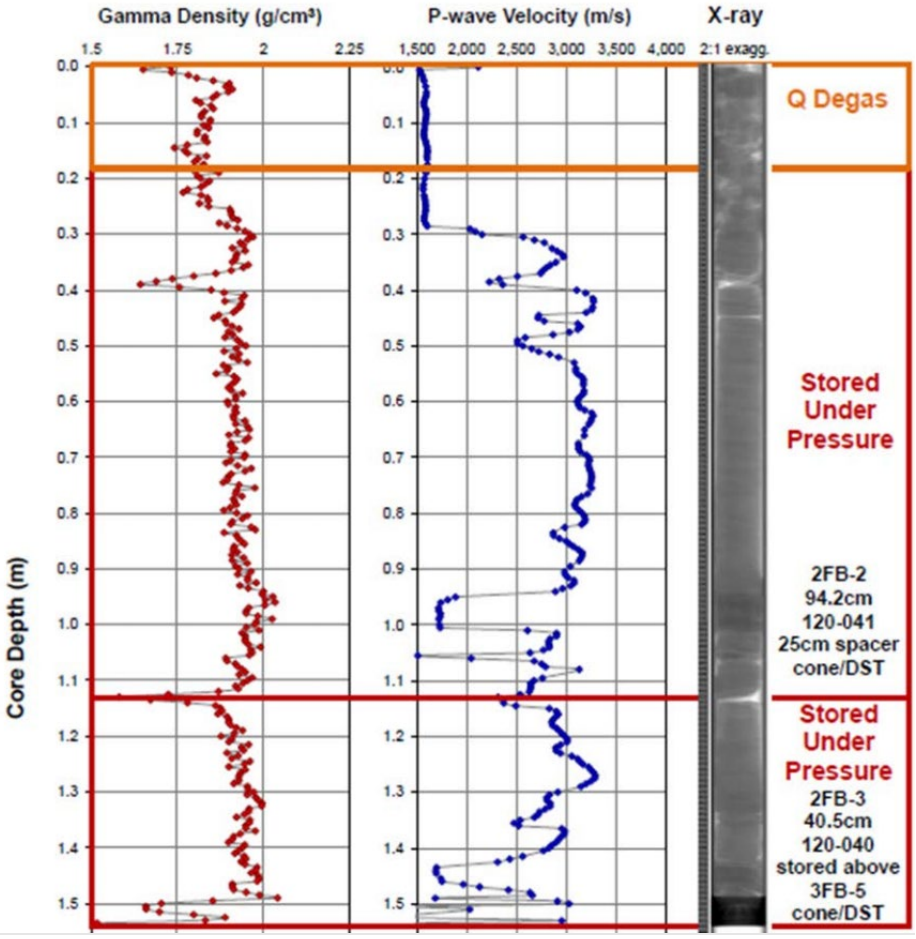

Figure 0-6. Example of Quick scan P-wave, Gamma Density, and 2D X-ray data from UT-GOM2-1-H005-05FB.

A.3.2.5 PCATS Quick Scan Analysis

During a quick scan, cores are logged (velocity, density) with 2 to 5 cm resolution and single scan 2D X-ray image is taken from the top of the sediment (or approximately 10 cm above the bottom of the rabbit) to just above the core catcher.

The Quick scan takes 3.5 hours to prepare PCATS to receive the autoclave, attach the autoclave, pull the core with the "grabber" into PCATS, remove the autoclave, log and image the core, attach the temporary storage chamber, cut off the grabber, push the core into storage, remove the storage chamber, and clean PCATS.

A.3.2.6 PCATS Full Scan Analysis

During a full scan, cores are logged (velocity, density) with 1 cm resolution. Two 2D x-ray images are captured at 0- and 90-degree rotations and a 3D x-ray CT scan is taken on part or the entire length of the core depending on the core quality, geological features, and available time.

A full scan can take up to 9.5 hours.

A.3.2.7 PCATS Water Contamination

PCATS and the pressure storage chambers are pressurized with fresh water (PCATS water). The drilling fluid surrounding the core in the autoclave mixes with PCATS water when a pressure core is moved from the autoclave into PCATS. The core is stored with this mixture when the pressure core is moved from PCATS into a storage chamber. Similarly, every time a pressure core is moved back into PCATS from a storage chamber, the storage fluid (a combination of drilling fluid and old PCATS water) mixes with the new PCATS water. Over time, the storage fluid mixture interacts with the drilling fluid trapped between the core and core liner. The trapped drilling fluid also interacts with the core pore water, modifying the pore water makeup. To quantify the level of pore water contamination, samples of the drilling fluid and samples of the mixed storage fluid must be collected. The mixed storage fluid must be collected from PCATS during depressurization just after the pressure core has been moved and sealed in a pressure storage chamber.

A.3.2.8 PCATS Data Collection

PCATS variables that will be tracked as part of the operation of PCATS include information about which storage chamber is being used, PCATS temperature and pressure, time-in and time-out, etc. In addition, contamination control samples will be taken periodically of the PCATS fill water from PCATS.

A.3.2.9 PCATS Schedule/Timing

A detailed assessment of the PCATS schedule for this expedition was completed for the science plan. The assessment helps optimize the use PCATS on-board and ensure that we bring enough equipment to safely store and process the cores. The assessment assumed 100% coring success. The assessment confirms that there is enough time for full scans and cutting of most pressure cores. Only 2 of 34 pressure cores will only have a quickscan. The assessment also shows that a minimum of 5 SC_{350} and 36 SC_{120} will be required on-board assuming 4 autoclaves and 3 degassing manifolds are available.

A.3.2.9.1 PCATS Schedule Assessment Method

Rig time estimates for H003 and H002 were used to estimate when each pressure core would arrive at PCATS. The coring plan was used to identify the type of pressure core: Type 1 background mud cores for dissolved methane and other studies; Type 2 bounding mud; Type 3. Sand cores

Long sections (1-1.3 m) of background and bounding mud core will receive a full scan logging with CT imaging and be quantitatively degassed. We assumed that background

mud cores would be cut into three large sections and that all three sections would undergo quantitative degassing on-board.

Red and Blue sand cores will receive a full scan and lithofacies specific cuts will be made for quantitative degassing, depressurization after freezing with liquid nitrogen, and rapid depressurization for pore water assessment.

Most Orange sand cores will receive a quickscan for an initial assessment of core quality and content followed later by full scan core logs and CT imaging with lithofacies specific cuts optimized to separate different lithofacies and features of interest.

Table 0-3 shows the amount of PCATS time and required number of storage chambers for each activity. A draft schedule was then prepared allocating time for fully scanning cores or quick scanning cores as allowed such that there was never a backlog of pressure cores in autoclaves waiting on PCATS at any given time. A backlog was defined as more than 2 pressure cores based on 4 available autoclaves and the time it takes to clean and return the autoclave to the rig floor. The draft also assumed 3 available degassing manifolds. For each half hour increment the number of autoclaves waiting at PCATS, the number of SC_{350} storage chambers in use, the maximum number of cores waiting for degassing, and the maximum number of SC_{120} storage chambers in use was calculated. The equipment required was then based on the highest number for each equipment type.

After the draft was generated, trade-offs were assessed for different processing and coring options.

Table 0-3. Required time and equipment for each PCATS activity A. Whether or not the pressure core is a new core coming from an autoclave or a previously processed pressure core coming from a storage chamber, B. PCATS activity/operations/core processing, C. Required amount of time for the activity., D. The resulting data, E. the required storage chambers for the activity.

A. PC	B. On-board Operation	C. Time (hr)	D. Data	E. Chambers
new	"quick scan and store"	3.5	0 deg X-ray, 1 cm res log	1 SC_{350}
new	"quick scan and cut"	5	0 deg X-ray, 1 cm res log	3 SC_{120}
new	"full scan and store"	6	0 and 90 degr X-ray, 0.5 cm log	1 SC_{350}
new	"full scan and cut"	7.5	0 and 90 degr X-ray, 0.5 cm log	3 SC_{120}
new	"full scan, CT, and cut"	9.5	0 and 90 degr X-ray, 0.5 cm log, CT	3 SC_{120}
returned	"full scan and cut"	5.5	0 and 90 degr X-ray, 0.5 cm log	3 SC_{120}
returned	"CT, and cut"	6.5	CT	3 SC_{120}
returned	"full scan, CT, and cut"	9	0 and 90 degr X-ray, 0.5 cm log, CT	3 SC_{120}
-	"added cut"	+1	-	+1 SC_{30} or SC_{120}
-	"added CT "	+1.15/m	-	+1 SC_{30} or SC_{120}

A.3.3 Rapid Degassing

Rapid Degassing will be done using one of two methods. Rapid degassing in PCATS or Rapid degassing in SC_{120} storage chambers.

A.3.3.1 Rapid degassing in SC_{120} storage chambers

Samples over 20 cm long will be cut and moved into one of the 1.2 m storage chambers, SC120. The chambers will be safely and incrementally depressurized over 1-2 hours. No gas will be collected.

A.3.3.2 Rapid degassing in PCATS

If the sample is less than 20 cm long, it is moved between the ball valves of PCATS and any other storage chamber. This space is also called the rapid depressurization chamber. The sample is then safely depressurized within a few minutes with no attempt to capture gas.

Some small sections of pressure core will be left in the PCATS grabber and will be depressurized as PCATS is prepared to receive the next pressure core. This sediment will be collected, bagged, and stored for dockside or post-expedition analysis.

A.3.4 Quantitative Degassing

Four quantitative degassing procedures are available to determine the concentration of hydrate in each pressure core successfully recovered from a suspected hydrate-bearing sand layer. This technique is also used to determine dissolved methane concentration in background cores. On-Board the procedure is completed in 6-12 hours. Post-expedition the procedure is completed in days to weeks.

On-board, initial indication of the presence of hydrate can be seen from PCATS P-wave velocity and 2D and 3D X-ray imaging of the pressure cores. With this information, 0.33 to 3.3’ (0.1 to 1.0 m) subsections of methane-hydrate bearing core will be cut on-board using PCATS. PCATS will transfer the sample into a 0.35 or 1.2 m storage chambers, described above, which will then be connected to a Geotek degassing/gas collection manifold (Figure 0-7) to determine the total methane extracted from and initial concentration of hydrate within the core (Dickens et al., 2000). The chamber is a simple chamber and does not include any thermocouples or electrodes and cannot be imaged during depressurization. The depressurization will occur inside a temperature-controlled room and will include a DST in the chamber above the core. Any extra space will be filled with solid plastic cylinders to minimize the dead volume. On-board, degassing of these sections will be conducted over 6 or 12 hours depending on the section length and amount of hydrate present in the section.

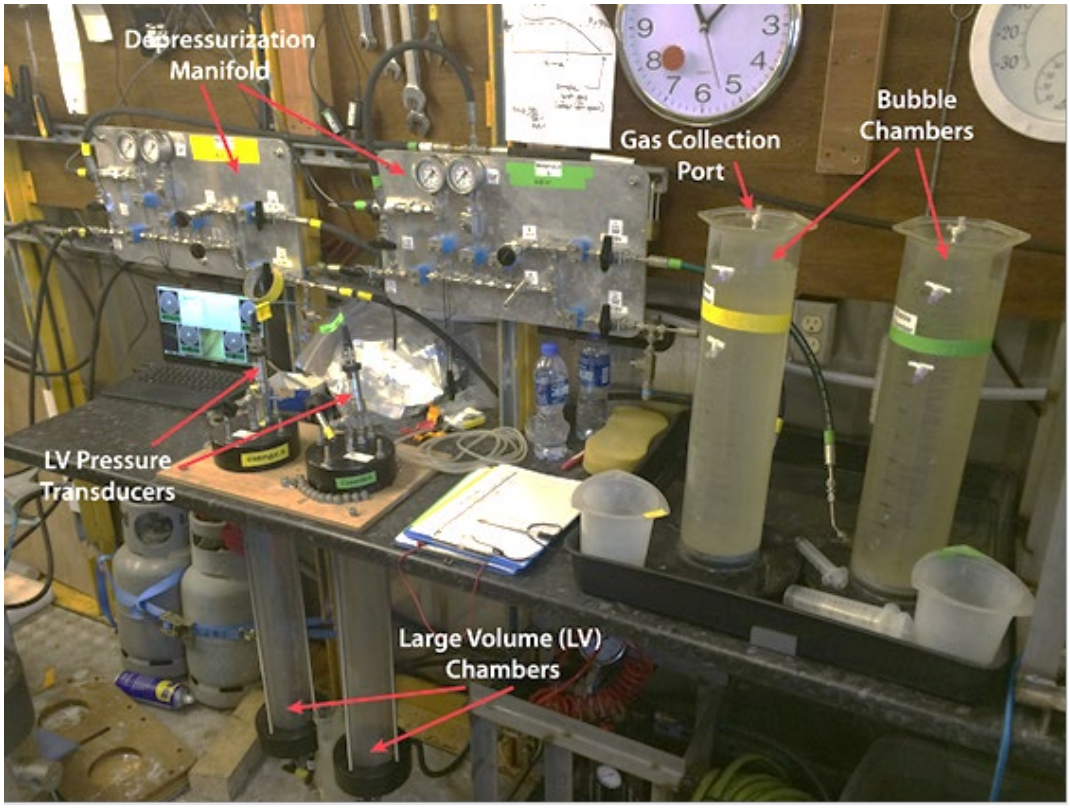

Figure 0-7. Photo of Geotek degassing manifolds and volume measuring equipment

During quantitative degassing samples will be quickly depressurized to just above hydrate stability (about 60 bar). Pressure will then be reduced by a standard increment of pressure and allowed to stabilize through hydrate dissociation. Gas forced out of the chamber during depressurization will be collected in a bubbling chamber built from an inverted graduated cylinder in a water column and measured, recorded, and sampled for compositional analysis. Water forced out of the chamber will also be measured and recorded. After pressure has re-stabilized, the process will be repeated until no pressure differential remains in the chamber. At the end of the experiment, the total amount of water forced from the chamber will be added to the last gas increment as this is an accurate assessment of the gas remaining inside the chamber which could not escape. The contents of the chamber will be removed intact in the liner if possible (and placed in a bag otherwise) for curation.

Methane concentration (along with C2-C5 hydrocarbons, O_2, and N_2) will be measured from gas collection each degassing step. Methane hydrate saturation will be calculated using the total amount of methane collected (moles) and the pore volume of the core section (calculated from porosity and core volume). Thermodynamic equilibrium will be assumed, and the calculation will be performed after Collett et al. (2008).

The initial measurements of core volume for the mass balance will be based on the inner diameter of the core liner, which will lead to an underestimation of hydrate saturation. Once final X-ray CT data from PCATS are available, the volumes will be adjusted for actual core volume based on CT slices. Samples containing the core catcher will be examined for sediment within. If the cores are disturbed by drilling or partial dissociation, or otherwise in a shape not quantifiable from the XCT data, the sample volume will be estimated by the internal diameter of the core liner. This value will yield a maximum core size and minimum hydrate saturation.

The porosity of the sample will be used to calculate the pore volume and hydrate saturation. If possible, this will be calculated from moisture and density analysis. Otherwise, porosity will be calculated based on the bulk density, measurements of grain density, and estimates of fluid density.

Quantitative degassing will be used to calculate hydrate saturation as described above and to calculate the dissolved methane concentration in background spot cores. Careful analysis of PCATS X-ray scans will be necessary to determine true background sediment intervals that do not contain hydrate-filled fractures or thin-bedded coarse-grained, hydrate-bearing layers. Due to the low solubility of methane, a larger sample will need to be cut for degassing and gas collection to obtain sufficient volume of gas. For example, a 10 cm sample from a hydrate reservoir with a porosity of 0.4 and 85% hydrate saturation will release 13 L of methane. A 10 cm background sample collected at 50 mbsf with a porosity of 0.6 and at the methane solubility of 0.05 mol CH_4/kg H_2O will only produce ~150 mL of methane. By degassing a longer section in a full 1.2 m storage section, we could increase the amount of methane produced to 1.9 L which will improve our ability to measure gas volume and methane concentration, leading to more accurate dissolved methane concentrations. Like the hydrate-bearing sediments, core volume and porosity measurements are essential for the dissolved methane calculations.

A.3.5 Slow, Quantitative Degassing

Quantitative degassing experiments of pressurized core samples are an effective approach to quantify the hydrate concentration in the core as well as hydrate composition (Bahk et al., 2013; Collett et al., 2008; Dickens et al., 1997; Dickens et al., 2000; Fujii et al., 2009; Heeschen et al., 2007; Holland and Schultheiss, 2014; Kim et al., 2013; Konno et al., 2016; Riedel et al., 2006; Santamarina et al., 2015). Degassing over 6 to 12 hours while tracking gas volume and composition will be a standard approach during shipboard operations during UT-GOM2-1. Using a similar technique over a longer period of depressurization (several days or more) of pressure cores can be used to estimate the in-situ salinity of hydrate samples (Milkov et al., 2004). Recent depressurization of laboratory-formed methane hydrate samples at UT confirm that a slow depressurization approach can determine in situ salinity within 0.5 wt. %. Characterization of molecular and isotopic composition of methane and other light hydrocarbon cases can provide insight into the relative contribution of microbial and thermogenic methane to gas hydrates (Whiticar, 1999). Due to kinetic isotope fractionation effects during dissociation (Winters, 2000), slow depressurization of pressure core samples can allow for quantification of carbon and hydrogen isotopes. A recently developed technique for measuring multiply-substituted isotopologues, aka “clumped isotopes”, of methane can further help discriminate relative microbial/thermogenic contributions and the formation temperature of methane (Stolper et al., 2014; Stolper et al., 2015).

Samples will be cut from pressure cores using PCATS or the UT Mini-PCATS. The sample will then be transferred into the degassing chamber, which will be connected to a gas collection system for analysis. We will slowly depressurize each sample over at least several days or up to 1-2 weeks) accurately observe the initial pressure of the onset of hydrate dissociation at constant temperature to calculate in situ salinity 2) observe the pressure response of the sample during perturbation and 3) collect gases over the course of dissociation to determine isotopic fractionation effects on bulk and clumped C and H isotopes. Gas volume and pressure will be monitored throughout each depressurization and multiple gas samples will be sent to Ohio State for molecular and bulk isotopic composition using FID/TCD gas chromatography and continuous flow isotope ratio mass spectrometry (C1-C5 and CO_2 gas concentration, δ13C of C1-C5 and CO_2

gases, δD of C1). From two hydrate-bearing samples we will collect gas samples to be measured for multiply substituted “clumped” isotopes (13CH3D and 12CH2D2) at the California Institute of Technology with an ultra-high-resolution isotope ratio mass spectrometer.

Depressurization/degassing may be done in conjunction with 3D CT scanning, 3D Micro-CT imaging, and/or 3D Micro-Raman Spectroscopy to further confirm and/or quantify hydrate levels.

A.3.6 LN2 Depressurization

We propose that the UT-GOM2-2 expedition use this technology to better recover whole rounds for microbiology. The overall goal of this approach is to be able to collect intact whole-rounds samples representing the range of lithofacies present in the recovered core, as well as being able to sample transitions between lithofacies. This approach will allow us to increase our understanding in several areas:

- Microbial community composition in varying lithofacies (e.g. high hydrate saturation reservoir sands-silts versus low hydrate saturation hemipelagic or interbedded mud)
- The effect of long-term pressure core storage on microbial communities
- Grain scale variation in sediment mineralogy and structure

Routine depressurization of sediment core sections (not maintaining effective stress on the specimen), consisting of mainly silt and sand sized grains hosting high saturations of gas hydrates, can cause major disturbance and disaggregation to the core sections as a result of dissociating hydrates and gas expansion of the produced gas. For example, during the UT-GOM2-1 expedition, sandy silt beds hosted methane hydrates occupying greater than 80% of the pore space. When this material was dissociated, the remaining sediments were disaggregated and fell out of the liner after the core storage chamber was opened. This precluded collection of any intact sediment cores from the reservoir lithofacies, and skewed whole round sampling towards clay-rich interbeds.

During the UT-GOM2-1 expedition, nine whole round samples were collected for microbiological analyses during offshore and dockside operations (May-June 2017). Every one of these samples were from clay-rich interbeds or overlying hemipelagic sediments that remained intact during degassing. We were unable to recover any whole rounds of the high-saturation reservoir lithofacies (sandy silt). These were recovered as slurries mixed with pore and storage chamber water and were unsuitable for microbiological analyses and only limited sedimentological analysis (grain size, X-ray diffraction).

It was not possible to recover microbiological samples from the sandy silt, high hydrate saturation lithofacies until August 2019 (over 2 years post-expedition) when the BIO chamber was used to collect one sample from this lithofacies. However, we do not know whether long periods of core storage affect the microbial communities in these sediments, though past studies have suggested changes to subsurface microbial communities following sample storage. Similarly, intact core sections from the coarse facies were not available to be analyzed for sediment fabric or lamination scale observations beyond initial PCATS X-ray scans. Only after samples measured in the K0 permeameter were depressurized under effective stress, were very limited thin sections or post dissociation X-ray CT images taken.

Figure 0-8 as a schematic of the LN2 depressurization method. First, a core with a specific lithofacies or transition between lithofacies is identified, cut in PCATS, and then transferred to a specialized 35 cm core storage chamber. In this chamber there is a weight positioned above the core liner. This core storage chamber is then attached at the flange to a specialized Dewar containing LN2 Then the water in the core storage chamber is purged at high pressure (10 MPa) with nitrogen gas. This purging process will likely only displace a minimal amount of the pore fluid, leaving most of the pore fluid, and associated microbes, intact. The ball valve on the core storage chamber is opened slightly to allow the pressures to equalize and the sample is allowed to cool. After approximately 20 minutes, the sample is frozen and then the ball valve is opened and the sample dropped from the core storage chamber into the specialized Dewar of LN2. Then, the entire system is depressurized once the sample is immersed in liquid nitrogen.

Once the samples are in LN2 they may be transferred to LN2 shipping vessels or transferred to a glove bag where they can be processed and then stored and shipped for microbiological analysis according to the standard core flow for microbiology samples. These samples will then be stored and shipped with the rest of the microbiology samples. The samples can be sublimated in a -20 C freezer while frozen allowing the methane to leave slowly leaving a hydrate-free (water ice frozen) core minimizing core disturbance from gas expansion.

This LN2 freezing approach has been successfully used to collect intact samples of coarse-grained, high saturation hydrate reservoirs. While there is the possibility of some cracks forming in the sample the cores remain as intact whole rounds and the sediment structure is largely preserved. These samples will allow for whole rounds of coarse-grained material to be collected so that the microbial communities from the reservoir materials only can be characterized. We can then compare the microbial communities from bounding muds to the reservoir materials. We would also have the opportunity to compare microbial analyses from cores collected soon after the expedition (LN2 freezing) or after long storage (BIO chamber) to learn about the effect of storage on microbes.

We can observe fine-scale structure (e.g. cross laminations, authigenic mineral bands) in the core materials that typically would be lost during core recovery and depressurization. These frozen core sections can be CT scanned post depressurization to document any structural changes during freezing and depressurization. It will likely be possible to split these core sections while frozen for description. It is then possible to thaw, dry, and saturate the sediments with epoxy and create thin sections, allowing for observation of microscopic structures and variations in minerals. This would allow for observations of gradation in grain or banding of authigenic minerals that would not be possible without freezing before hydrate dissociation. Some whole round sections could be retained under LN2 for SEM analysis of hydrate and sediment.

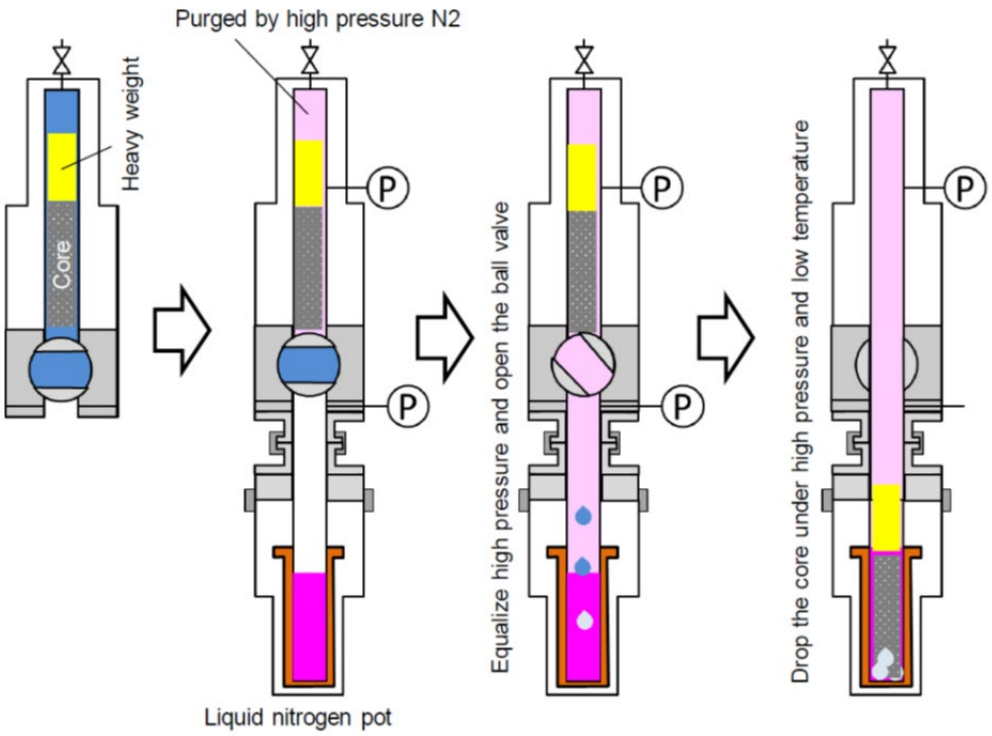

Figure 0-8. Schematic of liquid nitrogen (LN2) depressurization steps. From left to right: A. the LN2 depressurization chamber with weight, core sample, and PCATS water trapped in the closed ball valve. Prior to this a heavy weight is placed in the LN2 depressurization chamber, the chamber is pressurized with water, a core section is moved at pressure into the LN2 depressurization chamber using PCATS, the chamber is seal and removed from PCATS. A to B. The LN2 depressurization chamber is connected to a liquid nitrogen Dewar and the water in the chamber is purged with high pressure nitrogen gas. B to C. Pressure in the LN2 depressurization chamber is equalized with the liquid nitrogen Dewar and the LN2 depressurization chamber ball valve is partially opened. Water trapped in the ball valve drops into the liquid nitrogen and is frozen. C to D. The LN2 depressurization ball valve is fully opened. The pressure core drops into the liquid nitrogen and is frozen. After freezing the core, the chamber is depressurized, the depressurized frozen core remains in the Dewar, and the LN2 depressurization chamber is removed. The core can remain in the Dewar or moved to a freezer. Figure courtesy of Jun Yoneda, AIST

A.3.7 Pressure Core Transport over land

A.3.7.1 Transport to UT

1.2 m Pressure cores in storage chambers will be transferred inside the Geotek cold storage container on a shipping vessel from the rig to the port. The container will be kept cold until they can be moved to reefer container on a vibration limited truck. The storage chambers will be moved from the container to the overpack frame where they will be individually placed inside cold large DOT approved overpacks (shells capable of safely containing the storage chamber contents if the storage chamber was to fail). From Schultheiss et al (Schultheiss et al., 2017).

“Geotek pressurized storage chambers are secured and shock-mounted within the overpack cylinders in all dimensions by a machined support structure, so there is no risk of damaging the

overpack cylinder during transit. To avoid any possibility of creating an explosive mix within the overpack cylinder, the free volume within the overpack cylinder is purged with nitrogen once the pressurized storage chamber has been loaded. Any methane released into the overpack will mix with the nitrogen only."

The truck will then transport the cores from the port to SLC and later, SLC to the UT Pressure Core Center (UT PCC). The Data Storage Tag (as described above) from Star-Oddi for pressure and temperature tracking will be placed inside the storage chambers with the core to track pressure and temperature during transport and storage between the time the subsection of core is placed in the 1.2 m storage chamber on-board and the time the chamber is finally depressurized.

A. 

B. 

C. 

Figure 0-9. Pressure Core Transport. A. Overpack frame with individual overpacks mounted inside (Schultheiss et al., 2017). B. Pressure Core Storage Chambers waiting to be moving into the reefer truck and placed inside overpacks. C. Pressure core storage chamber being moved into place inside the UT Pressure Core Center.

A.3.7.2 Transport from UT to other institutions

Each receiving institution will provide their own DOT-certified transfer chambers or contract Geotek for use of their Overpak system.

A.4. Whole Round Conventional and Intact Depressurized Core

This section describes some of the detailed analysis methods of whole round conventional and intact depressurized cores.

A.4.1 Thermal Imaging

Thermal Imaging (MSCL-IR) – Geotek Ltd. The Geotek infrared imaging system (MSCL-IR) rapidly captures thermal images with a resolution of ±0.1˚C. IR scanning is performed as soon as the cores are received from the coring team. This IR will can scan up to 9.5 m core of any core diameter that Geotek provides (all conventional cores and depressurized PCTB cores).

IR scans are very quick (less than) 5 minutes per core.

A.4.2 Scanning

Whole core logging will be done using the Geotek MSCL-S and will include gamma density, P-wave, magnetic susceptibility, resistivity, and natural gamma. See https://www.geotek.co.uk/services/mscl_services/.

A.4.2.1 Gamma density

Gamma Density – Geotek Ltd.

A gamma ray source and detector are mounted across the core on a sensor stand that aligns them with the centre of the core. A narrow beam of collimated gamma rays is emitted from a 137-Caesium source with energies principally at 0.662 MeV. These photons pass through the core and are detected on the other side. At this energy level the primary mechanism for the attenuation of gamma rays is by Compton scattering. The incident photons are scattered by the electrons in the core with a partial energy loss. The attenuation, therefore, is directly related to the number of electrons in the gamma ray beam (core thickness and electron density). By measuring the number of transmitted gamma photons that pass through the core unattenuated the density of the core material can be determined.

To differentiate between scattered and transmitted photons the gamma detector system only counts those photons that have the same principal energy of the source. To do this a counting window is set which spans the region of interest around 0.662 MeV.

A.4.2.2 P-wave Velocity

Acoustic Rolling Contact (ARC) P-wave Velocity Geotek LTD

The Acoustic Rolling Contact (ARC) transducers for the Geotek MSCL systems incorporate the latest acoustic technology to achieve precise, high quality and repeatable results from cores of almost any composition in diameters from 50 to 150 mm. The ARC transducer uses a stationary

active transducer element which is made from a unique polymer material that combines high coupling with relatively low acoustic impedance.

ARC transducers have two distinct advantages for whole and split sediment cores: 1) ARC transducers couple directly to the core liner and hence do not require any coupling fluid between the transducer and the core liner. This eliminates the need for the user to wet the core liner and ensure that it stays wet during the logging process. 2) Spectral analysis of the received signal from the new ARC transducers will enable sediment characterisation work because of their improved pulse and coupling characteristics.

The stationary composite element is surrounded by an acoustic oil and a rotating soft deformable diaphragm. This arrangement enables the complete transducer assembly to rotate as the core is passed through the spring loaded opposing transducer pair. The careful internal design provides radiused internal locating lips which gives a wide contact area and positive repeatable location of the transducers over core diameters within the range 50mm to 150mm.

A.4.2.3 Magnetic susceptibility

Magnetic Susceptibility – Geotek LTD

Two sensors are available from Bartington Instruments (Bartington) for integration with a Geotek MSCL system, a loop sensor (MS2C) and a point sensor (MS2E) that are paired to the Bartington MS3 meter with a measuring range of 26 SI.

The Bartington loop sensor (MS2C) is used for volume susceptibility measurements of whole sediment and rock cores. It is available in a range of internal diameters from 30 mm to 150 mm. The Bartington point sensor (MS2E) is used for surface scanning and providing high-resolution surface measurements on split sediment or slabbed rock cores.

A.4.2.4 Resistivity

Non-contact resistivity – Geotek Ltd

The NCR [non-contact resistivity] technique operates by inducing a high-frequency magnetic field in the core from a transmitter coil, which in turn induces electrical currents in the core which are inversely proportional to the resistivity. Very small magnetic fields regenerated by the electrical current are measured by a receiver coil. To measure these very small magnetic fields accurately a difference technique has been developed which compares the readings generated from the measuring coils to the readings from an identical set of coils operating in air. This technique provides the requisite accuracy and stability required. Resistivities between 0.1 and 10 ohm-meters can be measured at spatial resolutions along the core of approximately 2cm.

A.4.2.5 Natural gamma

Natural Gamma Spectrometry = Geotek LTD

The entire sensor assembly comprises at least one, but generally three, 3” x 3” NaI(Tl) detectors housed in 6” diameter lead shields. Each detector unit has a NaI(Tl) crystal optically coupled to a photomultiplier tube and connected to an integrated bias base and MCA. Emitted gamma rays hit the NaI(Tl) crystals which produces a pulse of light. These photons strike the photomultiplier tube, producing a small electrical current to give a voltage pulse. The peak height of the voltage pulse is related to the energy of the gamma emission which is recorded by the multichannel analyser in one of 1024 channels.

A.4.3 3D CT imaging

From Standard X-ray CT System (XCT) – Geotek Ltd.

The versatile Geotek XCT system allows for the acquisition of both 2D X-ray transmission images and full 3D helical X-ray CT volumes of core samples through horizontal scanning techniques. The system is available with a 130 kV or 180 kV microfocus closed X-ray source to meet different industry needs and core types. Whether lined whole core, split core, or slabbed core sections, the XCT's flexible geometry allows for scanning cores of varying diameters. Adjustable X-ray source and detector positions can be optimised for image quality, resolution and core size.

See specifications at Standard X-ray CT System (XCT) – Geotek Ltd.

A.5. Split Core

A.5.1 High Resolution Magnetic Susceptibility

High resolution surface measurements of magnetic susceptibility on split core will be made with a Bartington point sensor (MS2E). The sensor will be integrated with the Geotek MSCL split core scanning system and paired to a Bartington MS3 meter with a measuring range of 26 SI.

From https://www.geotek.co.uk/sensors/magsusc/

An oscillator circuit in the sensor produces a low intensity of approximately 80 ampere per meter (A/m) root mean squared (RMS) non-saturating, alternating magnetic field (0.565 kHz for the MS2C sensor and 2 kHz for the MS2E sensor). Any material in the near vicinity of the sensor that has a magnetic susceptibility will cause a change in the oscillator frequency. The Geotek MSCL system electronics convert this pulsed frequency information into magnetic susceptibility values reported as SI or CGS.

The MS2C and MS2E sensors are electronically calibrated to measure a single standard of stable iron oxide tested and analysed by Bartington. All magnetic susceptibility sensors are supplied with a stable iron check piece, which can be used to check the long-term consistency of the calibration.

The data acquired using the MS2C and MS2E sensors can be presented as uncorrected, volume specific magnetic susceptibility, corrected volume specific magnetic susceptibility or mass specific magnetic susceptibility.

A.5.2 Photo scan

From https://www.geotek.co.uk/sensors/geoscan/

The Geotek Geoscan V line scan camera has one massive c.5340 pixel CCD. Incoming light is passed through a set of red, green, and blue filters to produce true independent colour separation. Averaged image data can be converted to these RGB values and saved in a separate file to facilitate quantitative comparisons between cores and other down-core measurements.

Images can be collected over the full core width between 100 and 1000 lines per centimetre, corresponding to 100 and 10 micron pixel sizes, respectively. Images are output as 48-bit RGB TIFF images but are quickly and easily converted to JPEG or other formats as required. Each Geotek core section image has a companion XML metadata file, containing important metadata pertaining to the core section and imaging conditions. A ruler can be generated next to the image, depicting either depth in core section or depth in core.

A.5.3 X-ray Fluorescence

Geotek offers two XRF spectrometers: the high resolution and ultra-sensitive Geotek XRF, and the popular hand-held Olympus Vanta, we will use the Vanta.

A.5.4 Color Reflectance

From https://www.geotek.co.uk/sensors/spectrophotometer/ :

The CM-700d spectrophotometer uses a diffused illumination, 8 degree viewing angle with a pulsed xenon lamp providing the illumination. The instrument detector collects light in 10 nm increments between 400 nm and 700 nm wavelength ranges. The spectrophotometric method utilizes multiple sensors to measure the spectral reflectance of the object at each wavelength or in each narrow wavelength range. The sensor's electronics then calculate the tristimulus values from the spectral reflectance data using integration. The measuring aperture is selectable between 8 mm (MAV) and 3 mm (SAV). For each measurement, data for the specular components included (SCI) and excluded (SCE) are recorded simultaneously to analyse the core surface.

See the Geotek website for more details.

A.5.5 Near-IR scanning

Near-IR scanning is not planned for this expedition.

A.6. Oregon State Microbiology of Conventional Core

The exact protocol for microbial analysis of sediment at Oregon State is still to be determined. Oregon State, with others, will identify challenges such as low biomass, lab contamination issues, and sediment constituents that interfere with molecular-based studies to determine strategies for this expedition.

Strategies will consider

1. Understanding both taxonomically diverse and functionally specific microbes from a range of geological materials associated with deep hydrate-bearing sediments;
2. Differentiating between microbes present in coarse vs. fine grained materials, and at interfaces
3. Optimizing data interpretation and integration of different science elements;
4. Biogeochemistry data to allow integration with porewater geochemistry and physical properties/sedimentological measurements.
5. judicious tracer strategy to enable contamination checks, on-board and dockside lab protocols;
6. sediment constituents that interfere with molecular-based studies
7. minimizing contamination

A.6.1 DNA sequencing-based microbial diversity

DNA extraction and sequencing to determine the dominant types of microbes in the sediments will be performed on 60 selected samples representative of key facies defined at least in part by grain size and total organic matter concentration. Grain size and TOC can define the types of

biological productivity present. Evidence of the presence and diversity of methanogens and other microbial community members that may contribute to the degradation of buried organic matter which ultimately results in the conversion of simple metabolic waste products into methane is key to understanding how methane accumulates in the system. Detection of key microbes in the sediments within or proximal to the hydrate stability zone would support the idea of methane generation relatively close to the location of hydrates. The absence of these microbes would lend support to the premise that methane is generated at some distance (e.g., deeper in the formation) and then migrates to a location where hydrates form.

A.6.2 RNA sequencing-based microbial activity

RNA extraction, conversion to cDNA, and then sequencing of the cDNA will be done on the same samples as for DNA sequencing as a way to indicate whether RNA transcripts specific to methanogenic processes and other related microbial metabolisms are present and active in the system. This will complement the DNA sequencing by indicating the functioning metabolic pathways of cells in the sediments.

A.6.3 DNA-based levels of selected functional genes

To establish whether key functional capabilities associated with methanogenesis reside within the microbial communities we will use droplet digital polymerase chain reaction (ddPCR) on the extracted DNA to determine the concentrations of specific genes associated with methane production. The measure is quantitative and should allow estimates of gene abundance in respective sediments and therefore a determination of whether the capability predominates in one sediment type or another, or whether it is present in the sampled strata at all.

A.6.4 Sample quality

Routine samples of the drilling fluids, make-up water, water used in pressure vessels, and exteriors of the cores to be used in comparative studies of the microbial communities that are found in the portions of the cores deemed to be of high quality. The microorganisms found in these potential contaminating materials, along with negative controls used in the lab, will be used as microbial tracers to determine the possible source and level of contamination. These analyses will be performed along with the aforementioned DNA sequence-based measurements. Inclusion of chemical tracers that are purposefully added to the drilling fluid or detected as a part of the porewater chemistry sampling program will also be included. This QA/QC program will allow us to analytically define the quality of samples acquired as a part of the UT-GOM2-2 expedition.

A.7. UT Compressibility, Permeability of Pressure Core

Lithofacies specific samples for Constant-Rate-of-Strain (CRS) measurements and Ko permeameter will be cut from the sand-rich hydrate-bearing and mud pressure core brought to UT.

UT will focus on establishing an estimate of the compression behavior with hydrate in place; an estimate of the compaction that will occur during dissociation; and a comparison of the compression behavior with and without hydrate in equivalent reservoir facies.

A.7.1 Compressibility

We will perform uniaxial compression on pressure cores of the reservoir-bearing facies with and without depressurization. On one set of pressure-core samples, we will perform constant-rate-

of-strain uniaxial consolidation tests on samples within the hydrate-bearing reservoir facies while keeping the sample within the hydrate stability zone. On a 2nd set of pressure-core samples, as equivalent as possible to the first two, we will load the samples to the in-situ stress state and then dissociate the hydrate and explore the compression behavior. On a 3rd set of depressurized samples, we will perform CRS compression tests.

A.7.2 Permeability

At UT we will measure the permeability of the core samples to water using our Effective Stress/Permeability Chamber (K0 Permeameter). The K0 Permeameter enables the analysis of petrophysical properties of pressure core samples while maintaining in situ hydrostatic pressures. Once the sample has been extruded into the rubber sleeve under a constant pressure, the cell will be isolated from the high pressure pump, the K0 Chamber separated from Mini-PCATS, and attached to an Axial Loading and Transfer System (ALTS). A separate pressure control system will control the confining and pore pressures. The ALTS can then be used to apply vertical loads up to 10 MPa (measured using an integral load cell) to the sample simulating in situ vertical stress conditions enabling consolidation testing to take place. Vertical displacement will be measured using the precise motion of the ALTS motor.

Direct flow/permeability tests will be performed using UT flow pump systems through the sample via pressure ports connected directly to permeable porous discs located at the top and bottom of the sample.

For permeability to water core length will be cut in the Mini-PCATS and then transferred into the K0-permeameter sample chamber into a flexible rubber sleeve under a constant pressure. The stress state will be increased to estimate in situ conditions. The sample will then be held within the hydrate stability zone while water is introduced at a steady rate to determine the permeability to water in the presence of the hydrate-bearing phase. Different rates will be used to look at permeability versus flow rate.

Depressurized cores will also be placed in our Ko-permeameter. We will increase stress state to estimate in situ conditions. We will flow water at a steady rate to determine the permeability to water in the presence of the hydrate-bearing phase.

A.8. UT Micro-CT of Natural Sediment with Synthesized Hydrate (TBD)

The UT Micro-CT experiments will seek to examine the influence of sediment hydrate growth, static permeability and evolution of permeability upon depressurization.

A.9. UT Very Slow Depressurization: Gas Hydrate Composition and Possible Pore Fluid Extraction without Hydrate Dissociation (TBD)

Pressure core samples will be subjected to very slow, quantitative degassing and gas sampling, and a new high pressure pore water extraction technique.

This slow approach combined with pore water analysis approach will allow us to better understand the hydrate and pore fluid chemistry and the in situ thermodynamic state of the reservoir. Slow degassing will be used to pinpoint the initial pressure of dissociation to calculate in situ salinity, while monitoring the pressure response to perturbation and collecting samples

for gas composition. Gases will be measured for bulk molecular and C and H isotopic composition, as well as 'clumped' methane isotopologues to estimate methane formation temperature. Pore waters may be extracted by displacing in situ fluids and collecting the expelled fluids. This will provide a first-ever analysis of pressure core interstitial water without alteration by dissociating hydrate.

A.10. Georgia Tech Sediment hydro-mechanical behavior under high effective stress (TBD)

2" whole round core from each of the six primary sediment types from hole 1 and/or hole 2 including: (1) overburden seal; (2) upper reservoir sand; (3) inter-bedded upper reservoir mud; (4) water-bearing sand; (5) lower reservoir sand; (and 6) inter-bedded lower reservoir mud. These samples will be transferred to Georgia Institute of Technology (Georgia Tech).

Georgia Tech will focus on quantifying the impacts of high effective stress (up to 25MPa) on the compressibility, sand crushing, and permeability (horizontal and vertical) of these GC955 sediments. Hydrate-bearing cores recovered from this GOM2 drilling are subjected to 24-25MPa total stress in situ (i.e., ~20MPa water pressure and ~4.1-4.5MPa effective stress depending on core recovery depth below sea floor). Due to low water permeability of the upper seal, depressurization for gas production from this deposit will cause significant pore pressure drop that takes a relatively long time period to recover; the decreased pore pressure will be transferred onto sediment skeleton to sustain the overburden total stress. Such stress transfer increases the effective stress that essentially governs soil behavior, including compressibility, sand crushing, and permeability. This information is essential to the evaluation of gas production potential and geomechanical instability of deep-water hydrate deposits.

A.10.1 Oedometer Tests

Post pressure core testing samples with THF hydrate will be studied measuring compressibility, the stress-volume response, and sand crushing.

A.10.2 Permeability

Post pressure core testing samples with THF hydrate will be studied looking at the change in horizontal and vertical permeability to water under high effective stress.

A.10.3 Index Properties

Index properties of the sediment will be measured after the hydro-mechanical behavior looking at grain size, particle shape and surface texture, specific gravity, and SEM/XRPD.

A.11. UT Micro-Raman (TBD)

Analysis of the 2D (3D) micro-Raman spectra before and during dissociation will allow identification of phases and molecules present in the samples including the interfaces between methane hydrate grains and sands in micro-submicron scale spatial resolution. 2D (3D) imaging in micro resolution will be reconstructed to illustrate the geometry, volume ratio, methane concentration, gas composition (methane, ethane, propane, etc.),and brine composition in the samples. Analysis of the Raman spectra with variations of pressure-temperature and composition on these samples as a function of time will be used to probe the kinetics of

methane release and migration from the methane hydrate phase into the surrounding sand and pore water.

Pressure core samples may be cut and moved to a Micro-Raman pressure chamber for Raman analysis at UT.

A.12. USGS PCCT Assessment of Pressure Core (TBD)

Hydrate-bearing pressure cores may be transferred from UT to USGS Woods Hole.

The Pressure Core Characterization Tool (PCCT) can be used to characterize the existing hydrate-associated formation and enable forward modeling of the reservoir response to methane extraction as an energy resource. Links between permeability, relative permeability, compressibility, stiffness, shear strength and gas chemistry for the major GC955 sediment types are also quantified.

Pressure Core sample are likely to be transferred to the USGS Woods Hole to be analyzed on the PCCT

A.12.1 ESC

Sub-sections of pressure core can be cut and moved to the Effective Stress Cell (ESC) using the PCCT manipulator (MAN). ESC will be used to measure water permeability and compressibility at in situ effective stress and hydrate-saturation conditions (site characterization) as well as at elevated effective stress (up to ~10MPa) in hydrate-free sediment for testing responses of the different sediment types to methane extraction via depressurization (reservoir modeling). Relative permeability measurements (water flowing through gas-bearing sediment) will be made after hydrate dissociation. ESC measurements of water permeability will be directly comparable with DOE/NETL and UT measurements made using their modified ESC devices. Specific measurements include Coefficient of consolidation; Coefficient of volume compressibility; Compression, recompression indices; Permeability (relative, hydrate-free, with gas + water); Permeability (water, with and without hydrate); and settlement during hydrate dissociation.

A.12.2 DSC

Sub-sections of pressure core can be cut and moved to the Direct Shear Chamber (DSC) using the PCCT manipulator (MAN). The DSC will be used to make measurements of direct shear strength and P-wave velocity at in situ vertical loads and hydrate saturations. P-wave velocity measurements will be compared to sediment morphology from micro CT imagery at DOE/NETL and/or UT. Strength measurements will be compared with the DOE/NETL extensional mode measurements and the UT triaxial shear measurements. Specific measurements include Coefficient of consolidation; Coefficient of volume compressibility; Compression, recompression indices; Compressional wave speed; Peak shear strength; and settlement during hydrate dissociation.

A.12.3 Hydrate Saturation and Gas Analysis

Each specimen tested in the DSC or ESC can be depressurized slowly under confinement to preserve sediment fabric and quantify the hydrate saturation. Analysis of gas released during dissociation via gas chromatography, flame ionization, thermal conductivity and pulsed discharge-helium ion detection will include the concentration of C1 - C6 hydrocarbons and the concentration of CO_2, N_2, O_2 and H_2. Stable carbon isotope analysis of methane via cavity ring-

down spectroscopy using the USGS discrete specimen analysis module, with analytical confirmation by isotope radio mass spectrometry will also be pursued. Similarly, gas chemistry and isotopic data can be linked back to shipboard and UT gas analyses to increase the completeness of those downhole profiles.

A.12.4 Oedometer Tests

Per the section above, re-molded sediments can be measured using a Standard Oedometer Cell after PCCT analysis.

A.12.5 Index Properties

Index property measurements, such as grain size, grain density, SEM and XRPD analyses can be made after PCCT analysis and can be linked back to OSU index properties to increase the completeness of their downhole profiles. XRPD samples should be sent to James Hutton, grain size samples to UNH.

A.13. NETL Core-Scale Characterization with Micro-Scale Visualization (TBD)

Pressure cores can be sub-cored and then measured in an effective stress chamber or scanned with the micro CT scanner (resolution up to 1 µm) at NETL to provide observation of hydrate pore habits and interactions with sediment matrix. These observations can then be linked to measured core properties based on physical/theoretical models and numerical simulations.

Pressure core samples may be transferred to NETL for Micro CT imaging.

A.14. BIO Chamber High Pressure Cultivation with Microbial Analysis (TBD)

Microbial assessment using the USGS Woods Hole BIO chamber can be used to help develop a conceptual model of the origin of methane in the system and be integrated into reactive transport models that allow reconstruction of the biogeochemical setting.

Pressure core samples of hydrate-bearing sediment stored at UT Pressure Core Center will be designated for Microbial cultivation under pressure.

A.14.1 BIO

The sampler for multiple bio reactor chambers (BIO) will be shipped to UT where sediment subsamples will be loaded into the BIO chamber using Mini-PCATS while maintaining *in situ* pressure by pumping the chamber full of argon gas as described in (Santamarina et al., 2015). A sterile (previously autoclaved for 40 min at 120°C and 100 kPa) exchangeable bio-reactor chamber will be attached to the BIO chamber (Fig. 1). To avoid contamination, 10 mm of surface sediment will be scraped from the circular face of the pressure core and discarded. Thereafter, uncontaminated sediment will be collected in 10 mm intervals at the head of the scraper and dropped into the sterile bio-reactor chamber, which will contain anoxic liquid growth medium designed to match *in situ* pore water composition (e.g. salinity) based on geochemical analysis and pressurized with methane to 25 mM. The incubations will run at 4°C for 6-12 weeks due to the expected slow growth rates of these deep subsurface microbes (Parkes et al., 2009). At the end of the incubation, samples will be preserved for microbial characterization by DNA, RNA, and microscopy, and transferred into fresh medium for further isolation efforts. Linkage of the

results to *in situ* microbiology and environmental parameters in matched samples from other analysis efforts as described in this plan will be made.

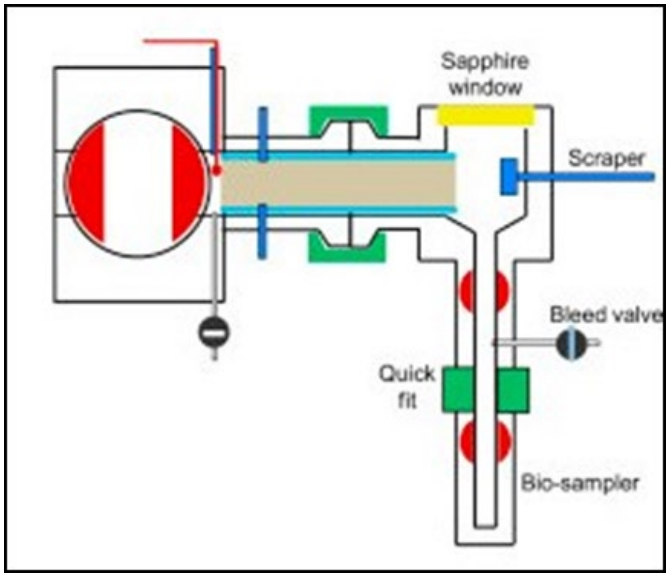

*Figure 0-10. Depiction of BIO chamber for sub-sampling of methane hydrate sediment cores into bio-reactor chambers for microbial cultivation at 35 kPa. Figure modified from Santamarina et al. (2015).*DNA sequences; microbial microscopy (direct cell counting)

High-pressure microbial cultivation experiments may be performed on methane hydrate-bearing sediment cores recovered and maintained at 20 MPa and *in situ* salinity using the BIO chamber (Santamarina et al., 2012) to enrich for piezo- and halo-philic microbes by Georgia Tech at UT.

A.15. Velocity Saturation Behavior under Pressure (TBD)

Best methods and location for analyzing the velocity saturation behavior of pressure cores, if done, are to be determined.

Appendix B. The Core Analysis and Distribution Technical Advisory Group

The GOM^2 PC Technical Advisory Group was charged with the following:

1) Identifying and prioritizing the key experiments to achieve science goals of the expedition
2) Identifying and reviewing the specific and optimal methodology/protocol for each experiment
3) Prioritizing experiments including review of submitted proposals for analysis and distribution of recovered cores

Participants:

Peter Flemings, UT

Ray Boswell, DoE

William (Bill) Waite, USGS Woods Hole

Steve Phillips, USGS Woods Hole

Yongkoo Seol, DOE - NETL

Sheng Dai, Georgia Tech

Tim Collett, USGS Devner

Carla Thomas, UT

Appendix C. UT-GOM2-2 Sample request form

A copy of the sample request form will be posted on our website once it has been released. [UT-GOM2-2: Gulf of Mexico Deepwater Hydrate Coring Expedition (utexas.edu)]

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