

# Oil & Natural Gas Technology

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## Core Procedures Manual (Draft Volumes 1-3)

# DRILLING AND PRODUCTION TESTING THE METHANE HYDRATE RESOURCE POTENTIAL ASSOCIATED WITH THE BARROW GAS FIELDS

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Office of Fossil Energy

**Detailed Core Procedures Manual**  
**Volume 1 – Core Acquisition**  
**Draft, Rev1.0 – September 21, 2009**

**INTRODUCTION, BACKGROUND**

This “Core Procedures Manual, Volume 1” contains a description of proposed best practices for use during coring operations for the Barrow Gas Hydrate Test Well Program. This document is designed to describe the responsibilities of well site operations personnel, primarily the core acquisition contractor and coring fluids contractor, Weatherford/Omni and Halliburton/Baroid respectively.

This well is being drilled as a gas hydrate test well within Phase 2B of the North Slope Borough (NSB) - US Department of Energy (DOE) Gas Hydrate Cooperative Research Project. This volume of the core procedures manual includes several written elements of the coring plan, including the following:

- Risk mitigation measures - Planning phase and Operations phase
- Personnel roster, roles, and responsibilities
- Pre-Core Planning
- Operational procedures covering mud specifications, core acquisition, and core retrieval

A total of four documents have been written to cover the complete scope of coring, core preparation, on-site core analysis, and subsequent decision criteria to complete the well. These documents (with volumes 2, 3, and 4 under separate cover) are as follows:

- ***Volume 1 – Core Acquisition***  
Discusses pre-well planning and coring operations up through core laydown
- ***Volume 2 – Core Handling, On-site Analysis***  
Discusses core handling, sub-sampling, preservation, and transportation
- ***Volume 3 – Post-Field Core Analysis***  
Discusses off-site core analysis tests and methods conducted by the Science Party
- ***Volume 4 - Decision Criteria to Complete Well***  
Discusses the test results that will trigger a decision to complete or abandon the well

Lessons learned from previous hydrate-bearing cored wells, including the Mallik (1998 and 2002), Hot Ice (2004), and Mt. Elbert (2007) arctic onshore wells, and certain offshore research programs are incorporated into these documents where applicable.

The program has been designed to deliver the key objectives identified by the Gas Hydrate project research team and the Barrow Field development team including members of DOE, USGS, NSB, PRA, and supporting service companies. This document will be reviewed and refined through a number of meetings leading up to well spud, including a coring budget workshop being planned for September 2009. All four volumes of this manual will then be used as an on-site policy and procedures manual to guide all aspects of the coring program. It is also intended to serve as the all-encompassing master reference document for all interested

stakeholders.

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# 1 PROJECT OVERVIEW

At least one stratigraphic test well will be cored in the East Barrow gas field, east of Barrow Alaska. If hydrates are confirmed at the first location, the well will be completed as an observation well and a horizontal well will be drilled nearby to produce free gas and dissociate the hydrate zone. If no hydrates are found at the East Barrow Gas Field, a second stratigraphic test well will be drilled and cored in the Walakpa gas field, south of Barrow. This manual is intended to describe all aspects of the coring operation at East Barrow, and if a second well is cored at Walakpa.

One of the primary objectives of this program is to obtain a minimally disturbed 3 inch whole core from gas hydrate-bearing sandstone beneath the permafrost, from a field that is currently producing gas at continuous and commercial rates. It would be the first well to confirm the presence of hydrates in a field undergoing depressurization and depletion. The combination of core data, log data, and long term production data represents a huge step forward in confirming the potential of hydrates as a commercial source of energy.

## 1.1 Project Background, Justification, Phasing

The DOE and NSB have teamed up to confirm the presence of hydrates at one of the Barrow gas fields. The Barrow gas fields, as shown in Figure 1, lies within the boundaries of the hydrate stability zone as mapped by the USGS. The DOE and other government agencies including the USGS will progress its understanding of hydrates while the NSB will gather critical information about the reservoir depletion mechanism of gas fields that provide heat and electricity to the community of Barrow.

**Figure 1: ANS Gas Hydrate Stability Zone Extent with Barrow Gas Fields Highlighted. (Similar to previous USGS map).**

Phase 2B represents the execution phase following a reservoir study phase (Phase 1) and well planning and AFE phase (Phase 2A). The first version of this document was drafted in the summer of 2009 during Phase 2A to understand coring tasks, personnel responsibilities, and equipment requirements in order to estimate the scope and costs of on-site core acquisition and on-site core analyses. A final draft of this document will be published shortly before the well is spud in fall of 2010.

The well site of this coring program will be the East Barrow field, and the Walakpa field if no hydrates are found at East Barrow. The two fields, and the location of planned core wells and production test wells is shown in Figure 2 below.

**Figure 2: East Barrow and Walakpa Gas Fields, with Postulated Outline of Gas Hydrates**

## **1.2 Coring Requirements**

The coring operation is intended to prove the presence of hydrates in one or more of the Barrow Gas fields and measure the nature and character of the hydrate and its host formation. Described below are the specifications for each well and the overall requirements of the program.

### **1.2.1 East Barrow Field**

The Savik No. 1 well, located in the East Barrow Field, is planned to have wireline-retrievable core taken from the Walakpa and Barrow sandstones. At the prognosed core depths, gas hydrate is expected in both sand intervals. Approximately 10 feet of the Walakpa sand and 100 feet of the Barrow sand, which includes both the Upper and Lower sands, will be cored. These sand packages are shown in the South Barrow No. 18 type log (Figure 3).

The thickness of the Walakpa sand at the Savik No. 1 location is expected to be approximately 10 feet thick. Therefore, to insure that a hydrate bearing core is captured, the coring operation is planned to start approximately 15 feet above the prognosed top of sand, and extend approximately 23 feet below the prognosed bottom of sand for a total cored interval of 48 feet, for a total of two core runs at 24 feet per core run.

The thickness of the Upper and Lower Barrow sands at the Savik No. 1 location is approximately 100 feet thick. To insure that a hydrate bearing core is taken, the coring operation is planned to start approximately 10 feet above the prognosed top of sand, and extend approximately 10 feet below the prognosed bottom of sand for a total cored interval of 120 feet, for a total of five core runs at 24 feet per core run.

Core points for Savik No. 1 will be picked by the wellsite geologists based on correlations from adjacent South Barrow wells. Logging-while-drilling (LWD) tools including near-bit gamma-ray (GR) and resistivity measurements will be used to minimize depth point prediction uncertainty. The Walakpa and Barrow sands are expected to be penetrated at 1715 ft tvdss and 1935 ft. tvdss, respectively, but both tops are subject to change following correlations from real-time LWD logs. Actual core points are 10 feet above the formation tops to ensure both the interval top and gas hydrate are captured. After the Barrow sandstone is cored, approximately 150 ft. of rathole will be drilled to prepare the well for wireline logging and eventual completion as an observation well, if hydrates are confirmed.

Well S BARROW 18  
 Well ID 500232001700  
 Field EAST BARROW  
 County BARROW  
 State/Prov  
 Country US  
 Location TWP: - Range: - Sec.  
 Status P&A-G

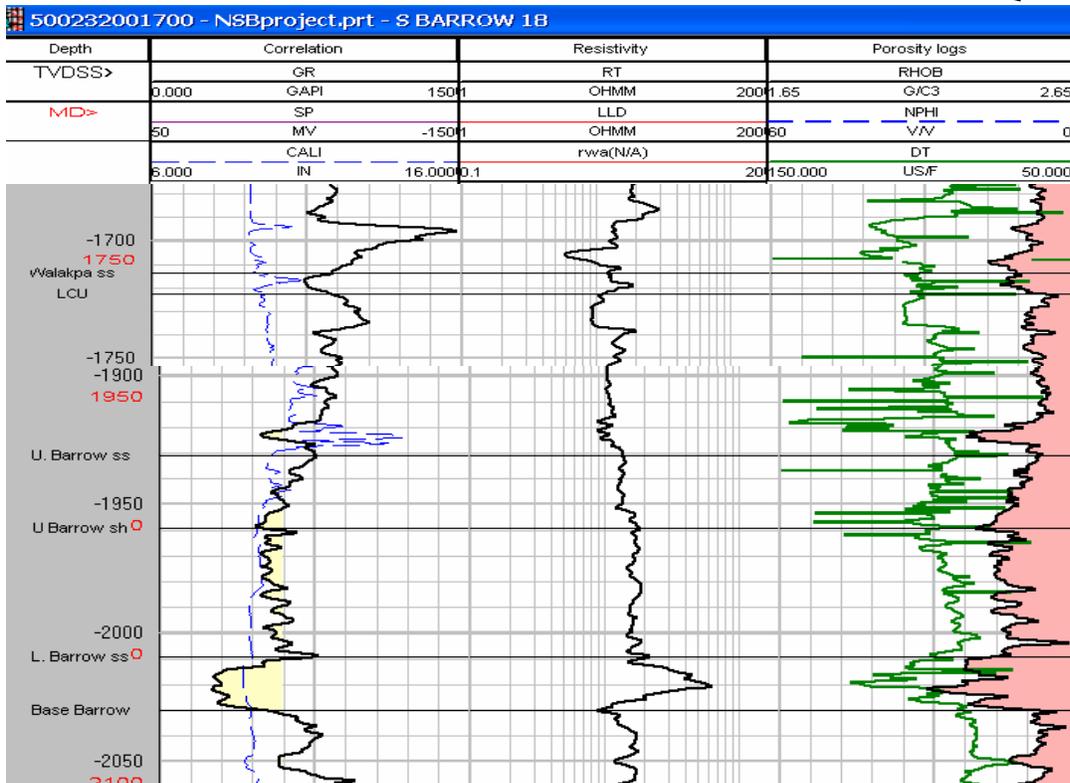
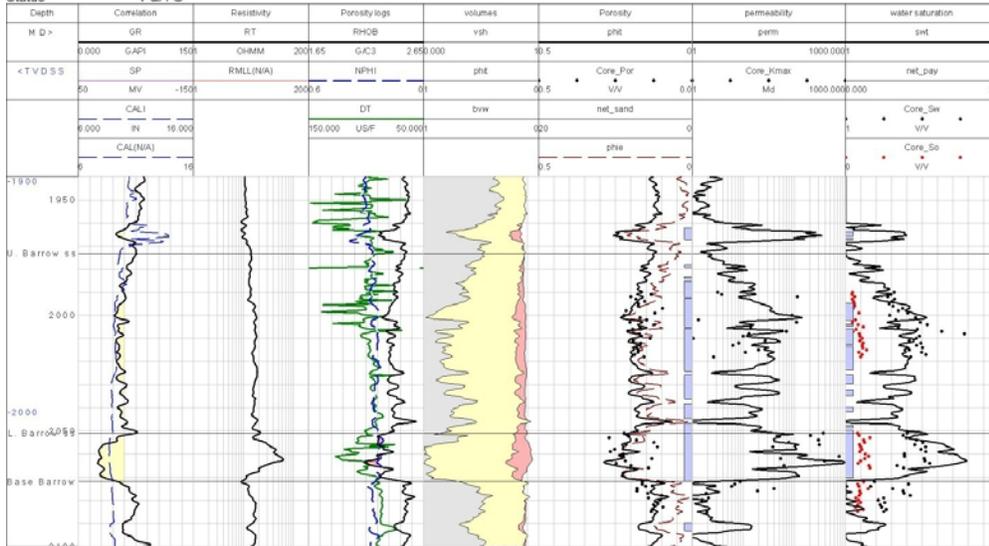


Figure 3: East Barrow Type Well (S. Barrow No. 18), Showing Anticipated Core Interval

The Walakpa core is intended to prove the presence of hydrates in this sand at a location that is known to be within the hydrate stability zone. The Walakpa sand is not productive in this area, so it is not the target of a long term production test.

Since the Barrow sandstone is productive in this area, it is the target of a long term production test, provided hydrates are confirmed to be present. Reservoir properties and lateral continuity of the Barrow sandstone are well understood, based on seismic and core data from numerous wells that penetrate the reservoir. The Barrow sandstone, interpreted to be a series of near-shore marine sands, contains relatively low porosity and permeability sands compared to other shallow sandstones on the North Slope such as Ugnu and West Sak. In the Upper Barrow sand, porosities range from 17% to 19% and permeabilities range from 13 to 97 md. In the Lower Barrow sand, porosities range from 19% to 24% and permeabilities range from 33-364 md. The Barrow sandstone, Jurassic in age, is believed to have been buried to a depth of around 10,000 feet subsea, which consolidated the sand packages. In Tertiary time, the sands were uplifted to their current depth near 2,000 feet subsea.

### **1.2.2 Walakpa Field**

If hydrates are not found at Savik No. 1, the rig and operating spread will be moved to the Walakpa field to conduct a second attempt at confirming hydrates. The sand thickness at the Walakpa hydrate test well location (Walakpa No. 15) is expected to be approximately 18 feet thick. Therefore, to insure that a core of the hydrate well is taken, a core is planned to start approximately 15 feet above the prognosed top of sand, and extend approximately 15 feet below the prognosed bottom of sand for a total cored interval of 48 feet, for a total of two core runs at 24 feet per core run. The interval to be cored is shown on the Walakpa No. 1 type log (Figure 4). At the prognosed core depth, the Walakpa sand is expected to be within the gas hydrate stability zone. The reservoir properties and lateral continuity of the Walakpa sandstone is less understood than the Barrow sandstone at East Barrow, due to less well control, but generally the sands thin to the north.

The core point for this well will be picked by the wellsite geologists based on correlations from adjacent Walakpa wells. Near-bit gamma-ray (GR) and resistivity measurements again will be used to minimize depth point prediction uncertainty. The projected formation top is 1950 ft tvdss, but is subject to change following correlations from real-time LWD logs. The core point will be 10 feet above the sand top to ensure both the interval top and gas hydrate are captured. Once the Walakpa sandstone is cored, approximately 150 ft. of rathole will be drilled to prepare the well for wireline logging and eventual completion as an observation well, if hydrates are confirmed.

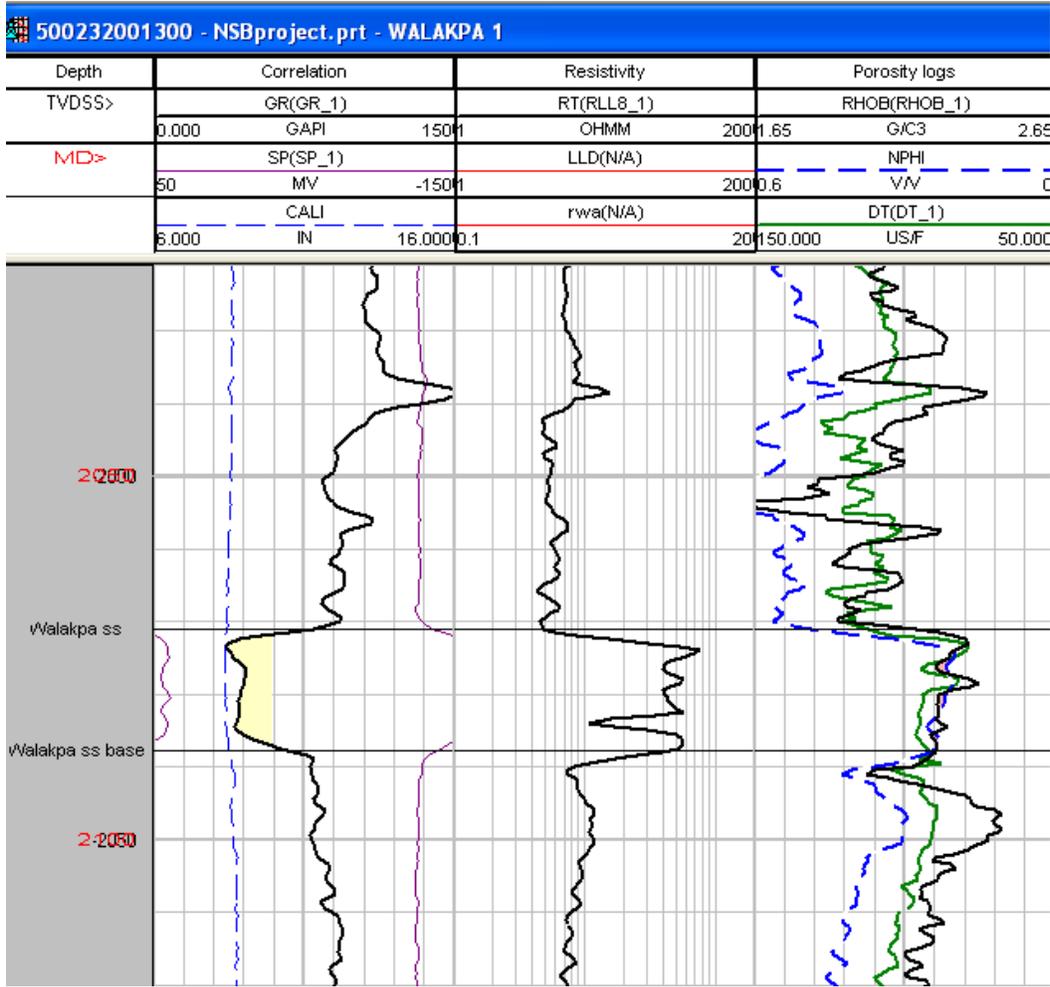
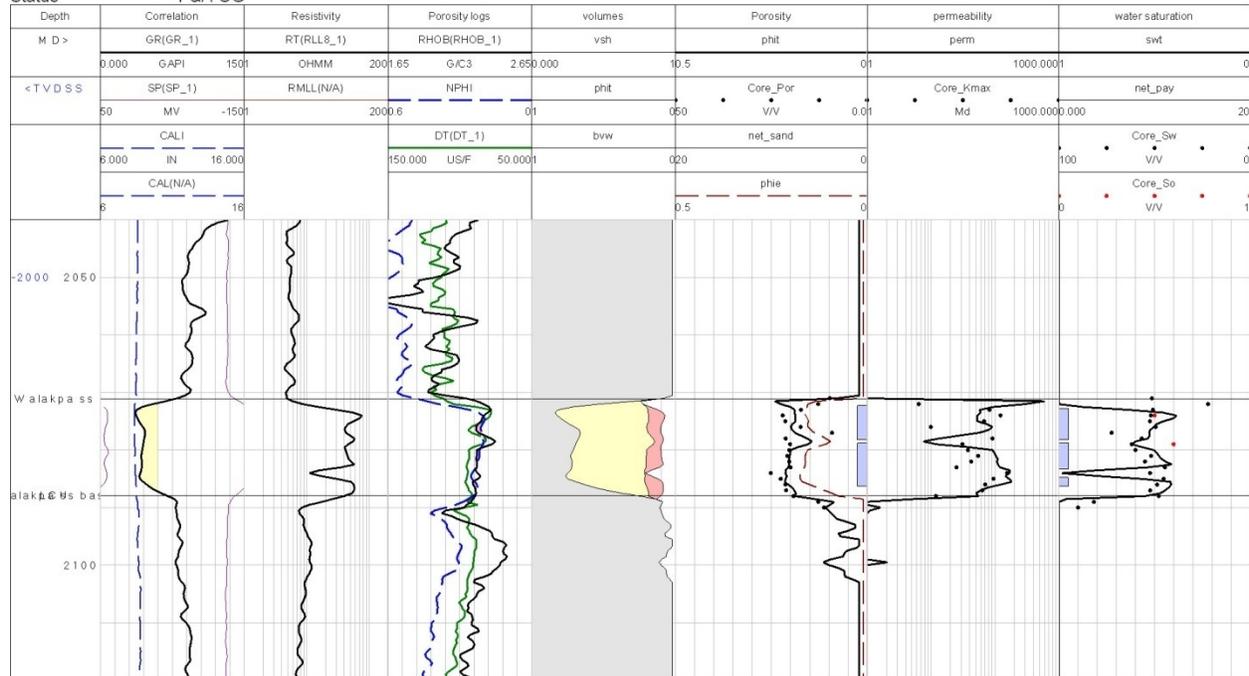


Figure 4: Walakpa Type Well (Walakpa No. 1), Showing Anticipated Core Interval

Well  
Well ID  
Field  
County  
State/Prov  
Country  
Location  
Status

WALAKPA 1  
500232001300  
WALAKPA

TWP: - Range: - Sec.  
P&A-OG



The Walakpa sandstones are interpreted to be near-shore marine sands with better porosity and permeability compared to the Barrow sandstones. Walakpa sand porosities range from 19.5% to 22% and permeabilities range from 95-362 md. The Walakpa sandstone, Cretaceous in age, is also believed to have been buried to a depth of around 10,000 feet subsea. In Tertiary time, these sands were uplifted to their current depth near 2,000 feet subsea.

For both wells, the wellbore orientation is planned to be vertical through the core interval at a maximum inclination not to exceed 10 degrees. Inclination will be measured real-time with mud-pulsed MWD tools. The core point will be picked by the wellsite coring leads from USGS (and DOE), and communicated with the lead operations geologist and Drilling Supervisor. Once core point is reached, the drilling assembly will be tripped out of the hole and replaced with the coring assembly. The coring operation will then commence.

The criteria for ending the planned core program are as follows:

- The full 48 feet (2 coring runs with 24 ft. barrels) of the Walakpa Sandstone in the Savik No. 1 well is gathered,
- The full 120 ft. (5 coring runs with 24 ft barrels) of Barrow Sandstone in the Savik No. 1 well is gathered,
- The full 48 feet (2 coring runs with 24 ft. barrels) of Walakpa Sandstone in the Walakpa No. 15 well is gathered, or

- If coring operations are unsuccessful due to core jamming and/or tool failure, the coring operation will cease once at least four or more attempts have been made and the on-site core supervisor team, lead by USGS and DOE supervisors, has determined that the continuation of coring at that specific interval is futile. These guidelines have been stated to remind the team that there are limits to how long a coring operation can continue when no progress is being made.

### **1.3 Purpose and Objectives**

The purpose of obtaining the core is, in combination with downhole log data, to confirm the existence of gas hydrates, measure the properties of the hydrate bearing reservoirs, and characterize reservoir and fluid properties to help reduce subsurface uncertainties.

The Barrow gas field onsite sub-sampling analysis objectives are summarized below:

- Confirm gas hydrate occurrence and characterize reservoir properties.
- Sample and determine mineralogy and lithology for log model calibration, and understanding formation physical and mechanical properties.
- Sample and measure gas hydrate and pore water geochemical and microbiological properties to understand the origin of gas hydrate.
- Obtain whole-round cores for later porosity, permeability, and fluid saturations for log model calibration, and resource assessments.

In terms of predicting future deliverability of the resource, core will also provide critical information on reservoir quality, interpreted reservoir lateral continuity, hydrocarbon in-place, resources, potential deliverability, and future well placement. Specific post-well core studies may include the following:

- Core-derived  $R_w/S_w$  (gas-hydrate-in-place)
- Sedimentology (for future well placement, reserves)
- Poro-perm (reserves, well productivity)
- Reservoir quality (well deliverability)
- Vertical and horizontal heterogeneity description (compartments, depletion plan)
- Coreflood tests (relative permeability)
- Petrophysical tests
- Geo-mechanical properties

### **1.4 Risk Identification, Risk Mitigation**

Given the critical nature of core acquisition to the overall objectives of this program, considerable effort has been and will continue to be expended to prevent any adverse impacts on the project schedule and objectives. To that end, a list of potential risks has been compiled to identify and address the risks of coring failure. Shown below is a summary of the main risks, with potential impacts of failure and mitigation plans to prevent failure. If additional risks are

identified during the planning process, they will be listed here with appropriate mitigation measures.

**Stage:** AFE Planning

**Risk:** Coring and/or core handling procedures are incomplete or lack clarity, resulting in inadequate funding of program.

**Impact:** Budget is exceeded or well-site program is de-scoped, resulting in a program that does not meet performance targets.

**Mitigation:** Proper front-end planning and documentation, prior to final AFE submittal.

**Stage:** Well Planning and Preparation

**Risk:** Coring equipment and/or personnel are not available or fully functional when needed. For example, Corion's wireline system, Weatherford/OMNI equipment, USGS/DOE supplies, or critical team members are unavailable or not fully functional at a critical point in the operation.

**Impact:** Unable to core well, possible rig standby waiting on equipment

**Mitigation Actions:** Prepare detailed coring plans and procedures. Work with vendors to confirm equipment/personnel are available and fully functional. Prepare checklists and distribute to all parties involved in the coring operation. Maintain a punch list of outstanding issues and resolve all issues prior to spud. Prepare checklist for training and North Slope clearance.

**Stage:** Operations

**Risk:** Mud chiller fails

**Impact:** Cannot proceed with drilling/coring well, poor data acquisition, poor borehole conditions, loss of borehole, potential well control issue

**Mitigation:** DrillCool equipment must be checked out and fully functional, with complete integration with rig's mud system, prior to spud.

**Stage:** Operations

**Risk:** Mud properties fail to meet specifications

**Impact:** Cannot proceed with drilling/coring well, poor data acquisition, poor borehole conditions, loss of borehole, potential well control issue

**Mitigation:** Mud delivery and mud mixing must meet target specifications, prior to critical time of coring.

**Stage:** Operations

**Risk:** Core point picked too shallow or too deep

**Impact:** Core the wrong interval. Pick too shallow and additional core is cut at added time and expense. Pick too deep and critical zone is drilled, not cored, resulting in loss of critical data.

**Mitigation:** Have rig-site geologists and USGS/DOE personnel in agreement for core point.

**Stage:** Operations

**Risk:** Swabbing while POOH with wireline retrievable core

**Impact:** Well control incident

**Mitigation:** Prepare tripping guidelines to include maximum speed per wireline run, which may include pumping down annulus while POOH. Model the swab and underbalance pressure prior

to coring and develop tripping schedule. Include all team members, when developing well control plan, as this is different than routine drilling operations.

**Stage:** Operations

**Risk:** Severe gas liberation at rig floor

**Impact:** HSE incident due to risk of explosion. May result in poor core quality

**Mitigation:** Prepare tripping guidelines to include maximum pulling speed. Minimize gas liberation by using chilled oil-based coring fluid.

**Stage:** Operations

**Risk:** Inadequate analysis resources or procedures are available for analyzing the core

**Impact:** Critical decisions regarding completion or abandonment of well cannot be made

**Mitigation:** Understand exactly what analysis needs to be completed at the wellsite so that real-time completion decisions about the well can be made in a timely manner

Additional concerns include, but are not limited to:

- Site access risk – Extended inclement weather, or failure of ice road contractor to perform, prevents the construction of ice roads and pads in time to access the surface location and drill the well
- HSE event(s) slows or halts the operation – a major spill or major injury prevents the plan from being executed
- Permits – failure to obtain all permits halts the operation
- Waste and cuttings disposal – failure to adequately handle mud cuttings, or other waste stream, prevents the plan from being executed
- Jamming of core, resulting in drilled-up section, and/or additional trips to pull core and replace barrel
- Poor recovery of the gas hydrate-bearing reservoir intervals
- Poor displacement of water-based drilling mud with oil-based coring fluid or excess water in oil-based coring fluid system,
- Borehole problems due to inadequate mud chilling or gas dissolution from gas hydrate or associated free gas-bearing formations,
- Core is obscured by mud additives, such as black gilsenite additive causing difficulty in sub-sampling.

Note: The risks that have been identified to date are to be addressed in the following sections of this document. This document is intended to be an evergreen document, with continuous improvements and additions to be incorporated as planning progresses up to the spud date of the well.

## **2 PRE-CORE PLANNING AND WELL SITE PREPARATION**

The Barrow Gas Hydrate project will obtain up to 200 feet of core, if both East Barrow and Walakpa field wells are cored. Due to the complex and sensitive nature of the core and core analysis, the operation must be thoroughly planned out prior to initiation of operations. Described in this section are the planning and well site preparation steps necessary to manage the project, personnel, and timelines which will be critical to success.

### **2.1 Objectives**

- Ensure that all core team members understand and agree with the detailed objectives of the core operation so a safe and effective operation can be successfully accomplished.
- Ensure that the core operations can be organized in concert with other essential rig operations in compliance with established HSE requirements.
- Ensure that all core processing and analysis equipment items are fully functional and tested prior to coring operations.

### **2.2 Safety Considerations**

- Coring and core handling is a non-standard drilling operation and is likely to be new to some members of the rig crew.
- All core operations will be discussed with the rig team to insure that all coring tasks are integrated with the traditional drilling tasks in a seamless manner.
- The number of people involved in handling the core will be kept to a minimum.
- NOV Corion is responsible for the core from acquisition through initial laydown in the pipeshed. Note: The primary focus of this document (Volume 1) is to describe these procedures.
- Weatherford/Omni is responsible for the core from the pipeshed to the core trailers and packaging. Note: These tasks are the primary focus of Volume 2 of this manual.

### **2.3 Operational Considerations**

This section discusses several facets of the operation including communication guidelines, equipment setup guidelines, roles, and responsibilities of the various team members.

#### **2.3.1 Communication Guidelines**

- The core team will meet with the on-site Drilling Supervisor, HSE Supervisor, and Toolpusher to discuss the proposed coring and core handling process and walk through a simulated operation prior to initial core acquisition.
- Roles and responsibilities should be reviewed and agreed upon. All hazards must be identified and discussed.
- Appropriate working-level detailed procedures should be written and reviewed prior to initiation of coring operations.

- Additional pre-coring meetings will be arranged at the beginning of each shift, and as required around drilling operations and rig schedules. Meetings will include presentation of coring objectives, discussion of best practices, post well evaluation, and means of mitigating operational and safety risks.

### 2.3.2 Equipment Setup and Testing

- The core team will setup, test, and evaluate all coring, core handling, core processing, core sub-sampling, and core preservation equipment as soon as possible after arrival at the wellsite.
- The core team and rig representatives, under supervision by the HSE Supervisor, will evaluate core handling equipment and procedures for compatibility with all rig systems as soon as possible after arrival at the wellsite.
- Laboratory equipment and its function are the responsibility of the core handling contractor, or responsible member of the Science Party, according to ownership of the equipment. The rig electrician can be used to help install and integrate the equipment, but is not responsible for insuring proper function or calibration of said equipment.

## 2.4 Core Team Members, Roles, and Responsibilities

This section is included in all three volumes to ensure all parties understand their roles and responsibilities.

The “core supervisors” listed below will be responsible for core lengths, core tops, meeting coring objectives, etc. and will provide 24 hour coverage with at least one person available at all times.

- 1 PRA/??? Operations Geologist, oversight
- 1 DOE Geologist, oversight
- 1 USGS Geologist, oversight

The contractor-based team members listed below will work 12 hour shifts with full-time coverage of the operation.

- 4 total, 2 per shift, NOV Corion lead core engineer and wireline engineer
- 4 total, 2/shift Weatherford/Omni core marking, breaking, and preservation staff

The on-site Science Party will consist of the following: (Note: the table below summarizes these responsibilities). *Note: The tentative roster of on-site core supervisory and Science Party team listed below will be revised and updated as coring operations near and individual’s schedules are determined.*

- 8 total, 4/shift USGS, USDOE, and Oregon State University core sub-sampling staff (pore waters, geochemistry, microbiology, gas hydrate-specific core properties analyses.)
- 2 total, 1/shift Organic Geochemistry and Microbiology sub-sampling staff

- 2 total, 1/shift Physical Properties sub-sampling staff
- 4 total, 2/shift Inorganic Geochemistry (water sampling)

<u>NAME</u>	<u>AFFILIATION</u>	<u>RESPONSIBILITIES</u>
TBD Ops geologist	PRA/???	Core point, TD, wellsite core shift supervisor
Tim Collett	USGS	Core point, TD, wellsite core shift supervisor
Ray Boswell	US DOE	Core Handling and swing shift supervisor
Kelly Rose	US DOE	Core Handling, core water chemistry
TBD	US DOE/TBD	Core Handling, water chemistry, thermal conductivity
Rick Colwell	Oregon State Univ	Core Sampling, geochemistry, microbiology
Marta Torres	Oregon State Univ	Core Sampling, water analyses lead
TBD	USGS	Core Sampling, physical properties
TBD	USGS	Core Sampling, geochemistry, microbiology
TBD	USGS	Core Sampling, physical properties
TBD	TBD	Core Sampling, water chemistry
TBD	TBD	Core Sampling, inorganic chemistry

- The Drilling Supervisor (“Company Man”) has ultimate responsibility and final say over all coring and drilling operations at the wellsite. Described below are the principal roles and responsibilities of the “core team” members, all of whom advise and report to the Drilling Supervisor while at the wellsite.
- The PRA Operations Geologist is responsible for facilitating and coordinating all communication between the scientific staff (“Science Party”) and the Drilling Supervisor.
- The HSE Supervisor will be included and consulted on all matters of an operational nature that have safety or spill risk elements.
- The Core shift supervisors/leads serve in an oversight role and are responsible for coordination of core data, technical guidance, team coordination, and assurance of coring objectives.
- NOV Corion technicians are responsible for supplying all equipment, manpower, and expertise for core acquisition through core lay-down in the pipe-shed.
- Weatherford/Omni staff will be responsible for inner core barrel marking, whole core measurement, marking, preservation, wellsite processing, assistance with USGS onsite sub-sampling, bulk core stabilization by freezing, onsite core storage, core transportation to Anchorage, and post-program routine and specialized core analyses if required.
- The Science Party, with support from Weatherford/Omni staff, will be responsible for on-site sub-sampling, on-site analysis including logging, lithostratigraphic descriptions, organic and inorganic chemistry, and physical property measurements.
- Weatherford/Omni staff will be responsible for shipping other frozen samples, mud samples, State of Alaska chip samples, and liquid nitrogen stored samples as required.
- The Science Party will be responsible for return shipment of laboratory equipment and certain specialized core subsamples.
- Canned cuttings and samples will be shipped by USGS to USGS at Menlo Park.

## 2.5 Selection and Set-up of Core Processing Area

- Two core processing trailers will be required including the cold Corion trailer and the warm geo-trailer needed for the onsite core sub-sampling, processing, and data

acquisition operations. One office trailer with lighting and power supply will also be needed.

- A refrigerated shipping container, provided by AES or COREHANCON, will be onsite for core storage and transportation.
- A core processing area will be selected which will not interfere with other rig activities, meet all HSE requirements, and allow for optimal onsite processing and sampling of gas hydrate-bearing core properties. As part of the commissioning procedure, the core supervisory team, Drilling Supervisor, and HSE Lead will conduct a safety assessment of the area, and insure that obstructions that might impair lay-down or handling of core be mitigated.
- Equipment in the core processing area should be arranged so that all plumbing, compressed air, and electrical piping and lines are routed in a safe and efficient manner.
- Gas sniffers (Hydrogen Sulfide (H<sub>2</sub>S) and Methane, (CH<sub>4</sub>)) and Oxygen (O<sub>2</sub>) sensors will be used in the core processing trailers to allow for use of non-intrinsically safe equipment operations during hot-work permitted operations; detailed protocols will be developed onsite during Task Risk Assessments (TRAs).
- The installation of all equipment is to be approved by the Drilling Supervisor, Toolpusher, and HSE Lead. The rig electrician is responsible for all electrical connections.
- The final layout of the core processing area will be reviewed and agreed upon by the lead coring supervisory team and Drilling Supervisor. The diagram below (Figure 5) shows the recommended layout to indicate the scale of equipment and trailers, and is included for general guidance purposes.

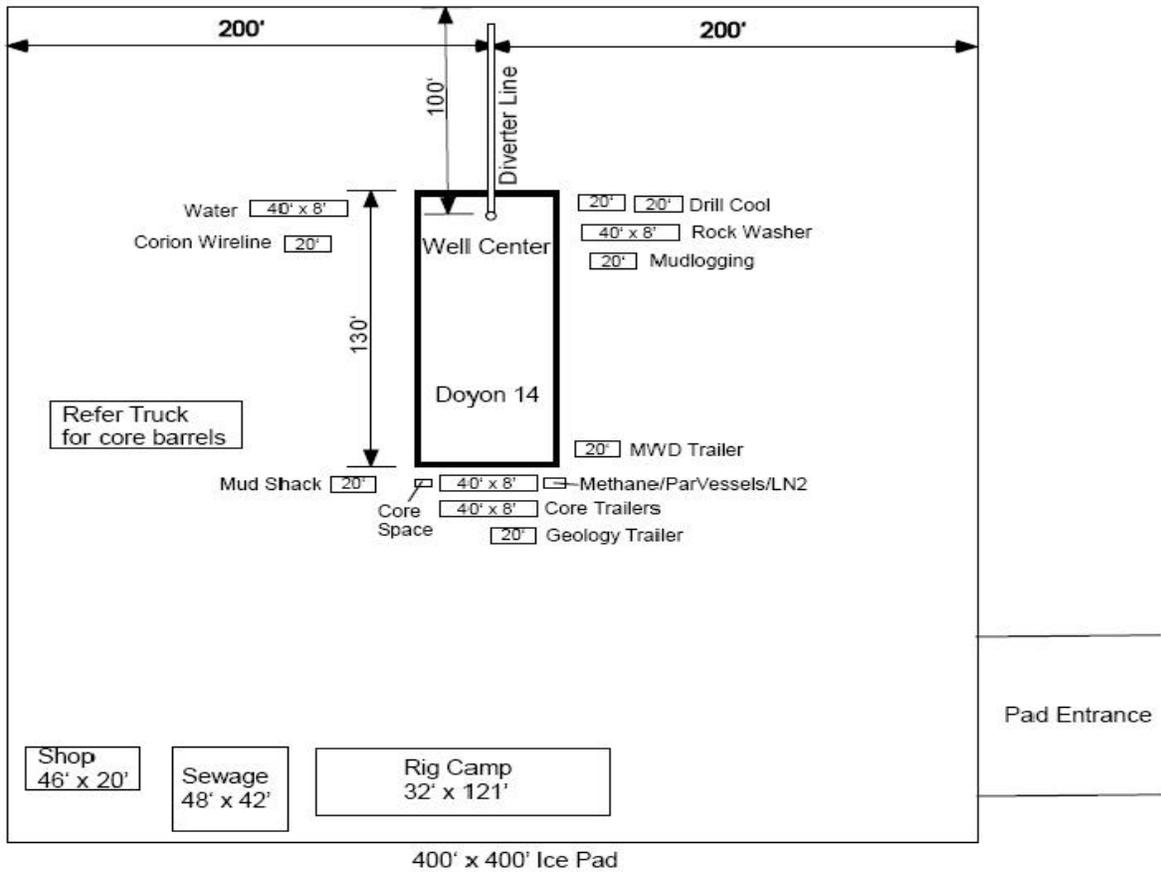


Figure 5: Ice Pad and Rigsite layout diagram. Note that Rig Camp and facilities will likely be staged on MPB-pad, approximately 1 mile to the north.

**Figure 5: Drillsite Pad and Rigsite Layout. Note the location of the Cold geo trailer(s) and the warm laboratory trailer.**

Note: This figure needs updating for the actual rig selected in late 2009. The figure will show how the core is moved from pipeshed to cold trailer.

### 3 MUD CHEMISTRY AND MUD-CHILLING SPECIFICATIONS

Proper mud chemistry and adequate mud-chilling are critical to maintain a gauge borehole, maximize core recovery, and minimize hydrate dissociation during core acquisition and retrieval. This chapter discusses the specifications and details surrounding the drilling fluid and cooling specifications.

#### 3.1 Objectives

The objectives of an optimal mud chemistry program include the following:

- Ensure that mud chemistry is maintained to maximize coring performance and minimize filtrate invasion and contamination of native fluids.
- Ensure that proper mud temperature is maintained to minimize gas dissociation. A mud temperature within 0 to 4 degrees Celsius is critical to maintain gas hydrate stability during coring and subsequent logging operations.
- Ensure that mud filtrate is non-damaging to the core and does not irreversibly alter core or core fluid properties.
- Ensure that major mud changes and/or treatments are not required during coring operations and that mud properties remain relatively constant throughout the operation.

#### 3.2 Safety Considerations

- MSDS sheets for mud components, contingency chemicals, and core sub-sampling chemicals must be available on-site for public review.
- Appropriate personal protective equipment (PPE) must be used by all personnel in proximity to mud system as oil-based mud is potentially harmful.
- For activities where significant mud contact can occur (e.g. breaking down core barrel on rig floor), mud proof slicker suits are to be worn.
- If any significant mud contact with skin occurs, the affected area should be washed immediately to prevent absorption into the skin.

#### 3.3 Background – Surface Hole

The 12-1/4” surface hole will be drilled with a conventional, water-based, surface hole drilling fluid system, similar to systems used near Prudhoe/Kuparuk. A table of mud properties for this hole section is given below:

Density (ppg)	PV	YP	pH	API Filtrate	Chlorides
<9.4	10 - 30	25 - 50	9.0 – 9.5	NC - 10	NC

A more detailed description of the mud system, and drilling precautions is provided in the well plan, under separate cover. Below are a few issues that warrant noting in this document:

- Maintaining mud temperatures at 60°F or cooler is always beneficial for wellbore stability in the permafrost section to minimize thawing and washout of the hole. Keep the mud as cool as practical with cold water make-up and dilution.
- Hydrates are not expected in this interval, however normal drilling precautions should be taken to monitor the flowline and pits for signs of gas influx.
- Some gravel is expected in the surface hole so cuttings carrying capacity of the mud is important. It is expected that funnel viscosities of 100 sec/qt will be adequate to lift the gravel to surface, but funnel viscosities as high as 150 – 200 sec/qt may be necessary.
- Bit balling with clay may cause swabbing and tight hole on short trips.
- This is a vertical hole, so lowside hole cleaning is not an issue, but viscous sweeps are beneficial. High flow rates and high rotary speeds are not necessary and not recommended. Circulation rates should be maintained between 400 and 500 gpm.

Once the surface hole is drilled, 9-5/8” surface casing will be run and cemented to surface. When it is time to displace cement, the chase fluid will be the mineral oil-based mud (MOBM) coring fluid used to drill the production hole section (as described below). Once the “plug is bumped” with MOBM, and the wellhead and BOPE equipment has been installed, an 8-1/2” mill-tooth rockbit and LWD tools will be run and used to drill float equipment and 20’ of new hole for a Formation Integrity Test (FIT). Upon a successful FIT, drilling will continue to the coring point.

### **3.4 Operational Details, Cored Section**

A primary objective of the Barrow gas hydrate coring program is to determine in-situ gas hydrate saturations and pore water chemistry. An oil-based mud system with zero water content is proposed for this core program to minimize mud filtrate invasion, and thus maximize the volume of virgin un-invaded inner section of the core. The oil-based mud system will provide a much lower spurt loss and lower permeability filter cake than a water-based mud, which should help ensure that fluid invasion is minimized and borehole and core stability is maintained. In addition to the low invasion benefits of oil-based mud, the use of an oil-based mud system should reduce the risk of core barrel jamming by reducing friction between the core and core barrel.

#### **3.4.1 Mud Requirements**

Successful low invasion coring is accomplished with the combination of proper bit selection as well as proper mud chemistry. The correct bit design helps minimize core invasion by:

1. Delivering high ROP
2. Not having throat discharge ports which force fluid into the core, and
3. Not having extra gauge cutters in the throat, which scrapes off filter cake and allows more invasion.

The correct mud system is designed to:

1. Build a tight (low permeability) filter cake very quickly, and
2. Limit filtrate invasion.

More than half of the filtrate that penetrates a core is generated at the core bit during the shaping of the core. Each time a PDC cutter exposes new rock surface, spurt-loss occurs on that surface. This spurt-loss is due to very rapid absorption of filtrate as the mud cake is forming. Minimizing the area on the core where cutting occurs is an important consideration to bit design and selection.

After an intact mud cake has formed the static-fluid loss properties of the coring fluid serve as the main deterrent to fluid loss. During this stage, fluid loss is much lower, and decreases with the square root of time as the filter cake thickness grows. Gradually, the pressure differential required to force filtrate into the core increases with core length and at several feet above the core bit decreases to near zero.

### **3.4.2 Coring Fluid Design Criteria**

The low invasion coring fluid criteria established at the DOE funded, BP operated Mt. Elbert well serves as the template for the design of oil-based coring fluid of the Barrow Gas-Hydrate test well. The Anadarko operated Hot Ice program used a water-based KCl mud system, but this system was found to be deficient in that it contributed to water contamination to the core and made drill cuttings handling difficult.

There are two opportunities to measure accurate water saturations and chemistry in the core when using an oil-based coring fluid. Preserving virgin or un-invaded rock is much preferred, but if invasion does occur, filtrate with a high interfacial tension relative to the native brine can often leave the native brine undisturbed. The coring fluid for the Barrow Gas Hydrate program is designed both for minimal invasion and to preserve the second option through careful selection of surfactants.

The coring fluid that has been chosen for this program is a mineral oil-based mud (MOBM) system, similar to other oil-based systems used in fields around Prudhoe Bay. The main design criteria for the MOBM fluid are as follows:

- The fluid must contain emulsifiers in adequate amounts to disperse any contaminating water. Only carboxylic acid emulsifiers should be used since these have been shown to have minimal effects on rock wettability, while effectively emulsifying water while maintaining relatively high interfacial tension. The emulsion stability (ES), as measured by a Fann 23D-type instrument, should be greater than 2,000 volts.
- The oil/brine interfacial tension (IFT) of the fluid must be 7-14 dynes/cm, or higher. Poly-amide emulsifiers and sulfonated surfactants should be excluded from the system, since they lower IFT's and can alter rock wettability. An IFT measurement will be made with filtrate from the final formulation.
- The fluid must have rheology that is acceptable for an effective drilling and coring fluid in a vertical hole. A yield point near 15-25 range is preferred, with 6 rpm and 3 rpm readings in the 7 to 12 range. The rheology must remain stable in the presence of small amounts of contaminating water. The fluid must develop sufficient initial viscosity at the mixing plant to suspend weighting material.

- The high temperature/high pressure (HTHP) static fluid loss (at 100°F) of the fluid must be in the range of 1 - 4 cc, and preferably 2 cc or less. When all of the components of a low invasion fluid are present, the spurt loss of the fluid tracks closely with the HTHP fluid loss. There must be no free water in the HTHP filtrate. This is more important than having an ES of 2000 volts, which is an indirect estimate of emulsion stability. Any invading water will complicate the measurement of in-situ native water saturation.
- The coring fluid should contain an adequate concentration of sized solids to lower the spurt loss of the mud and minimize mud filtrate invasion. Past experience has shown that 100 pounds per barrel (ppb) of calcium carbonate (CaCO<sub>3</sub>) with a median size of 5-10 microns works well. Calcium Carbonate size 10 has a median size in this range.
- The water phase of the mud should have minimal concentration of salt. This is to minimize gas hydrate dissociation, and to not interfere with the measurement of in-situ virgin chloride concentration in the connate water, to allow for accurate resistivity log interpretation. If the coring fluid does pick-up excess water, no (calcium, sodium, or potassium) chloride should be added.
- The mud must contain excess lime to ensure that the surfactants are calcium salt based. Absolutely no sodium-based salts (NaCl) should be used as part of the mud formulation. Sodium salts of carboxylic acid emulsifiers tend to lower interfacial tensions, and calcium salts are more effective in forming water-in-oil emulsions. 1 ppb excess lime is adequate.
- The coring fluid must have a water content of <1% by volume, preferably zero.
- The drilling mud will be chilled to a range of 32° to 38° F (0° to 3.3° degrees Celsius) to maintain borehole and gas hydrate stability. The mud properties at the actual circulating temperature (input at the suction tank and output at the possum belly), in addition to the standard tests at 70°F and 100°F, should be measured and monitored at all times.
- The mud may contain tracers. The tracer is 1- bromonaphthalene, and the recommended concentration is 100 parts per million (ppm).
- During coring, a microsphere tracer will be added to the mud system as detailed in Section 3.4 of this document.

### 3.4.3 Coring Fluid Formulation

The final mud weight of the coring fluid will be determined shortly before spud of the well. The mud weight that is chosen will be based on the estimated pore pressure of the coring interval, in addition to the pros and cons of a mud weight that might be considered too light or too heavy. The obvious disadvantage of a mud weight that is too low is the increased risk of a well control incident. The disadvantage of a mud weight that is too high is risk of higher filtrate loss and risk of contaminating the core.

At this time, a tentative mud weight of 9.5 ppg is chosen to serve as the basis of a recipe. A formulation of a fluid with 9.5 ppg weight and cold temperature viscosity/properties is given below: **(to be confirmed by mud company)**

LVT-200	0.787 bbl
Claytone EM	7.0 ppb
Lime	3.0 ppb

Versa-mod Emulsifier	3.0 ppb
Versa-Trol	25 ppb
Safe-Carb 10 (10.5 micron CaCO <sub>3</sub> )	90 ppb
Barite	75 ppb
1-bromonaphthalene	+/- 100 ppm (may be present if mud is reconditioned)

Note: A complete list of mud chemicals, with MSDS sheets, should be sent to Marta Torres at Oregon State University to confirm that no NaCl salts are in the proposed chemical set.

With <2% water, the formulation should give the following properties after being sheared on a Silverson mixer and hot-rolled for 12 hours at 150 degrees F.

	<u>70 °F</u>	<u>100°F</u>
600 rpm	145	97
300 rpm	84	57
200 rpm	60	42
100 rpm	36	27
6 rpm	8	7
3 rpm	7	6
PV, cp	61	40
YP, lb/100ft <sup>2</sup>	23	17
Gels (10sec/10min)	8/30	11/39
ES, volts	2000+	
HTHP (35μ), cc	1.2	2.4

#### 3.4.4 Discussion of Chemical Additives

- LVT-200 is the base mineral oil
- Claytone EM is a bentonitic-based? clay designed to yield in oil-based mud. It is specially modified so that it will yield prior to mud mixing and allow the mud to suspend barite. Most raw clays do not yield well in mineral oils, especially before they have been sheared through the bit. Also, most clays give huge viscosity increases when they come in contact with small amounts of water; Claytone EM is relatively stable in the presence of water.
- Lime is used to maximize the effects of calcium based surfactants.
- Versamod is a fatty acid that gives superior low shear rate rheology, high ES, and very high IFT.
- Vers-Trol is gilsonite (a natural asphalt), that controls spurt-loss and static fluid loss similar to blown asphalt used on prior North Slope wells. Blown asphalt is no longer carried by MI.
- Safe-Carb 10 (Calcium carbonate) is a finely ground marble with a median particle size of 10 microns. It is used to build filter cake and lower fluid loss.
- Barite is a weighting agent to raise mud weight to desired weight, primarily for hydrostatic well control.

Note: Mud properties must be measured and monitored on a minimum of twice daily basis by the on-site mud engineer, and reports should be made available to the Drilling Supervisor and Coring advisor.

### **3.4.5 Mud Mixing Suggestions**

The most critical step in mixing the mud at the mud plant is yielding the clay in a controlled environment. Time, heat, and shear are the most important factors in getting the clay to yield. The steps that are followed to mix the mud are given below:

1. Heat a 200 bbl mixing tank of LVT-200 and required concentration of Claytone EM to 130-140°F and supply extra shearing energy to the system via a SECO (Echols) Homogenizer pump. This should produce a fluid with consistent properties, allowing it to support the weighting material and solids. The clay and base oil will go through a visible “water-to-cream” transition as it yields.
2. The other products should not be mixed through the same shear device as it will reduce the average grind size of the calcium carbonate. The order of mixing is the same as the order the products are listed in the recommended formulation.
3. After mixing with strong paddle shearing, the fully mixed sample should have a (70°F) YP of 23 with gels of 8/30, and a 3 rpm reading of 7.

Since keeping water to a minimum is a critical objective, the mixing pits and lines should be cleaned and completely dried before mixing begins. It is recommended to send a slug of pure base oil through all pumps and lines to clear the system of possible contaminating fluids. The same care should be taken with the trucks that carry the mud to the rig. The tanks should be clean and dry.

### **3.4.6 Oil-based Mud Maintenance**

During coring it is preferred not to make additional make-up, dilutions, or treatments to the oil-based coring fluid, if possible. Since this project involves coring two relatively short intervals, the mud should not need mid-course treatments. However, if the viscosity deviates or fluid loss increases too high, or some other coring specification is breached, appropriate treatments should be made. If a treatment is required, the following guidelines should be followed:

- The system should be circulated until all chemicals are completely mixed and evenly distributed, and core fluid specifications are again met.
- A small amount of centrifuging may be used to control mud weight and rheology. It should be noted that calcium carbonate (used to control fluid loss) has nearly the same density as drilled solids and may be unavoidably reduced with centrifuging.
- When slugging the pipe to come out of the hole, small amount of calcium carbonate can be used in addition to barite. This allows fresh bridging material to be introduced.
- When drilling to core point and drilling the rat hole after coring, the simplest and most effective way to reduce viscosity will be to dilute with un-weighted mud. This pre-mix should have all products present in the proper amounts (emulsifier, fluid loss material,

etc), so the properties of the system do not fluctuate. The mix should be bled into the system smoothly over at least one full circulation.

- The amount of calcium carbonate in the system should be kept track of, by mass balance. If dilution lowers the concentration below 90 ppb, then more should be added before coring commences.

### **3.4.7 Mud Sampling**

Mud samples should be obtained during drilling and coring to ensure quality control and understanding of potential impacts on geochemistry and water chemistry. Samples will be acquired at the midway point (12 feet into) of each 24 foot core run while maintaining normal fluid circulation. Two samples should be acquired, one at suction tank and a second at the possum belly. This sampling frequency, technique, and interval should be coordinated with the mud engineer and mud-loggers. Samples will be obtained in 1-liter Nalgene bottles supplied by COREHANCON Lab and shipped to the following address for chemistry and microbiology analysis:

Marta Torres  
104 COAS Admin Building  
Oregon State University  
Corvallis, OR 97331-5503

## **3.5 Micro-spherical Tracer**

### **3.5.1 Introduction**

When collecting subsurface cores for microbial characterization it is essential that the microbiologists ascertain that the microbes that they detect in the samples from the interior of the recovered cores are native and authentic to the subsurface and not introduced as a part of the drilling process. Accordingly, scientific drilling teams in terrestrial and marine settings have devised various quality assurance/quality control tracers that can be deployed during the coring acquisition process to assess sample quality. For a number of years, carboxylated latex microsphere tracers have been used to assure the quality of subsurface cores for microbiological analyses in deep drilling efforts conducted in other locations. These microspheres are non-toxic, inert, and considered safe for environmental use. The microspheres are to be supplied by:

Rick Colwell  
104 COAS Admin Building  
Oregon State University  
Corvallis, OR 97331-5503

### **3.5.2 Objectives**

- Use a tracer in the mud system to trace the extent of infiltration of mud into the core.

- Estimate the extent of infiltration (i.e. contamination of microbial-sized particles) to the interior of core sections that will be examined for microbiological properties.

### **3.5.3 Approach**

- Carboxylated, latex microspheres (Polysciences Inc., Warrington, PA) nominally 0.9 micron in diameter, are shipped in a concentrated solution. These microspheres are conjugated with a fluorochrome (i.e., fluorescein) so they can be seen with a microscope capable of epi-fluorescent illumination. In the field prior to coring, the concentrated solution is diluted in sterile water to yield final numbers of approximately  $8.75 \times 10^9$  microspheres per ml in a total of 100 ml.
- When using wire-line coring, the diluted 100-ml microsphere solution is added to a sterile Whirlpak bag which is sealed using wire ties. The microbeads are placed in the Whirlpak bag, sealed with the heat sealer, which leaves the Whirlpak bag free of wire ties. The core engineer will be supplied with a box of prepared microbead bags before coring, which will be enough for all core runs.
- Between runs, the core engineer attaches the Whirlpak bag to the catcher (a 30 second step) as the next barrel is readied to be run in hole. Since this is done between core runs, by the core engineer on the rig floor, there is no interruption to the core process.
- The tracer bag is then attached to the inside of the shoe beneath the core barrel. The specific arrangement of the tracer bag is adapted to the type of shoe or core catcher used in the coring assembly. There may be several ways to attach the tracer bag; however, the method must protect the integrity of the tracer bag that contains the tracers during the trip to the bottom of the hole so that the tracer solution is only released when the core enters the core barrel and breaks the tracer bag.

Note: Since the microsphere tracers are used frequently (i.e., every core run), it is essential to have at least two core catcher/shoe assemblies on-site so that during any given core run in which one of the assemblies is being used, the second assembly can be fitted with the tracer bag to be used in the next core run.

## **4 PRE-CORING BOREHOLE CONDITIONING**

### **4.1 Objectives**

- Prepare the borehole so that hole is in gauge or slightly over gauge, straight, no sharp doglegs, and free of cuttings.
- Ensure that the borehole is prepared in such a way that the first coring run delivers good quality core with maximum core recovery.
- Obtain maximum useable core with minimum rig cost.

### **4.2 Safety Considerations**

- Well control requirements take precedence over any technical requirements.
- Hole geometry and mud weight should be designed to allow the drilling assembly to remain on or near bottom without risk of sticking during wireline retrieval of core barrel. If hole problems or sticking do occur, the drilling assembly may be tripped the 400 - 600 feet into the surface casing following each core run.
- The Corion (NOV) wireline-retrievable core system and Drillcool mud chilling system are relatively new to North Slope operations and care must be taken to ensure that tools and procedures are fully compliant with safe operations.
- The reservoir section should be penetrated with adequate but not excessive overbalance. Good drilling practices should be followed to avoid influx of reservoir fluids or differential sticking of the drillstring.

### **4.3 Operational Details**

The Walakpa and Barrow Sands will be cored using Corion's Wireline Express system with 3-inch by 24-foot core barrel assembly. It is critical that the pre-coring section of the hole be drilled in such a way that maximizes the chance of trouble-free coring and maximum core recovery.

The most effective method of drilling the hole to core point without extended reaming will depend on specific real-time conditions and experience with the area. The team will design the best pre-coring BHA to minimize hole angle (i.e. maintain vertical) and optimize ROP with proper bit mechanics and hole cleaning. The cored section will be purposefully cored with 7 7/8" bit and then reamed out to 8-1/2" prior to logging operations.

If there are indications of junk in the hole after pulling the pre-coring BHA, then the likely type, amount, and location of junk must be established. The impact of this junk on coring operations must be assessed and a clean-up run considered. A junk basket may be run in the BHA used to drill to core point if this is considered to be useful and acceptable.

## **5 CORE POINT AND CORE TERMINATION CRITERIA**

As mentioned in the introduction, The Savik No. 1 well is planning two 24' core barrel in Walakpa Sand, and five 24' core barrel runs in the Barrow Sand. If required, the Walakpa No. 15 well is planning two 24' core barrel runs in the Walakpa sand. This core initiation and termination protocol described in this section covers all three intervals.

### **5.1 Objective**

- Establish the core initiation point and core termination point to minimize the possibility of miscommunication.
- Allow all parties to agree on the criteria.

### **5.2 Safety Considerations**

- As core point approaches, the geologist, mud logger, and driller should work closely to ensure the pore pressure is accurately estimated and mud weight is adjusted accordingly.
- If a well control incident occurs, efforts to regain control of the well will take precedence over geological core acquisition and termination criteria.

### **5.3 Operational Details**

The onsite geologists will pick the core point based on extrapolated offset well log data as described below. The senior project geologists, Tim Collett and DOE designate, in consultation with the PRA/AES Operations Geologist, and the core team, will make all geological core point and coring termination decisions.

The one exception is in the case of budget constraints should the operation exceed the planned 2-3 days. In this case, the termination of coring will be based on financial factors as well as technical factors and involve discussions with NSB and DOE project managers to insure the budget is available to continue the operation, or cease the coring operation.

#### **5.3.1 Start of Coring – Core Point Selection.**

Savik No. 1 – Walakpa Sand

The thickness of the Walakpa sand at the Savik No. 1 location is expected to be approximately 10 feet. Therefore, to minimize the risk of missing the interval, the coring operation is planned to start approximately 15 feet above the prognosed top of sand, and extend approximately 23 feet below the prognosed bottom of sand for a total cored interval of 48 feet, for a total of two core runs at 24 feet per core run.

Core point will be picked 15 ft. above the top of the top of the Walakpa sand and extrapolated from offset well log data. The core point will occur at approximately 1700 TVDss, but may be subject to change if the well plan requires a final modification prior to spud. The Walakpa sand section is expected to consist of inter-bedded sandstone, siltstone, and shale sequences which may contain a variety of pore fluids including gas hydrate, water, and free gas. The gas hydrate-bearing sections will appear “cemented” by the hydrate; any water or free gas-bearing sections will be consolidated.

#### Savik No. 1 - Upper Barrow and Lower Barrow Sands

The Upper and Lower Barrow sands at the Savik No. 1 location are approximately 100 feet thick. To insure that a hydrate bearing core is taken, the coring operation is planned to start approximately 10 feet above the prognosed top of sand, and extend approximately 10 feet below the prognosed bottom of sand for a total cored interval of 120 feet, for a total of five core runs at 24 feet per core run.

Core point will be picked 10 feet above the top of the Upper Barrow sand and extrapolated from offset well log data. The core point will occur at approximately 1925 TVDss, but may be subject to change if the well plan requires a final modification prior to spud. This coring procedure starts at the top of the Upper Barrow sand and continues to the base of the Lower Barrow sand, for a total core length of 120 feet (5 core barrel runs using a 24 ft. core barrel) assuming coring operations commence as planned. The Barrow sand section will likely consist of inter-bedded sandstone, siltstone, and shale sequences which may contain a variety of pore fluids including gas hydrate, water, and free gas. The core is expected to be consolidated and well cemented. The gas hydrate-bearing sections may have visual indications of hydrate, or it may not, given the small pores and consolidated nature of this zone.

#### Walakpa No. 15 – Walakpa Sand

The sand thickness at the Walakpa hydrate test well location (Walakpa No. 15) is expected to be approximately 18 feet thick. Therefore, to insure that a core of the hydrate well is taken, a core is planned to start approximately 15 feet above the prognosed top of sand, and extend approximately 15 feet below the prognosed bottom of sand for a total cored interval of 48 feet, for a total of two core runs at 24 feet per core run.

The core point will be picked 15 ft. above the top of the Walakpa sand and extrapolated from offset well log data. The core point will occur at approximately 1935 TVDss, but may be subject to change if the well plan requires a final modification prior to spud. The Walakpa sand section will likely consist of thinly inter-bedded sandstone, siltstone, and shale sequences which may contain a variety of pore fluids including gas hydrate, water, and free gas. The gas hydrate-bearing sections will appear “cemented” by the hydrate; any water or free gas-bearing sections will be consolidated.

### **5.3.2 Core Point Termination**

The criteria for ending each core program are as follows:

- The full 48 feet of core (10 feet of Walakpa Sand) interval in the Savik No. 1 well core has been recovered (2 core trips using a 24 ft. core barrel length)
- The full 120 feet of Upper and Lower Barrow Sand interval in the Savik No. 1 well core has been recovered (5 core trips using a 24 ft. core barrel length per run)
- The full 48 feet of core (18 feet of Walakpa Sand) interval in the Walakpa No. 15 well core has been recovered (2 core trips using a 24 ft. core barrel length per run)
- If problems are encountered and continue during coring operations, and little progress has been made in 96 hours, at the discretion of the coring supervisors from USGS and DOE, coring operations may be discontinued. This decision will not be made lightly, but may be necessary if it appears that a core cannot be captured.
- If core operations are perceived to damage the borehole such that subsequent critically important planned logging operations cannot be effectively or safely implemented.

## **6 CORE ACQUISITION**

### **6.1 Objectives**

- Cut maximum length (up to 24 feet per run) with maximum recovery of good quality core within the target reservoir section.
- Do not endanger any subsequent objectives of the well (logging, completion) by excessive borehole damage, enlargement, etc.

### **6.2 Safety Considerations**

- If a well control incident occurs, efforts to regain control of the well will take precedence over geological core acquisition and termination criteria.
- Coring is not a routine activity, the PRA/AES Operations Geologist and the Drilling Supervisor will lead the Task Risk Assessments (TRAs).
- The core supervisory team will coordinate with the rig crew to ensure that safe and effective procedures are established and reviewed before core operations commence.
- TRAs will be reviewed with each new crew at shift change.

### **6.3 Operational Details – Coring Assembly**

#### **6.3.1 Core head Types and Selection**

It is intended that each section in the Savik No. 1 well and Walakpa No. 15 well will be cored using Corion's Wire line Express 3-inch by 24 foot core barrel assembly. This system was run in Alaska in the BP operated Mt. Elbert hydrate test well and the Mallik 5L-38 test well. The main difference between this system and conventional coring systems is the BHA is not tripped to surface after core is cut. The BHA is left on bottom, or tripped into the nearest casing string, and then the core is retrieved through the center of the drilling string via wireline.

There are a number of technical requirements for the Barrow Gas Field coring operation to enable accurate reservoir evaluation, as follows:

- The coring assembly should be tripped to bottom with no damage to bit, core barrel, or core assembly.
- The target intervals should be cored over its entire core barrel length of 24 feet per run.
- The time required to retrieve the core to surface should be minimized, without compromising safety due to hole swabbing; this will increase the chances of obtaining a core sample with intact gas hydrates.
- Coring should occur at an acceptable ROP that minimizes rig time while maximizing core recovery.
- Coring should occur with minimum torque and torque variation to avoid any chance of damaging, jamming, and/or losing the core barrel.

- Several different core heads will be available for the Barrow gas hydrate test wells. One possible core head for this application (7-7/8" X 3" core in oil based mud) is the CSS 513 core head, shown in Figure 6. Alternatives core heads, including one with shorter cutter arms, will be available for back-up. Final corehead selection will be made based on prevailing operational conditions by the lead Corion coring engineer, coring supervisory team, and drilling supervisor.



## CSS 513

### APPLICATION

- ◆ Designed for soft to medium formations with low to medium compressive strengths in consolidated and unconsolidated formations.
- ◆ Applications include vertical, directional, horizontal and Corion Express™ coring.

### FEATURES

- ◆ Low invasion bit design
- ◆ Core bit features fluid flow ports that direct the mud away from the core, an internal lip lower shoe to seal off mud flow from the throat of the core bit, and a flush inner diameter to prevent disruption of the core's filter cake
- ◆ Excellent cleaning features for shales and other soft formations
- ◆ Core bit features larger junk slots increasing cleaning ability and removal of foreign material down hole

### BIT DESIGN

Bit Profile: Flat, Rounded  
 Bit Body: Steel  
 Cutter Type: PDC (30 cutters)  
 Cutter Size: 13.0 mm  
 No. of Blades: 5  
 Gauge Protection: TSP  
 TFA: 0.98in<sup>2</sup> (613mm<sup>2</sup>)

### AVAILABLE SIZES

200 mm x 76 mm (Express)  
 200 mm x 89 mm (Express / Conv)  
 200 mm x 102 mm (Conv)

Figure 6: Corion CSS 513 Core Bit

### 6.3.2 Barrel Length and Core Run Plan

- The strength of the formation, and presence of sharp changes in rock mechanical properties usually controls the length of core that can be cut before the core breaks or the core barrel jams.
- For all planned intervals, core barrel length will be set to accommodate the longest core that can be cut with the Corion assembly (24 feet). There are clear advantages to taking this length of core including ease and speed of handling, core processing quality, etc.
- Rig operations must be designed to minimize operational risk. If drilling and coring operations are trouble free with no high erratic torque, stalling of the BHA, tight spots and/or sticking during coring or tripping, the Wireline Express coring system will remain in place to the completion of the well. In the event of operational problems with this wire-line retrievable system, it may be replaced with a conventional system supplied by NOV Corion, if so determined and time permits.

### 6.3.3 Core Size

- In the 7-7/8" hole, the actual core size will be 3 inches.
- The 7-7/8" hole will be reamed out to 8 1/2 inches prior to logging operations.

### 6.3.4 Inner Barrel Type

The inner barrel is composed of stainless steel and is 24 feet long (Figure 7). Placed inside of the steel inner barrel are two aluminum sleeves. Each sleeve is 12 feet long. The liners are slotted in order to distribute formation pressure along the length of the core barrel and to provide quick access to the core sample when it gets to surface. Caution and care must be taken when removing each liner and processing it.



**Figure 7: Corion Inner Core Barrel with Slotted Liner**

### **6.3.5 Core Catchers and Pilot Shoes**

The core catchers that will be utilized for the Hydrate section will utilize a dual catcher system. The core will enter through the pilot shoe and pass through a slip catcher that has a grit abrasive coating. The core will then pass through a finger basket catcher, with stiff fingers, (Figure 8) that has the ability to capture and hold a consolidated core. These two different style catchers work in conjunction with each other to greatly increase overall core recovery. Full core recovery, in excess of 95%, is expected in this application.

[Note: More flexible finger catchers are available to capture cores that are expected to be very friable or unconsolidated. The stiff fingers are recommended in this application.]



**Figure 8: Finger Basket Core Catcher**

The Design of the CSS-513 core head utilizes a back up gauge cutter should the primary cutter become damaged during coring operations.

All catchers & pilot shoes should be inspected by the coring engineer to ensure that they are in good condition before use. The primary plan is to use heavy duty spring catchers.

Extended lower shoe format pilot shoes (and core heads) are preferred. The CSS-513 is a Low Invasion face discharge core head. The lower lip of the shoe is extended into the throat of the bit to minimize the amount of fluid passing by the core while coring. The total flow area (TFA) of jets in the bit is normally set at 1.0 in<sup>2</sup> but can be adjusted in the manufacturing facility prior to shipping to the wellsite.

Pilot shoes must be compatible with the core head selected; i.e. the corehead TFA must not be significantly reduced when the core is pulled and the inner tube stretched. The design of the

CSS-513 makes it impossible to restrict the flow even if a failure occurs and the shoe completely bottoms out in the bit.

## 6.4 Picking Up Corion Express Drill Pipe

- Have a safety meeting with rig crews prior to picking up any tools to point out hazards and discuss safe operating practices.
- The Corion Express drill string is a nominal 5" OD (127mm) drill pipe with 4 ½" IF modified connections with a secondary seal area. Each drill pipe section is approximately 9.45m (~31 ft.) in length with an ID of 108mm (3.75 in.).
- The Corion Express drill pipe must have pin protectors on at all times that the pipe is being raised to the floor and must not be removed until the pipe is hanging vertically in the derrick.
- Pick up nubbins must be installed in the box end of each joint of Corion Express drill pipe to hoist the pipe to the rig floor and ensure that no damage to the seal area occurs.
- When picking up the drill pipe, remove the pin protector from the pipe and drift the pin end of the pipe as it is hanging vertically in the derrick prior to making the connection.



### 6.4.1 Coring BHA

- The number of drill collars in the coring BHA run should ideally be the minimum required to apply necessary WOB, with the neutral point below the top of drill collars.
- The coring BHA should employ stabilizers to minimize wobble and improve coring performance and minimize the chance of differential sticking.
- Consider use of roller reamers in the BHA to minimize torque.
- Although a float sub would be nice to have, to prevent cuttings backflow, there is no place in the string to place a sub that allows a core to be cut and retrieved via wireline.
- Figure 9 on the next page shows the planned core BHA.



HD Corion Express Collars  
OD=6.5"  
ID=4.25"  
Length=31 ft

Stabilizer  
Body OD=6.5"  
ID=4.25"  
Blade OD= 7.8"

Seat Sub  
OD=6.5"

Outer Tube  
Body OD=6.5"  
ID=4.25"  
Length=22ft

Core Bit  
Blade OD=7.8"  
Size= 7.875" x 3"  
CSS 513

Figure 9: Planned Core BHA

## 6.4.2 Assembling Coring Tools

Described in detail in the section below are the steps to be followed to assemble the coring tools. These steps are to be followed as written, unless the Corion coring engineer chooses to substitute with a procedure designed to overcome a specific problem that occurs.

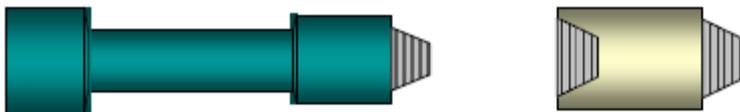
- 1) Pick up the Corion Express core barrel and hang it in the derrick with the elevators or top drive. The outer barrel typically arrives in the field with the seat sub, 1' spacing sub and a pick up sub attached to it.



- 2) Remove the pin protector from the outer barrel and screw the core bit on. Install the bit breaker onto the bit and torque the bit to 26,000 ft-lbs (DDD threads) or to 10,000 ft-lbs (4 1/2" IF threads).



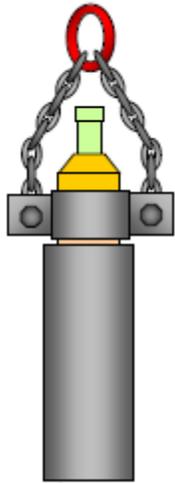
- 3) Lower the outer barrel assembly into the hole and set it in the slips with a collar clamp.
- 4) Remove the 1' spacing sub and the lifting sub from the outer core barrel. The seat sub will still be attached to the top of the outer barrel.



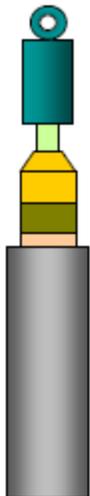
- 5) Pick up the inner barrel assembly from the catwalk.
  - a) Attach the rig's winch line to the pressure head clamp, which is fastened to the top of the inner barrel assembly.
  - b) Ensure that the wheeled cart is attached to the bottom of the inner barrel to protect it while it is being raised up the V-door.



- c) Slowly raise the inner barrel assembly up the V-door and remove the wheeled cart once the assembly is hanging vertical in the derrick.
- d) Slowly lower the inner barrel assembly into the outer core barrel in the table with the winch line. The pressure head clamp will rest on the box end of the seat sub attached to the outer barrel.



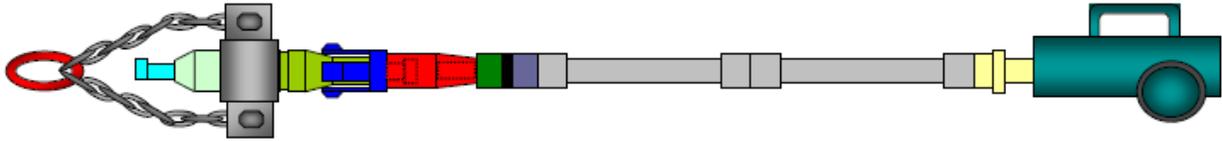
- e) Remove the rig's winch line from the pressure head clamp. Fasten the birdhouse clamp on the top of the rope socket of the inner barrel assembly and attach the rig's winch line.



- f) Slightly raise the inner barrel assembly in order to remove the pressure head clamp from the assembly using a 1 ¼" wrench.
  - g) Once the pressure head clamp is removed, lower the inner barrel assembly into the outer barrel. Once seated, remove the birdhouse clamp.
- 6) After the inner barrel assembly is seated, insert the pick-up sub and 1' spacing sub into the seat sub attached to the outer barrel.
- 7) Latch the elevators to the pick-up sub.
- 8) Check the lead between the inner barrel's bottom shoe and the bit.
- a) Hoist the core barrel assembly.
  - b) Remove the collar clamp from the outer barrel, remove the slips and hoist the outer barrel upwards out of the hole with the bit at chest height. Install the hole cover.
  - c) The core hand will check the lead in order to ensure that the inner barrel assembly is free to move within the outer barrel. A gap of approximately ¼" (6mm) must be between the inner barrel's bottom shoe and the bit.
- 9) Remove the hole cover and lower the outer barrel back into the hole. Set the slips and install the collar clamp on the outer barrel.
- 10) Pressure test the inner barrel assembly.
- a) With the core assembly in the slips and collar clamp in place, remove the pick-up sub but do not remove the 1' spacing sub.
  - b) Attach the kelly (or top drive) to the outer barrel assembly. Bring the rig pumps up to speed to achieve a flow rate that is between 190 gal/min and 260 gal/min.
  - c) The Corion coring engineer will record these numbers (SPM, flow rate, operating pressures, etc.) and will ensure that the inner barrel assembly is properly seated.
  - d) Turn off the rig pumps. Break the connections between the kelly (or top drive), the 1' spacing sub and the seat sub.
  - e) Remove the 1' spacing sub from the outer core barrel and put it aside.
- 11) Remove the inner barrel assembly from the outer barrel.
- a) Attach the birdhouse clamp to the rope socket of the inner barrel assembly.
  - b) Attach the rig's winch line to the birdhouse clamp and lift the assembly upwards approximately 1 foot (0.30m).
  - c) Attach the pressure head clamp to the inner barrel assembly. Lower the assembly back into the outer barrel and have the pressure head clamp rest on the box end of the seat sub attached to the outer barrel.
  - d) Remove the birdhouse clamp from the rig's winch line. Attach the winch line to the pressure head clamp on top of the inner barrel assembly.
  - e) Slowly raise the inner barrel assembly and remove it from the outer barrel in the table.
  - f) Attach the wheeled cart to the bottom of the inner barrel and lower it down the V-door onto the catwalk.

12) Pick up the inner drilling assembly from the catwalk.

- a) Attach the rig's winch line to the pressure head clamp, which is fastened to the top of the inner drilling assembly.
- b) Ensure that the wheeled cart is attached to the bottom of the inner drilling assembly to protect the insert bit while it is being raised up the V-door.



- c) Slowly raise the inner drilling assembly up the V-door and remove the wheeled cart once the assembly is hanging vertical in the derrick.
  - d) Slowly lower the inner drilling assembly into the outer core barrel with the winch line. The pressure head clamp will rest on the box end of the seat sub attached to the outer barrel.
  - e) Remove the rig's winch line from the pressure head clamp. Fasten the birdhouse clamp on the top of the rope socket of the inner drilling assembly and attach the rig's winch line.
  - f) Slightly raise the inner drilling assembly in order to remove the pressure head clamp from the assembly using a 1 1/4" wrench.
  - g) Once the pressure head clamp is removed, lower the inner drilling assembly into the outer barrel. Once seated, remove the birdhouse clamp.
- 13) After the inner drilling assembly is seated, insert the pick-up sub and 1' spacing sub into the seat sub attached to the outer barrel.
- 14) Latch the elevators to the pick-up sub. Check the lead between the insert bit and the core bit.
- a) Hoist the core barrel assembly.
  - b) Remove the collar clamp from the outer barrel, remove the slips and hoist the outer barrel upwards out of the hole so the core bit is chest height. Install the hole cover.
  - c) The coring engineer will check the lead in order to ensure that the pilot bit is seated and flush with the core bit.
- 15) Remove the hole cover and lower the outer barrel back into the table. Insert the slips and install the collar clamp on the outer barrel.
- 16) Pressure test the inner drilling assembly.
- a) Set the core barrel assembly back in the table with the slips and a collar clamp. Remove the pick-up sub but do not remove the 1' spacing sub.
  - b) Attach the kelly (or top drive) to the outer barrel assembly. Bring the rig pumps up to speed to achieve a flow rate that is between 330 gal/min and 380 gal/min.
  - c) The core hand will record these numbers (SPM, flow rate, operating pressures, etc.) and will ensure that the inner drilling assembly is properly seated.
  - d) Turn off the rig pumps. Break the connections between the kelly (or top drive), the 1' spacing sub and the seat sub.

- e) Remove the 1' spacing sub from the outer core barrel and put it aside.
- 17) Remove the inner drilling assembly from the outer barrel.
- Attach the birdhouse clamp to the rope socket of the inner drilling assembly.
  - Attach the rig's winch line to the birdhouse clamp and lift the assembly upwards approximately 0.30m.
  - Attach the pressure head clamp to the inner drilling assembly. Lower the assembly back into the outer barrel and have the pressure head clamp rest on the box end of the seat sub attached to the outer barrel.
  - Remove the birdhouse clamp from the rig's winch line. Attach the winch line to the pressure head clamp on top of the inner drilling assembly.
  - Slowly raise the inner drilling assembly and remove it from the outer barrel in the table.
  - Attach the wheeled cart to the bottom of the inner drilling assembly and lower it down the V-door onto the catwalk.
- 18) Attach the top stabilizer to the seat sub on the core barrel. Important Note: Do not run the 1' spacing sub in the BHA.
- 19) Pick up the required amount of Corion Express core collars and assemble them above the outer core barrel. Torque connections up to 26,000 ft-lbs (DDD threads) or to 10,000 ft.lbs. (4 1/2" IF threads).
- 20) All Corion Express collars are picked up in the same manner as conventional drill collars.
- 21) Drift all Corion Express core collars in the same fashion as drifting Corion Express drill pipe.
- 22) Run in the hole open-ended with Corion Express drill pipe. Ensure that the drill pipe is drifted prior to making up connections. Torque drill pipe to 10,000 ft-lbs (4 1/2" IF threads).

## **6.5 Temperature Gauge and Rabbit**

- Corion will supply a temperature gauge and rabbit connection similar to that utilized in the 2002 Mallik program. This is used to monitor the temperature while the core is acquired and retrieved to surface.
- USGS will also provide additional micro pressure-temperature sensors as backup to the Corion system, along with additional instrumentation.

## **6.6 Connections while Coring**

- There can be no connections while coring. Pup joints will be utilized to ensure there is adequate spacing to cut a full 24' core barrel. Pup joints available in 5 ft, 10 ft and 15 ft lengths.

## 6.7 Core Run Termination

- Coring should be terminated when the barrel is full (i.e. the core has reached top of the inner core barrel) or when evidence of jamming has been observed. In event of a jam, the inner core barrel will unlatch itself from its seat and an immediate drop of pressure will be observed on the rig pumps. At this point, core will be tripped to surface using the defined pulling out of hole protocol (POOH), described below.
- If coring is about to be terminated and ROP is high, consider reducing the flowrate (to a minimum safe rate) in order to minimize possible core erosion and ensure that there is full gauge core in catcher. This is not a typical operation when using the CSS-513. Once the core is in the catcher and in the inner barrel, it should not be affected by the flow rate. Normal operations would be to allow the weight to drill off at the desired depth, reduce pump to an idle, reduce rotary to 0 RPM and then stop the pump.
- Coring mechanics may be slightly different due to consolidated nature of these sands. Requires discussions with NOV Corion.
- It is not recommended to spin the barrel at higher rpm to "burn core in" as is traditional in some applications; the inner barrel may rotate with the outer, causing core damage. It is never a recommendation to "burn in" core. Since the Corion Express inner barrel is held in place with hydraulic thrust, it is impossible to reduce volume to "burn in".
- If low ROP suggests that the core is finishing in harder rock or has packed off, then coring should be terminated at the normal flowrate.
- If torque and pressure indicate that bottom hole conditions are prone to sticking, and stopping the rotary with the barrel on bottom poses as significant sticking risk, the core can be broken with rotation, but it is not recommended.
- Once the core is broken, the new section of hole should be reamed up and down at low rotary speeds until the hole is completely clean of cuttings and torque is steady and low before POOH.
- Circulation after coring should be performed in a way that minimizes the risk of soft sandstone core being washed from the catcher. The preferred option is to circulate the minimum amount for well control needs before tripping out. Clean the hole by washing down and circulating bottoms-up before starting the next core run. Reciprocating during wireline core retrieval is also possible.
- With circulation being possible while wire-line retrieving the inner barrel, some thought should be given to the amount of circulation needed before retrieving the inner barrel.

## **7 CORE RETRIEVAL WITH WIRELINE**

### **7.1 Objectives**

- Retrieve core in the minimum time without swabbing the well, dropping the core, or damaging the core by the expansion of escaping fluids.

### **7.2 Safety Considerations**

- The retrieval of a large core barrel within a small annulus is a prescription of swabbing. The core engineer should calculate acceptable wireline-retrievable tripping rates to prevent swabbing.
- During wireline retrieval of core, care must be taken to not swab pore fluids up the drill-string. This interval has not been penetrated at this location and the exact nature of the pore fluids, while interpreted to contain gas hydrate, is not known; pore fluids may include water, gas hydrate, and/or free gas.
- Normal drillfloor procedure for safe tripping and wire-lining is required.
- The reservoir sections may be cored with moderate overbalance so the adoption of procedures to avoid differential sticking of the coring assembly is essential until BHA is safely tripped into the surface casing.

### **7.3 Optimal Wireline Pulling Speed - Tradeoffs**

The optimal pulling speed is a tradeoff of hydrate preservation, rig time, risk of swabbing, and the negative effect of rapid fluid expansion in the core as it is being de-pressured. There are four basic concerns that should be addressed in the core retrieval procedure, as follows:

- Minimize the time it takes to get the core to surface to allow intact hydrate to be observed and measured.
- Prevent the influx of gas saturated fluids into the wellbore (i.e. swabbing).
- Prevent damage to the core due to rapid fluid or gas expansion as hydrostatic pressure is lowered.
- The fourth concern is mechanical damage, which can occur during tripping the drill string and handling the core.

#### **7.3.1 Hydrate Preservation**

To preserve as much of the hydrate as possible, the time it takes to retrieve the core from its native temperature and pressure state (downhole), to the surface should be minimized.

Because the hydrate will exit the hydrate stability zone as the pressure is reduced below approximately 200 psi (a depth of approximately 550 feet), any hydrate that is present in the core will begin to dissociate. To minimize the amount of dissociation that takes place, before it is observed on the surface, the core retrieval time should be minimized.

### **7.3.2 Swabbing**

Because the OD of the core will be only slightly smaller than the ID of the drill collars and drillpipe, when retrieving the core via wireline, a negative swabbing pressure (due to a syringe effect) will be induced on the reservoir interval.

There is some amount of flexibility of pulling speed versus risk of swabbing. If the top valve on the diverter sub is closed, a pulling speed of up to 200 feet per minute (fpm) can be attained with little to no swab pressure induced on the formation. If the valve is left open, swabbing is more likely to occur. It is recommended that the diverter valve stay closed so that pulling speeds of 200 fpm can be attained.

### **7.3.3 Expanding Pore Fluids**

Expanding pore fluids, which are unable to escape from the core while tripping out of hole, may induce whole-core expansion and possible fracturing. This damage mechanism is most common in poorly consolidated or friable sediments containing gas or water, or with core that has suffered high amounts of mud filtrate invasion. If hydrates are present, and the core fluid has a low filtrate rate, this phenomenon should not occur. Thus, in the expected case of gas hydrate-bearing core, this core should be tripped to surface at maximum possible rates while avoiding excess swabbing. If however free gas is suspected, than pulling speeds may need to be lowered.

### **7.3.4 Mechanical Damage**

Care should be taken to treat the core as carefully as possible so not to damage the core or prevent accurate analysis of the core. Cores and core assemblies should not be treated as “dumb iron”.

## **7.4 Operational Considerations**

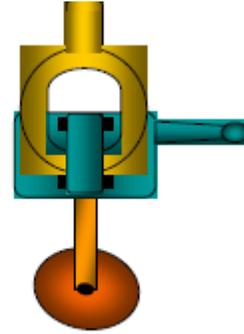
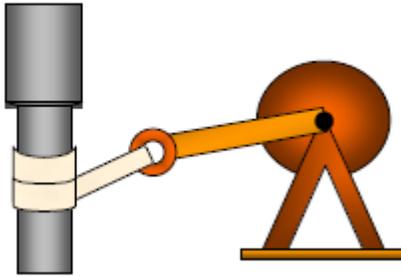
Described below are details about the wireline retrieval process that should be followed.

### **7.4.1 Rig-floor Precautions**

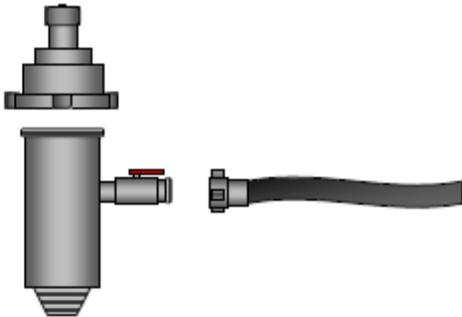
- If tripping the assembly up into casing, braking and slip setting should be performed without jarring the core. Sudden vertical shocks to the drill string can result in substantial core damage and may result in loss of core.
- Minimize rotation of the core barrel when breaking connections. Top drive, iron roughneck, or chain tongs should be used to rotate the pipe above the connection, rather than using the rotary table to rotate the string below the connection.
- When recovering the core it will be done via wire-line. There will be no pipe conveyed in core retrieval. The wire line movement is very constant and the stretch of the line actually absorbs some of the shock of starting and stopping.

## 7.4.2 Rigging Up the Wire Line Unit

- 1) Have a safety meeting with the rig crews and wireline operators prior to rigging up wireline unit.
- 2) Have the drill pipe stump sticking up approximately 0.75m above the slips. Sling one sheave to the stump with a double wrap.



- 3) Attach the second sheave to the elevators or the top drive with the supplied slings.
- 4) Screw the diverter sub into the drill pipe stump in the table and attach the 2" high pressure hose.



- 5) The 2" high pressure hose will either be used to drain fluid to the trip tank as it is displaced, or it will be connected to the standpipe in order to pump down the inner drilling assembly.
- 6) String the wire line through the sheaves and slowly raise the top sheave with the elevators. Watch for loops in wireline and for tangles on the drilling floor and catwalk.
- 7) Raise the top drive elevators approximately 13 feet (4 M) above the diverter sub in the stump.
- 8) The wireline running gear will be hoisted and lowered into and out of the diverter sub by the wireline operators.

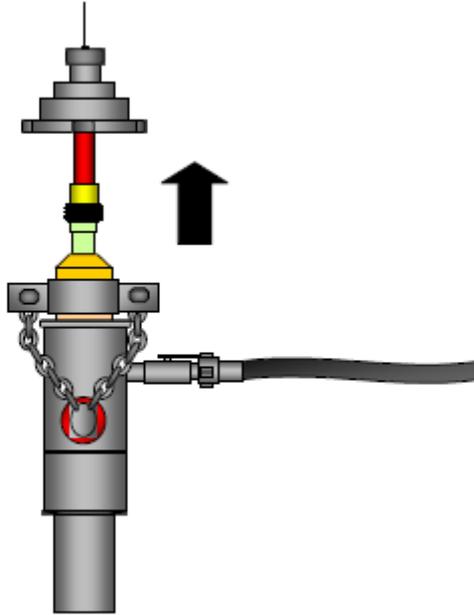
- 9) Ensure that all hands are off the drilling floor when running the wireline in and out of the drill string. Maintain proper communication between the driller, core hands and wireline operators at all times.

### **7.4.3 Retrieving Core and Tripping Rate (out of hole)**

- 1) Remove the kelly (or top drive) and rack back any pipe required to keep the BHA above the reservoir zone.
- 2) Install the diverter sub into the drill pipe stump and attach the 2" diverter hose.
- 3) Rig up the wireline equipment as described above.
- 4) Slowly lower the running gear into the diverter sub and attach the stripping head to the diverter sub.
- 5) Lower the running gear and overshot to the bottom of the hole and latch onto the inner barrel.

The following recommendations are based on good results during prior gas hydrate-bearing coring operations:

- Retrieve wireline core from 2500-ft to surface at normal controlled rate up to 200 feet per minute, preventing swabbing by slowing wireline speed if free gas is suspected to occur, depending on operational and gas show observations.
  - Recognize that the gas hydrate-bearing core will exit from the gas hydrate stability field above approximately 500-600 feet TVDs. From this point (or from an agreed depth below this point as determined by onsite operations), the core should be retrieved via wireline as quickly as possible to avoid excessive gas hydrate dissociation. This assumes the mud, hole, and well control conditions are good.
  - Provision should be made to increase or decrease the tripping rates if necessary.
- 6) Remove the stripping head and pull the running gear out of the stump until the pressure head is above the diverter sub.
  - 7) Place the pressure head clamp onto the top of the inner barrel assembly. Lower the inner barrel assembly. The pressure head clamp will rest on the box end of the drill pipe stump.



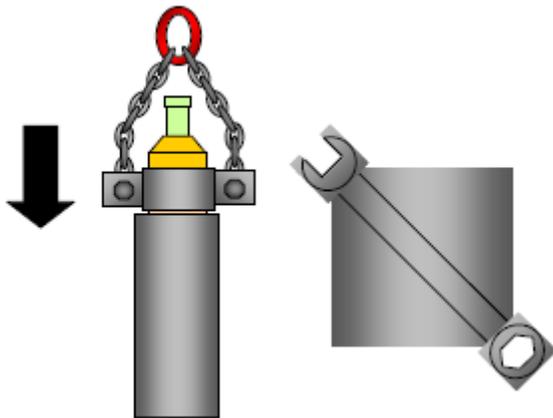
- 8) Allow the wire line to become slack and remove the overshot from the inner barrel.
- 9) Set the wire line equipment off to the side where it won't be an obstruction or a hazard.
- 10) Lay down the inner barrel assembly.
  - a) Cautiously hoist the inner barrel assembly with the rig's winch line and attach the wheeled cart to it.
  - b) Be cautious in order to prevent deflection of the core inside.
  - c) Carefully lower the inner barrel assembly down the V-door using the rig's winch line.
  - d) The shift-lead geologist will examine, measure, and record the annulus between the core and the inner barrel at each cut core face during processing and chip sampling in order to identify possible damage during the pulling of the core. Adjustment to pulling speed will be made accordingly.
- 11) If additional core is to be cut, connect the kelly (or top drive) to the drill pipe stump and circulate the hole (at approximately 0.75 m<sup>3</sup>/min) while recovering core and preparing a new inner barrel assembly.
- 12) Repeat required steps as listed above for each cored section.

#### **7.4.4 Installing the Inner Barrel**

Once the core has been removed from the drill string, a second empty inner core barrel assembly can then be pumped to the coring BHA to re-dress the tool.

- 1) Circulate bottoms up for approximately 5 minutes to allow for any obstruction to be cleared from the drill string.

- 2) Remove the kelly (or top drive).
- 3) Pick up the inner barrel assembly from the catwalk.
  - a) Attach the rig's winch line to the pressure head clamp, which is fastened to the top of the inner barrel assembly.
  - b) Ensure that the wheeled cart is attached to the bottom of the inner barrel to protect it while it is being raised up the V-door.
  - c) Slowly raise the inner barrel assembly up the V-door and remove the wheeled cart once the assembly is hanging vertical in the derrick.
  - d) Slowly lower the inner barrel assembly into the drill pipe stump in the table with the winch line. The pressure head clamp will rest on the box end of the drill pipe stump.
  - e) Remove the rig's winch line from the pressure head clamp.
- 4) Holding onto the pressure head clamp chain with one hand, drop the inner barrel assembly into the drill string by loosening the pressure head clamp bolt using a 1 ¼" wrench.



- 5) Once the pressure head clamp is dropped into the drill string, make up the kelly (or top drive).
- 6) Start the rig pumps up and circulate the inner barrel assembly to bottom with the pumps at an idle. The flow rate should be between 80 GPM to 160 GPM.
- 7) The pressure will increase to a minimum of surface test pressure indicating that the inner barrel assembly has reached bottom.
- 8) Once the inner barrel has seated, increase the pumps to the listed operating parameters (200 GPM and 275 GPM) and wash to bottom.
- 9) Begin coring at the discretion of the coring engineer.

## **8 RIGFLOOR CORE HANDLING AND CORE LAYDOWN IN PIPESHED (CATWALK)**

### **8.1 Objective**

- To safely move the inner barrel and core from the drill floor to the processing area without damaging the core.
- Handle the core in optimal time at minimal cost.

### **8.2 Safety Considerations**

- Core laydown is not a routine activity. The coring engineer will lead a Task Risk Assessment (TRA) and discussion with the rig crew involved to ensure that safe and effective procedures are used before beginning the core lay-down.
- TRAs will be reviewed with each crew as changes occur.
- Any misalignment of the inner tube during the cutting and the application of the shear boot may result in dropping the core onto the drill floor. This activity must therefore be conducted with great care.
- Stringent precautions for heavy lifting must be followed with care – this is one of the most potentially dangerous parts of the whole coring operation.

### **8.3 Operational Considerations**

Described below are guidelines to follow and issues to consider when laying down the core.

#### **8.3.1 Requirement for Specialized Surface Handling**

- Once at the surface, the core must be quickly removed from the drill floor to allow critical path drilling operations to be carried out without delay.
- The 24 foot steel inner barrel which will contain two 12 foot aluminum sleeves (liners) will be laid down using the rig's winch line or crane jib.
- Care should be taken during surface handling and processing of core to avoid core damage by inner barrel flexure and impact.
- Care must be taken to contain the oil-based mud during core assembly retrieval to surface and during subsequent transport to pipeshed and core trailer. Absorbent material will be required; plastic wrap may also be required during transport from rig floor to pipeshed.

#### **8.3.2 Inner Barrel Separation**

- Gentle core handling is essential. The rig crew input is critical to a safe and successful coring operation. Core team supervisors and members should have informal discussions with the rig crew to emphasize the importance of gentle core handling.
- The rig floor extraction of the core assembly, breakdown of the core barrel, laydown of the inner barrel, and breaking of the catcher will be led by the coring engineer.

- If there are any sections of the inner core barrel not filled with core, these will likely be full with MOB. The mud bucket should be attached to minimize mud spillage on the drillfloor during removal of these sections.
- The joint in the 24-foot core assembly is threaded and unscrewed into two 12-foot sections
- The lower section of inner barrel is clamped and the inner tube alignment device is fitted to the inner tube above and below the joint.

## 8.4 Core Lay-down Procedure

A core cradle is not planned (or being pre-assembled) for transporting the core barrel assembly from rig-floor to pipeshed as core damage is not expected. If however damage occurs during the initial core run, the need for a core cradle will be reassessed and one may be constructed on-site using pipe joints.

After removal from the core barrel assembly, the two 12-foot inner aluminum sleeves inside the two 12 foot steel outer barrels must be quickly and safely transferred to the core trailer processing area using wooden boxes, without bending or breaking the core. The core trailer provides a safe environment for the core processing team, and minimizes disruption to drilling operations.

The procedure to laydown the core barrel is as follows:

1. Using the rig crane or winch line, gently lower the core assembly (inner and outer barrel) down the pipe skid (V-door) to the catwalk/pipeshed
2. With the core assembly in the pipeshed and using absorbent material and proper PPE, extrude the inner core sleeve from the inner core barrel and unthread the inner assembly into two 12-foot sections.
3. Place each of the 12 foot sections into wooden boxes lined with absorbent material to contain any residual MOB. Boxes are supplied by Weatherford/Omni.
4. Transfer the core to the core receiving trailer in wooden boxes. Due to the weight of each 12 foot core section and the tall height of the pipeshed relative ground level, a fork-lift will be required to bring the inner barrels down to ground level and to the core trailer.
5. When the core arrives in the core trailer, the 12 foot section will be laid out on the table with the taps positioned on the side of the core. The tabs will then be cut and the top of liner removed. Sub-sampling and core descriptions will take place on the full 12 foot long section.
6. After sub-sampling and core description is completed, the top of liner will be replaced over the core and the two liner halves tape wrapped together. The full section will then be cut with pipe cutters into 3 foot segments at the pre-cut tabs.
7. Subsequent sub-sampling and analysis methods are described in detail in Volumes 2 and 3 respectively.

The exact best handling method and safest way of laying down the core assembly and inner barrels will be decided by the drilling personnel, core team, and core technicians on location,

depending on the specific rig equipment. This detailed procedure will be described in the TSA for the activity.

## **8.5 Laying Down Drill Pipe and the BHA (post wireline coring)**

- Hold a safety meeting with rig crews prior to laying down any tools to point out hazards and discuss safe operating practices.

### **8.5.1 Laying Down the Corion Express Drill Pipe.**

1. The Corion Express drill pipe must have pin protectors on at any time the pipe is being lowered down the V-door onto the catwalk. Failure to follow these procedures could damage the drill string.
2. Pick-up nubbins should be installed in the box end of each Corion Express drill pipe to ensure that no seal damage occurs.
3. Repeat the above steps for the remaining drill pipe in the hole.

### **8.5.2 Laying Down the Corion Express Core Collars.**

1. Lay down the core collars in the same manner as conventional drill collars using a lay down line or lay down machine.
2. Ensure that protectors are attached to each core collar prior to laying them down the V-door.

### **8.5.3 Laying down the Corion Express Core Barrel.**

1. Set the core barrel in the slips with a collar clamp.
2. Break the connections between the top stabilizer, the double pin seat sub, and the core barrel.
3. Remove the top stabilizer from the outer core barrel. Put the top stabilizer aside with protectors on it. Keep the pick-up sub as it will be required again.

### **8.5.4 Remove the Inner Drilling Assembly from the Outer Barrel.**

1. Attach the birdhouse clamp to the rope socket of the inner drilling assembly.
2. Attach the rig's winch line to the birdhouse clamp and lift the assembly upwards approximately 0.30m.
3. Attach the pressure head clamp to the inner drilling assembly. Lower the assembly back into the outer barrel and have the pressure head clamp rest on the box end of the seat sub attached to the outer barrel.
4. Remove the birdhouse clamp from the rig's winch line. Attach the winch line to the pressure head clamp on top of the inner drilling assembly.
5. Slowly raise the inner drilling assembly and remove it from the outer barrel in the table.
6. Attach the wheeled cart to the bottom of the inner drilling assembly and lower it down the V-door onto the catwalk.

7. Attach the lifting sub and the 1' spacing sub to the seat sub on the core barrel and hoist the barrel so the bit is out of the hole.
8. Attach the bit breaker and carefully remove the bit.
9. Attach the bottom protector to the core barrel and lay it down the same way as the Corion Express core collars.

## **9 DATA RECORDING REQUIREMENTS OF CORING ENGINEER**

Described in this section are the reporting requirements of the Coring Engineer for the various phases of coring.

### **9.1 During each Core Run**

- Continuously monitor all coring parameters and trends and maintain constant communication with the Driller to make sure that drilling does not continue after core jams and/or pack-offs.
- If the torque, ROP, or stand pipe pressure deviate substantially from the baseline, a real-time plot of coring parameters (described below) should be reviewed with the Driller and shift lead geologist to determine if core is being cut, or adjustments need to be made.
- Tripping parameters should be continually monitored and compared with the theoretical fill rates and the general tripping plan. This should be done for the usual well control reasons and in order to safeguard against departure from tripping plan.
- If tripping parameters vary substantially from the plan, the Driller should be notified immediately.

### **9.2 After each Core Run**

- The following real-time coring parameters should be plotted (.pdf file and paper plot) at 16 cm/hr scale from the time ball is dropped to the time the core barrel is tripped:
  - Hook Height (ft.)
  - Hook Load - WOH (klbs)
  - Weight on Bit - WOB (klbs)
  - Rotary speed, RPM
  - Rotary Torque (ft.lbs)
  - Pump pressure (psi)
  - Flowrates (gpm)
  - ROP (m/hr)
  - Total Gas (%)
- Trip monitoring data (depth of bit vs. trip rate in minutes/stand) is to be provided in Excel format and given to the core specialist immediately after each core trip. A paper plot of trip performance should also be produced for immediate discussion with the Drilling Supervisor, Operations Geologist, core shift lead geologist, and core specialist, in case the tripping schedule requires modification.

### **9.3 On Completion of Coring**

- Excel file of coring parameters for each core on high-resolution regular depth spacing (reading every 0.5 meter) to be given to the core shift lead geologist on completion of the last core, including the following parameters:
  - ROP (ft/hr & m/hr),

- WOB (klbs.)
- RPM, Average, Maximum and Minimum Torque (ft.lbs.)
- Flowrates in/out (gpm)
- Mud temperature, in/out
- Pump pressure (psi)
- Temperature (from Rabbit gauge)
- GammaRay (if run).
- Details of the coring BHA and core barrel run for each core.

## **Core Procedures Manual**

### **Volume 2: Core Handling, Sub-Sampling, On-Site Analysis, Preservation, Transportation Draft, Rev1.0 – September 21, 2009**

## **INTRODUCTION**

This “Core Procedures Manual, Volume 2” contains a description of proposed best practices of the on-site core handling and testing portion of the Barrow Gas Hydrate Test Well Program. These procedures are the primary responsibility of the core handling contractor and on-site Science team.

This well is being drilled as a gas hydrate test well within Phase 2B of the North Slope Borough (NSB) - US Department of Energy (DOE) Gas Hydrate Cooperative Research Project. This volume of the manual includes several written elements of the coring plan, including the following:

- Personnel roster, roles, and responsibilities
- Operational procedures covering:
  - Equipment and Supplies
  - Core handling
  - On-site sub-sampling
- Core preservation
- Transportation
- Storage

A total of four documents have been written to cover the complete scope of coring, core preparation, on-site core analysis, and subsequent decision criteria to complete the well. These documents (with volumes 1, 3, and 4 under separate cover) are as follows:

- ***Volume 1 – Core Acquisition***  
Discusses pre-well planning and coring operations up through core laydown
- ***Volume 2 – Core Handling, On-site Analysis***  
Discusses core handling, sub-sampling, preservation, and transportation
- ***Volume 3 – Post-Field Core Analysis***  
Discusses off-site core analysis tests and methods conducted by the Science Party
- ***Volume 4 - Decision Criteria to Complete Well***  
Discusses the test results that will trigger a decision to complete or abandon the well

Lessons learned from previous hydrate-bearing cored wells, including the Mallik (1998 and 2002), Hot Ice (2004), and Mt. Elbert (2007) arctic onshore wells, and certain offshore research programs are incorporated into these documents where applicable.

The program has been designed to deliver the key core objectives identified by the Gas Hydrate project research team and the Barrow Field development team including members of DOE, USGS, NSB, PRA, and supporting service companies. This document will be reviewed and refined through a number of meetings leading up to well spud, including a coring budget workshop being planned for September 2009. This manual will then be used as an on-site policy and procedures manual to guide all aspects of the coring program.

## **Document Overview**

This document describes the procedures used to handle, sub-sample, preserve, and transport the core. It also discusses the on-site analysis that will be done to support wellsite decisions. The document is into six categories, as follows:

1. Planning and Preparation
2. Core Processing and Sub-sampling
3. On-site Analysis
4. Core Preservation and Freezing
5. Core Transportation
6. Core Storage

On-site analysis in this application is required for two reasons:

1. To observe and measure properties as close to native state as possible, and
2. To determine if hydrates are in fact present in the reservoir, so that the decision to complete the well as a monitoring well can be made.

The detailed procedures described in this document are meant to represent a “super-set” or master set of procedures. In some cases, only a sub-set of these procedures will apply, depending on the specifics of the well and core that is gathered.

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# 1 OVERVIEW AND GENERAL GUIDELINES

This chapter describes personnel responsibilities and anticipated work shifts, in addition to a general overview of the core handling and core analysis process. The methods described in this document and *Volume 3 – Core Analysis* are intended to follow the Integrated Ocean Drilling Program (IODP) protocols, where applicable, but have been modified to meet the requirements of an on-shore, Arctic hydrate project.

## 1.1 Background

### Savik No. 1

**Hole TD:** Permitted to ??? feet TVDss, Operational TD expected at ??? feet TVDss

**Core point:** Approximately 1715 ft TVDss in the Walakpa Sand  
Approximately 1935 ft TVDss in the Upper and Lower Barrow Sandstone

#### Primary core targets:

Two 24-ft. cores to completely capture a 10 foot thick gas-hydrate-saturated sandstone section in the Walakpa sandstone, located above the base of the gas hydrate stability zone. This interval is predicted to be at a depth of approximately 1715 ft (TVDss). No free-gas-bearing sections expected.

Five 24-ft cores to completely capture a 100 ft. thick gas-hydrate sandstone section in the Barrow sandstone, just above or coincident to the base of the gas hydrate stability zone which is predicted to be at a depth of approximately 1935 ft (TVDss). Free gas is not expected, but remains a possibility.

### Walakpa No. 15

**Hole TD:** Permitted to ??? feet TVDss, Operational TD expected at ??? feet TVDss

**Core point:** Approximately 1950 ft TVDss in the Walakpa Sand

#### Primary core targets:

Two 24-ft-cores to completely capture an 18 foot thick gas-hydrate-saturated sandstone sections in the Walakpa sandstone, located above the base of the gas hydrate stability zone. This interval is predicted to be at a depth of approximately 1950 ft (TVDss). No free-gas-bearing sections expected, but is possible.

## 1.2 Core Team Members, Roles, and Responsibilities

This section is included in all three volumes to ensure all parties understand their roles and responsibilities.

The “core supervisors” listed below will be responsible for core lengths, core tops, meeting coring objectives, etc. and will provide 24 hour coverage with at least one person available at all times.

- 1 PRA/??? Operations Geologist, oversight
- 1 DOE Geologist, oversight
- 1 USGS Geologist, oversight

The contractor-based team members listed below will work 12 hours shifts with full-time coverage of the operation.

- 4 total, 2 per shift, Nov Corion lead core engineer and wireline engineer
- 4 total, 2/shift Weatherford/Omni core marking, breaking, and preservation staff

The on-site Science Party will consist of the following: (Note: the table below summarizes these responsibilities). *Note: The tentative roster of on-site core supervisory and Science Party team listed below will be revised and updated as coring operations near and individual’s schedules are determined.*

- 8 total, 4/shift USGS, USDOE, and Oregon State University core sub-sampling staff (pore waters, geochemistry, microbiology, gas hydrate-specific core properties analyses.)
- 2 total, 1/shift Organic Geochemistry and Microbiology sub-sampling staff
- 2 total, 1/shift Physical Properties sub-sampling staff
- 4 total, 2/shift Inorganic Geochemistry (water sampling)

<u>NAME</u>	<u>AFFILIATION</u>	<u>RESPONSIBILITIES</u>
TBD Ops geologist	PRA/???	Core point, TD, wellsite core shift supervisor
Tim Collett	USGS	Core point, TD, wellsite core shift supervisor
Ray Boswell	US DOE	Core Handling and swing shift supervisor
Kelly Rose	US DOE	Core Handling, core water chemistry
TBD	US DOE/TBD	Core Handling, water chemistry, thermal conductivity
Rick Colwell	Oregon State Univ.	Core Sampling, geochemistry, microbiology
Marta Torres	Oregon State Univ.	Core Sampling, water analyses lead
TBD	USGS	Core Sampling, physical properties
TBD	USGS	Core Sampling, geochemistry, microbiology
TBD	USGS	Core Sampling, physical properties
TBD	TBD	Core Sampling, water chemistry
TBD	TBD	Core Sampling, inorganic chemistry

- The Drilling Supervisor (“Company Man”) has ultimate responsibility and final say over all coring and drilling operations at the wellsite. Described below are the principal roles

and responsibilities of the “core team” members, all of whom advise and report to the Drilling Supervisor while at the wellsite.

- The PRA Operations Geologist is responsible for facilitating and coordinating all communication between the scientific staff (“Science Party”) and the Drilling Supervisor.
- The HSE Supervisor will be included and consulted on all matters of an operational nature that have safety or spill risk elements.
- The Core shift supervisors/leads serve in an oversight role and are responsible for coordination of core data, technical guidance, team coordination, and assurance of coring objectives.
- NOV Corion technicians are responsible for supplying all equipment, manpower, and expertise for core acquisition through core lay-down in the pipe-shed.
- Weatherford/Omni staff will be responsible for inner core barrel marking, whole core measurement, marking, preservation, wellsite processing, assistance with USGS onsite sub-sampling, bulk core stabilization by freezing, onsite core storage, core transportation to Anchorage, and post-program routine and specialized core analyses if required.
- The Science Party, with support from Weatherford/Omni staff, will be responsible for on-site sub-sampling, on-site analysis including logging, lithostratigraphic descriptions, organic and inorganic chemistry, and physical property measurements.
- Weatherford/Omni staff will be responsible for shipping other frozen samples, mud samples, State of Alaska chip samples, and liquid nitrogen stored samples as required.
- The Science Party will be responsible for return shipment of laboratory equipment and certain specialized core subsamples.
- Canned cuttings and samples will be shipped by USGS to USGS at Menlo Park.

### **1.3 Work Shifts, Time Estimates**

A 12-hour work shift system will be implemented for all team members. The expectation is that no one should need to work longer than 12 hour days with an absolute maximum of 16 hours in a day. The basic analysis plan for 24-foot core sections requires two 6 person teams for processing the core as documented in the procedures and time estimates of the next section of this document. This team of 12 (two 6 man shifts) is needed to maintain safe work hours for 1-2 days of successive 24 foot cores with approximately 90-120 minutes between cores. A 30-90 minute shift change-over time will be required during each shift change, depending on operations and difficulties.

Core acquisition turn-around is expected to take 75 to 90 minutes per 24 foot core with the Corion system at optimal efficiency. Core processing and sub-sampling is estimated to require 60 minutes per 24 foot core. Core analysis will take as long as necessary, but is expected to be completed by the time the well is logged so decisions regarding the completion of the well can be made without having to place the operation on standby time.

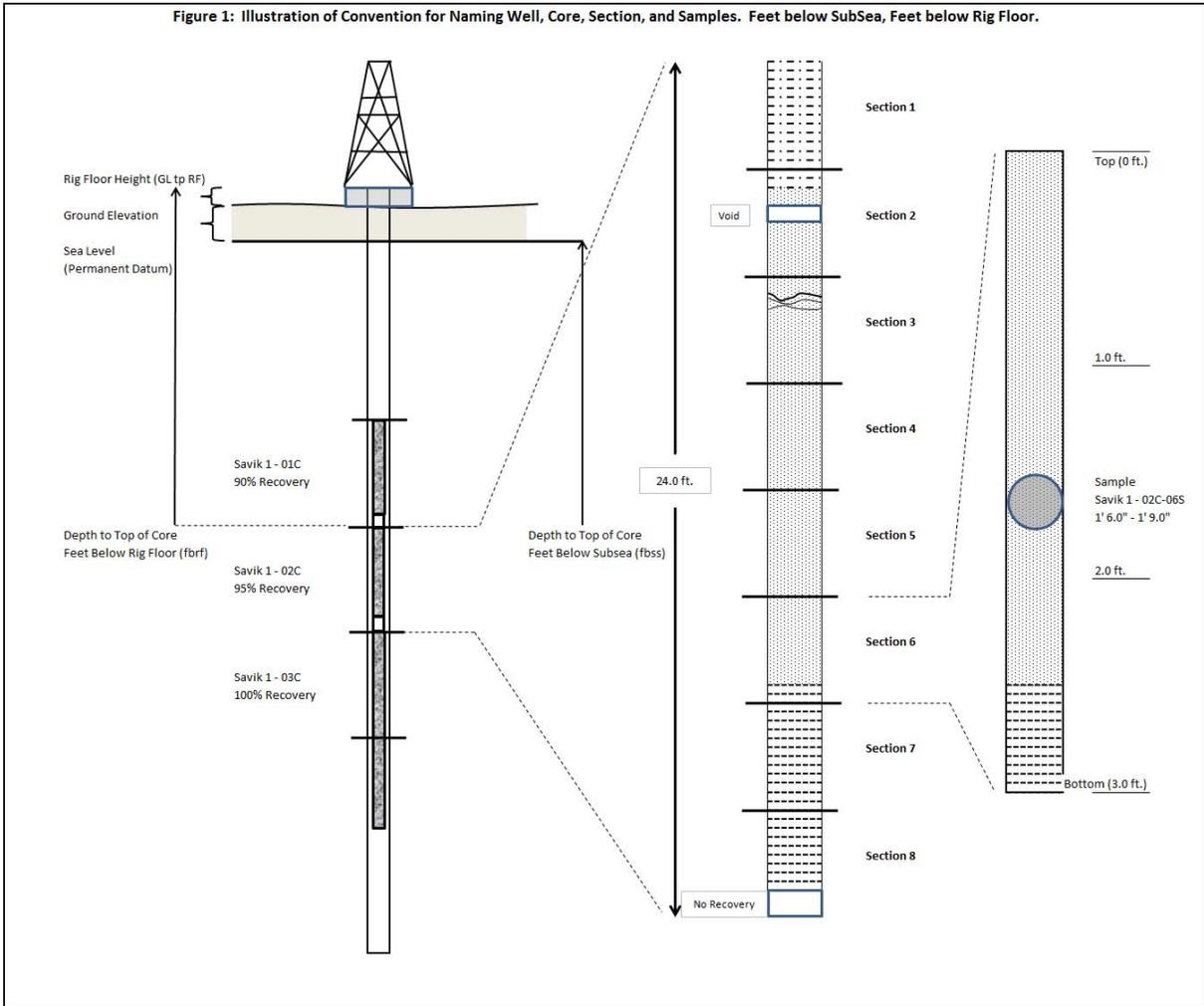
### **1.4 Core Curation and Sample Depth Calculation**

Cores will be obtained with the NOV Corion Express wireline retrievable coring system as described in *Volume 1 – Core Acquisition* of this Coring Procedures Manual. In addition to

cores, a variety of data will be obtained through LWD/MWD, and through wireline logging in cored vertical holes. The data collection and interpretation methods that govern the logging program are described in detail in a separate document, entitled “Downhole Logging.”

As is the standard convention in U.S. oilfields, drilling depth is measured in terms of feet below rig floor (fbrf) as determined from a strap of the drillstring and shared amongst all wellsite parties including mud loggers, directional drillers, loggers, and the Science Party. All depths will be kept and maintained in English (Imperial) units below rig floor for all measurements that pertain to the core. The core top in feet below rig floor (fbrf) measurement is the ultimate depth reference for all other depth calculations.

In addition to relative position within a core section, specific intervals and horizons are also described in “curatorial” feet below the rig floor (fbrf). The fbrf of a sample or horizon is calculated by adding the interval depth of the sample and the lengths of all upper sections to the core top datum as measured with the drill string. Often, cores expand upon recovery, leading to more than 100% recovery (for example, 9.8 meters of core recovery whereas bit advance may have been only 9.6 meters). Likewise, because the core top is marked at the highest recovered sediment within a core, there is often an unquantifiable coring gap between cores. As a result, discrepancies may exist between the drilling depth and curatorial depth, including instances where the curatorial depth at the bottom of one core may be greater than that for the top of the subsequent (deeper) core.



**Figure 1 – Conventions for noting holes, cores, section, and samples, with depths**

In cores with less than complete recovery, the uppermost recovered sediment is assumed to mark the top of the core. Furthermore, all other recovered sediment within the core is assumed to mark a continuous section downward. Therefore, in most cases when sediment is missing from a core, that missing section is curated to occur at the base of the core. This convention results in a necessary sampling uncertainty that should be taken into consideration when working with depth data (e.g. core to log correlation).

## 1.5 Drilling-Induced Core Deformation

Many cores show signs of disturbance. Bedding deformation, particularly along the outer edge, is a common effect of the coring process, as are sediment mixing (particularly at the tops of cores), fluidization (“biscuit and slurry”), and liquid injection. Core deformation such as creation of partings also occurs commonly as a result of the depressurization associated with core retrieval. Dissociation of gas hydrate and gas expansion also result from core depressurization (and warming), and can further disturb the sediment.

## 1.6 Core Handling and Analyses Overview

Cores acquired during the Barrow Hydrate Test well program are intended to be handled according to ODP/IODP procedures. Modifications to these procedures, similar to those made in Mt. Elbert and Mallik 5L-38 projects, are designed to enable quick identification of gas hydrate intervals and maintain aseptic conditions for microbiological sampling.

It should be noted that Infrared (IR) scanning techniques are not useful in Arctic conditions as there is little contrast between a cold core from endothermic reaction, and the naturally cold core that is removed from the well. Consequently, full-length IR scans are not planned for this well.

Priority should be placed on the quick determination and sub-sampling of zones suspected to contain gas hydrate. Digital thermometers and IR guns can provide temperature measurements during processing and sub-sampling. Observations of decreasing temperature during processing would be attributed to ongoing endothermic effects of gas hydrate dissociation. In addition, small samples will be removed and immersed in water; active bubbling of gas from these samples provides confirmation of gas hydrate occurrence.

As part of the handling process, the following steps are taken:

- Core subsamples will be sent to the warm geotrailer (geochemistry laboratory) for inspection and testing (i.e. extraction of interstitial waters for geochemical analyses). The core will then be:
  - Sampled and stored for post-well microbiological studies, and
  - Sampled and sealed in pint-size cans for headspace gas analysis.
- Gas hydrate samples will be taken and stored in liquid nitrogen.
- A series of non-anomalous background samples will be taken from each core for immediate geochemical analyses.
- The suite of pre-planned samples are then cut, capped, labeled, and stored as appropriate for physical and petrophysical properties.
- The remainder of the core will then be measured for sectioning, and labeled with a permanent marker with up orientation, core number, core type, and section number.
- The remaining core is then cut into sections 3.0 feet in length, wrapped in plastic, capped, and prepared for transport.

Once in the laboratory (warm geotrailer), the remaining whole round core sections are tested for thermal conductivity. Then, if it was determined that the MSCL machine is required for on-site analysis then the core will be passed through the multi-sensor core logger (MSCL) to obtain measurements of *P*-wave velocity, non-contact resistivity, gamma ray attenuation bulk density, and magnetic susceptibility. After these tests are completed, the core is preserved and prepared to transport for off-site analysis, as described in Volume 3. Offsite analysis includes the following:

The cores are split, from bottom to top, creating separate archive and working halves. Investigators should keep in mind that splitting, using either a pulled wire or a fixed circular saw blade, may further disturb the core and result in the transport of material upward along the surface of each core half. The working half is then sampled for further physical properties

testing (moisture and density, shear strength, split-core acoustic velocity, and contact resistivity) including MSCL measurements if not conducted at the wellsite. Next, the working half will be further sub-sampled. Sub-sampling includes the regular collection of background material for later sedimentological, x-ray diffraction, carbon dating, and paleomagnetism studies, as well as targeted sampling of notable features such as clay, mineral, or carbonate nodules, macrofossils, and other features. The archive half sections are to be scanned on the digital imaging system (DIS). Visual core descriptions (VCDs) of the archive halves are prepared, augmented by microscopic analyses of smear slides. Digital close-up photographs are taken of particular features for illustrations. Both halves of the core are then placed in labeled plastic tubes and transferred to cold storage for transportation to final location.

## **2 CORE HANDLING PLANNING AND PREPARATION**

Considerable front-end planning will be required to insure that all equipment, supplies, and procedures are in place to handle and process the core as soon as it arrives on the surface. This section is designed to capture the long lead items that need to be worked and communicated prior to well spud.

This section will continue to be a work in progress. It is intended to serve as a “catch all” of outstanding issues and other details that arise during the detailed planning phase.

### **2.1 Long Lead Items**

The following items will take considerable time to locate, secure, and have delivered to the wellsite and ready for the core handling operation.

#### **2.1.1 Cold Core Trailer (handling, cutting, sub-sampling the core)**

At least one 40’ long X 8’ wide trailer, will be required to handle, cut, and sub-sample the core in a cold environment. This will likely be supplied by Corion. The trailer should be intrinsically safe (explosion proof, Cl I, Div I) as methane gas is expected to be present during routine core operations. The trailer should be outfitted with adequate lighting and ventilation, LEL detection, and two exits for safe egress in case of emergency.

#### **2.1.2 Warm Geo-Trailer (conduct core analyses)**

A “geo-trailer” or geological laboratory trailer, at least 20’ long X 8’ wide, will be required to conduct core analysis tests in. This would likely be supplied by Weatherford/Omni or AES. The trailer should be intrinsically safe (explosion proof, Cl I, Div I) as methane gas is expected to be present during routine core operations. The trailer should be outfitted with adequate lighting and ventilation, LEL detection, and two exits for safe egress in case of emergency.

#### **2.1.3 Liquid Nitrogen Supply**

Approximately ?? gallons of liquid nitrogen will need to be secured and delivered to the wellsite for preserving certain core samples.

#### **2.1.4 Shipping Containers**

Various sizes and types of shipping containers will need to be secured.

### **2.2 Mud Logger collected Gas Samples**

The mud logging contractor will be capturing cutting and gas samples that will be analyzed by the Science Party.

- Canned drill cuttings: The contract mud logging company collects and cans drilling cuttings as follows:
  - With cuttings being collected at 30 foot spacing from about 1500 feet TVDss (surface casing point) to TD. The canned drill cuttings should be treated with an extra heavy dose of table salt and frozen.
  - Note: this is the same technique used to store core headspace samples in pint-sized cans, as described earlier.
- Flowed gas samples: The contract mud logging company should also collect flowing free gas samples from the mud trap installed in the drill mud return tank on the shaker table (near the location where the canned drill cuttings were collected).

### **2.3 Other**

### **3 CORE PROCESSING AND SUBSAMPLING FOR ON-SITE ANALYSIS**

This section describes the steps and procedures to process the core from the pipeshed to the geologic laboratory trailer (geo-trailer) where on-site analyses will be conducted.

This section may need some work to make sure it is consistent, but not redundant with *Volume 3 – Core Analysis*. This will require communication between Omni/Weatherford and Science party, facilitated primarily by Tim Collett, Mike Dunn, and Bob Hunter.

#### **3.1 Objectives**

- Rapid description of core samples and core fluids to support real-time operational decisions.
- Isolate and sub-sample the central portion of the core as soon as possible after coring to obtain samples with minimum (ideally zero) mud filtrate invasion and to minimize time-temperature-pressure dependent gas hydrate dissociation.
- Seal and protect time and temperature-dependent samples so that they arrive at the laboratory for analysis in good condition.
- Maintain core integrity to the extent possible so that subsequent off-site analyses yield valuable data.
- Perform on-site analysis to support real-time decision to complete the well

#### **3.2 Equipment Setup and Testing**

This document gives a detailed list of all equipment and laboratory apparatus that will be used for on-site analysis. Below are a few guidelines about setup and testing.

- The core team/Science Party will setup, test, and evaluate all coring, core handling, core processing, core sub-sampling, and core preservation equipment as soon as possible after arrival at the wellsite.
- The core team and rig representatives will evaluate core handling equipment and procedures for compatibility with all rig systems as soon as possible after arrival at the wellsite.
- Laboratory equipment and its function are the responsibility of the core handling contractor, or responsible member of the Science Party, according to ownership of the equipment. The rig electrician can be used to help install and integrate the equipment, but is not responsible for insuring proper function or calibration of said equipment.

#### **3.3 Safety Considerations**

Core handling, by its nature, presents manual handling risks in the form of lifting, pinching, and exposure to chemicals. Core handling operations will be carefully reviewed with the team, and

all risks eliminated or minimized. All core processing activities must be reviewed with the Drilling Supervisor and HSE Officer before work begins to make sure it does not conflict with any other activities. Proper hot work and/or general work permits must be obtained for any specialized procedures and equipment. Only authorized users are allowed to use specialized equipment such as power saws, rock press, etc.

Note: The core from the Walakpa and Barrow sandstones are much harder and more consolidated than cores taken at Mt. Elbert, Hot Ice, and Mallik. As such, different cutting techniques may need to be used. A rock chop saw will likely be needed to cut the core.

- Core processing is a non-routine activity. Pre-job briefings and training will be given to any staff member who temporarily assists the core analysis team (e.g. rig crew member, mud loggers, etc.).
- Core handling will involve cleaning the oil-based mud from the outside surface of the core. Proper PPE, wiping rags, and rag disposal procedures must be used to minimize health exposure and environmental impacts of this operation.
- The aluminum inner core sleeve has tabs which require cutting using a small pneumatic powered abrasion saw which must only be used by qualified operators with appropriate PPE including gloves, goggles, dust mask, and earplugs. All non-essential staff should stand clear.
- The core itself will be cut with a rock chop saw and/or chisel and hammer; proper PPE and precaution must be used to avoid rock chipping hazard and potential eye injuries.
- Care and caution should be a priority when operating the hydraulic press to collect interstitial water. Extreme pressure and forces will be exerted when squeezing the core samples. Note: The rock press may not be able to extract water in this core, as is typical of unconsolidated core. A centrifuging procedure is included as a back-up.
- Appropriate caution and care should be taken with the compressed air line for presses in the geo trailer. Note: Air lines exposed to ambient cold temperatures require special protection and procedures.
- Appropriate caution should be used with the outdoor nitrogen station near the core trailer. The methane and nitrogen bottles should be stabilized and strapped using a standard bottle rack assembly and protected from the elements by placing them on the leeward side of the trailer and constructing a temporary shelter, if needed.
- A hot-work permit must be pulled for electrical equipment in the presence of potential out-gassing from hydrate dissociation of the core.

### **3.4 Required Equipment, Instruments, and Supplies**

- 12 foot long Wooden Boxes
- Handheld abrasion saw, air powered for cutting aluminum tabs
- Rock “chop” saw – preferably air powered (vs. electric powered) for cutting core
- Pipe cutter
- Rubber squeegee, spatula, rags for wiping core
- Hand-held digital thermometers – minimum of four (4)
- Hand held Infrared IR Thermometer gun – minimum of four (4) of the type at <http://www.sperdirect.com/cgi-bin/category.cgi?item=800103&type=store> or equivalent

- Foam core fillers/spacers
- Core Analysis form on clipboard
- Tape measure – decimal feet
- Hammer and chisel for collecting core sub-samples
- Liquid nitrogen vessels
- Quart-sized cans for storing sub-samples
- Rags for cleaning inner barrel, solvent for erasing marking errors, paint pens that will indelibly mark inner barrel under rigsite conditions, good quality steel tape measure greater than 30-ft long.
- Pneumatic abrasion saw for cutting aluminum tabs. The saw must be designed to present minimum risk to the operator and core team and be capable of cutting through the inner barrel tabs in the slotted liner and the core safely in one pass with minimal vibration. Spare saw blades must be supplied.
- As a backup, a 7-1/4” circular “skil” saw with steel blade
- Equipment used to protect core faces: 2 end caps and 2 clips required per cut section. Approximately 4 caps and 4 clips should be available for each 3-foot core section (including those for temporary capping of samples awaiting preservation).
- Plastic wrap to wrap inside of core liner and prevent core from freezing to liner.
- Plastic bag wrap for wrapping core sections,
- Black electrical tape and Duct tape to wrap core
- Sharpie markers and labels for labeling core
- Core racks sufficient to hold multiple 3-foot sections of core to be provided by Weatherford/Omni (4X4 containers)
- Sealable sample bags and sampling equipment (spoon for soft sandstone and hammer and screwdriver or small chisel for hard sections). Paint scrapers and spatulas for cleaning core faces for inspection
- Sampling lists and box logs.

### **3.5 Core Receiving, Handling, and Processing in the Cold Core Trailer**

Described in this section is a step-by-step procedure of handling and sub-sampling the core to prepare samples for on-site core analysis. It should be noted that *Volume 3 – Core Analysis* provides a detailed description of the subsequent tests that will be run on the core and core samples.

#### **3.5.1 Initial Core Receiving in the Cold Core Trailer**

This procedure is expected to take approximately 10 minutes per 24’ core section. Omni/Weatherford core technicians will conduct this work.

1. Move the core from the pipeshed to the Corion cold core trailer in 12 foot wooden boxes. Use forklift if necessary. Use small service door to access the Corion trailer.
2. Pick up the core and move to trays on the work bench.
3. Clip tabs on the aluminum sleeve with small abrasion saw
4. Lay out full 12 foot length of core and conduct initial inspection

5. Wipe off and mark the core (top, bottom, mark each 3 foot increment)

### **3.5.2 Core Measuring and Logging**

This procedure is expected to take approximately 15 minutes per 24' core section. Weatherford/Omni core technicians will direct or conduct this work with direct supervision from members of the Science Party.

1. Set tape measure at top of core, extend full length alongside core.
2. Position marker (index) card at top depth in decimal feet.
3. Cleave core to match pre-cut foam inserts for sub-sampling requirements.
4. The core will be slid in the core tray to remove any small gaps in the core; however, any large voids will be filled with labeled foam fillers.
5. Quickly scrape the core with a rubber squeegee or spatula, observe, and identify the following:
  - a. Gas hydrate occurrence
  - b. Physical properties
  - c. Gross sedimentology and structural geology
  - d. Grain-size, grading, etc.
6. Enter observed information into standard Core Analysis Form
7. Install thermometers (about four hand-held digital type) and record temperature of inner core in degrees F where inner core is available to accept thermometer.
8. For this (consolidated, hard) core, IR thermometer guns (as specified above) should be used to measure temperature of core surface at multiple points.
9. Pull core data P-T logger ("Temperature Rabbit"), replace memory module, and return to Corion coring engineer for next core run.

### **3.5.3 Photo Imaging and Video Recording**

This procedure is expected to take approximately 15 minutes per 24' core section. Weatherford/Omni core technicians will conduct this work with direct supervision from members of the Science Party.

- Set up and conduct plain light photo imaging. Two digital cameras (10 Gb and 6.2 Gb system), along with portable lights, copy stand, tripod, and system computer for still photography will be used. The USGS will provide all of the listed photo equipment.
- Photo images will be taken on an ad-hoc basis and not as complete core scans. Images will consist of standard core images and image logs of samples taken from the core.
- Setup and have available digital (DVD) video camera system, to be provided by the USGS. Handheld video of each core is useful for archive purposes.

### **3.5.4 Sub-Sampling – Identification, Creation of Samples**

This procedure is expected to take approximately 10 minutes per 24' core section. Weatherford/Omni core technicians and Core Shift Supervisor will conduct this work with direct oversight from members of the Science Party.

1. Note the depth of each sub-sample and record sample depths on master clip-board form. Sample depths will be marked and recorded in decimal feet.
2. Core shift supervisor cuts each subsample with rock chop saw, and/or hammer and chisel.
3. Collect and move each sample to the designated sample work area (organic chemistry, in-organic chemistry, micro-biological, physical property, etc.)
4. Physical Property representative will supervise Weatherford/Omni technician while gas hydrate-bearing cores are being preserved in liquid nitrogen.

### **3.6 Disposition of Whole Round Core (WRC) Sampling for On-site Analysis**

This section discusses the disposition of samples for on-site, real time analysis. The detailed analysis procedures are described in the following section, *On-site Analysis*.

- Pore water sampling for field processing, WRC samples (3-4 WRC samples per core placed in air tight sample glove bags; blow open bag using nitrogen, then insert sample), sampled as directed, from reservoir sand and shale sections, samples should not be frozen and are transferred to warm trailer for squeezing.
- Thermal testing - 3" to 6" long whole, round core (WRC) samples will be received in insulated containers. The core will be pushed through the container; sheared off to expose a new clean, flat surface; the surface irregularities will be removed; and then the thermal properties will be analyzed.
- Physical properties (MSCL), a total of about 2 WRC samples per 24' core section, sampled as directed, from reservoir sand and shale sections.

**Note:** It is not been finalized whether MSCL testing will be done at the wellsite, or conducted post-field. As currently written, the MSCL testing procedure is included in both volumes.

### **3.7 Disposition of Whole Round Core (WRC) Sampling for Off-site Analysis**

This section discusses where the various samples are expected to be used in subsequent analysis procedures. The detailed analysis procedures are described in *Volume 3 – Post Field Core Analysis*.

- Head space gas samples, WRC samples (3-4 WRC samples per core placed in quart size cans; 50 quart cans will be available; sampled as directed, from reservoir sand and shale sections, samples should be and remain frozen; will transport in igloo coolers.
- Microbiological samples, WRC samples in bag, partial heat-seal, seal with nitrogen (approximately 2 samples per core); duplicate sample directly tied to every-other head-space gas sample; Spacing will be determined.

- Pore water sampling for field processing, WRC samples (3-4 WRC samples per core placed in air tight sample glove bags; blow open bag using nitrogen, then insert sample), sampled as directed, from reservoir sand and shale sections, samples should not be frozen and are transferred to warm trailer for squeezing.
- Physical properties (MAD and grain size analysis), single WRC samples (4-6 WRC samples after clean-up edges to avoid contamination per core), for grain density analysis, sampled as directed, from reservoir sand and shale sections, samples should not be frozen.
- Physical properties (geotechnical and strength testing), a total of about 10 WRC samples for the entire well, sampled as directed, from reservoir sand and shale sections, samples will be both frozen and non-frozen; wrapped in foil and plastic wrap and placed in zip-lock or COREHANCON 3 foot zip-lock (if available) bags.
- Petrophysical properties (intrinsic porosity-permeability studies, no gas hydrate phase), a total of about 20 WRC samples for the entire well, sampled as directed, from reservoir sand and shale sections, samples should be frozen.
- A Weatherford/Omni representative will bag small chips from the top faces of each 1-foot core as taken by shift-team lead length as required by the State of Alaska, per the diagram below.

## **4 ON-SITE CORE ANALYSIS (ON-SITE AND POST-FIELD)**

Described in this section are the on-site tests that will be done as soon as the core is retrieved to surface. The results of these tests will help determine whether hydrates are in fact present, and whether the well should be completed as a monitoring well.

### **4.1 Gas Hydrate Tests, Cold Trailer**

This procedure is expected to take approximately 10 minutes per 24' core section. Weatherford/Omni core technicians will conduct this work with direct supervision from members of the Science Party.

- Quick gas hydrate dissociation test. Gather small pieces of core – typically core edge-chips (2-4 samples per core), 3 different systems:
  - Volume yield in water (2 gallon igloo cooler to insulate water from freezing),
  - Syringe system – most reliable (2-5cc in 100ml syringe; place by core), and
  - Small pressure vessel (rare samples, large chunks of gas hydrate only); concurrent activity.
- Gas hydrate samples (noted best by visual observation of bubbling core)
- Whole-round-core (WRC) samples from apparent gas-hydrate-bearing sections (20 plus per project), to be stored in
  - Liquid nitrogen shippers (WRC lengths 12cm or 25 cm long), 25cm long cups, wrapped in Aluminum foil, put in cloth bags, and labeled.

### **4.2 Inorganic Geochemistry (Interstitial Water Analyses, Warm Geo-Trailer)**

The measurement of inorganic geochemistry is critical to understanding the make-up and origin of interstitial water, which is key to understanding the presence and nature of gas hydrates. This chapter describes the processes used to sample, preserve, and measure the inorganic geochemistry of the core fluids.

**Note:** This section is a work in progress, pending consultation with Marta Torres at Oregon State University (OSU) and possible testing of existing Barrow and Walakpa cores. The methodology and sampling equipment needed in support of the centrifuge method and rhizon method is uncertain at this time and will need to be developed in consultation with OSU.

Given the hard, consolidated nature of these cores, conventional “squeezing” of the core may not be possible. This issue will require considerable discussion with USGS geologists and experts at Oregon State University, and others. Existing Barrow field and Walakpa field cores will be examined and tested to develop a suitable water extraction method. Options being considered include the following:

- 1) Centrifuge method

- 2) Rhizon method – the rhizon consists of a micro-porous tube with a pore size of 0.1  $\mu\text{m}$  supported by a nylon wire. The material and size of the tube allows it to soak up the pore fluid with capillary action. Whether this method can be used to obtain a sample of pore fluid from a consolidated core needs to be tested and addressed.
- 3) Hydraulic press method

If the centrifuge method is used, the procedure would read as follows:

1. Store WRC in glove bag while waiting to be centrifuged
2. Clean WRC to remove outer layer contamination
3. Examine and describe sample
4. Take sub-sample for grain size
5. Load the WRC into a tube that is sized to fit into the centrifuge
6. Load the tube (and core) into the centrifuge
7. Spin the sample for a minimum time of ?? minutes at a minimum of ?? rpm
8. Press and collect the water into syringe (10 or 30 cc, depends on expected volume)
9. .... (to be edited and completed after working with OSU).

If the rhizon method is used, the procedure would read as follows:

1. Store WRC in glove bag while waiting to be rhizon sampled
2. Clean WRC to remove outer layer contamination
3. Examine and describe sample
4. Take sub-sample for grain size
5. Load the WRC into ..... that allows a rhizon strand to be inserted
6. ....(to be edited and completed after working with OSU).

If a hydraulic press were to be used, the procedure would read as follows:

These samples will be squeezed to destruction using a hydraulic press to obtain pore water samples. While obtaining pore fluids is a simple operation, samples must be properly handled to avoid contamination and oxidation of some chemical species, and the squeezers must be adequately cleaned between samples to avoid cross sample contamination or interference by small amounts of oil-based mud that may be present.

1. Store WRC in glove bag while waiting to be squeezed
2. Clean WRC to remove outer layer contamination
3. Examine and describe sample
4. Take sub-sample for grain size
5. Load the WRC into the hydraulic squeezer
6. Press and collect the water into syringe (10 or 30 cc, depends on expected volume)
7. Filter through a 2  $\mu\text{m}$  acrudisk (on line filter)
8. Measure salinity using refractometer and record in logbook
9. Take a  $R_w$  measurement using the probe
10. Record total volume of water collected

Field laboratory: Squeezed water samples will be immediately analyzed for salinity, alkalinity, and ammonium, and sulfide will be precipitated in order to subsequently (lab-based) obtain correct sulfate and sulfide concentrations.

#### **4.2.1 Required Equipment, Instruments, Supplies, and Chemicals**

##### ***4.2.1.1 All Methods***

- Whatman No. 1 filter
- 0.45 µm Nalgene disposable filter
- Goldberg optical hand-held refractometer
- Metrohm autotitrator
- Ultrapure HCl, minimum of xx ml.
- 5% HgCl<sub>2</sub>
- Various glass vials, including 2 mL and 10 mL sizes
- Amber colored glass septum vials
- Cryovials, 5mL sizes precharged with ultrapure HNO<sub>3</sub>
- Glass ampoules
- Vacutainers – 5 ml size
- Agilent vials
- Sulfate analysis preservative Cadmium Nitrate - Cd(NO<sub>3</sub>)<sub>2</sub>
- 10 mM sodium bicarbonate and 20 mM sodium carbonate standards for salinity calibration
- Metrohm 761 (861) ion chromatograph
- Silver nitrate - AgNO<sub>3</sub>
- Ever-Pure RT-3 water filter cartridge
- Barnstead NANOpure® Diamond™ Analytical (Model D11901) deionization system

##### ***4.2.1.2 Hydraulic Press Method***

- Hydraulic press Titanium squeezer, with forces up to 30,000 lbs.

##### ***4.2.1.3 Centrifuge Method***

In addition to the equipment listing above, the centrifuge method will require the following:

- Centrifuge
- Test tubes
- ..... (to be edited and completed after working with OSU).

##### ***4.2.1.4 Rhizon Method***

In addition to the equipment listing above, the centrifuge method will require the following:

- Rhizon tubes
- Test tubes

- ..... (to be edited and completed after working with OSU).

#### **4.2.2 Collection of Sub-samples for On-site Analyses**

The majority of on-site interstitial water (IW) samples are obtained on 5- to 30-cm-long whole-round core using the process described below:

- Interstitial water is passed through a pre-washed dry Whatman No. 1 filter fitted above a titanium screen, filtered through a 0.45  $\mu\text{m}$  Nalgene disposable filter, and subsequently extruded into a new, plastic syringe attached to the bottom of the squeezer assembly.
- In most cases, 25–40  $\text{cm}^3$  of pore water is collected from each sample, which requires squeezing the sediment for 5–90 minutes, depending on lithology.

#### **4.2.3 Collection of Sub-samples for Off-site Analyses**

Sub-samples for both alkalinity and sulfate determinations are taken and analyzed as soon after interstitial water collection as possible. The procedure for collecting sub-samples for off-site analyses is described below:

- A sub-sample for ammonium analysis (1.00 mL) is pipetted as soon after squeezing as practical into a glass ampoule, acidified with 0.010 mL ultrapure HCl, and flame-sealed.
- The remaining interstitial water is placed into a new polypropylene (PP) centrifuge tube and split for on-site and off-site analyses.
- Total dissolved inorganic carbon (DIC) samples (1 mL) are preserved with 0.100 mL 5%  $\text{HgCl}_2$  in 2 mL glass vials with septum caps.
- Samples for dissolved organic carbon (DOC) and volatile fatty acids (0-3 mL) are stored in amber glass septum vials and frozen.
- Samples for oxygen and hydrogen isotopes (1-3 mL) and major ions (2-8 mL) are flame-sealed in glass ampoules.
- Minor element samples are pipetted into 5 mL cryovials that have been pre-charged with 0.040 mL ultrapure  $\text{HNO}_3$ .
- DIC isotope samples are syringed into 5 mL vacutainers that are pre-loaded with 0.100 mL 5%  $\text{HgCl}_2$ .
- 2 mL samples for off-site sulfate analyses are pipetted into agilent vials and preserved with 100  $\mu\text{L}$   $\text{Cd}(\text{NO}_3)_2$  to precipitate sulfide out of solution.
- The remaining water is used for on-site chloride, bromide and sulfate measurements.

#### **4.2.4 On-site Interstitial Water Analyses**

Routine on-site measurements are conducted according to standard procedures (Gieskes et al., 1991), as described below:

- Salinity is measured using a Goldberg optical hand-held refractometer.
- pH is determined by ion-selective electrode.
- Alkalinity is determined by Gran titration with a Metrohm autotitrator. Accuracy and precision are monitored by repeated calibrations using analyses of International

Association of the Physical Sciences of the Ocean (IAPSO) standard seawater, as well as 10 mM sodium bicarbonate and 20 mM sodium carbonate standards. The ion-selective electrode is calibrated and standards analyzed every ten samples. The average external accuracy and precision were based on the multiple analyses of the standards <2% and ~1%, respectively.

- Sulfate (SO<sub>4</sub><sup>2-</sup>) concentration is determined by manual dilution and manual injection into a Metrohm 761 (861) ion chromatograph with eluent suppression, on aliquots to which Cd(NO<sub>3</sub>)<sub>2</sub> is added as soon as possible after interstitial water collection.
- Sulfate to chloride and bromide to chloride ratios are determined by comparison of peak heights to those measured for IAPSO standard seawater. Sulfate and bromide concentrations are determined by multiplying the respective ratios by the chloride concentration determined by titration. Based on replicate analyses of IAPSO the standard deviation of the sulfate/chloride ratio determination should be less than 0.25%. Combined with the uncertainty of the chloride concentration, the resulting confidence limit for sulfate concentration is 0.3% at seawater concentrations based on repeated analysis of IAPSO. Aliquots for shore-based analyses are processed following the sampling plan given in Table T1 below.

Table T1: Sample division plan for interstitial waters.

PORE FLUID DIVISION SCHEME													
NGHP Expedition 01													
	Alkalinity	SO <sub>4</sub> /H <sub>2</sub> S	DIC Concentrations	Acetate	NH <sub>4</sub>	DIC isotopes	DOC/VFA	O/H	Majors/Cl	Minors SiO	Minors OSU (Leg 4)	Shipboard	Total
code	IWPA	IWPSO	IWGTC	IWGTLA	IWGN	IWGIC	IWGOC	IWGI	IWGSM	IWPSM	IWPOM	IWPS	
subsample container	plastic test tube w cap	1.5 ml centrifuge tube	2 ml agilent vial	2 ml agilent vial	1 ml ampoule	5 ml vacutainer or 2 ml agilent vial	4 ml amber autosampler vial	2 or 5 ml ampoule	2, 5, 10, or 20 ml ampoule	5 ml plastic cryotube	Torres Nalgene bottle	1.5 ml centrifuge tube	
treatment		100µl Cd(NO <sub>3</sub> ) <sub>2</sub>	100µl HgCl <sub>2</sub>		10µl Optima HCl	100µl HgCl <sub>2</sub>	freeze			40µl Optima HNO <sub>3</sub>	40µl Optima HNO <sub>3</sub>		
>35ml	3	1	1	0.5	1	3	3	3	7	5	8	1.5	37.0
30ml	3	1	1	0.5	1	2	2	2	6	5	5	1.5	30.0
25ml	3	1	1	0.5	1	2	2	2	4	3	4	1.5	25.0
20ml	3	1	1	0.5	1	2	1	1	3	2	4	1	20.5
15ml	0	1	1	0.5	1	2	0	2	2	2	3	1	15.5
10ml	0	1	1	0	0	1	0	1	1	2	2	1	10.0
5ml	0	1	0	0	0	0	0	0	1	1	1	1	5.0

- High-precision chloride concentrations are determined by Mohr titration using silver nitrate (AgNO<sub>3</sub>) for most samples. Quantification is based on comparison with IAPSO standard seawater. Most chloride concentrations are determined in duplicate. The reproducibility of the chloride titrations, expressed as 1 σ relative standard deviations, is evaluated by replicate analyses of IAPSO standard seawater and should be <0.2%. If the reagent needed for Cl titration is exhausted, Cl<sup>-</sup> can be analyzed by IC, with a precision of 0.7%. These samples will be re-analyzed off-site by titration.
- Bromide concentration is analyzed by manual injection into a Metrohm 761(861) ion chromatograph with eluent suppressor used for sulfate concentration. The precision based on duplicate analyses of IAPSO standard seawater, expressed as 1 σ relative standard deviations should be 1-2%.

All figures are plotted based on concentration data that are not corrected for drillwater contamination?

#### **4.2.5 On-site Processing to Create High-Purity Water**

Water used for all well site dilutions, solution preparations, and final washing of equipment (stir bars, spatulae, core squeezer parts, etc.) is produced by passing the wellsite's potable water through an Ever- Pure RT-3 water filter cartridge followed by a Barnstead NANOpure® Diamond™ Analytical (Model D11901) deionization system. The conductivity of the output of the NANOpure system is monitored and should have a conductivity of 18.2 Mohm-cm.

As an alternative to processing potable water, it may be necessary to purchase high-purity water in bulk and transport to the well site.

Field laboratory: Squeezed water samples will be immediately analyzed for salinity, alkalinity, and ammonium, and sulfide will be precipitated in order to subsequently (lab-based) obtain correct sulfate and sulfide concentrations. [See above description about all analysis post field]

Lab-based analyses will consist of the following major, minor, and trace components:

- -Majors: Cl, Na, K, Ca, and Mg concentrations;
- -Minors and traces: Sr, Br, Fe, I, Ba and Mn concentrations
- -Dissolved inorganic carbon (DIC) will be analyzed for  $\delta^{13}\text{C}$  values

#### **4.2.6 Disposition of Pore Water Samples**

- 2 ml in 4-ml glass vials glass vial for  $\text{NH}_4$  analyses, frozen
- 1 ml for sulfate analyses in 2 ml-microcentrifuge tubes pre spiked with  $\text{CdNO}_3$ -residue for sulfide work to 10 ml for halogens in glass vials (4 or 20 ml).
- 2 ml in agilent vials for  $\delta^{13}\text{C}$ , preserved with  $\text{HgCl}_2$
- 2 ml in agilent vials for  $\delta^{13}\text{C}$ , frozen (in case the  $\text{HgCl}_2$  results in salting out effects due to low salinity of the fluids)
- 2 ml in glass vials for acetate, frozen
- 2 ml in agilent vials for D/O- no treatment
- The rest in 8 ml nalgene bottles for major and minor analyses

#### **4.2.7 Other Notes**

- Store and seal the squeeze cakes in plastic bags.
- Clean and dry the squeezer for next sample
- Sample and process drill fluid samples and cuttings on a regular basis
- Drilling fluid that is uncontaminated by water or chemicals can be recycled back into the rig's mud tanks
- Proper site disposal procedures of other materials should be followed

### 4.3 Thermal-Properties Tests on Whole Round Core

The temperature history of a core sample from *in situ* to surface conditions is needed to understand the impact on recovered microbiology, gas hydrate, and other samples. During the coring process, frictional heat is generated, warming the cores by unknown and variable amount. Frictional heat is also generated by rotary drilling, even though the bit is being cooled by coring fluid that is pumped downhole at cool temperatures (4-5 °C). During core recovery, the core should not be exposed to warmer temperatures, with the minimum temperature likely in the pipeshed or on the catwalk.

Dissociation and endothermic cooling starts as the core is retrieved to surface above a depth of approximately 500 ft. and hydrostatic pressure of 250 psi. Because of these and other complications discussed below, the temperature history of the core is an important clue to the measurement of hydrates at the surface.

Since the process of core retrieval is fairly uniform, the temperatures at which cores arrive in the core trailer should be relatively consistent unless there are additional heat sources or sinks. Dissociation of gas hydrate, which is an endothermic process, represents one such heat sink, resulting in anomalous cold spots in the core. Other processes that can lead to cold spots in cores include gas evolution from pore water and adiabatic expansion of gas.

However, it should be noted that given the ambient cold temperature of the rig floor, catwalk, and core trailer, there may be little or very subtle differences between the natural core temperature, and any temperature drop due to endothermic cooling. Nevertheless, IR temperature scanning with handheld guns will be done.

The IR handheld gun is quick and simple to use and has a much higher spatial resolution than an array of thermocouples. For the Barrow Hydrate Test Well project, on-site analyses will be performed immediately to estimate the presence of gas hydrates.

#### 4.3.1 Preparation of Core Samples

The basic procedure to prepare cores for measurement of physical properties, are as follows:

- Soon after whole cores arrive in the cold core trailer, they are wiped with cloth rags to remove excess drilling fluid or sediment residue.
- After inspecting the 12 foot long sections, IR sample scans are performed on the core cross sections. Cold spots are interpreted to represent dissociating or dissociated gas hydrate, are used to select the location of sub-sample plugs for interstitial water, headspace gas, and gas hydrate measurements.
- The core sections are then moved into the warm trailer laboratory to equilibrate to room temperature which typically requires 2-4 hours according to core-end-temperature probes.

#### 4.3.2 Procedure

3” to 6” long whole, round core (WRC) samples will be received in insulated containers and placed into a cooler for sample preservation. The core will be pushed through the container; sheared off to expose a new clean, flat surface; the surface irregularities will be removed; and then the thermal properties will be analyzed.

Note: The procedure below assumes that thermometer probes will gather accurate temperatures. If this appears impractical because of the hardness of the core, handheld IR gun measurements can be substituted.

Thermal property measurements will be made by pressing the thermal property probe against the smoothed hydrate surface. Each measurement will take on the order of 30 seconds. For each core sample that is received, this measurement process will be repeated at approximately every inch. Efforts will be made to conserve core material by combining this WRC with water analyses and/or physical properties sub-sampling. Likely 1 sample per core, some gas hydrate-bearing, others non-hydrate-bearing.

Equipment used for these analyses will include:

- Laptop computer
- Agilent E364A Dual Output DC Power Supply
- National Instruments cDAQ-9172, compact data acquisition chassis
- Sensor assembly and circuitry, including insulated resistor
- Basic tools for assembly and multi-meter
- Core sample holders and ice chest for sample receiving/holding/preserving.

#### **4.3.3 Monitoring the Catwalk/Pipeshed or Cold Trailer Environment**

A total of four HOBO pendant temperature measurement devices will be deployed on the catwalk or in the cold trailer (depending on where IR measurements are taken), as follows:

- One on the rig floor
- One on the catwalk, in the pipe shed
- One outside the core unit between pipe shed and trailer
- One inside the core unit

#### **4.3.4 Monitoring Core Equilibration by Direct Contact Measurement of Core Temperature**

Direct measurement of core temperature is made on a routine basis using the following devices and procedure:

- The device used is a Onset Computer Corporation manufactured HOBO weatherproof temperature loggers (HOBO Outdoor/industrial 4-channel External Temperature Logger, H08-008-4, Onset Computer Corporation) and stainless steel sheathed thermocouples (TMC6-HC,  $\pm 0.5$  °C. accuracy,  $\pm 0.41$  °C precision, 3 min response time in air, 15 sec response time in stirred water.
- The probes are inserted ~6 cm into the center of the bottom of three to four sections per core.

- Temperature measurements are started after sections are brought into the core lab for thermal equilibration. Thermocouples are checked for accuracy in water-ice baths prior the beginning of coring.
- Full thermal equilibration of cores typically take about 2-4 hours and temperature probes are left in the cores until the temperature is  $>\sim 18.5$  °C.
- Direct contact temperature probes are typically inserted 8 cm into the bottom of cores. These probes are left in place as long as possible, usually about ten minutes, while the core is being sampled and cut into sections. The typical temperature probe arrangement has three probes in a straight horizontal line from the perimeter to the center of the core and a fourth probe at the top of the core.

## 4.4 Physical Property Testing

As noted earlier, the MSCL will likely be performed post-field, based on discussions with GeoTech and a more complete cost-benefit analysis. At this time, the coring manual has included this procedure in both the on-site section (Volume 2), and the post-field section (Volume 3). If it is determined that MSCL machine will not be mobilized to the wellsite, this section will be deleted from Volume 2.

### 4.4.1 Preparation of Core Samples

The basic procedure to prepare cores for measurement of physical properties, are as follows:

- The core sections are moved into the warm trailer laboratory to equilibrate to room temperature which typically requires 2-4 hours according to core-end-temperature probes.
- Gamma density, *P*-wave velocity, non-contact electrical resistivity, and magnetic susceptibility are measured using a Multi-sensor Core Logger-Standard (MSCL-S).
- After MSCL logging and thermal conductivity measurements are completed on whole-round core, the core sections were split in half.
- Several other stand-alone measurements are conducted including:
  - Electrical resistivity using a four-pin Wenner array,
  - *P*-wave velocity, and
  - Shear strengths by mini-vane, Torvane, and Pocket Penetrometer.

Moisture content and grain density are determined on subsamples after drying. Bulk density, porosity, and unit weights are determined from phase relations described in Winters et al. (in press).

### 4.4.2 Multi-Sensor Core Logger (MSCL)

The Multi-Sensor Core Logger – Standard (MSCL-S) has four physical property sensors mounted on an automated track as shown in Figure 7 and Figure 8 below, that sequentially measures the following properties:

- Bulk density using gamma ray attenuation (GRA)
- Compressional *P*-wave velocity (*V<sub>P</sub>*)
- Non-contact electrical resistivity (NCR)

- Magnetic susceptibility (MS)

Figure F7: Multisensor Core Logger (MSCL) with calibration standards on the track and measuring sensors on the left.

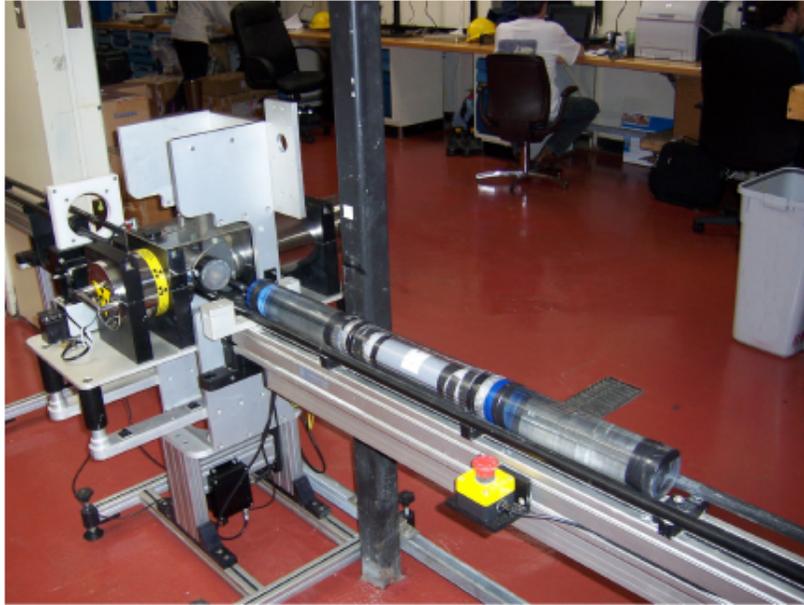


Figure 7 – MSCL-S System mounted on Track

Figure F8: Instruments on the Multisensor Core Logger (MSCL) used during NGHP Expedition 01, include (right to left) gamma ray densitometer (right), *P*-wave velocimeter, non-contact electrical resistivity device (below the core end cap), and magnetic susceptibility loop.

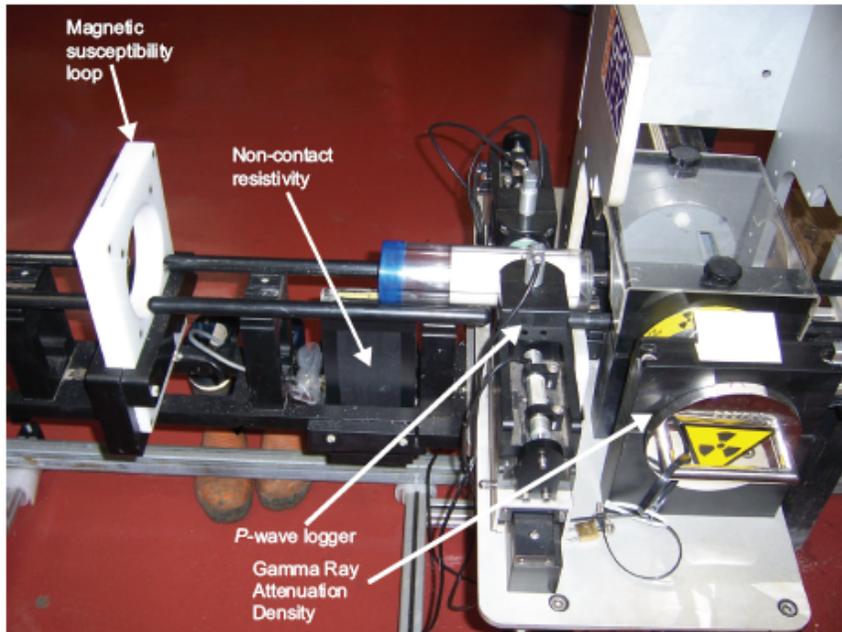


Figure 8 – MSCL-S System mounted on Track

MSCL measurements are nondestructive to sediment fabric and are used to measure and compare sediment properties along the length of the core. Data quality is a function of both core quality and sensor precision. Optimal MSCL measurements require a completely filled core liner with minimal drilling disturbance. Precision is a function of measurement time for MS, non-contact electrical resistivity, and GRA density but not for  $V_p$ . Technical notes on the Multi Sensor Core Logger and sensors are available from Geotek, Ltd., (2007).

Core sections logged on the MSCL are measured according to the following specifications:

- At an interval of 2 cm for gamma ray attenuation (GRA) density and  $P$ -wave velocity.
- A spatial resolution of 4 cm is used for the non-contact resistivity and magnetic susceptibility.
- For each measurement, magnetic susceptibility and NCR data are collected for 10 seconds while GRA density data is collected for 6 seconds.

Calibrations for each sensor are completed prior to logging each core section. Three standards: deionized (DI) water, Bartington Magnetic Susceptibility check piece, and water with a salinity of 17.5 ppt are run at the beginning of each core to confirm measurement accuracy and check for sensor drift.

#### **4.4.3 Gamma Ray Attenuation Density**

Gamma density is measured as follows:

- Measurements are made through the center of the core using a 10mCi  $^{137}\text{Cs}$  source and a NaI scintillation detector.
- The detection energy window is set to measure only primary (unscattered) gamma photons (0.662 MeV), providing raw gamma attenuation data in counts per second. Gamma density is derived from gamma attenuation and is reported as  $\text{g}/\text{cm}^3$ .
- The gamma beam is collimated through a 5-mm hole providing a downcore spatial resolution of about 1 cm. The precision is a direct function of total counts and hence is dependent upon the count time used (1 s) and the core thickness and density.

Although the empirical calibration procedure for GRA is based on bulk density measurements (i.e., of a known graduated aluminum and water standard), the measurements will vary from true gravimetric bulk density because of variations in mineralogy. Gamma attenuation coefficients for different materials vary as a function of atomic number. Fortunately, most earth-forming minerals have similar and low atomic numbers (similar to aluminum). Consequently, the correlation of GRA density and bulk density is usually very good.

GRA data are of highest quality when measured on non-gassy whole cores because the liner is typically filled with sediment. GRA measurements may exhibit some variability because of disturbance caused by rotary coring which tends to form alternating hard “biscuits” and softer groundup “infilling” material.

#### **4.4.4 Compressional P-wave Velocity**

Compressional-wave velocity ( $V_p$ ) measurements are gathered as follows:

- $V_P$  is measured on the MSCL track with the  $P$ -wave logger (PWL).
- The PWL transmits a 500-kHz  $P$ -wave pulse through the core at a specified repetition rate.
- Ultrasonic velocity is measured using a pair of Geotek Acoustic Rolling Contact Transducers.
- The travel time for wave pulse propagation through the core diameter is measured with a precision of 50 ns.
- At the same time, the core diameter is measured using a set of displacement transducers (precision 0.02 mm) that are mechanically coupled to the acoustic transducers.
- This produces an ultrasonic velocity with a precision of  $\pm 1.5$  m/s and a likely accuracy of  $\sim 5$  m/s.
- Core temperatures are obtained with a platinum resistance temperature probe (precision 0.05 °C) and a measured velocity is corrected to a velocity at a reference temperature (20 °C).
- Ultrasonic velocity is reported in m/s and has a typical downcore resolution of about 2 cm.

#### 4.4.5 Non-Contact Resistivity (NCR)

Electrical resistivity is measured as follows:

- The instrument used is a Geotek, Ltd. NCR sensor, containing inductive coil arrays which make resistivity measurements through whole plastic core liner.
- The NCR 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 that are inversely proportional to the resistivity.
- A receiver coil measures very small magnetic fields that are regenerated by the electrical current.
- To measure these very small magnetic fields accurately, a difference technique has been developed that compares the readings generated from the measuring coils to the readings from an identical set of coils operating in air.
- As with other parameters, the measurements are sensitive to core temperature and should be obtained in a stable temperature environment for best results. Electrical resistivity is reported in ohm-m with an accuracy of about  $\pm 5\%$  and integrates over a core length of  $\sim 3$ - 4 cm.

#### 4.4.6 Magnetic Susceptibility

Magnetic susceptibility is measured as follows:

- Using either a 13.0 cm or 8.0 cm diameter Bartington loop sensor.
- The frequency of the low-intensity, alternating- magnetic field produced by the sensor is sensitive to changes in the magnetic susceptibility of material within about 6.0 cm (for the 13.0 cm diameter loop) or 4 cm (for the 8.0 cm loop) on either side of the loop.
- Data is masked out to account for this effect at section ends and at locations of significant voids determined by both visual inspection of the cores as well as by anomalously low gamma density values ( $<1.3$  g/cc).

- Magnetic susceptibility, a dimensionless number, is reported as corrected volume susceptibility in SI units with an accuracy typically  $\sim \pm 4\%$ .

## **5 BULK CORE PRESERVATION BY FREEZING**

This section discusses the guidelines and procedures for preserving the core by freezing.

This section is ready to accept more detailed procedures, once Omni/Weatherford has been brought on board.

### **5.1 Objectives**

- To ensure that the remaining (non-subsampled) core is preserved by freezing to provide a flexible and unbiased sample of all rocktypes cored for subsequent, off-site conventional and special core analysis requirements.
- To ensure that all preserved core is sealed, supported and later packaged so that it arrives at the laboratory without drying, contamination or structural damage and in good condition for special core studies.
- To ensure that all preserved core remains in suitable condition for analysis for a significant length of time (sufficient to support all anticipated appraisal requirements).

### **5.2 Core Archiving and Storage Guidelines**

- Gaps left by removing core samples will be filled with sample labeled foam fillers.
- Plastic wrap core and reposition core linearly into the previously cut 3-foot sections.
- Place end caps, seal with tape (end caps and split down the liner) and label.

### **5.3 Whole Core Preparation**

- Pipe-cutter and/or chop saws for cutting 3-foot intervals within inner core barrel liners
- Wrap with plastic wrap and/or heavy plastic bags
- Place end caps
- Seal with tape
- Place in card-board boxes
- Store (freeze cores)

### **5.4 Core Freezing/Storing Procedures**

- 3-Foot plastic wrapped core sections will be stored in 3-foot card-board box
- 2 Weatherford/Omni representatives will carry boxes to onsite refrigerated truck
- Boxes will be placed into 4X4 container with Styrofoam inserts for shock protection
- 80 to 100 3-foot boxes can fit into one 4X4 container.
- Liquid nitrogen vessels will be stored in this truck

### **5.5 Miscellaneous Notes**

- Gas monitoring and ventilation will be provided for in the truck.
- Access to truck will be controlled via secure entry (key or other). Arctic Gear and anti-slip wear must be removed at entry to truck; which may require a receiving crew inside truck.
- If Weatherford/Omni staff needed for second core, may require different group to handle the core storage if causing delay between cores.

Although the technique is straightforward to apply, it is particularly susceptible to poor quality control, both in procedure and execution. The initial handling and wrapping of soft or friable sections will require special care.

## **6 CORE TRANSPORTATION**

Transporting the core back to Anchorage and beyond is a logistical and quality control challenge. This section discusses the processes and guidelines that should be followed to insure that cores arrive at their final destination in good order. Actual detailed procedures will be developed at a later date, once all the shipping parties and details have been worked.

**This section needs more detail, once Omni/Weatherford has been brought on board.**

### **6.1 Objectives**

- To ensure that all core samples arrive at the Anchorage site storage by DOT-approved containers in the shortest and most cost-effective time without loss or damage.
- Ensure that full chain-of-custody is handled by COREHANCON Lab staff.
- To ensure that good communications exists at all times between handling agents.
- To ensure that all shipping participants are briefed on the need for careful core handling to prevent damage.

### **6.2 Safety Considerations**

- Heavy lifting procedures must be followed when moving core boxes and equipment.
- All core and equipment must be securely loaded into containers to prevent shifting during transit, which could damage the core and endanger handlers.
- It will be required to have continuous gas monitoring and ventilation during transportation. This will require a remote monitor that can be checked during transportation.

### **6.3 Operational Procedures**

The stabilization and freeze-preservation techniques used to protect the core will reduce sensitivity to mechanical shock, however it is essential that all parties involved with core handling be briefed as to the fragile nature of the core. The following are some general guidelines, though transportation details will be site specific.

#### **6.3.1 Onsite Storage Facility**

- Carlisle (or equivalent) refrigerated truck will provide onsite storage.
- The truck will be running at all times to maintain climate controlled environment. Note: truck will need to be fueled periodically).
- Alternative onsite storage options may include open-air storage until monitoring and ventilation requirements can be met.
- There will be clearly established chain-of-custody requirements for core transportation.

### 6.3.2 Transport from Rig to Anchorage Storage Facility

- Once coring is completed, this truck will be used to transport bulk core, and liquefied nitrogen vessels, head-space gas, and frozen samples to a refrigerated facility in ????. A logical storehouse is with the Mt. Elbert cores at the AES facility in Anchorage. **These details will be worked with DOE once Omni/Weatherford has been brought on board.** Shipping documentation is signed off onsite and will involve identification of hazardous materials.

### 6.3.3 Subsequent Core Transport for Special Studies

Customs requirements must be checked for core that will have to leave the country, if it is likely it will be required to open individual core pieces, then some means of re-sealing the core before drying can occur should be available.

- The flight details and airway bill number and a copy of the airway bill must be transmitted to recipients in advance of shipment.
- An inventory of the core should accompany all shipments. A separate copy of this inventory should be sent to recipients via Fax or email with an estimated time of arrival.
- Transport of oil and gas samples (including core samples containing hydrocarbons) by commercial airfreight must be carried out in accordance with special procedures. Only specially trained personnel may perform these duties and sign the official declarations. All core samples must therefore be packaged in accordance with the IATA "Dangerous Goods Regulations Manual".
- Direct flights should be used to minimize unsupervised core handling.
- With international airfreight transportation, the storage cabin may not be pressurized. The associated temperature and pressure changes can have an adverse effect on the integrity of the applied core preservation. It is therefore recommended that aircraft with pressurized holds be used for transporting preserved core samples. Commercial carriers, which are unaccustomed to transporting fragile materials, should not be used, or used with caution.
- Below freezing temperatures should be maintained at all times by shipping in freezer containers or dry-ice.

## 6.4 Operational Guidelines

This section reviews some of the basic guidelines for delivering the various core samples to Anchorage and beyond.

- The core-shift lead geologists, logistics team and core specialist should review this plan. The agreed transportation route should be secure, should minimize handling steps and maintain freezing temperature.
- All individual core boxes should be braced into 4X4 Omni/Weatherford shipping containers for transport to Anchorage. Each crate or box should be clearly labeled with the well number and sample number on the lid and ends of the boxes.

- All communications regarding the core shipment should be channeled through the core shift lead geologists (USGS, DOE).
- It should be well communicated to all parties that the core is fragile, and special handling procedures are necessary.
- One of the core shift lead geologists (USGS, DOE) should observe loading and offloading of core containers to ensure cores are handled with care and procedures are followed.
- An Operations Geologist or Omni/Weatherford representative must meet the core at Anchorage storage facility at the Anchorage Yard and witness offloading.

#### **6.4.1 Bulk Frozen Core Shipment to Anchorage.**

Because Barrow is not connected to the road system, all of the bulk frozen core will be transported in a refrigerated airline-approved cargo container (temperature -10 deg C) to the "frozen" core storage facility in Anchorage. It is assumed that these cores will be stored at the drill site in the same container that will be used for shipping to Anchorage. Omni/Weatherford will be responsible for this shipment.

#### **6.4.2 Frozen Sub-sample Shipments to Anchorage.**

Frozen subsamples will be shipped with the bulk frozen core to Anchorage under direction of DOE. These samples will subsequently be sent to Lower-48 labs in dry-ice shippers through Alaska Airlines as our climate controlled shipping company. **The details surrounding the handoff between Omni/Weatherford and Alaska Airlines still needs to be worked.** Dry ice temperatures (-78 C) with the P samples may require further thought - can we keep them at a higher temperature, but still ensure they stay frozen?

#### **6.4.3 Non-Frozen Sub-sample Shipments to Anchorage.**

Subsamples that cannot be frozen (i.e., pore waters samples, and some WRC samples etc.), must be shipped by air or ground under climate controlled conditions, keeping the samples cold but not frozen. Probably be best to ship directly from the Slope to the Lower-48. Need to identify the responsible parties and shipping process for this sample type. This would perhaps apply to fine-grained consol and strength samples but not coarse-grained samples.

#### **6.4.4 Liquid Nitrogen Shipments to Anchorage.**

Liquid nitrogen shippers will be shipped with the bulk frozen core to Anchorage (Weatherford/Omni control). These samples will be sent to Lower-48 labs in the LN2 shippers via Alaska Airlines as our climate controlled shipping company. Need to work with Weatherford/Omni to understand the Alaska Airlines hand off procedure.

## **7 CORE STORAGE**

This section discusses where and when core will be stored for various post-well analyses and final disposition.

This section is a blank sheet of paper. The core team/Science Party will determine where core and core samples will be sent or stored. These details will be worked once all contractors have been selected and brought on board.

*Note: As of the date of this version of this document (Rev 1.0, September 21, 2009), PRA, as the well planning and prime technical contractor for the North Slope Borough and DOE, has reviewed previous work and assembled a first draft of a set of integrated procedures for off-site core analysis. This volume is designed to accompany volumes 1 and 2 as described below.*

*PRA's role is to insure that well-site acquisition, handling, or analysis procedures do not negatively impact the off-site analysis. PRA is proposing this first draft of the laboratory procedures planned by the Science Party, but should not be considered the owner or final editor of this document. PRA will continue its efforts to make sure the Science Party receives exactly what is specified. PRA wishes to remain in the loop, in case a change in the off-site analysis necessitates an adjustment at the wellsite. However, PRA does not expect to have a significant role after the point in time that the core is delivered to its final testing location in the condition that it was specified.*

## **Core Procedures Manual**

### **Volume 3 – Off-site Core Analysis and Science Party Tasks**

**Draft, Rev1.0 – September 21, 2009**

## **INTRODUCTION, BACKGROUND**

This “Core Procedures Manual, Volume 3” contains a detailed description of proposed methods and best practices of the off-site, post-field core analysis portion of the Barrow Gas Hydrate Test Well Program. This document is designed to outline the responsibility of the “Science Party” – those individuals or institutions that will use the core and/or data for subsequent research and analysis.

This well is being drilled as a gas hydrate test well within Phase 2B of the North Slope Borough (NSB) - US Department of Energy (DOE) Gas Hydrate Cooperative Research Project. This volume of the manual includes several written elements of the coring plan, including the following:

- Personnel roster, roles, and responsibilities
- Coring laboratory equipment and supplies checklist
- Recommended Practices and Methods for:
  - On-site sub-sampling
  - On-site core analysis
- Station checklists and recording logs for on-site sub-sampling.

A total of four documents have been written to cover the complete scope of coring, core preparation, on-site core analysis, and subsequent decision criteria to complete the well. These documents (with volumes 1, 2, and 4 under separate cover) are as follows:

- *Volume 1 – Core Acquisition*

- Discusses pre-well planning and coring operations up through core laydown
- ***Volume 2 – Core Handling, On-site Analysis***  
Discusses core handling, sub-sampling, preservation, and transportation
- ***Volume 3 – Post-Field Core Analysis***  
Discusses off-site core analysis tests and methods conducted by the Science Party
- ***Volume 4 - Decision Criteria to Complete Well***  
Discusses the test results that will trigger a decision to complete or abandon the well

Lessons learned from previous hydrate-bearing cored wells, including the Mallik (1998 and 2002), Hot Ice (2004), and Mt. Elbert (2007) arctic onshore wells, and certain offshore research programs are incorporated into these documents where applicable.

The program has been designed to deliver the key core objectives identified by the Gas Hydrate project research team and the Barrow Field development team including members of DOE, USGS, NSB, PRA, and supporting service companies. This document will be reviewed and refined through a number of meetings leading up to well spud, including a coring budget workshop being planned for September 2009. This manual will then be used as an on-site policy and procedures manual to guide all aspects of the coring program. It is also intended to serve as the all-encompassing master reference document for all interested stakeholders.

## **Document Overview**

This document is meant to describe the instruments and methods used to analyze core sub-samples offsite, post field.

The off-site analysis can be categorized into five categories as follows:

1. Lithostratigraphy
2. Physical Properties
3. Inorganic Chemistry
4. Organic Chemistry
5. Microbiology
6. Petrophysics

The detailed procedures described in this document are meant to represent a “super-set” of tests and measurements that may be run. In some cases, only a sub-set of these tests will be performed.

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# 1 PROJECT OVERVIEW

The DOE and NSB have teamed up to confirm the presence of hydrates at one of the Barrow gas fields. The DOE, other government and agencies including the USGS, and academic institutions will progress its understanding of hydrates while the NSB will gather critical information about the reservoir depletion mechanism of gas fields that provide heat and electricity to the community of Barrow. One of the primary objectives of this program is to obtain a minimally disturbed 3 inch whole core from gas hydrate-bearing sandstone beneath the permafrost, from a field that is currently producing gas at continuous and commercial rates. It would be the first well to confirm the presence of hydrates in a field undergoing depressurization and depletion. The combination of core data, log data, and long term production data represents a huge step forward in confirming the potential of hydrates as a commercial source of energy.

At least one stratigraphic test well will be cored in the East Barrow gas field east of Barrow Alaska. If hydrates are confirmed at the first location, the well will be completed as an observation well and a horizontal well will be drilled nearby to produce free gas and dissociate the hydrate zone. If no hydrates are found at the East Barrow Gas Field, a second stratigraphic test well will be drilled and cored in the Walakpa gas field, south of Barrow. This manual is intended to describe all aspects of the coring analysis at East Barrow, and if a second well is cored at Walakpa.

## 1.1 On-Site Core Team Members, Roles, and Responsibilities

This section is included in all three volumes to ensure all parties understand their roles and responsibilities.

The “core supervisors” listed below will be responsible for core lengths, core tops, meeting coring objectives, etc. and will provide 24 hour coverage with at least one person available at all times.

- 1 PRA/??? Operations Geologist, oversight
- 1 DOE Geologist, oversight
- 1 USGS Geologist, oversight

The contractor-based team members listed below will work 12 hours shifts with full-time coverage of the operation.

- 4 total, 2 per shift, NOV Corion lead core engineer and wireline engineer
- 4 total, 2/shift Weatherford/Omni core marking, breaking, gamma, photo, and preservation staff

The on-site Science Party will consist of the following: (Note: the table below summarizes these responsibilities). *Note: The tentative roster of on-site core supervisory and Science Party team*

*listed below will be revised and updated as coring operations near and individual's schedules are determined.*

- 8 total, 4/shift USGS, USDOE, and Oregon State University core sub-sampling staff (pore waters, geochemistry, microbiology, gas hydrate-specific core properties analyses.)
- 2 total, 1/shift Organic Geochemistry and Microbiology sub-sampling staff
- 2 total, 1/shift Physical Properties sub-sampling staff
- 4 total, 2/shift Inorganic Geochemistry (water sampling)

<u>NAME</u>	<u>AFFILIATION</u>	<u>RESPONSIBILITIES</u>
TBD Ops geologist	PRA/???	Core point, TD, wellsite core shift supervisor
Tim Collett	USGS	Core point, TD, wellsite core shift supervisor
Ray Boswell	US DOE	Core Handling and swing shift supervisor
Kelly Rose	US DOE	Core Handling, core water chemistry
TBD	US DOE/TBD	Core Handling, water chemistry, thermal conductivity
Rick Colwell	Oregon State Univ	Core Sampling, geochemistry, microbiology
Marta Torres	Oregon State Univ	Core Sampling, water analyses lead
TBD	USGS	Core Sampling, physical properties
TBD	USGS	Core Sampling, geochemistry, microbiology
TBD	USGS	Core Sampling, physical properties
TBD	TBD	Core Sampling, water chemistry
TBD	TBD	Core Sampling, inorganic chemistry

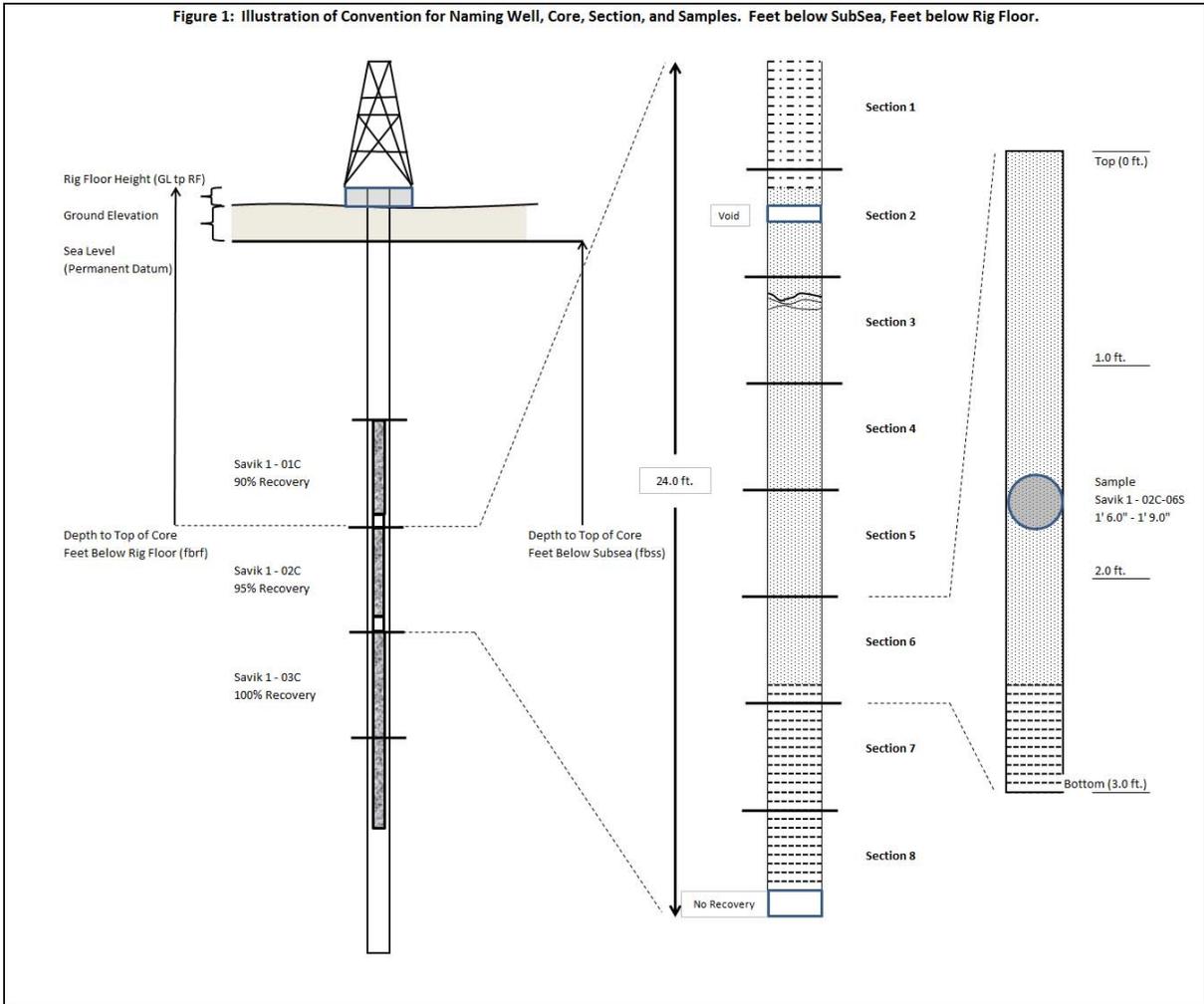
- The Drilling Supervisor (“Company Man”) has ultimate responsibility and final say over all coring and drilling operations at the wellsite. Described below are the principal roles and responsibilities of the “core team” members, all of whom advise and report to the Drilling Supervisor while at the wellsite.
- The PRA Operations Geologist is responsible for facilitating and coordinating all communication between the scientific staff (“Science Party”) and the Drilling Supervisor.
- The HSE Officer will be included and consulted on all matters of an operational nature that have safety or spill risk elements.
- The Core shift supervisors/leads serve in an oversight role and are responsible for coordination of core data, technical guidance, team coordination, and assurance of coring objectives.
- NOV Corion technicians are responsible for supplying all equipment, manpower, and expertise for core acquisition through core lay-down in the pipe-shed.
- Weatherford/Omni staff will be responsible for inner core barrel marking, whole core measurement, marking, preservation, wellsite processing, assistance with USGS onsite sub-sampling, bulk core stabilization by freezing, onsite core storage, core transportation to Anchorage, and post-program routine and specialized core analyses if required.
- The Science Party, with support from Weatherford/Omni staff, will be responsible for on-site sub-sampling, on-site analysis including logging, lithostratigraphic descriptions, organic and inorganic chemistry, and physical property measurements.
- Weatherford/Omni staff will be responsible for shipping other frozen samples, mud samples, State of Alaska chip samples, and liquid nitrogen stored samples as required.
- The Science Party will be responsible for return shipment of laboratory equipment and certain specialized core subsamples.

- Canned cuttings and samples will be shipped by USGS to USGS at Menlo Park.

## **1.2 Core Curation and Sample Depth Calculation**

As is the standard convention, drilling depth is measured in terms of feet below rig floor (fbrf) as determined from a strap of the drillstring and shared amongst all wellsite parties including mud loggers, directional drillers, loggers, and the Science Party. The core top in feet below rig floor (fbrf) measurement is the ultimate depth reference for all other depth calculations.

In addition to relative position within a core section, specific intervals and horizons are also described in “curatorial” feet below the rig floor (fbrf). The fbrf of a sample or horizon is calculated by adding the interval depth of the sample and the lengths of all upper sections to the core top datum as measured with the drill string. Often, cores expand upon recovery, leading to more than 100% recovery (for example, 9.8 feet of core recovery whereas bit advance may have been only 9.6 feet). Likewise, because the core top is marked at the highest recovered sediment within a core, there is often an unquantifiable coring gap between cores. As a result, discrepancies may exist between the drilling depth and curatorial depth, including instances where the curatorial depth at the bottom of one core may be greater than that for the top of the subsequent (deeper) core.



**Figure 1 – Conventions for noting holes, cores, section, and samples, with depths**

In cores with less than complete recovery, the uppermost recovered sediment is assumed to mark the top of the core. Furthermore, all other recovered sediment within the core is assumed to mark a continuous section downward. In other words, when sediment is missing from a core, that missing section is curated as occurring at the base of the core. This convention results in a necessary sampling uncertainty that should be taken into consideration when working with depth data (e.g.) core to log correlation.

### 1.3 Drilling-Induced Core Deformation

Many cores collected show signs of disturbance. Bedding deformation, particularly along the outer edge, is a common effect of the coring process, as are sediment mixing (particularly at the tops of cores), fluidization (“biscuit and slurry”) and liquid injection. Core deformation such as creation of partings also occurs commonly as a result of the depressurization associated with core retrieval. Dissociation of gas hydrate and gas expansion also result from core depressurization (and warming), and can further disturb the sediment. The “Lithostratigraphy” section of this

document provides a further discussion on the nature and interpretation of core characteristics, including those that may serve as indicators of the previous presence of gas hydrate.

## 1.4 Core Handling and Analyses Overview

Cores acquired during the Barrow Hydrate Test well program are intended to be handled according to ODP/IODP procedures. Modifications to these procedures, similar to those made in Mt. Elbert and Mallik 5L-38 projects, are designed to enable quick identification of gas hydrate intervals and maintain aseptic conditions for microbiological sampling.

Inspection of the core is to begin immediately upon recovery. Selection of sub-samples will be based on:

- 1) Visual observations
- 2) Water in watch glass
- 3) Core temperatures

Sites will be based on evidence of recent or ongoing gas hydrate dissociation within the enclosed sediments. Samples are to be immediately cut from the core and either

- Sent to the geochemistry lab for visual inspection and extraction of interstitial waters for geochemical analyses, as described in detail in the Inorganic Geochemistry Section.
- Sampled and stored for post-well microbiological studies, or
- Gas hydrate samples will be taken and stored in liquid nitrogen.
- A background sample (non-anomalous) will be taken from each core for immediate geochemical analyses.
- Gas samples will be sampled and sealed in pint-sized cans for headspace gas analysis.

In this particular project, the well will be cored using drilling fluids containing microbead tracers (see “Microbiology” section) to aid in the identification of uncontaminated sections.

- The remainder of the core will then be measured for sectioning, and labeled with a permanent marker with up orientation, core number, core type, and section number.
- The remaining core is then compressed with a plunger to remove all voids, when possible, and cut into sections 3 feet or less in length. The most appropriate section or sections were then selected for sampling (typically the most coherent, least disturbed, section that appears to be representative of the background condition of the core).
- The suite of pre-planned samples are then cut, capped, labeled, and stored as appropriate for physical property and petrophysical properties.

Once in the warm trailer laboratory, the remaining whole round core sections are first tested for thermal conductivity. Then, if the MSCL machine was mobilized to the wellsite, the core is passed through the multi-sensor core logger (MSCL) to obtain measurements of *P*-wave velocity, non-contact resistivity, gamma ray attenuation bulk density, and magnetic susceptibility as described in the “Physical Properties” section.

From this point on, all analysis will be done off-site, in a less expensive, more controlled environment. A brief summary of the handling process for offsite work is given below:

The cores are then split, from bottom to top, creating separate archive and working halves. Investigators should keep in mind that splitting, using either a pulled wire or a fixed circular saw blade, may further disturb the core and result in the transport of material upward along the surface of each core half.

The working half is then plugged and sub-sampled for further physical properties testing (moisture and density, shear strength, split-core acoustic velocity, and contact resistivity according to the “Physical properties” Section. Next, the working half of the core is further sub-sampled. Sub-sampling includes the regular collection of background material for later sedimentological, x-ray diffraction, carbon dating, and paleomagnetism studies, as well as targeted sampling of notable features such as clay, mineral, or carbonate nodules, macrofossils, and other features.

The archive half sections are to be scanned on the digital imaging system (DIS). Visual core descriptions (VCDs) of the archive halves are prepared, augmented by microscopic analyses of smear slides. Digital close-up photographs are taken of particular features for illustrations, as described in the “Lithostratigraphy” Section. Both halves of the core are then placed in labeled plastic tubes and transferred to cold storage for transportation to final location.

At the end of the project, selected cores and samples are shipped to selected post-well laboratories in the United States, or to the USGS facility in Woods Hole, Massachusetts, for further analysis.

## **2 LITHOSTRATIGRAPHY (POST-FIELD)**

The techniques and procedures used to identify, describe, and analyze the lithostratigraphy (lithology and stratigraphy) in cores recovered for the Barrow Hydrate Test Well are described below. They are based on the methodology employed in ODP Leg 204 and IODP Expedition 311 with additions from ODP Legs 172 and 202 and adapted to the specific conditions and equipment available during NGHP Expedition 01. The techniques and procedures described here include visual core descriptions, smear slide and coarse fraction descriptions, and high-resolution digital color core imaging.

### **2.1 Required Equipment, Instruments, and Supplies**

- Microscope – standard. At least x – x power, dual eyepiece
- Microscope – transmitted light polarizing petrographic type
- Microscopic scale - micrometers
- Hand Scale - millimeters
- Udden-Wentworth grain-size scale
- Terry and Chilingarian percentage comparison chart
- Visual Core Description (VCD) sheets
- Gretag- Macbeth ColorEye XTH hand-held color spectrophotometry instrument with spectral range between 360 and 750 nm, and a resolution of 10 nm (or visual comparison with the Munsell soil color chart - Munsell Color Co., 1975)
- Advanced Logic Technology © WellCAD Version 4.0 software package
- 1 in. X 3 in. glass slides – count of 100
- 2.2 cm. X 3.0 cm. glass covers – count of 100
- Norland optical adhesive #61
- Sieves - 63  $\mu$  mesh, ...
- Geotek X-Y digital imaging system (DIS)

### **2.2 Sediment Classification**

The naming conventions follow the ODP sediment-classification scheme of Mazzullo et al. (1988), modified for terrestrial sediment. Principal names were assigned to sediments based on composition, texture, and degree of lithification as determined primarily from visual description and smear slide analyses. Modifiers to the principal name are determined based on both the abundance and type of the non-principal component or components (e.g., siliciclastic or biogenic). Major modifiers are listed in order of increasing abundance and tagged with “bearing” or “rich” (see below).

#### **2.2.1 Siliciclastic Sediments**

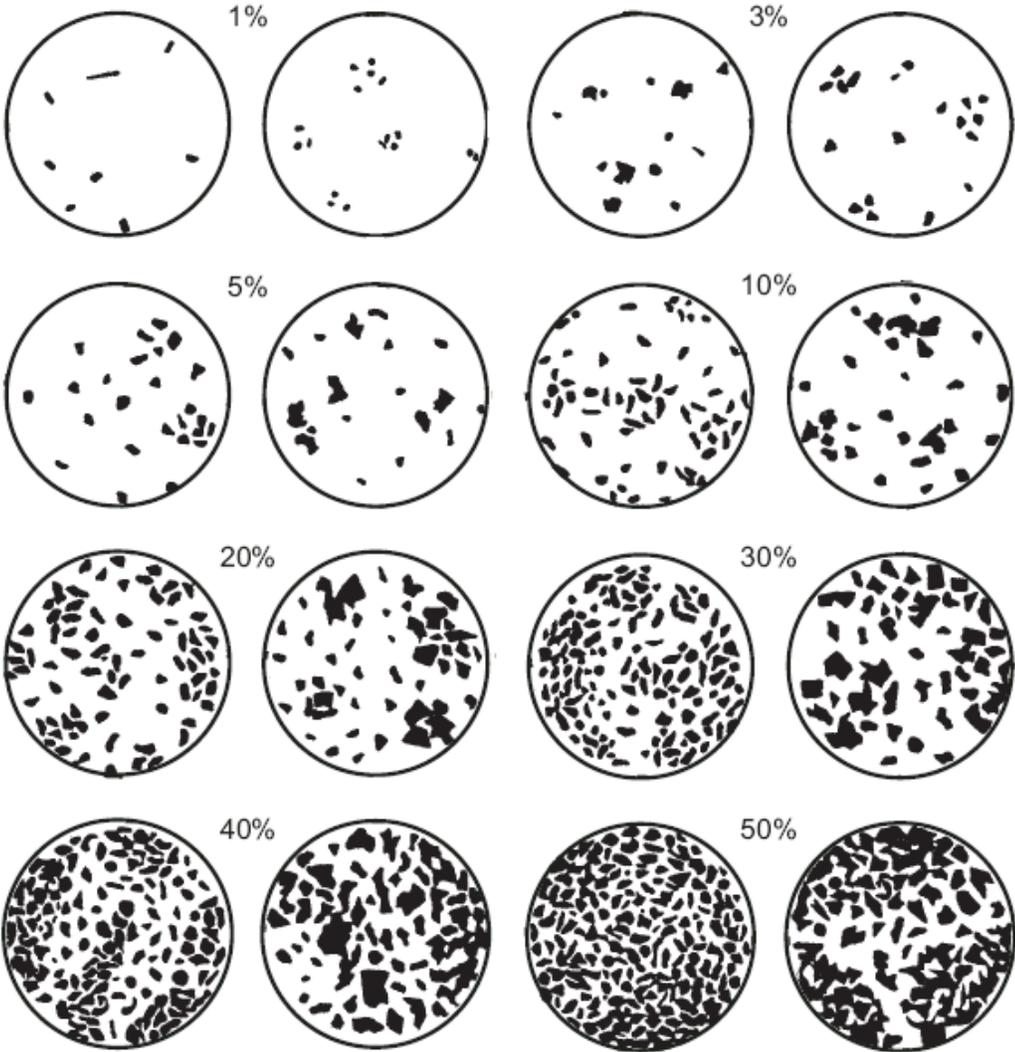
For sediments and rocks composed of >60% siliciclastic components, the principal name is determined by the size of the grains (sand, silt, and clay). Textural names are derived from the Udden-Wentworth grain-size scale (Wentworth, 1922), as shown in Figure 2 below.

**Figure F2:** Grain size divisions for sedimentary rocks (adapted from Wentworth, 1922).

Millimeters (mm)	Micrometers ( $\mu\text{m}$ )	Phi ( $\phi$ )	Wentworth size class	Rock type
4096		-12.0	Boulder	Conglomerate/ Breccia
256		-8.0	Cobble	
64		-6.0	Pebble	
4		-2.0	Granule	
2.00		-1.0		
1.00		0.0	Very coarse sand	Sandstone
1/2	500	1.0	Coarse sand	
1/4	250	2.0	Medium sand	
1/8	125	3.0	Fine sand	
1/16	63	4.0	Very fine sand	
1/32	31	5.0	Coarse silt	Siltstone
1/64	15.6	6.0	Medium silt	
1/128	7.8	7.0	Fine silt	
1/256	3.9	8.0	Very fine silt	
0.00006	0.06	14.0	Clay	Claystone

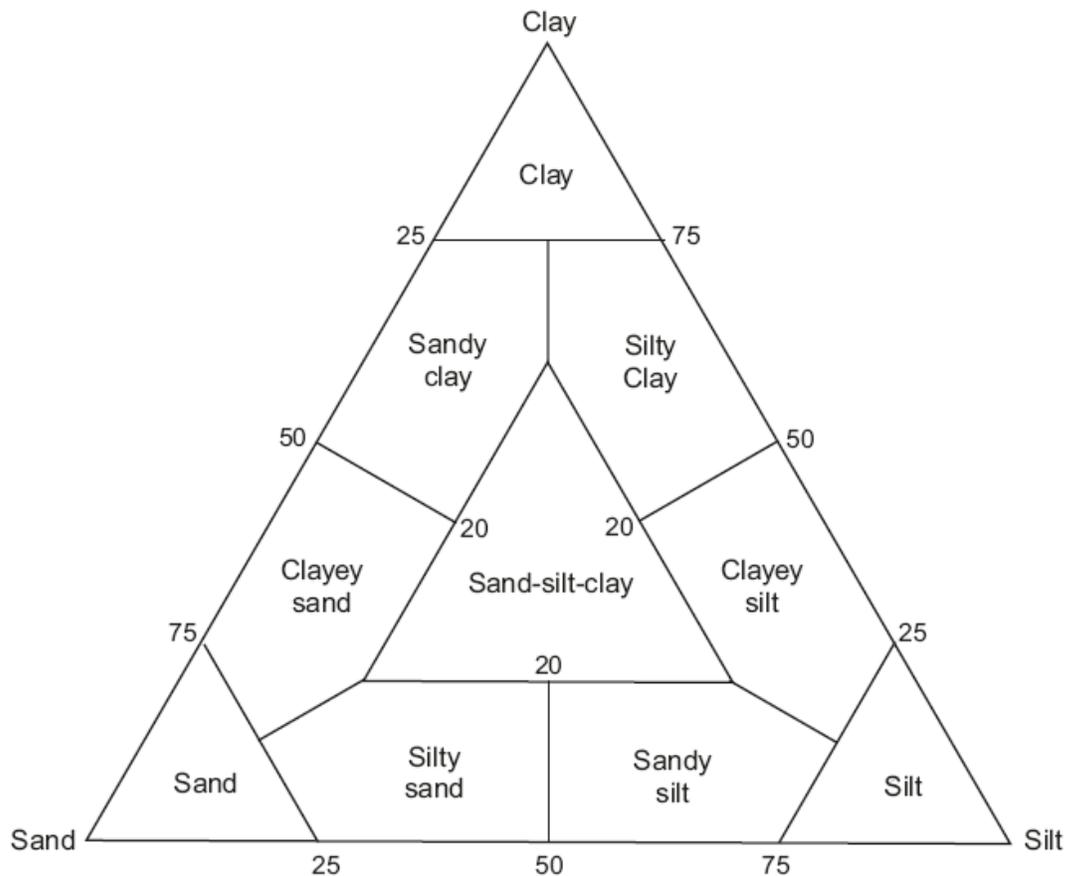
In this classification scheme, the term clay is independent of mineralogy and refers to all siliciclastic grains <3.9  $\mu\text{m}$  in size, regardless of composition. The relative proportion of different grain sizes is determined by visual percentage estimation using the comparison chart of Terry and Chilingarian (1955), as shown in Figure 3 below.

Figure F3: Comparison chart for volume percentage estimation (after Terry and Chilingar, 1955).



Once the relative proportions are determined, a modified Shepard (1954) classification scheme is used to assign the principal name, as shown in Figure 4 below.

Figure F4: Ternary diagram for siliciclastic textural classification. Numbers indicate percentages.



Clay, silt, and sand are the principal names in the Shepard diagram. If any component exceeds 25% of the total siliciclastic grains, it becomes a modifier to the principal name. For example, sediment composed of 10% clay and 90% silt is simply a silt, whereas sediment composed of 30% clay and 70% silt is a clayey silt.

Where diagnostic minerals (e.g., glauconite) or unusual components (e.g., volcanic glass) compose >5% of the sediment, the naming conventions of biogenic and mixed sediments are adopted. Thus, if the mineral component represents 5%–10% of the sediment, it is hyphenated with the suffix “-bearing” and precedes the major siliciclastic component name. If the component is 11%–40% of the sediment, it is hyphenated with the suffix “-rich,” instead. For example, sediment composed of 15% glauconite sand grains, 30% silt, and 55% clay is called a glauconite-rich silty clay. Where volcanic glass composed >40% of the sedimentary components, the name volcanic ash is used.

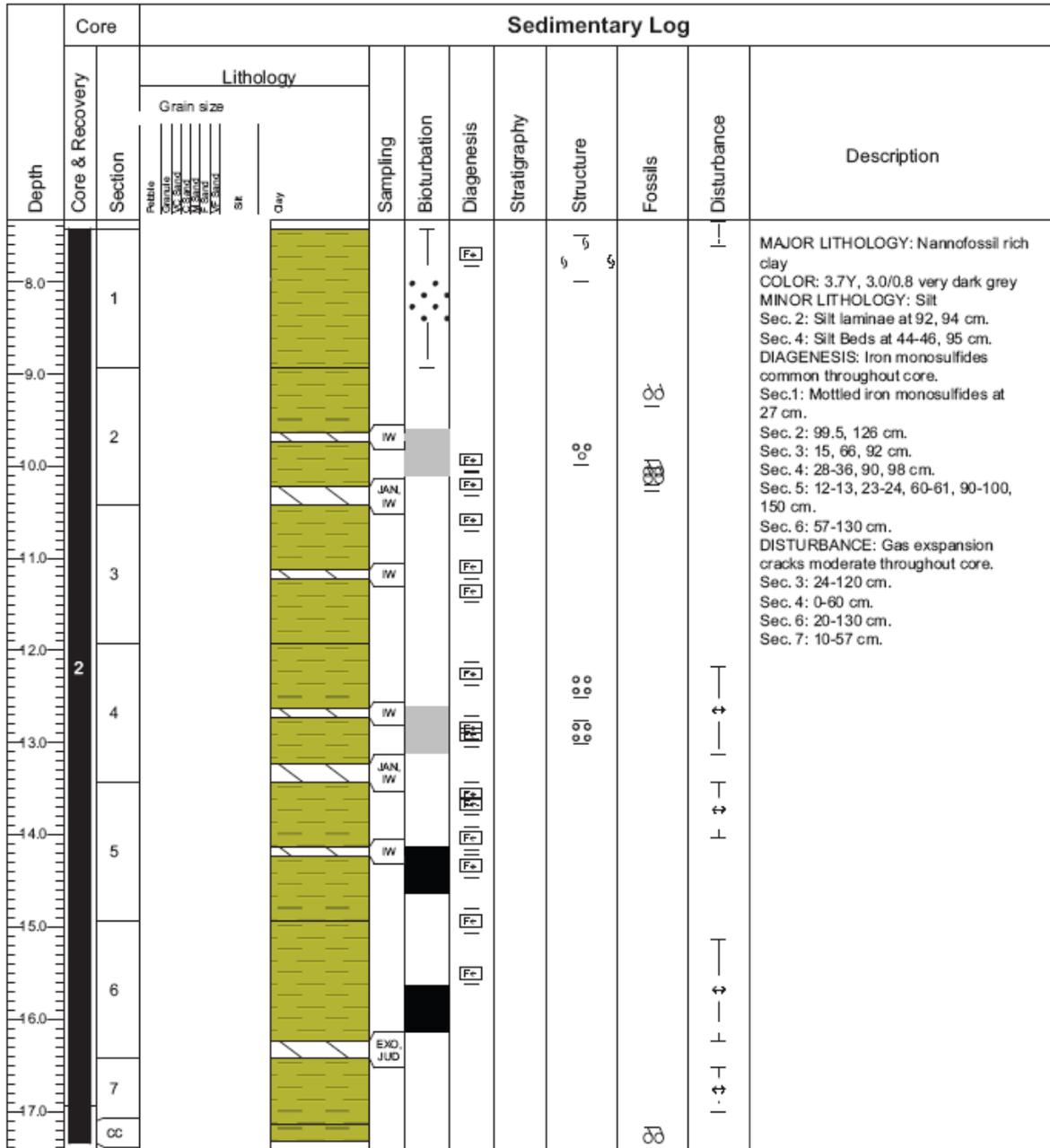
### 2.3 Visual Core Descriptions

Detailed sedimentologic observations and descriptions will be recorded manually for each core section on the visual core description (VCD) sheets. A wide variety of features that characterize

the sediments are recorded, including lithology, sedimentary structures, color, diagenetic precipitates, and core disturbance.

Compositional data are obtained from smear slides. The Munsell color designation (hue, value and chroma) of the sediments is determined by either color spectrophotometry (using a Gretag-Macbeth ColorEye XTH hand held instrument) or by visual comparison with the Munsell soil color chart (Munsell Color Co., 1975). This information is synthesized for each core in the Advanced Logic Technology © WellCAD Version 4.0 software package, which generates a one-page graphical description of each core, as shown in Figure 5 below.

Figure F5: WellCAD core description log example.



Of particular interest during hydrate test well projects are the visual indications of disruption to the sediment caused by the dissociation of gas hydrate in the recovered cores.

In consolidated formations, the presence of hydrates or the effect of hydrate dissociation are subtle to non-existent. Scientists will need to rely on visual observations (bubbles), dissociation in water glasses, and core temperature changes.

Remarks should be made on the digital core description sheet for each core showing potential indications of gas hydrate near the sampled intervals, including the presence of dry, flaky sediment that may have been dewatered by the formation of gas hydrate nearby.

### **2.3.1 Lithology and Grain Size**

The lithology and grain size of the described sediments are represented graphically in the WellCad generated descriptions. Intervals that are a few centimeters or greater in thickness can be portrayed accurately in the lithology column. Percentages are rounded to the nearest 10%, and lithologies that constitute <10% of the core are generally not shown but are listed in the “Description” column.

### **2.3.2 Bioturbation**

Visible bioturbation is classified into four intensity levels based on the degree of disturbance of the physical sedimentary structures:

1. Rare = isolated trace fossils; up to 10% of physical sedimentary structures are disrupted.
2. Moderate = ~10%–40% disrupted physical sedimentary structures; burrows are generally isolated but may overlap locally.
3. Common = ~40%–60% disrupted by burrows, sedimentary structures are disrupted
4. Abundant = >60% bedding completely disturbed; burrows are still intact in places.

These categories are based on the ichnofossil indices of Droser and Bottjer (1986) and are illustrated with graphic symbols in the “Bioturbation” column on the WellCAD core description sheets. Visual recognition of bioturbation is often limited in homogeneous sediments, particularly in hemipelagic clay zones without iron sulfide precipitates.

### **2.3.3 Sedimentary Structures**

Each type of sedimentary structure and its exact location are displayed in the “Structure” column on the WellCAD core description sheets. Symbols are used to note the wide variety of sedimentary structures that may be encountered, and these are listed in the legends for each WellCAD core description sheets included within each site chapter.

### **2.3.4 Fossils**

The presence of macroscopic fossils (e.g. shell fragments, preserved whole shells, bivalves, gastropods etc.) is displayed in a separate column on the barrel sheets.

### **2.3.5 Sediment Disturbance**

Drilling-related sediment disturbance that persists over intervals of ~10 cm or more is recorded in the “Disturbance” column. Separate terms are used to describe the degree of drilling disturbance in soft and firm sediments, as follows:

1. Slightly disturbed = bedding contacts are slightly deformed.
2. Moderately disturbed = bedding contacts have undergone extreme bowing.

3. Very disturbed = bedding is completely deformed as flow-in, coring/drilling slough, and other soft sediment stretching and/ or compressional shearing structures attributed to coring/drilling (e.g., gas expansion).

Soupy intervals of unconsolidated sediments are liquid saturated and have lost all primary sedimentary structures. When the soupy texture is related to gas hydrate dissociation, it is noted in the description on the WellCAD core sheet.

The degree of fracturing in indurated or semi-lithified to lithified sediments is described using the following categories

1. Slightly fractured = core pieces in place and broken.
2. Moderately fractured = core pieces are in place or partly displaced, but original orientation is preserved or recognizable.
3. Highly fractured = core pieces are probably in correct stratigraphic sequence (although they may not represent the entire sequence), but original orientations are lost.
4. Drilling breccia = core pieces (small and angular pieces) have lost their original orientation and stratigraphic position and may be mixed with drilling slurry.
5. Drilling biscuits and drilling slurry surrounding an intact or slightly fractured drilling biscuit.

Cores recovered from gas and gas hydrate-bearing sediments are often disturbed by gas expansion that causes fracturing. In cases where it is possible to distinguish between disturbance of the core resulting from drilling and disturbance resulting from gas expansion, notes are made in the description section of the WellCAD sheets listing the depths at which gas fracturing or gas expansion cracks are observed.

### **2.3.6 Sub-Samples – required notation**

The position and designation of all whole-round sub-samples removed from the core on the catwalk or in the core trailer are indicated in the “Sample” column on the WellCAD core description sheets. The abbreviations used can be found in the core samples database.

### **2.3.7 Diagenesis**

The relative positions of features that are related to diagenesis are displayed in the “Diagenesis” column on the WellCAD core description sheets. These are mineral precipitates (e.g., pyrite and authigenic carbonates).

## **2.4 Analysis of Smear Slides**

Smear slides are prepared from the archive halves of the cores. The procedure for creating a smear slide is given below:

- With a toothpick, a small amount of sediment is taken and put on a 1 in × 3 in glass slide, homogenized, and dispersed over the slide with a drop of deionized water.
- The sample is then dried on a hot plate at the lowest effective temperature.
- A drop of Norland optical adhesive #61 and a 2.2 cm × 3.0 cm glass cover is added.
- The cover slip is fixed to the slide in an ultraviolet light box.

- With a transmitted light polarizing petrographic microscope, both the grain size and abundance of dominant components in a sample is determined.
- Abundance is estimated with the help of a comparison chart for visual percentage estimation (after Terry and Chilingarian, 1955).

Note that smear slide analyses tend to underestimate the amount of sand-sized and larger grains because these grains are difficult to incorporate into the slide. The smear slide tables include information about the location of samples, their grain-size distribution, and whether the sample represents the dominant (D) or the minor (M) lithology in the core. Additionally, they provide estimates of the major mineralogical and biological components from the examination of each smear slide. The presence of authigenic minerals or other noticeable components such as woody debris or unique trace minerals are noted in the “Comments” column.

## 2.5 Coarse Fraction Descriptions

To aid in sediment description the samples are sieved, using a 63  $\mu$  mesh, into ~5 cc of sediment and described the coarse fraction. In combination with the smear slide results, which are slightly biased toward finer grained components, the coarse fractions is used to identify the relative abundances of the larger species and components (e.g. foraminifera, diatoms, radiolarians, silicoflagellates, pyrite, quartz, mica, feldspar etc.). Together these data provide the most complete description of the presence and distribution of sedimentary components throughout the core.

## 2.6 Color Reflectance Spectrophotometry

If color reflectance spectrophotometry measurements are requested, a hand-held Gretag-Macbeth ColorEye® XTH with a spectral range between 360 and 750 nm and a resolution of 10 nm is used in the Munsell mode to obtain Munsell color readings for major color zones in cores. Names for these color readings are selected from a Munsell color chart. Freshly split cores are covered with clear plastic wrap (Glad Cling® brand).

Additional detailed information about the measurement and interpretation of spectral data can be found in Balsam et al. (1997, 1998), Balsam and Damuth (2000), and Giosan et al. (2002).

## 2.7 Digital Color Imaging

All core sections are imaged using the Geotek X-Y digital imaging system (DIS) immediately after being split and scraped. The standard procedure that should be followed is described below:

- It is useful to scrape the cores immediately prior to imaging in order to capture the ephemeral nature of some sedimentary features, particularly sulfide precipitates, which become oxidized within minutes of core splitting.
- All images are acquired at a cross-core and down-core resolution of 100 pixels/cm.
- In order to retain the relative variability in core color within each core, it is expedient to fix the aperture of the camera at a value that would image all cores without the need for further adjustment.

- Care should be taken to ensure that the system is correctly calibrated using the “white tile” procedure and that the camera positioned correctly.
- A digital ruler is added to the images.
- Output from the DIS includes an uncompressed TIFF file for each scanned section with a digital ruler on the left side of the image. Red-greenblue (RGB) profiles for all images are also automatically saved.

### **3 PHYSICAL PROPERTIES (POST-FIELD)**

The determination of physical properties of cored sediments complements sedimentologic studies, petrophysical analyses, and the interpretation of well logs. They are important to relate gas hydrate occurrences to geologic controls and the physical nature of the host material. These data are also used to provide modeling parameters, to predict reservoir behavior, and to provide baseline information on sediment attributes. Porous media effects have been found to play an important role in the concentration of gas hydrate and physical characteristics of the subsurface influence the nature of fluid and gas migration.

#### **3.1 Required Equipment, Instruments, and Supplies**

- Multi-Sensor Core Logger – Standard (MSCL-S), manufactured by Geotek, Ltd with four physical property sensors (GRA, VP, NCR, MS).
- Geotek Acoustic Rolling Contact transducers
- Bartington Loop sensor of 13.0 or 8.0 cm for measuring magnetic susceptibility
- TK04 (Teka Bolin) thermal conductivity measurement system
- ER device - four-pin Wenner Array and temperature probe, with Fluke voltmeter.
- Velocity Test Unit (VTU) for measuring P-wave, with Fluke 45 dual-display Multimeter using Labview software program.
- Wykeham-Farrance model 23500 Vane Shear Machine
- Torvane Shear Strength measuring device in diameters of 19, 25, and 48 mm.
- Pocket Penetrometer
- Two Scientech 202 electronic balances

#### **3.2 Multi-Sensor Core Logger, MSCL (Post-Field)**

The Multi-Sensor Core Logger – Standard (MSCL-S) has four physical property sensors mounted on an automated track as shown in Figure 7 and Figure 8 below, that sequentially measures the following properties:

- Bulk density using gamma ray attenuation (GRA)
- Compressional *P*-wave velocity (VP)
- Non-contact electrical resistivity (NCR)
- Magnetic susceptibility (MS)

Figure F7: Multisensor Core Logger (MSCL) with calibration standards on the track and measuring sensors on the left.

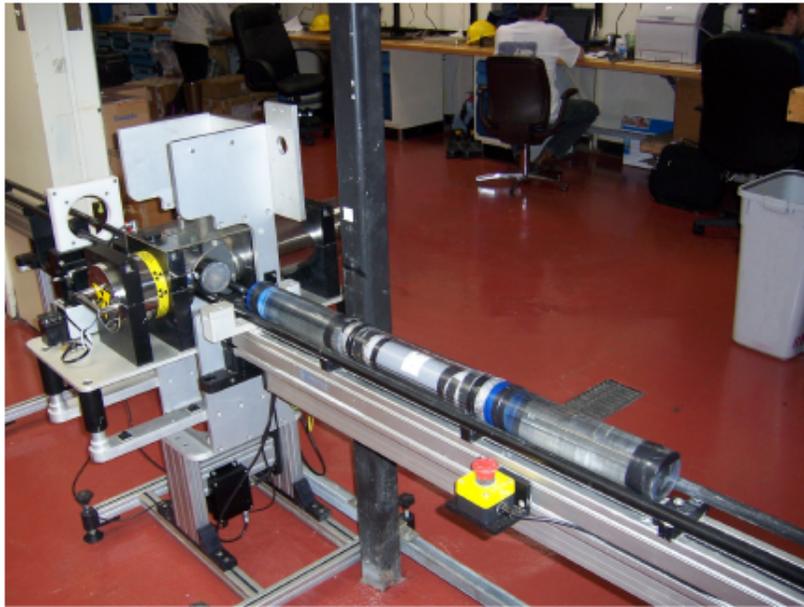


Figure 7 – MSCL-S System mounted on Track

Figure F8: Instruments on the Multisensor Core Logger (MSCL) used during NGHP Expedition 01, include (right to left) gamma ray densitometer (right), P-wave velocimeter, non-contact electrical resistivity device (below the core end cap), and magnetic susceptibility loop.

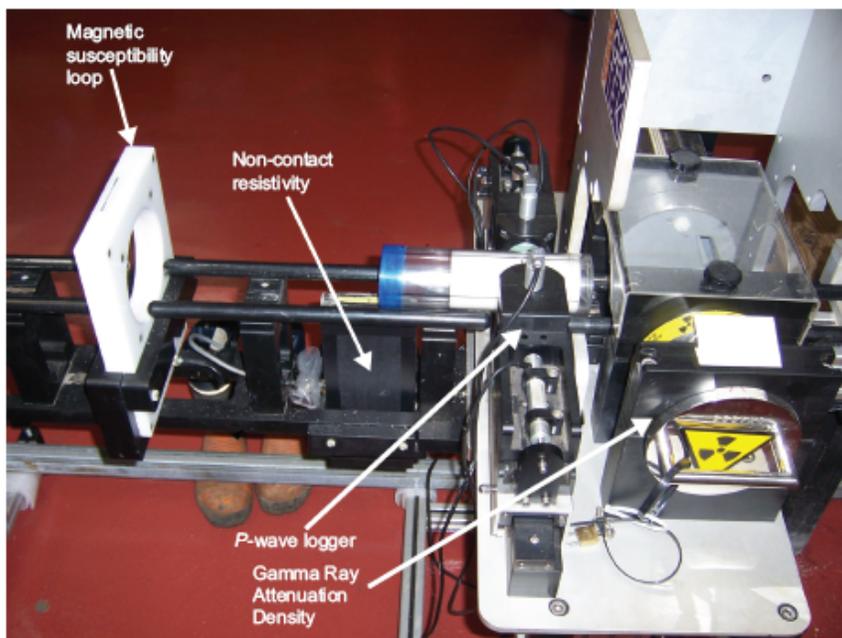


Figure 8 – MSCL-S System mounted on Track

MSCL measurements are nondestructive to sediment fabric and are used to measure and compare sediment properties along the length of the core. Data quality is a function of both core

quality and sensor precision. Optimal MSCL measurements require a completely filled core liner with minimal drilling disturbance. Precision is a function of measurement time for MS, non-contact electrical resistivity, and GRA density but not for VP. Technical notes on the Multi Sensor Core Logger and sensors are available from Geotek, Ltd., (2007).

Core sections logged on the MSCL are measured according to the following specifications:

- At an interval of 2 cm for gamma ray attenuation (GRA) density and *P*-wave velocity.
- A spatial resolution of 4 cm is used for the non-contact resistivity and magnetic susceptibility.
- For each measurement, magnetic susceptibility and NCR data are collected for 10 seconds while GRA density data is collected for 6 seconds.

Calibrations for each sensor are completed prior to logging each core section. Three standards: deionized (DI) water, Bartington Magnetic Susceptibility check piece, and water with a salinity of 17.5 ppt are run at the beginning of each core to confirm measurement accuracy and check for sensor drift.

### **3.2.1 Gamma Ray Attenuation Density**

Gamma density is measured as follows:

- Measurements are made through the center of the core using a 10mCi <sup>137</sup>Cs source and a NaI scintillation detector.
- The detection energy window is set to measure only primary (unscattered) gamma photons (0.662 MeV), providing raw gamma attenuation data in counts per second. Gamma density is derived from gamma attenuation and is reported as g/cm<sup>3</sup>.
- The gamma beam is collimated through a 5-mm hole providing a downcore spatial resolution of about 1 cm. The precision is a direct function of total counts and hence is dependent upon the count time used (1 s) and the core thickness and density.

Although the empirical calibration procedure for GRA is based on bulk density measurements (i.e., of a known graduated aluminum and water standard), the measurements will vary from true gravimetric bulk density because of variations in mineralogy. Gamma attenuation coefficients for different materials vary as a function of atomic number. Fortunately, most earth-forming minerals have similar and low atomic numbers (similar to aluminum). Consequently, the correlation of GRA density and bulk density is usually very good.

GRA data are of highest quality when measured on non-gassy whole cores because the liner is typically filled with sediment. GRA measurements may exhibit some variability because of disturbance caused by rotary coring which tends to form alternating hard “biscuits” and softer groundup “infilling” material.

### **3.2.2 Compressional P-wave Velocity**

Compressional-wave velocity (VP) measurements are gathered as follows:

- VP is measured on the MSCL track with the *P*-wave logger (PWL).
- The PWL transmits a 500-kHz *P*-wave pulse through the core at a specified repetition rate.

- Ultrasonic velocity is measured using a pair of Geotek Acoustic Rolling Contact Transducers.
- The travel time for wave pulse propagation through the core diameter is measured with a precision of 50 ns.
- At the same time, the core diameter is measured using a set of displacement transducers (precision 0.02 mm) that are mechanically coupled to the acoustic transducers.
- This produces an ultrasonic velocity with a precision of  $\pm 1.5$  m/s and a likely accuracy of  $\sim 5$  m/s.
- Core temperatures are obtained with a platinum resistance temperature probe (precision 0.05 °C) and a measured velocity is corrected to a velocity at a reference temperature (20 °C).
- Ultrasonic velocity is reported in m/s and has a typical downcore resolution of about 2 cm.

### 3.2.3 Non-Contact Resistivity (NCR)

Electrical resistivity is measured as follows:

- The instrument used is a Geotek, Ltd. NCR sensor, containing inductive coil arrays which make resistivity measurements through whole plastic core liner.
- The NCR 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 that are inversely proportional to the resistivity.
- A receiver coil measures very small magnetic fields that are regenerated by the electrical current.
- To measure these very small magnetic fields accurately, a difference technique has been developed that compares the readings generated from the measuring coils to the readings from an identical set of coils operating in air.
- As with other parameters, the measurements are sensitive to core temperature and should be obtained in a stable temperature environment for best results. Electrical resistivity is reported in ohm-m with an accuracy of about  $\pm 5\%$  and integrates over a core length of  $\sim 3$ - 4 cm.

### 3.2.4 Magnetic Susceptibility

Magnetic susceptibility is measured as follows:

- Using either a 13.0 cm or 8.0 cm diameter Bartington loop sensor.
- The frequency of the low-intensity, alternating- magnetic field produced by the sensor is sensitive to changes in the magnetic susceptibility of material within about 6.0 cm (for the 13.0 cm diameter loop) or 4 cm (for the 8.0 cm loop) on either side of the loop.
- Data is masked out to account for this effect at section ends and at locations of significant voids determined by both visual inspection of the cores as well as by anomalously low gamma density values ( $<1.3$  g/cc).
- Magnetic susceptibility, a dimensionless number, is reported as corrected volume susceptibility in SI units with an accuracy typically  $\sim \pm 4\%$ .

### 3.3 Thermal Conductivity (Post-Field)

Thermal conductivity measurements are made as follows:

- Measurements on whole-core samples are made using a TK04 (Teka Bolin) system described by Blum (1997)
- One measurement is made on each core.
- The measurement system employs a single needle probe (von Herzen and Maxwell, 1959) heated continuously in “full-space configuration.”
- The thermal conductivity needle, containing a heater wire and calibrated thermistor, is calibrated before leaving the manufacturer. It is tested on a material of known thermal conductivity (red rubber) prior to use and produced results within acceptable specifications.
- Four measurements are taken on each measured section and final thermal conductivity values are the average of the best three out of four measurements.

At the beginning of each measurement, temperatures in the samples are monitored automatically, without applying a heater current, until the background thermal drift is  $<0.04$  °C/min. Once the samples are equilibrated, the heating circuit is turned on and the temperature rise in the probe is measured. The temperature of the probe has a linear relationship with the natural logarithm of the time after the initiation of heating, per the equation below:

$$T(t) = (q/4k)\ln(t) + C$$

Where

T = temperature,  
q = heat input per unit length per unit time,  
k = thermal conductivity, t = time after the start of heating, and  
C = constant.

The thermal conductivity (k) is determined by fitting the temperatures measured during the first 150 s of each heating experiment (for details see Kristiansen, 1982; Blum, 1997). Data are reported in W/ (m·K), with measurement errors of 5–10% in high quality cores. Measured values are compared to a best-fit equation for thermal conductivity of sediment from the Cascadia accretionary prism and Nankai Trough (Davis et al., 1990):

$$k = 1.07 + (5.86 * 10^{-4} * D) - (D^2 * 3.24 * 10^{-7})$$

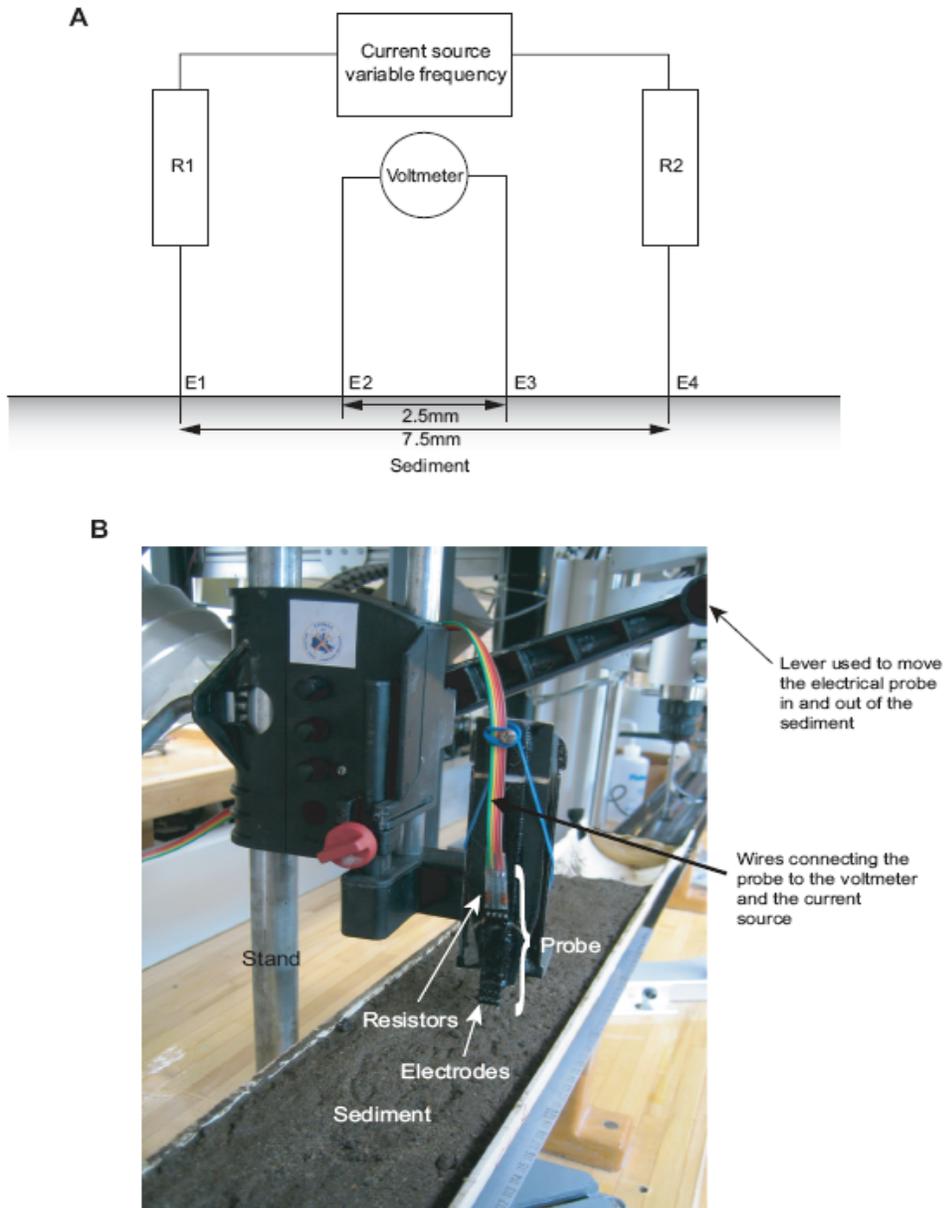
where

k = thermal conductivity, and  
D = depth below the seafloor [m].

### 3.4 Contact Electrical Resistivity Measurements (Post-Field)

Within the physical properties laboratory, electrical resistivity is the first of the contact measurements made on the working half of split cores. This prompt processing minimizes evaporation of porewater. Split cores are sometimes wrapped in cellophane after cutting to further decrease water loss. The measuring device and setup is shown in Figure 9 below:

Figure F9: (A) Split-core electrical resistivity measurement system. Current is passed between electrodes E1 and E4 and the potential difference is measured between E2 and E3. R1 and R2 are 5 kOhm resistors. (B) Photo of the electrical resistivity setup. In this photo, the probes are shown perpendicular to the core. During NGHP Expedition 01 the probes were oriented parallel to the axis of the core.



**Figure 9 – Electrical Resistivity Measurement System**

The measuring device is described as follows:

- The device consists of a four-pin Wenner array and a digital temperature probe (Fig. F9).
- The pins were gold plated and approximately 3 mm in length, separated from each other by 2.5 mm.
- The outer two pins were connected to a circuit board with an AC voltage source acting through current limiting resistors.
- The inner two pins were connected to a Fluke voltmeter.
- The entire instrument is connected to a PC through an RS-232 output thus allowing all raw data processing and display to be automated.

The procedure is described as follows:

- The probe is pushed into the sediment and a direct current (DC) 90 Hz square wave of 18 volt amplitude with a 10 k-ohm resistance (i.e. 1.8 mA current) is sent between the outer two electrodes.
- The sediment resistivity is derived by measuring the voltage between the two inner electrodes.
- An alternating current (AC) is used rather than a DC current to prevent charge build-up around the electrodes and unwanted electrochemical effects.
- The temperature of the sediment is also recorded so that the resistivity of the sediment can be corrected to a temperature of 20 °C.

Electrical resistivity,  $R$ , is defined by the following formula:

$$R = V / I * C$$

where

- V = voltage,
- I = current, and
- C = a cell constant.

The cell constant is determined using standard seawater with a known resistivity,  $R_w$ , which can be described by the following formula:

$$R_w = (2.8 + 0.1 * T)^{-1}$$

where:

- T = temperature in °C.

Measurement of the temperature, voltage, and current allows the cell constant to be determined. The instrument is thus calibrated by adjusting the cell constant until  $R_w$  equals 0.209 ohm-meters (the resistivity of Standard Mean Ocean Water (SMOW) with a salinity of 34.992 ppt) for at temperature of 20 °C.

Sample resistivity,  $R_o$ , is derived using the following formula:

$$R_o = R * (1 + 0.025 * (T - 20))$$

where:

- R = the measured sample resistivity, uncorrected for temperature,  
T = the temperature in °C.

Electrical resistivity is measured along split cores every 10-20 cm in the top of cores and less frequently in the presence of expansion cracks and voids deeper in the core. The probe is set up so that it is perpendicular to the bedding (i.e. parallel to the core). Measurements are omitted if the sediment is visibly altered during core recovery or if evidence of gas expansion is present.

When results became erratic, the electrodes are replaced. The probe electrodes are also washed in distilled water and dried before calibrating. The system is calibrated daily.

### 3.5 Directional *P*-wave Measurement (Post-Field)

The *P*-wave test measurement system is shown in Figure 10 below:

Figure F10: *P*-wave velocimeter with spade probes about to be inserted into a section of split core. The right spade contains a *P*-wave transmitting transducer and the left spade contains a receiving transducer.

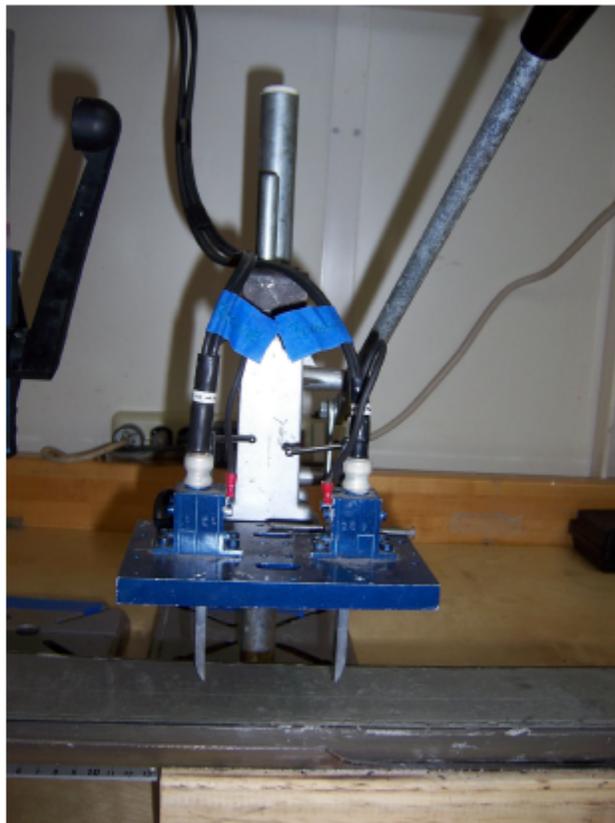


Figure 10 – *P*-wave Measurement System

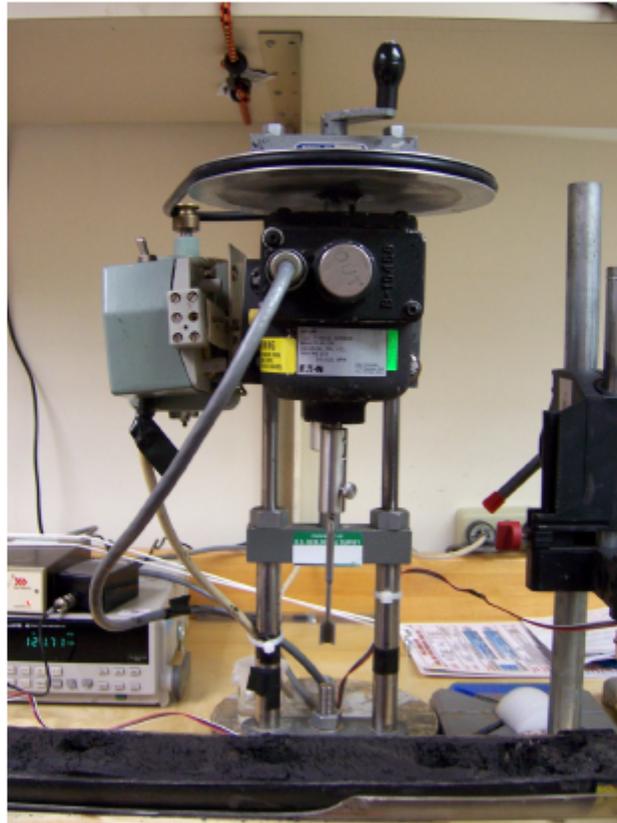
*P*-wave velocity is measured as follows:

- The measurement is taken parallel to the longitudinal axis of split cores by measuring the travel time between two fixed-distance, spade-mounted ultrasonic transducers that were inserted into the sediment.
- A Velocity Test Unit (VTU) provides an impulse-type bi-phase excitation voltage to the transmitting transducer and conditions the received signal.
- Resonant frequencies of the transducers is approximately 425 kHz.
- Acoustic signals and core temperature measured using a Fluke 45 dual-display multimeter are displayed using a Labview program which allows the first motion to be manually picked.
- Once-a-day calibrations using distilled water, adjusted for temperature, accounts for system-induced time delays.
- *P*-wave measurements are made on each split core section until the acoustic signal can not be accurately detected by the receiving transducer.
- *P*-wave velocities are generally more reliable in soft or shallow core sections. Cores that are consolidated, brittle, or from deeper in the subsurface often contain cracks and voids due to gas expansion, causing the instrument to give unreliable results.

### **3.6 Mini-vane-shear Strength (Post-Field)**

The Mini-vane Shear Strength measurement system is shown in Figure 11 below:

Figure F11: Mini-vane-shear machine with a 12.7-mm diameter by 12.7-mm high vane attached to a torque sensor. A test is performed by inserting the vane into the sediment and applying a torque to the top of the torque sensor through a drive belt attached to an electric motor. Recorded torque is proportional to the sediment shear strength.



**Figure 11 – Mini-vane Shear Machine**

Miniature-vane shear tests are performed as follows:

- Measurements are made at approximately 1.25-m intervals down core with a Wykeham-Farrance model 23500 vane shear machine and a 12.7-mm diameter by 12.7-mm high four-bladed vane..
- The vane shear strength tests are performed proximal to the sites of the electrical resistivity and  $V_p$  measurements, as shown Figure 12 below.
- The vane is inserted one vane height deep into the sediment and is turned at  $90^\circ$  per minute by applying a constant rotation rate to the top of a Lebow torque sensor.
- Voltages representative of torque values are recorded in analog form using a Daytronic strain-gage conditioner.
- Peak, residual, and remolded vane shear strengths are determined at each test location.
- Residual strength is determined after  $90^\circ$  of vane rotation and remolded strength is determined after quickly rotating the vane through an additional complete  $360^\circ$  rotation.
- If cracking, which reduces strength of the sediment, is observed during shear, the measurement should be omitted.

Figure F12: Artifacts of physical property testing in a core section. Test performed (from left to right) are: electrical resistivity using a four-pin Wenner array, *P*-wave velocity (left spade), Torvane (regular adapter), vane-shear strength, Pocket Penetrometer (no adapter), right spade of the *P*-wave velocimeter, and another electrical resistivity using a four-pin Wenner array.



Figure 12 – Locations of Physical Property Measurements on Core Slab

### 3.7 Torvane Strength (Post-Field)

The Torvance shear strength measurement device is shown in Figure 13 below:

Figure F13: Torvane with regular adapter attached. Notice the blade orientations in the surface of the sediment. This test was not actually performed at this location in the core.



**Figure 13 – Torvane Shear Strength Device**

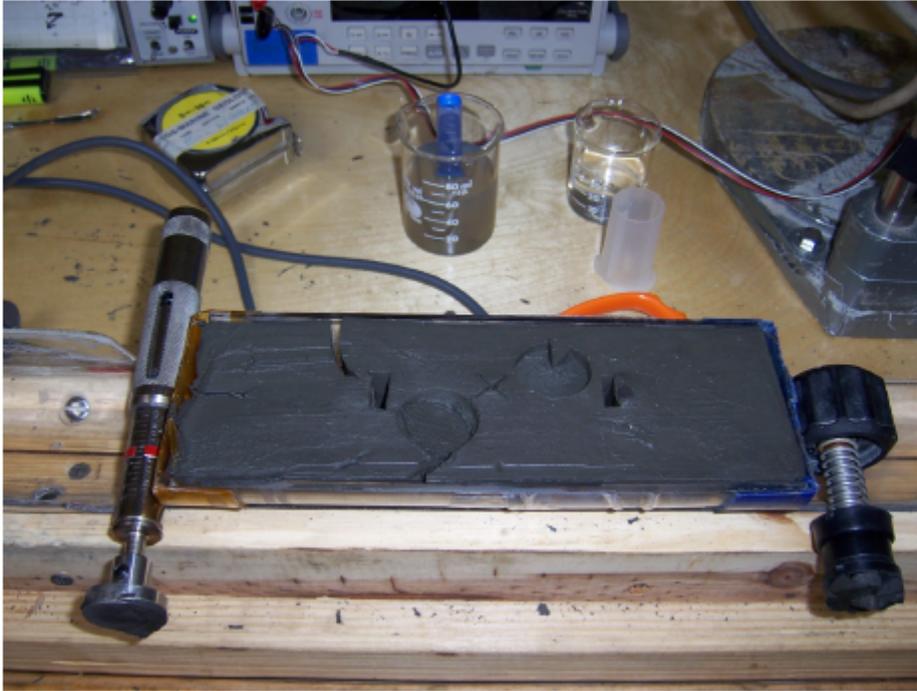
A Torvane device is used to measure shear strength near the exposed sediment surface of split cores, as follows:

- This device is operated by inserting adapters 5 mm into the exposed sediment surface.
- The top of the spring-loaded Torvane is rotated thereby producing a torque that shears the sediment.
- A pointer records the maximum torque value, which is proportional to the shear strength.
- One full revolution of the Torvane top produces a shear strength value of approximately 100 kPa.
- The Torvane comes in three diameters, 19, 25, and 48 mm, which measure a maximum shear stress up to 20, 100, and 250 kPa, respectively.
- Each size records on a continuous scale of 0-10 units, and measurements are multiplied by approximately 2, 10, and 25, respectively to obtain shear strength in units of kPa.

### **3.8 Pocket Penetrometer Strength (Post-Field)**

The Pocket Penetrometer, for measuring compressive strength, is shown in Figure 14 below.

Figure F14: Pocket penetrometer (left) and Torvane (right) strength-measuring devices. The pocket penetrometer is pushed straight down into the sediment thereby depressing a plunger. The amount of force required to penetrate the sediment is related to its strength. The top of the Torvane is rotated and the torque required to shear the sediment in contact with the blades is related to the shear strength.



**Figure 14 – Pocket Penetrometer**

In addition to the mini-vane and Torvane tests, a Pocket Penetrometer is also used to determine compressive strength and shear strength, follows:

- This device consists of a 6.35-mm diameter spring-loaded plunger that is pushed to a depth of 6.35 mm into the exposed sediment surface.
- A direct reading scale indicates the unconfined compressive strength (UCS) in kg/cm<sup>2</sup>.
- The maximum shear strength that can be determined with this device is 220 kPa.

Pocket penetrometer shear strength ( $S_{pp}$ ) is determined from:

$$S_{pp} \text{ (kPa)} = \text{UCS (kg/cm}^2\text{)} * 49$$

where:

UCS = the unconfined compressive strength reading from the pocket penetrometer strength scale.

If very soft sediment is tested, a 25.4-mm diameter adapter is applied to the end of the plunger. The maximum shear strength that can be determined with the adapter is 13.8 kPa.

If this adapter is used, the shear strength,  $S_{pp}$ , is determined from:

$$S_{pp} \text{ (kPa)} = \text{UCS (kg/cm}^2\text{)} * 3.1$$

where:

UCS = the unconfined compressive strength reading from the pocket penetrometer strength scale.

### 3.9 Moisture and Density Analysis (Post-Field)

Figure F15: Marine-going balance systems incorporating a dual load-cell design. A reference mass is placed on one cell and a beaker containing a sediment sample of unknown mass is placed on the other.



Figure 15 –

Moisture and density analysis (MAD) is used to measure wet mass, dry mass, and dry volume to determine moisture content, grain density, bulk density, porosity and void ratio, as described in Blum (1997). The procedure used to gather these measurements is outlined below:

- Sample plugs of ~10 cm<sup>3</sup> are placed in 10- mL beakers.
- Care is taken to sample undisturbed parts of the core and to avoid drilling slurry.
- One sample per section is taken at or near the location of other physical property measurements.
- Immediately after the samples are collected, wet sediment mass is measured.
- Dry mass and volume are measured after samples are heated in an oven at 105 ±5 °C for 24 hours and allowed to cool in a desiccator.

- Sample mass is determined to a precision of 0.01 g using two Scientech 202 electronic balances.
- After drying, the volume of dried solids is determined with two automatic gas pycnometers using helium as the purge and expansion gas (ASTM, 1997).
- The grain density of the pycnometer specimen is calculated using the mass of solids that was determined immediately prior to insertion of the sample into the pycnometer.
- All mass determinations are made quickly to prevent moisture in the air from being adsorbed by clay minerals.

The balance system is calibrated prior to shipment to the wellsite. Calibrations are performed daily on the two pycnometers and a sample of known density is run periodically to evaluate instrument performance.

All physical property calculations, except those specified, are corrected for the presence of residual salt left on the solid particles after driving off the pore fluid by oven drying. In the natural environment, salt and other particles that are dissolved in the pore fluid behave as part of the aqueous phase. The calculations remove the salt precipitate mass and volume from the solids and add it back to the fluid phase. Average salinity values for each core are used for the corrections. A default 35 ppt value was assumed for cores without salinity measurements. Sediment phase relations are back calculated assuming 100% water saturation of the pore voids. Visible drainage from the core sections are rarely observed, primarily because of the fine grained nature of the sediment.

The following six equations are used in calculating the physical property values (from Winters et al., in press):

$$\rho_d = M_s/V_t$$

where:

$\rho_d$  = the dry bulk density,  
 $M_s$  = the mass of solid sediment grains, and  
 $V_t$  = the calculated total specimen volume;  $\rho_w = M_t/V_t$  where:  $\rho_w$  = the wet bulk density,  $M_t$  = the total mass of the specimen, and  
 $V_t$  = the calculated total specimen volume;

$$\rho_s = M_s/V_s$$

where:

$\rho_s$  = the corrected grain density,  
 $M_s$  = the mass of solid sediment grains without salt, and  
 $V_s$  = the volume of the sediment grains without salt measured with a gas pycnometer;

$$n = V_{sw}/(V_s + V_{sw})$$

where:

$n$  = the porosity based on calculated specimen volume,

$V_{sw}$  = the volume of seawater, and

$V_s$  = the volume of solid sediment grains;

$$e = V_v/V_s$$

where:

$e$  = the void ratio,

$V_v$  = the volume of voids, and

$V_s$  = the volume of solid sediment grains;

$$WC_t = M_{sw}/M_t$$

where:

$WC_t$  = the corrected water content based on the total specimen mass,

$M_{sw}$  = the mass of sea water in the void space, and

$M_t$  = the total mass of the specimen;

$$WC_s = M_{sw}/M_s$$

where:

$WC_s$  = the corrected water content based on the solid grain mass,

$M_{sw}$  = the mass of sea water in the void space,

$M_s$  = the mass of the solid sediment grains without residual salt.

### 3.10 In-Situ Temperature

In previous hydrate drilling projects, a downhole sub has been used to measure and monitor downhole temperature. The most common technique is the use of an Advanced Piston Corer Temperature Tool (APCT), which fits into the cutting shoe of the core barrel and measures temperature during regular coring operations.

The coring tools that will be used at the Barrow project do not lend itself to this type of temperature measuring device. Instead, the well will be completed with a fiber optic Distributed Temperature Survey (DTS) to measure, in real time, the temperature of the interval. Fiber optic cable will be strapped along the casing or liner, across the producing interval, to monitor the formation for endothermic cooling. The completion design and monitoring procedure is discussed in a separate document.

## 4 INORGANIC GEOCHEMISTRY (ON-SITE AND POST-FIELD)

The measurement of inorganic geochemistry is critical to understanding the make-up and origin of interstitial water, which is key to understanding the presence and nature of gas hydrates. This chapter describes the processes used to sample, preserve, and measure the inorganic geochemistry of the core fluids.

**Note:** This section is a work in progress, pending work by Oregon State University (OSU) on existing core.

Given the hard, consolidated nature of these cores, conventional “squeezing” of the core may not be possible. This issue will require considerable discussion with USGS geologists and experts at Oregon State University, and others. Existing Barrow field and Walakpa field cores will be examined and tested to develop a suitable water extraction method. Options being considered include the following:

- 1) Centrifuge method
- 2) Rhizon method – the rhizon consists of a micro-porous tube with a pore size of 0.1  $\mu\text{m}$  supported by a nylon wire. The material and size of the tube allows it to soak up the pore fluid with capillary action. Whether this method can be used to obtain a sample of pore fluid from a consolidated core needs to be tested and addressed.
- 3) Hydraulic press method

If the centrifuge method is used, the procedure would read as follows:

1. Store WRC in glove bag while waiting to be centrifuged
2. Clean WRC to remove outer layer contamination
3. Examine and describe sample
4. Take sub-sample for grain size
5. Load the WRC into a tube that is sized to fit into the centrifuge
6. Load the tube (and core) into the centrifuge
7. Spin the sample for a minimum time of ?? minutes at a minimum of ?? rpm
8. Press and collect the water into syringe (10 or 30 cc, depends on expected volume)
9. .... (to be edited and completed after working with OSU).

If the rhizon method is used, the procedure would read as follows:

1. Store WRC in glove bag while waiting to be rhizon sampled
2. Clean WRC to remove outer layer contamination
3. Examine and describe sample
4. Take sub-sample for grain size
5. Load the WRC into ..... that allows a rhizon strand to be inserted
6. ....(to be edited and completed after working with OSU).

If a hydraulic press were to be used, the procedure would read as follows:

These samples will be squeezed to destruction using a hydraulic press to obtain pore water samples. While obtaining pore fluids is a simple operation, samples must be properly handled to avoid contamination and oxidation of some chemical species, and the squeezers must be adequately cleaned between samples to avoid cross sample contamination or interference by small amounts of oil-based mud that may be present.

1. Store WRC in glove bag while waiting to be squeezed
2. Clean WRC to remove outer layer contamination
3. Examine and describe sample
4. Take sub-sample for grain size
5. Load the WRC into the hydraulic squeezer
6. Press and collect the water into syringe (10 or 30 cc, depends on expected volume)
7. Filter through a 2 um acrudisk (on line filter)
8. Measure salinity using refractometer and record in logbook
9. Take a  $R_w$  measurement using the probe
10. Record total volume of water collected

Field laboratory: Squeezed water samples will be immediately analyzed for salinity, alkalinity, and ammonium, and sulfide will be precipitated in order to subsequently (lab-based) obtain correct sulfate and sulfide concentrations.

#### **4.1.1 Required Equipment, Instruments, Supplies, and Chemicals**

##### **4.1.1.1 All Methods**

- Whatman No. 1 filter
- 0.45  $\mu\text{m}$  Nalgene disposable filter
- Goldberg optical hand-held refractometer
- Metrohm autotitrator
- Ultrapure HCl, minimum of xx ml.
- 5%  $\text{HgCl}_2$
- Various glass vials, including 2 mL and 10 mL sizes
- Amber colored glass septum vials
- Cryovials, 5mL sizes precharged with ultrapure  $\text{HNO}_3$
- Glass ampoules
- Vacutainers – 5 Ml size
- Agilent vials
- Sulfate analysis preservative Cadmium Nitrate -  $\text{Cd}(\text{NO}_3)_2$
- 10 mM sodium bicarbonate and 20 mM sodium carbonate standards for salinity calibration
- Metrohm 761 (861) ion chromatograph
- Silver nitrate -  $\text{AgNO}_3$
- Ever-Pure RT-3 water filter cartridge
- Barnstead NANOpure® Diamond™ Analytical (Model D11901) deionization system

#### **4.1.1.2 Hydraulic Press Method**

- Hydraulic press Titanium squeezer, with forces up to 30,000 lbs.

#### **4.1.1.3 Centrifuge Method**

In addition to the equipment listing above, the centrifuge method will require the following:

- Centrifuge
- Test tubes
- ..... (to be edited and completed after working with OSU).

#### **4.1.1.4 Rhizon Method**

In addition to the equipment listing above, the centrifuge method will require the following:

- Rhizon tubes
- Test tubes
- ..... (to be edited and completed after working with OSU).

### **4.2 Collection of Sub-samples for On-site Analyses**

The majority of on-site interstitial water (IW) samples are obtained on 5- to 30-cm-long whole-round core using the process described below:

- Interstitial water is passed through a pre-washed dry Whatman No. 1 filter fitted above a titanium screen, filtered through a 0.45  $\mu\text{m}$  Nalgene disposable filter, and subsequently extruded into a new, plastic syringe attached to the bottom of the squeezer assembly.
- In most cases, 25–40 cm<sup>3</sup> of pore water is collected from each sample, which requires squeezing the sediment for 5–90 minutes, depending on lithology.

### **4.3 Collection of Sub-samples for Off-site Analyses**

Sub-samples for both alkalinity and sulfate determinations are taken and analyzed as soon after interstitial water collection as possible. The procedure for collecting sub-samples for off-site analyses is described below:

- A sub-sample for ammonium analysis (1.00 mL) is pipetted as soon after squeezing as practical into a glass ampoule, acidified with 0.010 mL ultrapure HCl, and flame-sealed.
- The remaining interstitial water is placed into a new polypropylene (PP) centrifuge tube and split for on-site and off-site analyses.
- Total dissolved inorganic carbon (DIC) samples (1 mL) are preserved with 0.100 mL 5% HgCl<sub>2</sub> in 2 mL glass vials with septum caps.
- Samples for dissolved organic carbon (DOC) and volatile fatty acids (0-3 mL) are stored in amber glass septum vials and frozen.

- Samples for oxygen and hydrogen isotopes (1-3 mL) and major ions (2-8 mL) are flame-sealed in glass ampoules.
- Minor element samples are pipetted into 5 mL cryovials that have been pre-charged with 0.040 mL ultrapure HNO<sub>3</sub>.
- DIC isotope samples are syringed into 5 mL vacutainers that are pre-loaded with 0.100 mL 5% HgCl<sub>2</sub>.
- 2 mL samples for off-site sulfate analyses are pipetted into agilent vials and preserved with 100 mL Cd(NO<sub>3</sub>)<sub>2</sub> to precipitate sulfide out of solution.
- The remaining water is used for on-site chloride, bromide and sulfate measurements.

#### 4.4 On-site Interstitial Water Analyses

Routine on-site measurements are conducted according to standard procedures (Gieskes et al., 1991), as described below:

- Salinity is measured using a Goldberg optical hand-held refractometer.
- pH is determined by ion-selective electrode.
- Alkalinity is determined by Gran titration with a Metrohm autotitrator. Accuracy and precision are monitored by repeated calibrations using analyses of International Association of the Physical Sciences of the Ocean (IAPSO) standard seawater, as well as 10 mM sodium bicarbonate and 20 mM sodium carbonate standards. The ion-selective electrode is calibrated and standards analyzed every ten samples. The average external accuracy and precision were based on the multiple analyses of the standards <2% and ~1%, respectively.
- Sulfate (SO<sub>4</sub><sup>2-</sup>) concentration is determined by manual dilution and manual injection into a Metrohm 761 (861) ion chromatograph with eluent suppression, on aliquots to which Cd(NO<sub>3</sub>)<sub>2</sub> is added as soon as possible after interstitial water collection.
- Sulfate to chloride and bromide to chloride ratios are determined by comparison of peak heights to those measured for IAPSO standard seawater. Sulfate and bromide concentrations are determined by multiplying the respective ratios by the chloride concentration determined by titration. Based on replicate analyses of IAPSO the standard deviation of the sulfate/chloride ratio determination should be less than 0.25%. Combined with the uncertainty of the chloride concentration, the resulting confidence limit for sulfate concentration is 0.3% at seawater concentrations based on repeated analysis of IAPSO. Aliquots for shore-based analyses are processed following the sampling plan given in [Table T1 below](#).

Table T1: Sample division plan for interstitial waters.

PORE FLUID DIVISION SCHEME													
NGHP Expedition 01													
	Alkalinity	SO <sub>4</sub> /H <sub>2</sub> S	DIC Concentrations	Acetate	NH <sub>4</sub>	DIC isotopes	DOC/VFA	O/H	Majors/Cl	Minors SIO	Minors OSU (Leg 4)	Shipboard	Total
code	IWPA	IWPSO	IWGTC	IWGTLA	IWGN	IWGIC	IWGOC	IWGI	IWGSM	IWPSM	IWPOM	IWPS	
subsample container	plastic test tube w cap	1.5 ml centrifuge tube	2 ml agilent vial	2 ml agilent vial	1 ml ampoule	5 ml vacutainer or 2 ml agilent vial	4 ml amber autosampler vial	2 or 5 ml ampoule	2, 5, 10, or 20 ml ampoule	5 ml plastic cryotube	Torres Nalgene bottle	1.5 ml centrifuge tube	
treatment		100µl Cd(NO <sub>3</sub> ) <sub>2</sub>	100µl HgCl <sub>2</sub>		10µl Optima HCl	100µl HgCl <sub>2</sub>	freeze			40µl Optima HNO <sub>3</sub>	40µl Optima HNO <sub>3</sub>		
>35ml	3	1	1	0.5	1	3	3	3	7	5	8	1.5	37.0
30ml	3	1	1	0.5	1	2	2	2	6	5	5	1.5	30.0
25ml	3	1	1	0.5	1	2	2	2	4	3	4	1.5	25.0
20ml	3	1	1	0.5	1	2	1	1	3	2	4	1	20.5
15ml	0	1	1	0.5	1	2	0	2	2	2	3	1	15.5
10ml	0	1	1	0	0	1	0	1	1	2	2	1	10.0
5ml	0	1	0	0	0	0	0	0	1	1	1	1	5.0

- High-precision chloride concentrations are determined by Mohr titration using silver nitrate (AgNO<sub>3</sub>) for most samples. Quantification is based on comparison with IAPSO standard seawater. Most chloride concentrations are determined in duplicate. The reproducibility of the chloride titrations, expressed as 1  $\sigma$  relative standard deviations, is evaluated by replicate analyses of IAPSO standard seawater and should be <0.2%. If the reagent needed for Cl titration is exhausted, Cl<sup>-</sup> can be analyzed by IC, with a precision of 0.7%. These samples will be re-analyzed off-site by titration.
- Bromide concentration is analyzed by manual injection into a Metrohm 761(861) ion chromatograph with eluent suppressor used for sulfate concentration. The precision based on duplicate analyses of IAPSO standard seawater, expressed as 1  $\sigma$  relative standard deviations should be 1-2%.

All figures are plotted based on concentration data that are not corrected for drillwater contamination?

#### 4.5 On-site Processing to Create High-Purity Water

Water used for all wellsite dilutions, solution preparations, and final washing of equipment (stir bars, spatulae, core squeezer parts, etc.) is produced by passing the wellsite's potable water through an Ever- Pure RT-3 water filter cartridge followed by a Barnstead NANOpure® Diamond™ Analytical (Model D11901) deionization system. The conductivity of the output of the NANOpure system is monitored and should have a conductivity of 18.2 Mohm-cm.

As an alternative to processing potable water, it may be necessary to purchase high-purity water in bulk and transport to the well site.

## **5 ORGANIC GEOCHEMISTRY (POST FIELD)**

The Barrow/Walakpa organic geochemistry research program will require access to gases, pore waters, and sediment samples. The USGS will collaborate on these analyses.

The off-site organic geochemistry program for Barrow Hydrate Test Well program consists of three routine sets of analyses:

- Analysis for volatile hydrocarbons, CO<sub>2</sub>, N<sub>2</sub>+Ar, H<sub>2</sub>S, and O<sub>2</sub>, in sediment by the headspace method;
- Measurement of free gas (FG) or gas voids (CO<sub>2</sub>, N<sub>2</sub>+Ar, H<sub>2</sub>S, and O<sub>2</sub>); and
- Analysis of sediment samples.

### **5.1 Required Equipment, Instruments, Supplies, and Chemicals**

- Sample supplies to be provided by USGS include:
- Quart-size cans for core and cuttings samples
- Glass vials, syringes, vacuum tubes, iso-tubes, etc.

### **5.2 Gas Sampling**

Samples for headspace (HS) analysis are collected on the opposite core end facing the interstitial water sample to integrate the interstitial water and gas datasets. The sampling frequency is increased at the first visible evidence of gas bubbles in sediment to achieve a high depth resolution in the hydrate bearing zone.

The sampling procedure is as follows:

- Upon core retrieval, a 2-3 inch whole round core sample is removed and placed in a quart can and sealed.
- Sediment volume was determined by weight, recorded to nearest 0.1 gm and known density as determined by physical properties.
- Air blanks incubated with septum fragments inside should confirm that no hydrocarbons (C<sub>1</sub>-C<sub>3</sub>) are released by the septum or glassware. The concentrations reported represent minimum proxy measurements of the actual concentrations due to limitations of the gas headspace method due to degassing during core recovery (Kvenvolden and Lorenson, 2000).

### **5.3 Gas Analysis**

- The vials are then frozen to preserve the sample for TOC and Rock Eval for post-well analyses.
- Analysis of methane, ethane, and propane (C<sub>1</sub>-C<sub>3</sub>) from headspace and flowed gas samples captured by the mod loggers.
- Precision analysis of higher molecular weight hydrocarbons (C<sub>3</sub>+), fixed natural gases (H<sub>2</sub>, O<sub>2</sub>, CO<sub>2</sub>, N<sub>2</sub>, and Ar) from headspace and flowed gas samples

- 2H, 18O of interstitial water and gas hydrate dissociation water in cooperation with OSU
- 13C of C1 and C2 gases, 13C and concentration of acetated and DOC, and extractable organics, alkanes, alkanols, alkenones, and polyaromatic hydrocarbons (PAHs).

#### 5.4 **Sediment**

Approximately 200 gm of wet sediment are collected for every 12' (4 m) cored section in organically-clean 60 mL jars and frozen to preserve sediment for post well-site studies of solvent extractable organics.

## 6 MICROBIOLOGY (POST-FIELD)

The microbiological make-up of the pore fluids is an important clue in determining the presence and source of methane hydrates. This section describes the methods used to measure the microbial characteristics of the core fluids.

### 6.1 Required Equipment, Instruments, Supplies, and Chemicals

- Filter-sterilized (0.2  $\mu\text{m}$ ) 2% formaldehyde in 3.5% NaCl.60 mL syringes
- Whirlpak bags
- Nitrogen ( $\text{N}_2$ )
- Cryostorage bags
- Oxygen scrubbers - Anaerocult® A mini, from EM SCIENCE
- Polycarbonate filters (0.2- $\mu\text{m}$  pore size)

### 6.2 Cell Enumeration

Sediment in 1  $\text{cm}^3$  plugs are taken for post well-site direct microscopic determination of bacterial numbers. These plugs are taken from the end of selected core sections immediately after the sections are cut in the cold trailer. The procedure is as follows:

- On average, one sample per core is collected.
- Potentially contaminated sediment is removed with a sterile scalpel, and a sterile 3  $\text{cm}^3$  plastic syringe with the luer end removed is used to take a 1  $\text{cm}^3$  plug.
- The 1  $\text{cm}^3$  plug is extruded into a sterile plastic centrifuge tube containing 9 mL of filter-sterilized (0.2  $\mu\text{m}$ ) 2% formaldehyde in 3.5% NaCl.
- The tube is shaken vigorously to disperse the sediment particles and subsequently stored at 4 °C.

### 6.3 Relationship of Microbial Characteristics to Hydrate

In order to determine whether fine-scale microbial characteristics in the sediments (e.g., microbial community dynamics, numbers of methanogens) are dictated by the presence of gas hydrate, or some other factor(s), a series of whole round core (WRC) plugs are collected from core sections that show evidence of gas hydrate and from adjacent sections that apparently lack gas hydrate.

WRCs removed from the single gas hydrate/non-gas hydrate bearing core units are wrapped in Saran wrap, double-bagged in Whirlpak bags, labeled, flushed with  $\text{N}_2$ , and then sealed in cryostorage bags. Subsequently, WRC samples are refrigerated (4 °C) or frozen (-80 °C) as soon as possible.

### 6.4 Hydrogenase Activity

WRCs with a length of 5 cm are collected for post well analyses hydrogenase activity determinations (Soffientiono et al, 2006). Whole rounds are cut in the cold trailer (or on the catwalk), capped, bagged with oxygen scrubbers (Anaerocult® A mini, EM SCIENCE) and stored at  $-80^{\circ}\text{C}$ .

## **6.5 Contamination Assays**

To confirm the suitability of the core material for microbiological research, contamination assays are conducted to quantify the intrusion of drill water using a fluorescent microsphere tracer technique described in Volume 1 of the Coring Procedures Manual. The solution is centrifuged and the supernatant containing the microspheres is filtered through polycarbonate filters (0.2- $\mu\text{m}$  pore size). The microspheres are then counted, and data are reported as number of microspheres per gram of sediment. The isolation and counting of the microspheres will take place off-site.

## **7 POST FIELD PETROPHYSICS**

In addition to measuring properties specific to gas hydrates and the native pore fluids, it is important to measure the intrinsic properties of the reservoir rock, without the effects of hydrates. As part of the Mt. Elbert project, core samples had native fluid removed and replaced with a known fluid type. The following conventional core analysis was then conducted:

- Grain size
- Water content
- Porosity
- Grain Density
- Bulk Density
- Conventional Permeability (gas type)
- Mercury Porosimetry Analysis
- X-ray Diffraction
- Nuclear Magnetic Resonance (NMR)
- Petrographic Analysis
- Permeability with mini-permeameter measurements

## 8 REFERENCES

### Primary Reference

NGHP Expedition 01 Scientists, Collett, T., Riedel, M., Cochran, J., Boswell, R., Presley, J., Kumar, P., Sathe, A., Sethi, A., Lall, M., Sibal, V. 2007. Methods. *In National Gas Hydrate Program Expedition 01 Initial Reports*.

### References of the Primary Reference

American Society for Testing and Materials (ASTM), 1997. Standard test method for specific gravity of soil solids by gas pycnometer D 5550-94; *in*

American Society for Testing and Materials, *Annual Book of ASTM Standards*, v. 04.09, Soil and Rock, West Conshohocken, PA, p. 380-383.

Aldred, W., Cook, J., Bern, P., Carpenter, B., Hutchinson, M., Lovell, J., Rezmer-Cooper, I., and Leder, P.C., 1998. Using downhole annular pressure measurements to improve drilling performance, *Oilfield Review*, Winter 1998: 40-55.

Archie, G.E., 1942. The electrical resistivity log as an aid in determining some reservoir characteristics. *J. Pet. Technol.*, 5:1-8.

Balsam, W.L., and Damuth, J.E., 2000. Further investigations of shipboard vs. shore-based spectral data: implications for interpreting Leg 164 sediment composition. In Paull, C.K., Matsumoto, R., Wallace, P., and Dillon, W.P. (Eds.), *Proc. ODP, Sci. Results, 164*: College Station, TX (Ocean Drilling Program), 313-324.

Balsam, W.L., Damuth, J.E., and Schneider, R.R., 1997. Comparison of shipboard vs. shore-based spectral data from Amazon-Fan cores: implications for interpreting sediment composition. In Flood, R.D., Piper, D.J.W., Klaus, A., and Peterson, L.C. (Eds.), *Proc. ODP, Sci. Results, 155*: College Station, TX (Ocean Drilling Program), 193- 215.

Balsam, W.L., Deaton, B.C., and Damuth, J.E., 1998. The effects of water content on diffuse reflectance measurements of deep-sea core samples: an example from ODP Leg 164 sediments. *Mar. Geol.*, 149:177-189.

Blum, P., 1997. Physical properties handbook: a guide to the shipboard measurement of physical properties of deep-sea cores. *ODP Tech. Note*, 26 [Online]. Available from World Wide Web: <<http://www.odp.tamu.edu/publications/tnotes/tn26/INDEX.HTM>>.

Bonner, S.D., Tabanou, J.R., Wu, P.T., Seydoux, J.P., Moriarty, K.A., Seal, B.K., Kwok, E.Y., and Kuchenbecker, M.W., 1995. New 2-MHz multiarray borehole-compensated resistivity tool

developed for MWD in slim holes [paper presented at Annu. Tech. Conf. Exhib. Soc. Pet. Eng., Dallas, October 1995], Pap. SPE 30547. Available from: Society of Petroleum Engineers.

Bonner, S., Fredette, M., Lovell, J., Montaron, B., Rosthal, R., Tabanou, J., Wu, P., Clark, B., Mills, R., and Williams, R., 1996, Resistivity while drilling—images from the string, *Oilfield Review*, Spring 1996: 4-19.

Bullard, E.C., 1954. The flow of heat through the floor of the Atlantic Ocean. *Proc. R. Soc. London A*, 222:408-429.

Collett, T.S., 1993. Natural gas hydrates of the Prudhoe Bay–Kuparuk River area, North Slope, Alaska. *AAPG Bull.*, 77:793–812 1998a.

Collett, T.S., 1998. Well log characterization of sediment porosities in gas-hydrate-bearing reservoirs [paper presented at Annual. Tech. Conf. Exhib. Soc. Pet. Eng., New Orleans, September 1998], Pap. SPE 49298. Available from: Society of Petroleum Engineers. 1998b.

Collett, T.S., 2000. Well log evaluation of gas hydrate saturations. In Trans. SPWLA 39th Annu. Logging Symp.: Houston (SPWLA), 39:MM., 2000. A review of well-log analysis techniques used to assess gas-hydrate-bearing reservoirs: In Paull, C.K., and Dillon, W.P. (Eds.), *Natural Gas Hydrates: Occurrence, Distribution, and Detection*. Geophys. Monogr., Am. Geophys. Union, 189–210.

Davis, E.E., Hyndman, R.D., and Villinger, H., 1990, Rates of fluid expulsion across the northern Cascadia accretionary prism: constraints from new heat flow and multichannel seismic reflection data. *Journal of Geophysical Research*, v. 95, p. 8869-8889.

Davis, E.E., Villinger, H., MacDonald, R.D., Meldrum, R.D., and Grigel, J., 1997. A robust rapid-response probe for measuring bottom-hole temperatures in deepocean boreholes. *Mar. Geophys. Res.*, 19:267–281.

Dickens, G. R., C. K. Paull, P. Wallace, and the ODP Leg 164 Scientific Party 1997. Direct measurement of *in situ* methane quantities in a large gashydrate reservoir. *Nature*, 385, 426-428.

Dickens, G.R., Borowski, W.S., Wehner, H., Paull, C.K., and the ODP Leg 164 Scientific Party, 2000a. Data Report: Additional shipboard information for the pressure core sampler (PCS). In Paull, C.K., Matsumoto, R., Wallace, P.J., and Dillon, W.P. (Eds.), *Proc. ODP, Sci. Results, 164*, 439–443 [CDROM]. Available from: Ocean Drilling Program, Texas A&M University, College Station, TX 77845- 9547, U.S.A.

Dickens, G.R., Wallace, P.J., Paull, C.K., and Borowski, W.S., 2000b. Detection of methane gas hydrate in the pressure core sampler (PCS): volume-pressure-time relations during controlled degassing experiments. In Paull, C.K., Matsumoto, R., Wallace, P.J., and Dillon, W.P. (Eds.), *Proc. ODP, Sci. Results*,: College Station, TX (Ocean Drilling Program), 113–126.

Dickens, G.R., Schroeder, D., Hinrichs, K.-U., and the Leg 201 Scientific Party, 2003. The pressure core sampler (PCS) on Ocean Drilling Program Leg 201: general operations and gas release. In D'Hondt, S.L., Jørgensen, B.B., Miller, D.J., et al., *Proc. ODP, Init. Repts.*, 201, 1–22 [CD-ROM]. Available from: Ocean Drilling Program, Texas A&M University, College Station TX 77845-9547, USA.

Droser, M.L., and Bottjer, D.J., 1986. A semiquantitative field classification of ichnofabric. *J. Sediment. Petrol.*, 56:558-559. Duan, Z., Møller, N., Greenberg, J., and Weare, J.H., 1992. The prediction of methane solubility in natural waters to high ionic strengths from 0° to 250°C and from 0 to 1600 bar. *Geochim. Cosmochim. Acta*, 56:1451–1460.

Dunham, R.J., 1962. Classification of carbonate rocks according to depositional texture. In Ham, W.E. (Ed.), *Classification of Carbonate Rocks*: AAPG Mem., 108-121.

Ellis, D.V., 1987. *Well Logging for Earth Scientists*: New York (Elsevier).

Embry, A.F., and Klovan, J.E., 1971. A late Devonian reef tract on northeastern Banks Island, Northwest Territories. *Bull. Can. Pet. Geol.*, 19:730- 781.

Expedition 311 Scientists, 2005. Cascadia margin gas hydrates. *IODP Prel. Rept.*, 311.  
[doi:10.2204/iodp.pr.311.2005](https://doi.org/10.2204/iodp.pr.311.2005)

Gealy, E.L., Winterer, E.L., and Moberly, R., Jr., 1971. Methods, conventions, and general observations. In Winterer, E.L., Riedel, W.R., et al., *Init. Repts. DSDP*, 7 (Pt. 1): Washington (U.S. Govt. Printing Office), 9-26.

Gieskes, J.M., Gamo, T., and Brumsack, H., 1991. Chemical methods for interstitial water analysis aboard *JOIDES Resolution*. *ODP Tech. Note*, 15.

Giosan, L., Flood, R.D., and Aller, R.C., 2002. Paleooceanographic significance of sediment color on western North Atlantic drifts: I. Origin of color. *Mar. Geol.*, 189:25-41.

Goldberg, D., 1997. The role of downhole measurements in marine geology and geophysics. *Rev. Geophys.*, 35:315–342.

Goldberg, D., Collett, T.S., and Hyndman, R.D., 2000. Ground truth: in-situ properties of hydrate. In Max, M.D. (Ed.), *Natural Gas Hydrate in Oceanic and Permafrost Environments: The Netherlands* (Kluwer Academic Publishers), 295–310.

Graber, K.K., Pollard, E., Jonasson, B., and Schulte, E. (Eds.), 2002. Overview of Ocean Drilling Program Engineering Tools and Hardware. *ODP Tech. Note*, 31 [Online]. Available from World Wide Web: <<http://www-odp.tamu.edu/publications/tnotes/tn31/INDEX.HTM>>. [Cited 2006-Aug-04]

Graber, K.K., Pollard, E., Jonasson, B., and Schulte, E. (Eds.), 2002. Overview of Ocean Drilling Program Engineering Tools and Hardware. *ODP Tech. Note*, 31 [Online]. Available from World

Wide Web: <<http://www-odp.tamu.edu/publications/tnotes/tn31/INDEX.HTM>>. [Cited 2005-10-05].

Guerin, G., and Goldberg, D., 2002. Sonic attenuation measurements in the Mallik 2L-38 gas hydrates research well, MacKenzie Delta, NWT Canada. *J. Geophys. Res.*, 107:10.1029/2001JB000556.

Guerin, G., Goldberg, D., and Meltser, A., 1999. Characterization of in situ elastic properties of gashydrate bearing sediments on the Blake Ridge. *J. Geophys. Res.*, 104:17781–17795.

Heeseman, M., H. Villinger, A.T. Fisher, A.M. Trehu, S. Witte, Data report: testing and deployment of the new APCT-3 tool to determine in situ temperatures while piston coring, in M. Riedel, T.S. Collett, M.J. Malone and the Expedition 311 Scientists, *Proceedings of the Integrated Ocean Drilling Program*, vol. 311, 2006.

Helgerud, M.B., Dvorkin, J., and Nur, A., 2000. Rock physics characterization for gas hydrate reservoirs: elastic properties. In Holder, G.D., and Bishnoi, P.R. (Eds.), *Gas Hydrates: Challenges for the Future*. Ann. N.Y. Acad. Sci., 912:116–125.

Horai, K., and Von Herzen, R.P., 1985. Measurement of heat flow on Leg 86 of the Deep Sea Drilling Project. In Heath, G.R., Burckle, L.H., et al., *Init. Repts. DSDP*, 86: Washington (U.S. Govt. Printing Office), 759–777.

Hunt, J.M. 1979. *Petroleum Geochemistry and Geology*. San Francisco (W.H. Freeman).  
Kleinberg, R.L., Flaum, C., and Collett, T.S., 2005. Magnetic Resonance Log of Mallik 5L-38: Hydrate Saturation, Growth Habit, and Relative Permeability, in Dallimore, S.R. and Collett, T.S., Scientific Results from the Mallik 2002 Gas Hydrate Production Research Well Program, Mackenzie Delta, Northwest Territories, Canada, *Geological Survey of Canada*, Bulletin no. 585.

Kristiansen, J.I., 1982. The transient cylindrical probe method for determination of thermal parameters of earth materials [Ph.D. dissert.]. Aarhus Univ. Kvenvolden, K. A., and Lorensen, T. D. (2000). Methane and other hydrocarbon gases in sediment from the southeastern north American continental margin; In Paull, C. K., Matsumoto, R., Wallace, P. J., & Black, N. R. et al. proceedings of the ocean drilling program; volume 164; scientific results; gas hydrate sampling on the Blake ridge and Carolina rise; covering leg 164 of the cruises of the drilling vessel *JOIDES Resolution*, Halifax, Nova Scotia, to Miami, Florida, sites 991-997, 31 October-19 December 1995. *Proceedings of the Ocean Drilling Program, Scientific Results, 164*, 29-36.

Lamont-Doherty Earth Observatory Borehole Research Group, 2001. *ODP Logging Services Electronic Manual*, Version 2.0 [Online]. Available from World Wide Web: <<http://www.ldeo.columbia.edu/BRG/ODP/LOGGING/MANUAL/index.html>>. [Cited 2005-09-28]

Luthi, S. M., 2001. *Geological Well Logs: Their Use in Reservoir Modeling*, Springer-Verlag, Berlin. Manheim, F.T., and Sayles, F.L., 1974. Composition and origin of interstitial waters of

marine sediments, based on deep sea drilled cores. In Goldberg, E.D. (Ed.), *The Sea* (Vol. 5): *Marine Chemistry: The Sedimentary Cycle*: New York (Wiley), 527–568.

Mathews, M., 1986. Logging characteristics of methane hydrate. *The Log Analyst*, 27:26–63.

Mazzullo, J., and Graham, A.G. (Eds.), 1988. Handbook for shipboard sedimentologists. *ODP Tech. Note*, 8.

Mazzullo, J.M., Meyer, A., and Kidd, R.B., 1988. New sediment classification scheme for the Ocean Drilling Program. In Mazzullo, J., and Graham, A.G. (Eds.), *Handbook for Shipboard Sedimentologists*. *ODP Tech. Note*, 8:45-67.

Mikada, H., Becker, K., Moore, J.C., Klaus, A., et al., 2002. Proc. ODP, Init. Repts., 196 [Online]. Available from World Wide Web: [http://www.wodp.tamu.edu/publications/196\\_IR/196ir.htm](http://www.wodp.tamu.edu/publications/196_IR/196ir.htm). [Cited 2005-09-28]

Milkov, A.V., G.R. Dickens, G.E. Claypool, Y.-J. Lee, W.S. Borowski, M.E. Torres, W. Xu, H. Tomaru, A.M. Tréhu, P. Schultheiss, 2004. Co-existence of gas hydrate, free gas, and brine within the regional gas hydrate stability zone at Hydrate Ridge (Oregon margin): evidence from prolonged degassing of a pressurized core. *Earth and Planetary Science Letters*, 222, 829-843.  
Munsell Color Company, Inc., 1975. *Munsell Soil Color Charts*: Baltimore, MD (Munsell).  
Paull, C.K., Matsumoto, R., Wallace, P.J., et al., 1996. *Proc. ODP, Init. Repts.*, 164: College Station, TX (Ocean Drilling Program).

Paull, C.K., Matsumoto, R., Wallace, P.J., et al., 1996. *Proc. ODP, Init. Repts.*, 164: College Station, TX (Ocean Drilling Program).

Paull, C.K., and Ussler, W., III, 2000. History and significance of gas sampling during DSDP and ODP drilling associated with gas hydrates. In Paull, C.K., and Dillon, W.P. (Eds.), *Natural Gas Hydrates: Occurrence, Distribution, and Detection*. Am. Geophys. Union, Geophys. Monogr. Ser., 124:53–66.

Pettigrew, T. L. 1992. The design and operation of a wireline pressure core sampler (PCS). *ODP Technical Note*, 17, Ocean Drilling Program, College Station, TX.

Pettigrew, T.L., 1992. The design and operation of a wireline pressure core sampler (PCS). *ODP Tech. Note*, 17.

Pimmel, A., and Claypool, G., 2001. Introduction to shipboard organic geochemistry on the *JOIDES Resolution*. *ODP Tech. Note*, 30 [Online]. Available from World Wide Web: <http://www.wodp.tamu.edu/publications/tnotes/tn30/INDEX.HTM>. [Cited 2005-09-30]

Rider, M., 1996. *The Geological Interpretation of Well Logs* (2nd ed.): Caithness (Whittles Publishing). Sloan, E.D., 1998. *Clathrate Hydrates of Natural Gases* (2nd ed.): New York (Marcel Dekker).

Schlager, W., and James, N.P., 1978. Low-magnesian calcite limestones forming at the deep-sea floor, Tongue of the Ocean, Bahamas. *Sedimentology*, 25:675-702. Schlumberger, 1989. *Log Interpretation Principles/ Applications: Houston* (Schlumberger Educ. Services), SMP-7017. ———, 1994. *Log Interpretation Charts: Sugar Land* (Schlumberger), SMP-7006.

Serra, O., 1984. Fundamentals of Well-Log Interpretation (Vol. 1): The Acquisition of Logging Data: *Dev. Pet. Sci.*, 15A. ———, 1986. Fundamentals of Well-Log Interpretation (Vol. 2): The Interpretation of Logging Data: *Dev. Pet. Sci.*, 15B. ———, 1989. *Formation MicroScanner Image Interpretation: Houston* (Schlumberger Educ. Services), SMP-7028.

Schultheiss, P.J., Francis, T.J.G., Holland, M., Roberts, J.A., Amann, H., Thjunjoto, Parkes, R. J., Martin, D., Rothfuss, M., Tyunder, F., & P.D. Jackson. 2006. Pressure coring, logging and subsampling with the HYACINTH system. In "New Ways of Looking at Sediment Cores and Core Data," Rothwell R.G. ed., *Geological Society*, London. Special Publications 267:151-165.

Shepard, F., 1954. Nomenclature based on sand-siltclay ratios. *J. Sediment. Petrol.*, 24:151-158. Shipboard Scientific Party, 1998. Introduction. In Becker, K., Malone, M.J., et al., *Proc. ODP, Init. Repts., 174B*: College Station, TX (Ocean Drilling Program), 3–9.

Shipboard Scientific Party, 2003. Explanatory notes. In Tréhu, A.M, Bohrmann, G., Rack, F.R., Torres, M.E., et al., *Proc. ODP, Init. Repts., 204*, 1-102 [CD-ROM]. Available from: Ocean Drilling Program, Texas A&M University, College Station TX 77845-9547, USA.

Smith, D.C., Spivack, A.J., Fisk, M.R., Haveman, S.A., Staudigel, H., and ODP Leg 185 Scientific Party, 2000. Methods for quantifying potential microbial contamination during deep ocean coring. *ODP Tech. Note*, 28 [Online]. Available from World Wide Web: <<http://www-odp.tamu.edu/publications/tnotes/tn28/INDEX.HTM>>.

Soffientino B., Spivack A.J., Smith D.C., Roggenstein E.B., D Hondt S.J., 2006. A versatile and sensitive tritium-based radioassay for measuring hydrogenase activity in aquatic sediments. *Microbiol Methods*. Jul;66(1):136-46. Epub 2005 Dec 13.

Terry, R.D., and Chilingarian, G.V., 1955. Summary of "Concerning some additional aids in studying sedimentary formations" by M.S. Shvetsov. *J. Sediment. Petrol.*, 25:229-234.

Tréhu, A.M., 2006. Subsurface temperatures beneath southern Hydrate Ridge. In Tréhu, A.M., Bohrmann, G., Torres, M.E., and Colwell, F.S. (Eds.), *Proc. ODP, Sci. Results*, 204: College Station, TX (Ocean Drilling Program), 1–26. [doi:10.2973/odp.proc.sr.204.114.2006](https://doi.org/10.2973/odp.proc.sr.204.114.2006)

Tréhu, A.M, Bohrmann, G., Rack, F.R., Torres, M.E., et al., 2003. *Proc. ODP, Init. Repts.*, 204: College Station, TX (Ocean Drilling Program). [doi:10.2973/odp.proc.ir.204.2003](https://doi.org/10.2973/odp.proc.ir.204.2003) Ussler, W., III, Paull, C.K., McGill, P., Schroeder, D., and Ferrell, D., 2006. A test of the temperature, pressure, and conductivity tool prototype at Hydrate Ridge. In Tréhu, A.M., Bohrmann, G.,

Torres, M.E., and Colwell, F.S. (Eds.), *Proc. ODP, Sci. Results*, 204: College Station, TX (Ocean Drilling Program), 1–41. [doi:10.2973/odp.proc.sr.204.112.2006](https://doi.org/10.2973/odp.proc.sr.204.112.2006) von Herzen, R.P., and Maxwell, A.E., 1959. The measurement of thermal conductivity of deep-sea sediments by a needle-probe method. *J. Geophys. Res.*, 64:1557–1563.

Wallace, P.J., Dickens, G.R., Paull, C.K., and Ussler III, W., 2000. Effects of core retrieval and degassing on the carbon isotope composition of methane in gas hydrate– and free gas–bearing sediments from the Blake Ridge. In Paull, C.K., Matsumoto, R., Wallace, P.J., and Dillon, W.P. (Eds.), *Proc. ODP, Sci. Results*, 164, 101–112 [CD-ROM]. Available from: Ocean Drilling Program, Texas A&M University, College Station, TX 77845-9547, U.S.A.

Wentworth, C.K., 1922. A scale of grade and class terms of clastic sediments. *J. Geol.*, 30:377-392.

Winters, W.J., Novosel, I., Boldina, O., Waite, W.F., and Jablonski, S., in press, Physical Properties of Sediment Obtained During the IMAGES VIII/PAGE 127 Gas Hydrate and Paleoclimate Cruise on the R/V *Marion Dufresne* in the Gulf of Mexico, 2-18 July 2002; in Winters, W.J., Lorenson, T.D., Paull, C.K., eds., Initial report of the IMAGES VIII/PAGE 127 gas hydrate and paleoclimate cruise on the R/V *Marion Dufresne* in the Gulf of Mexico, 2-18 July 2002, *U.S. Geological Survey Open-File Report 2004-1358*.

Westbrook, G.K., Carson, B., Musgrave, R.J., et al., 1994. *Proc. ODP, Init. Repts.*, 146 (Pt. 1): College Station, TX (Ocean Drilling Program). UNESCO, 1981: Tenth report of the joint panel on oceanographic tables and standards. UNESCO Tech. Paper in *Marine Science* 36, 25 pp.

Xu, W., 2002. Phase balance and dynamic equilibrium during formation and dissociation of methane gas hydrate, in *Proc. 4th International Conference on Gas Hydrates*, pp. 195-200, Yokohama, Japan. Xu, W., 2004. Modeling dynamic marine gas hydrate systems, *American Mineralogist*, 89, 1271- 1279.

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