

# Oil & Natural Gas Technology

DOE Award No.: DE-FE0023919

## Phase 1 Report

*Type: Other*

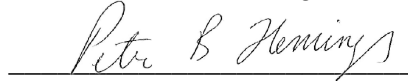
**(Period Ending 09/30/2015)**

## Deepwater Methane Hydrate Characterization and Scientific Assessment

Project Period 10/01/2014 – 09/30/2018

Submitted by:

Peter B. Flemings



Signature

The University of Texas at Austin

DUNS #:170230239

101 East 27<sup>th</sup> Street, Suite 4.300

Austin, TX 78712-1500

e-mail: [pflemings@jsg.utexas.edu](mailto:pflemings@jsg.utexas.edu)

Phone number: (512) 475-9520

Prepared for:

United States Department of Energy  
National Energy Technology Laboratory

Revision Submitted: February 1, 2016



Office of Fossil Energy



“This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.”

## Contents

|  |           |
|--|-----------|
| 1. Executive Summary.....  | 1         |
| 2. Introduction .....  | 1         |
| 3. Summary of Tasks .....  | 1         |
| 3.1. Task 1: Project Management and Planning .....   | 1         |
| 3.2. Task 2: Site Analysis and Selection.....  | 3         |
| 3.3. Task 3: Develop Pre-Expedition Drilling/Logging/Coring/Sampling Operational Plan.....   | 8         |
| 3.4. Task 4: Complete and Update IODP CPP Proposal.....  | 9         |
| 3.5. Task 5: Pressure Coring and Core Analysis System Modification and Testing.....  | 10        |
| 4. Products Developed .....  | 12        |
| 4.1. Publications, conference papers, and presentations.....   | 12        |
| 4.2. Website(s) or other Internet site(s) .....  | 13        |
| 5. References.....   | 13        |
| 6. Acronyms .....  | 13        |
| <b>Appendix A: Operational Plan .....</b>  | <b>17</b> |
| <b>Appendix B: IODP Complementary Project Proposal for Genesis of Methane Hydrate in Coarse-Grained Systems:<br/>Northern Gulf of Mexico Slope (GOM^2) .....</b> | <b>21</b> |
| <b>Appendix C: Methane Hydrate Pressure Coring and Analysis: Gulf of Mexico Scientific Planning Workshop Report ....</b>   | <b>71</b> |
| <b>Appendix D: Hybrid Pressure Coring Tool with Ball Valve (PCTB) 2015 Laboratory Test Program .....</b>   | <b>95</b> |

## 1. Executive Summary

The objective of this project is to gain insight into the nature, formation, occurrence and physical properties of methane hydrate-bearing sediments for the purpose of methane hydrate resource appraisal through the planning and conduct of drilling, coring, logging, testing and analytical activities to assess the geologic occurrence, regional context, and characteristics of marine methane hydrate deposits in the Gulf of Mexico and / or other areas of the US Outer Continental Shelf. This effort includes the planning and execution a state -of -the-art deepwater, methane hydrate - drilling program targeting methane hydrate reservoirs on the US continental margin.

In Phase 1, potential research expedition sites were identified and each site was appraised using available geophysical and geologic data and ranked relative to one another using criteria developed in conjunction with DOE.

Following site selection, a pre-expedition drilling, coring, logging and sampling operation plan (Operational Plan) was developed. At the culmination of Phase 1, a Complementary Project Proposal (CPP), based on this Operational Plan, was submitted to the International Ocean Discovery Program (IODP) as a primary method of accessing a suitable drill ship / science vessel. Concurrently during Phase 1, there was bench testing and modification of the DOE Pressure Coring System (the PCTB) and planning for a Land Test of this system.

## 2. Introduction

This project “Deepwater Methane Hydrate Characterization and Scientific Assessment” was funded from the Department of Energy in October of 2014. Phase 1 of this project encompasses the first year; October 1, 2014 to September 30, 2015. This report provides a summary of activities in Phase 1 of the project and a collection of key deliverables.

This report will pay particular attention to the Site Location and Ranking Report, Preliminary Field Program Operational Plan Report, PCS Scientific Workshop Report, and PCTB Lab Test Report.

**Table 1: Phase 1 Project Milestones**

| Task | Milestone Description                                  |
|------|--|
| 1.00 | M1A: Project Management Plan                           |
| 1.00 | M1B: Project Kick-off Meeting                          |
| 2.00 | M1C: Site Location and Ranking Report                  |
| 3.00 | M1D: Preliminary Field Program Operational Plan Report |
| 4.00 | M1E: Updated CPP Proposal Submitted                    |
| 2.00 | M1F: Demonstration of a viable PCS Tool (Lab Test)     |
| n/a  | M1G: Document results of BP1/Phase 1 Activities        |

## 3. Summary of Tasks

### 3.1. Task 1: Project Management and Planning

#### A. Goal:

The recipient will manage and control project activities in accordance with their established processes and procedures to ensure subtasks and tasks are completed within schedule and budget constraints defined by the PMP. This includes tracking and reporting progress and project risks to DOE and other stakeholders.

**B. Activities Phase 1:****B1. Assembled team to meet project needs.**

| Title                           | Status | Name             | Date Hired             |
|---------------------------------|--------|------------------|------------------------|
| Postdoc                         | Filled | Stacey Worman    | August 2015            |
| Postdoc                         | Filled | Stephen Phillips | September 2015         |
| Graduate Research Associate     | Filled | Kevin Meazell    | March 2015             |
| Sr. Program Coordinator         | Filled | Colleen Morgan   | September 2015         |
| Project Manager                 | Filled | Tessa Green      | June 2015              |
| Project Manager                 | Filled | Carla Thomas     | June 2015              |
| Research Scientist Associate II | Open   | TBD              | Expected Start Phase 2 |
| Research Scientist Associate V  | Open   | TBD              | Expected Start Phase 2 |
| Graduate Research Associate     | Open   | TBD              | Recruiting for Phase 2 |

**Table 2: UT Austin team recruiting status****B2. Coordinated the overall scientific progress, administration and finances of the project**

- Managed the compilation and delivery of data in support of the IODP CPP
- Negotiated land test location details and started associated contracts
- Coordinated meetings with ship vendors in preparation for Phase 2 Marine Test
- Developed Marine Test Scope of Work
- Managed the compilation and delivery of revised CPP
- Coordinated logistics of land test procedure, tool delivery, and personnel
- Finalized details of BP1 continuation application
- Monitored costs

**B3. Communicated with project team and sponsors**

- Project Management Plan created
- Kick-Off Meeting was held on December 11, 2014
- Several communication tools were established to ensure successful collaborative work and to exchange information
- Organized regular team meetings
  - Monthly Sponsor Meetings
  - Monthly Team Meetings
- Actively monitored project risks and as needed reported to project team and stakeholders.
- SharePoint sites developed for each project team to facilitate online communication and collaboration
- Established email list serves for key project teams
- Managed SharePoint sites developed for each project team to facilitate online communication and collaboration
- Managed email list serves for key project teams
- Managed archive/website for project deliverables
- Completed Reports (as of the end of budget period one):
  - 4 Quarterly Research Performance Progress Reports
  - Monthly Cost Accrual Reports
  - Monthly SF-425 Federal Financial Reports

#### **B4. Coordinated and supervised all subcontractors and service agreements to realize deliverables and milestones according to the work plan**

- Negotiated subawards with: Consortium for Ocean Leadership, Columbia University - Lamont-Doherty Earth Observatory, & Ohio State University School of Earth Sciences
- Actively managed subcontractors and service agreements.
- Coordinated subcontractor Statements of Work

### **3.2. Task 2: Site Analysis and Selection**

#### *A. Subtask 2.1: Site Analysis*

##### **A1. Goal:**

The Recipient will evaluate and compare prospective drilling locations using data available to the project.

The Recipient will establish (within 45 days of the award start date) and consult with an Advisory Team (expected to include, but not limited to, DOE, BOEM and the USGS) to identify and evaluate sites (each site may contain multiple potential locations), which have a high probability of containing high-saturation natural gas hydrate reservoirs and for which there is significant existing data to complement the planned research expedition.

The Recipient will collect, compile and integrate geological and geophysical data, reports and publications to constrain potential research expedition sites.

The Recipient will compile site / location information including, but not limited to, geographic location, and water depth and will use all the available information to estimate lithology, in-situ pressure and temperature conditions, approximate base of hydrate stability, gas hydrate occurrence / saturation and natural gas in-place at each potential site / location.

The Recipient will convene internal and external project workshop(s), as needed, in support of research expedition site / location screening and review.

##### **A2. Activities Phase 1:**

###### *A2.1. Established Advisory Team:*

There will be a myriad of technical decisions during the project. We will be guided in our decisions by an the Project Advisory Team (Figure 1) that will include the Project Team (UT Austin and sub-contractors), the US Department of Energy, the Bureau of Ocean Energy Management (BOEM), United States Geological Survey (USGS), and industry.

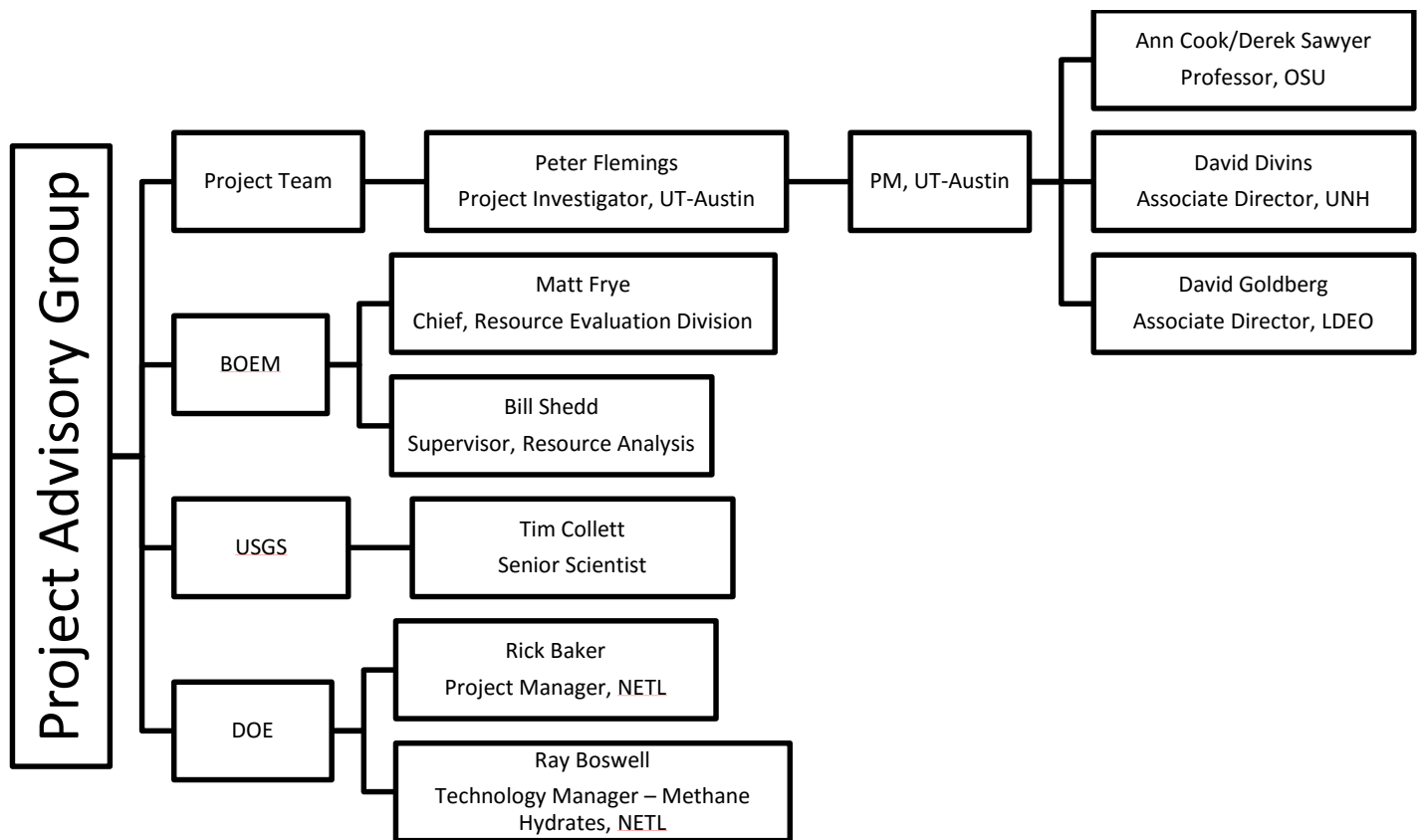


Figure 1: Project Advisory Group

*A2.2. Collected Data:*

Purchased access to the data used to interpret existing 3-D seismic data over prospective drilling locations. Seismic agreements were executed with Schlumberger WesternGeco covering the Orca Basin and Perdido areas. Schlumberger WesternGeco also provided seismic data over the former DOE – Chevron JIP project (DE-FC26-01NT41330) sites (WR313 and GC955).

Non-disclosure agreement (NDA) amendment negotiated with BP for project’s use of Mad Dog data under existing UT/Mad Dog Parties NDA

*A2.3. Compiled site / location information*

UT began building ArcGIS model encompassing data over all sites

11 sites identified – 9 primary, 2 alternate. See Figure 2 for map of site locations.

- Terrebonne (WR313) – 3 sites
- Sigsbee (GC955) – 1 site
- Mad Dog – 2 sites
- Orca – 4 sites (2 primary, 2 alternate)
- Perdido – 1 site

#### *A2.4. Project Workshops/Meetings*

- A. Site Review was held in Austin, Texas on January 27 and 28, 2015. Site Review Team members, BOEM representatives, and DOE sponsors attended
  - a. This meeting was to review the proposed sites for inclusion in the Complementary Project Proposal (CPP). The team summarized strengths and weaknesses of each proposed location and created the first draft of the CPP.
  - b. Results of this meeting contributed the results of Subtask 2.2, Task 4, Appendix A, and Appendix B.
- B. Weekly meetings held in March with proponents to discuss seismic interpretation, drilling locations, etc. Site scientific objectives, desired measurements, and tools required outlined for each potential site.

#### *B. Subtask 2.2: Site Ranking*

##### **B1. Goal:**

The Recipient, in consultation with its Advisory Team, will develop specific criteria for ranking and review of prospective research expedition sites and specific locations within each site, with a focus on achieving the overall objectives of the project in the most cost effective and efficient manner.

The Recipient, in consultation with the Project Advisory Team, will evaluate and compare all prospects identified under Subtask 2.1 using the developed ranking criteria with the intent that, at a minimum, the 3 highest ranking prospects will serve as primary drilling targets and other targets may be considered as available time / funding allows.

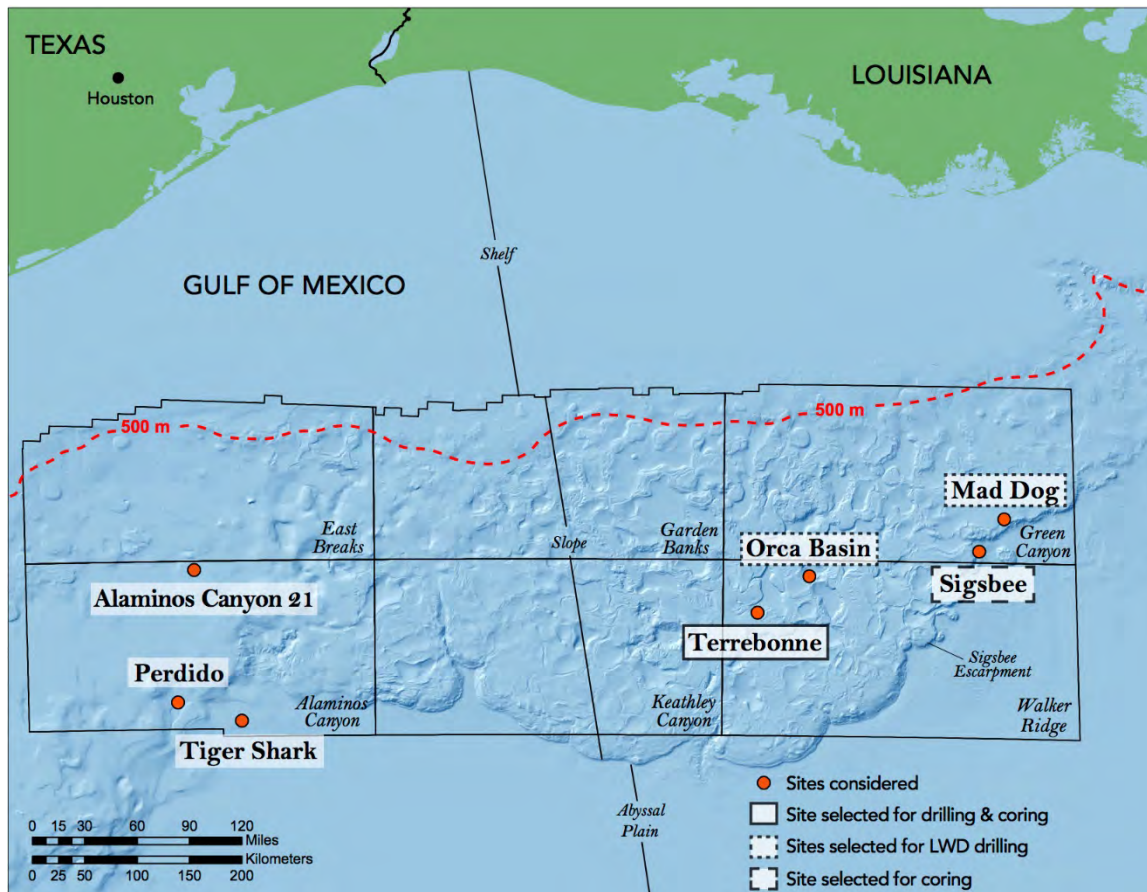
The Recipient will document both the process and resulting findings of the site analysis and ranking as a dedicated site location and ranking report.

##### **B2. Activities Phase 1:**

Over the past year, the GOM<sup>2</sup> team considered seven possible drilling and coring locations in the northern Gulf of Mexico: Alaminos Canyon 21 (AC 21), Perdido, Tiger Shark, Terrebonne, Orca Basin, Sigsbee and Mad Dog (Figure 2, Table 3). These sites were identified by a variety of different methods. Several sites, AC 21, Terrebonne and Sigsbee were previously drilled during the Gulf of Mexico Gas Hydrate Joint Industry Project (JIP) (Boswell et al., 2012) Leg 2. Mad Dog was also identified by the JIP, but not drilled (Hutchinson et al., 2010). Shedd et al. (2012) identified Orca Basin as having a prominent, discontinuous BSR. Perdido and Tiger Shark (Boswell et al., 2009) were previously drilled by the oil and gas industry and measured well logs suggested significant gas hydrate accumulations.

During the fall and winter of 2014-2015, we collected and assessed scientific well log data, public industry log data, published articles and public reports on each available site. ArcGIS maps were compiled at Ohio State of the hydrate and industry wells drilled at each site, the extent of known BSRs from Shedd et al., (2012), and previous seismic data extent. The maps, available data, and published papers and reports were put on SharePoint for the project advisory group to assess. Several discussions and presentations were held on each site, within PI meetings in the fall of 2014 and at the proposal writing meeting at UT Austin in January of 2015. Two criteria was selected as important for the drilling project: 1) the likelihood of finding high saturation gas hydrate in sand systems at the site and 2) the potential to provide further understanding about gas hydrate in sand systems in the Gulf of Mexico. From this data and from the subsequent discussions. From this data and from the subsequent discussion, we decide to eliminate two sites: AC 21 and Tiger Shark.





**Figure 2: The seven sites considered for drilling and/or coring as a part of GOM2**

The JIP Leg 2 drilled two logging-while-drilling (LWD) holes at AC 21 in 2009 (Boswell et al., 2012). The target was a prominent, extensive, positive amplitude reflector within the gas hydrate stability zone (GHSZ), which could indicate a gas hydrate filled sand layer. A thick sand layer was identified in both AC 21 LWD holes, however, it still remained unclear of the sand contained gas hydrate due to a severe wash out in the target sand. A washout in a hydrate-filled sand is unusual, as hydrate generally contributes to sediment stability and an in-gauge borehole. It could be that a low-saturation of hydrate occurs in the layer, as a small increase in both resistivity and compressional velocity was measured in the layer; Lee et al., (2012) suggest this layer contains ~20% gas hydrate saturation. Others argue that the small increase in resistivity and compressional velocity can be explained by a lower porosity in the sand layer and gas hydrate likely does not occur (Cook and Tost, 2014). Aside from the uncertainty surrounding the occurrence of gas hydrate, the GOM<sup>2</sup> team was also concerned about the ability to collect and recover sand sediment in a core barrel, as this is often an issue for scientific ocean drilling (Kominz et al., 2011). Further LWD or wireline logging would most likely not be able to resolve whether gas hydrate occurs in the sand layer. For these reasons, AC 21 was removed from the list of potential sites.

Chevron drilled the Tiger Shark exploration well in Alaminos Canyon Block 818 in 2004 (Boswell et al., 2009). A 13-m thick sand was encountered near the base of the GHSZ that contained high saturations of gas hydrate. Hydrate was not recovered from the well, but sidewall cores were taken in the hydrate-bearing formation. The sidewall cores confirmed the hydrate-bearing unit is part of the Frio Sand, an Oligocene volcanoclastic sand. The Frio is considerably older and a significantly different lithologic type (as it is composed of volcanoclastic sand with a low grain density) than most shallow sand units in the northern Gulf of Mexico within the GHSZ. This suggested that the information learned by further drilling at Tiger Shark might not be applicable to other areas within the Gulf of Mexico. In addition, the Tiger Shark gas hydrate accumulation is positioned on the top of a large, steeply dipping anticlinal fold raising serious concerns that

drilling Tiger Shark could be dangerous without a riser. For these reasons, Tiger Shark was eliminated from possible drilling sites.

Following the elimination of the first two sites, seismic data and industry well log data was ordered or obtained over our five remaining sites of interest: Perdido, Terrebonne, Orca Basin, Sigsbee and Mad Dog. At each site, extensive mapping of each of the prospects took place. Maps of the aerial extent and the amplitude of the BSR were produced, maps of interest horizons (especially, horizons with phase reversals), and maps of the gas legs below the BSR. In areas with multiple horizons of interest, such as Orca Basin and Terrebonne, amplitude maps were overlaid to see where optimal drilling locations could be placed to target several sands with one hole. Based on these amplitude maps and integration with the well data, potential drilling locations were selected by the team in mapping team discussions and each site was included as a primary site in our March 2015 (initial submission) proposal submission to the International Ocean Discovery Program (IODP).

In July of 2015, we received the reviews of our March 2015 IODP proposal. There were two main comments that affected our site selection process. First, the reviewers thought it was not always clear what hypothesis or questions we were asking at each site because our priorities at each site were varied. Second, the reviewers noted that including all five sites would result in a cruise that was much too long, and that further refinement of the sites and drilling plan was needed.

To address these concerns, we chose to focus our IODP proposal questions on methane migration mechanisms within sand and we identified sites with observable seismic patterns in dipping sand bodies suggestive of gas hydrate occurrence for the October 2015 IODP proposal resubmission. The observable seismic patterns consist of these three traits: a strong, leading-positive reflection above the GHSZ or bottom simulating reflector (BSR), a phase reversal at the BSR, and a leading negative below the BSR. The Terrebonne location, drilled by JIP Leg 2 in 2009 and originally named 'WR 313', has two dipping sand bodies, the Blue Sand and the Orange Sand, that have both the observable seismic pattern as well as confirmed high gas hydrate saturations in the GHSZ (Boswell et al., 2012; Frye et al., 2012). It is highly likely that the observable seismic pattern is caused by high saturation of gas hydrate within the GHSZ, and free gas in the sand below the GHSZ.

Terrebonne is considered our main drilling and coring site because high gas hydrate saturations occur in dipping sand units. In the October 2015 IODP proposal, we propose to drill 2-3 locations at Terrebonne to recover gas hydrate samples from the Blue and Orange Sands preserved in pressure cores, to study the geochemical, microbiological and physical properties of these samples. We will also measure basic physical properties in the Blue and Orange Sands in hole, including permeability, shear velocity, temperature and pore pressure.

Two other sites, Orca Basin and Mad Dog, also have similar observable seismic patterns as Terrebonne, where a leading-positive reflector changes phase at the BSR. This suggests that Orca Basin and Mad Dog also have gas hydrate filled sands within the gas hydrate stability zone, however, this hypothesis is unconfirmed. To confirm that gas hydrate occurs in sand at Mad Dog and Orca Basin, we proposed in the October 2015 IODP proposal to drill two LWD holes at each site, targeting the strong positive reflectors with the GHSZ. If either Mad Dog or Orca Basin yields high saturation gas hydrate within coarse-grained units, we may return to the site for further coring instead of drilling and coring a third hole at Terrebonne.

The Sigsbee location, first drilled by JIP Leg 2 in 2009 and called 'GC 955', also has a sand unit with high saturation gas hydrate (Boswell et al., 2012). Unlike Terrebonne, Sigsbee does not have a dipping sand layer that causes a phase reversal at the BSR. Instead, Sigsbee is an uplifted, highly faulted 4-way closure. For this reason, we have relisted Sigsbee on our October 2015 IODP proposal as an alternate site. More importantly, Sigsbee has been selected as the primary location to test the pressure-coring tool before the IODP cruise. Currently, 10 pressure cores are planned to be collected in the Sigsbee sand reservoir in spring or fall of 2016.

Lastly, the Perdido location was removed from the possible sites. It is unclear if a gas hydrate filled sand occurs at Perdido, as one interpretation of the measured industry well logs suggest Perdido may have a gas hydrate filled sand, while another interpretation of the well log patterns indicate Perdido may only contain gas hydrate in fractured mud. Moreover, Perdido does not have the observable seismic pattern indicating gas hydrate in a dipping sand unit, and thus, lacks similarity to the main sites in the October 2015 IODP proposal: Terrebonne, Mad Dog and Orca Basin.

In October 2015, additional 3D seismic data was ordered for the Orca Basin, because the optimal drilling locations selected were near the edge of the seismic data set. The new seismic data has several promising locations and the proposed drilling locations will likely be moved from those proposed in the October 2015 drilling proposal.

| Sites                     | Previous drilling in area of interest | Gas hydrate in sand confirmed | Gas hydrate suspected in sand | Outcome   |
|---------------------------|---------------------------------------|-------------------------------|-------------------------------|---|
| <b>Alaminos Canyon 21</b> | JIP Leg 2, AC 21                      | possibly                      | possibly                      | Eliminated because of lack of confirmed hydrate in sand. There were additional concerns that coring in the potential hydrate sand may be difficult because the sand easily washes out.          |
| <b>Perdido</b>            | Statoil, 2001                         | possibly                      | possibly                      | Eliminated because of lack of confirmed hydrate in sand and no phase reversal. There is some evidence that this site contains only gas hydrate in fractured muds.                               |
| <b>Tiger Shark</b>        | Chevron, 2004                         | yes                           |                               | Eliminated because of drilling safety questions, deep water, and strong concerns that Tiger Shark does not represent a typical GOM hydrate accumulation because of reservoir age and lithology. |
| <b>Terrebonne</b>         | JIP Leg 2, WR 313                     | yes                           |                               | Selected for drilling & coring because high saturation gas hydrate found in multiple sand layers.   |
| <b>Orca Basin</b>         |                                       |                               | yes, phase reversal           | Selected for LWD drilling because of strong phase reversal and high amplitudes within stability zone.   |
| <b>Sigsbee</b>            | JIP Leg 2, GC 955                     | yes                           |                               | Selected for pressure coring because gas hydrate was found in high saturation in a thick, ~30 m sand unit.  |
| <b>Mad Dog</b>            |                                       |                               | yes, phase reversal           | Selected for LWD drilling because of strong phase reversal and high amplitudes within stability zone.   |

Table 3: Summary of sites considered for GOM<sup>2</sup> including reasons for elimination or selection.

### 3.3. Task 3: Develop Pre-Expedition Drilling/Logging/Coring/Sampling Operational Plan

#### A. Goal:

For the three sites / locations identified in Subtask 2.2, The Recipient will develop, in consultation with the project Advisory Team, pre - expedition drilling / logging / coring / sampling Operation Plan.

This Operational Plan will use the geologic and geophysical information compiled during Subtask 2.1 (as well as any additional information considered to be necessary) to identify appropriate equipment and determine whether there are any gaps in technology that must be addressed for drilling of wells, the collection of pressure cores, conventional cores,

sidewall cores and in situ temperature & pressure measurements and will identify the specific shipboard science and measurements that will need to be performed.

The developed Operational Plan for each site will include, but not be limited to, a time estimate for vessel transit, drill string assembly, the performance of planned drilling, logging, coring and sampling operations and the shipboard science and measurements to be performed.

#### *B. Activities Phase 1:*

The operational plan describes how this project will drill for and test the geothermal resources at those sites defined in Subtask 2.2 as part of the CPP. See Appendix A for Preliminary Operational Plan. This is a preliminary operational plan and will change in Phase 2 of this project.

### **3.4. Task 4: Complete and Update IODP CPP Proposal**

#### *A. Goal:*

The Recipient will complete and update, as necessary, a Complementary Project Proposal (CPP) for the International Ocean Drilling Program (IODP) based on the sites identified in Task 2 and the preliminary Operational Plan developed in Task 3. The goal of this CPP proposal will be to gain access to a research vessel suitable for conducting the planned research expedition. This CPP proposal will include all content required by the CPP proposal process. The proposal will include, at a minimum, a summary of the science proposed, detailed identification of the sites / locations to be drilled, and specification of the planned scientific program to be conducted.

The Recipient will develop the CPP proposal in consultation with the established Project Advisory Team.

The initial CPP proposal will be submitted in accordance with deadlines established by the IODP (estimated to be in late March 2015) and modified or updated as necessary or as requested by IODP.

The Recipient will document the submitted IODP CPP proposal and relevant content to DOE via normal project quarterly progress reporting.

#### *B. Activities Phase 1:*

Science in IODP is driven by community-generated proposals targeting the research themes outlined in the program's overall Science Plan, Illuminating Earth's Past, Present, and Future ([www.iodp.org/program-documents](http://www.iodp.org/program-documents)). Because the level of investment per expedition goes beyond an individual researcher or research group, the proposal structure, review and planning processes are comprehensive and differ from those applied to typical grant applications. The biggest difference is that the IODP process is somewhat iterative and open to communication between the science proponents, the advisory panels, and the drilling platform operators. It is a process designed to transform exciting science into successful expeditions.

A Complementary Project Proposal (CPP) is a Full Proposal that has a commitment from a third party source for a substantial amount of financial support. Expeditions arising from such proposals follow the normal IODP rules and the IODP Sample, Data and Obligations Policy that defines the data moratorium, data access, and publication responsibilities. The level of scientific staffing for the entity contributing the CPP funds is negotiated on a case-by-case basis.

An IODP CPP is a desirable option to conduct our field program. The detailed technical planning permitting, implementation, and financial responsibilities for ship operations are managed within IODP. In addition, CPPs can receive fast-track consideration if required by the situation (e.g., funding source, operational plans, etc.).

The Science Evaluation Panel (SEP) reviews all IODP proposals, together with external reviews, and any additional information requested and provided by the proponents. They also review all available site survey data to characterize the completeness and adequacy of the data. The SEP then decides whether the proposal should advance to the JR Facility Board for possible implementation.

The final decision whether a proposal will be implemented is made by the JR Facility Board. During consideration by the Facility Board, the Full Proposal may be subject to additional requirements (for example, safety review by the Environmental Protection and Safety Panel) and must satisfy all additional conditions made by the Facility Board before it can be implemented.

Below are summarized the activities accomplished during Phase 1 with respect to the submission of the IODP CPP proposal.

Held meetings to develop text of proposal, coordinate progression of analysis, documentation, and submittal.

1. Various phone/web meetings held throughout Phase 1
2. Proposal Writing Workshop, January 2015
3. CPP Writing Workshop, August 2015

Submitted CPP to IODP, uploaded and categorized the data available for the IODP SSDB, created materials for IODP CPP meeting. Revised seismic, log and bathymetric data at Terrebonne and Sigsbee locations in support of CPP proposal.

1. First submittal of CPP, April 2015
2. Uploaded data to IODP SSDB, May 2015
3. Revised submittal of CPP, October 2015
4. Data upload for revised submittal to be completed in Phase 2

Final submittal from October 2015 attached: Appendix B: Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope (GOM<sup>2</sup>)

### **3.5. Task 5: Pressure Coring and Core Analysis System Modification and Testing**

The Recipient will initiate planning, modification or upgrade, lab, and/or land-based testing of pressure coring and core analysis tools, as deemed necessary by mutual agreement of the Recipient, DOE and the Project Advisory Team, to assure the readiness of the systems for use in the planned research expedition or post-expedition analysis efforts, as defined within the initial Operational Plan (Task 3). Continuation of these efforts may extend into Phase 2 of the project.

#### ***A. Subtask 5.1: Pressure Coring Tool with Ball (PCTB) Scientific Workshop***

##### **A1. Goal:**

The Recipient will organize a Scientific Planning workshop to assist with planning the pressure coring strategy and scientific objectives for the Task 5.0. The goals of the workshop are to:

- review scientific, technical and logistical goals of the DOE drilling experiment;
- review recent scientific achievements in pressure coring;
- review current pressurized coring capabilities;

- develop Science Plan for DOE Drilling;
- shipboard science, and sampling
- shore-based analysis
- develop a project team composed of scientists and institutions enthused with participating in research program.

## **A2. Activities Phase 1:**

On March 9th and 10th, 2015, 27 scientists and engineers from around the world held a Scientific Planning workshop to assist with planning the pressure coring strategy and scientific objectives for the Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope Project (“GOM<sup>2</sup>”). For details and results of that workshop, see Appendix C: Methane Hydrate Pressure Coring and Analysis: Gulf of Mexico Scientific Planning Workshop Report

### *B. Subtask 5.2: Pressure Coring Tool with Ball (PCTB) Lab Test*

#### **B1. Goal:**

The Recipient will perform a Lab Test of the PCTB tool to obtain a high degree of confidence in overall PCTB operation with focus on pressure retention. The Recipient will carry out full function lab test using bench test apparatus, recording PCTB internal pressures using fish pills, and recording retained pressure using pressure gauges or pressure transducers. The function test will be repeated until a high degree of confidence in overall PCTB operation, with focus on pressure retention, is obtained.

#### **B2. Activities Phase 1:**

Completed lab testing of the PCTB tool see Appendix D: Hybrid Pressure Coring System (PCTB) 2015 Laboratory Test Program Final Report

### *C. Subtask 5.3: Pressure Coring Tool with Ball (PCTB) Land Test Prep*

#### **C1. Goal:**

The Recipient will perform all necessary activities in preparation for the Land Test of the PCTB tool. This includes but is not limited to; contracting vendors, tool modifications, developing a test plan, and shipping tool.

#### **C2. Activities Phase 1:**

Performed all necessary activities in preparation for the Land Test of the PCTB tool. Activities included contracting vendors, tool modifications, developing a test plan, and shipping tool.

- Finalized Statement of Work for Schlumberger Cameron Test and Training Facility (CTTF). Completed necessary University paperwork to execute contract.
  - Schlumberger provided use of CTTF a full-scale test facility and staff to test Pressure Coring Tool with Ball (PTBC) coring and logging technology under actual wellsite conditions in a controlled and confidential environment. The knowledge gained by this rigorous assessment helps meet the requirements of the Department of Energy project objectives and improve tool design prior to actual wellsite execution.
- Finalized Statement of Work for Geotek Coring Inc. (GCI)

- Geotek Coring Inc. provided fabrication, engineering development, and testing of the Department of Energy (DOE) Hybrid Pressure Coring System (HPCS) as it relates to the project scientific assessment and production potential. This includes sea trial, staff training, and a feasibility study.
- Finalized Statement of Work for Pettigrew Engineering, PLLC
  - Pettigrew provided engineering development, and testing of the Department of Energy (DOE) Hybrid Pressure Coring System (HPCS) as it relates to this project objectives.
- Refined experimental plan.
- Tool Modifications
  - Meetings were held with subject experts and vendors to review tool issues and discussed possible modifications. We discussed possible modifications, the effects of mud weight, and filtrates.
  - Envisioned Plan Forward: First, we will determine which modifications can be performed prior to the field test. Second, we will work with subject matter experts to decide which of the longer term modifications to pursue.
- Tool Fabrications
  - Concurrent to discussions of tool modification we are moving forward with fabricating a 9 7/8 bit, bit sub, and stabilizer to be used in the BP2 Land Test.
  - There is a broad consensus that a narrower bit is advisable. Pettigrew calculated the annular velocities for a 10-5/8 BHA and a 9-7/8 BHA and got some interesting results. The annular velocity past the 8-1/2 drill collars is increased ~60% by going to a 9-7/8 BHA. Similarly, the annular velocity past 5-1/2 drill pipe is increased ~20% by going to a 9-7/8 BHA. Therefore, it is advisable we go with a 9-7/8 BHA for the sea trial and full deployment.
  - In terms of the ID (inner diameter), nothing changes. Thus, there is no impact on the ability to pass other tools.
  - The outer diameter (OD) of the main bit will change from 10-5/8" to 9-7/8". The bit sub will be rebuilt so that its apparent OD will drop 10-5/8" to 9-7/8".
  - We will build a 9-7/8" stabilizer that will go right on top of the outer core barrel assembly.
  - We are adding the new stabilized bit sub (4' long), the new bit (order 18" long), and adding the stabilizer.
- Created service van and pipe shipping plan

## 4. Products Developed

### 4.1. Publications, conference papers, and presentations

Cook, A., Sawyer, D., Accepted, August 31, The mud sand crossover on marine seismic data, Geophysics.

Cook, A., Hillman, J., Sawyer, D., 2015, Gas migration in the Terrebonne Basin gas hydrate system, Abstract OS23D-05 to be presented at 2015, Fall Meeting, AGU, San Francisco, CA, 14-18 Dec.

Meazell, K., 2015, Methane hydrate-bearing sediments in the Terrebonne basin, northern Gulf of Mexico , Abstract OS23B-2012 to be presented at 2015 Fall Meeting, AGU, San Francisco, CA., 14-18 Dec.

Phillips, S.C., Flemings, P.B., Meyer, D.W., You, K., Kneafsey, T.J., Germaine, J.T., Solomon, E.A., and Kastner, M., 2016, Extraction of pore fluids at in situ pressures from methane hydrate experimental vessels, Poster to be presented at 2016 Gordon Research Conference from Feb28 to Mar04 in Galveston, TX, United States.

#### 4.2. Website(s) or other Internet site(s)

Project Website: <http://ig.utexas.edu/energy/genesis-of-methane-hydrate-in-coarse-grained-systems/>

Project SharePoint: <https://sps.austin.utexas.edu/sites/GEOMech/doehd/teams/>

## 5. References

- Boswell, R., Collett, T. S., Frye, M., Shedd, W., McConnell, D. R., and Shelander, D., 2012, Subsurface gas hydrates in the northern Gulf of Mexico: Marine and Petroleum Geology, v. 34, no. 1, p. 4-30.
- Boswell, R., Shelander, D., Lee, M., Latham, T., Collett, T., Guerin, G., Moridis, G., Reagan, M., and Goldberg, D., 2009, Occurrence of gas hydrate in Oligocene Frio sand: Alaminos Canyon Block 818: Northern Gulf of Mexico: Marine and Petroleum Geology, v. 26, no. 8, p. 1499-1512.
- Cook, A. E., and Tost, B. C., 2014, Geophysical signatures for low porosity can mimic natural gas hydrate: An example from Alaminos Canyon, Gulf of Mexico: Journal of Geophysical Research: Solid Earth, v. 119, no. 10, p. 2014JB011342.
- Frye, M., Shedd, W., and Boswell, R., 2012, Gas hydrate resource potential in the Terrebonne Basin, Northern Gulf of Mexico: Marine and Petroleum Geology, v. 34, no. 1, p. 150-168.
- Hutchinson, D., Boswell, R., Collett, T., Chun Dai, J., Dugan, B., Frye, M., Jones, E., McConnell, D., Rose, K., Ruppel, C., Shedd, W., Shelander, D., and Wood, W. T., 2010, Gulf of Mexico Gas Hydrate Joint Industry Project Leg II: Green Canyon 781 Site Selection.
- Kominz, M. A., Patterson, K., and Odette, D., 2011, Lithology Dependence of Porosity In Slope and Deep Marine Sediments: Journal of Sedimentary Research, v. 81, no. 10, p. 730-742.
- Lee, M. W., Collett, T. S., and Lewis, K. A., 2012, Anisotropic models to account for large borehole washouts to estimate gas hydrate saturations in the Gulf of Mexico Gas Hydrate Joint Industry Project Leg II Alaminos Canyon 21 B well: Marine and Petroleum Geology, v. 34, no. 1, p. 85-95.
- Shedd, W., Boswell, R., Frye, M., Godfriaux, P., and Kramer, K., 2012, Occurrence and nature of “bottom simulating reflectors” in the northern Gulf of Mexico: Marine and Petroleum Geology, v. 34, no. 1, p. 31-40.

## 6. Acronyms

|        |  |
|--------|--|
| AIST   | National Institute of Advanced Industrial Science and Technology |
| APC    | Advanced Piston Corer  |
| APCT-3 | Advanced Piston Corer Temperature-3                              |
| BHA    | Bottom Hole Assembly   |
| BOEM   | Bureau of Ocean Energy Management                                |
| BP1    | Budget Period 1  |
| BP2    | Budget Period 2  |
| BSR    | Bottom Simulating Reflector                                      |
| CKOH   | Carolyn A. Koh   |



|                  |   |
|------------------|---|
| cm               | Centimeter  |
| CPP              | Complementary Project Proposal  |
| CRS              | Constant Rate of Strain   |
| CT scan          | Computed Tomography scan  |
| CTTF             | Cameron Test and Training Facility  |
| DOE              | Department of Energy  |
| FFF              | Free-Fall Funnel  |
| FMI              | Formation Microimager   |
| GC               | Gas Chemistry   |
| GCI              | Geotek Coring Inc.  |
| GH               | Gas hydrate   |
| GHSZ             | Gas hydrate stability zone  |
| GOM <sup>2</sup> | Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope |
| HBS              | Hydrate-Bearing Sediments   |
| hr               | Hour  |
| HYACE            | Hydrate Autoclave Coring Equipment  |
| ID               | Inner Diameter  |
| IODP             | International Ocean Discover Program  |
| IPTC             | Instrumented Pressure Testing Chamber   |
| JIP              | Joint Industry Project  |
| JOGMEC           | Japan Oil, Gas and Metals National Corporation                                      |
| JR/JRSO          | Joides Resolution/Joides Resolution Science Operator                                |
| KIGAM            | Korean Institute of Geoscience and Mineral Resources                                |
| LBNL             | Lawrence Berkeley National Laboratory   |
| LDEO             | Lamont–Doherty Earth Observatory  |
| LPSA             | Laser particle size analyzer  |
| LWD              | Logging-While-Drilling  |
| m                | Meter   |
| MAD              | Moisture and Density  |
| MAS-NMR          | Magic-angle spinning in nuclear magnetic resonance                                  |
| mbsf             | Meters below sea floor  |
| MDT              | Modular Dynamics Test   |
| micro-CT         | Micro Computed Tomography   |

|         |   |
|---------|---|
| MPU     | Milne Point Unit  |
| MSCL    | Multi-sensor core logger                                  |
| NDA     | Non-disclosure agreement                                  |
| NEPA    | National Environmental Policy Act                         |
| NETL    | National Energy Technology Laboratory                     |
| NMR     | Nuclear magnetic resonance                                |
| OD      | outer diameter  |
| OSU     | Ohio State University                                     |
| PBU     | Prudhoe Bay Unit  |
| PCATS   | Pressure Core Analysis and Transfer System                |
| PCB     | Pressure Core Barrel                                      |
| PCCT    | Pressure Core Characterization Tools                      |
| PCS     | Pressure Core Sampler                                     |
| PCTB    | Pressure Coring Tool with Ball                            |
| PMP     | Project Management Plan                                   |
| PNATs   | Pressure-core Nondestructive Analysis Tools               |
| TACTT   | Transparent Acrylic Cell Triaxial Testing System          |
| PTCS    | Pressure Temperature Core Sampler                         |
| RCB     | Rotary Core Barrel  |
| RIH     | Run in hole   |
| SCIMPI  | Simple Cabled Instrument for Measuring Parameters In-Situ |
| SEM     | Scanning Electron Microscopy                              |
| SSDB    | Site Survey Data Bank                                     |
| T2P     | Temperature 2 Pressure Probe                              |
| TCF     | Trillion Cubic Feet                                       |
| TD      | Total depth   |
| UNH     | University of New Hampshire                               |
| US      | United States   |
| USGS    | United States Geological Survey                           |
| UT      | The University of Texas                                   |
| UTIG    | The University of Texas Institute for Geophysics          |
| XCB     | Extended Core Barrel                                      |
| XRD/XRF | X-ray diffraction/X-ray fluorescence                      |



## Appendix A: Operational Plan

### Introduction

The purpose of the preliminary Operational Plan is to define and scope the technical activities required to achieve the scientific goals of our proposal. The selected set of measurement technologies included in this plan draws on the operational and technical successes of the previous industry, academic, and national methane hydrate field programs, including the International Ocean Discovery Program (and its predecessors), the U.S. Gulf of Mexico Joint Industry Projects (JIP Legs 1 and 2), the India National Gas Hydrate Program NGH01/NGH02 expeditions, the Mallik drilling and testing project in northern Canada, and the Mount Elbert and Ignik-Sikumi drilling projects in northern Alaska. Based on these experiences, we determine the critical measurements and technologies required to address the scientific objectives at each of the target sites to be drilled, including but not limited to, the collection of pressure cores, conventional cores, conventional and enhanced wireline logging, Logging-While-Drilling (LWD) operations, in situ temperature measurements, in situ fluid sampling, and pressure drawdown and permeability testing. In many of these previous hydrate drilling projects, LWD operations preceded coring and wireline logging to allow for detailed pressure coring and well testing plans to be designed. This Operational Plan makes use of the same approach at all of the proposed sites and has been partitioned into Stage 1 (LWD) and Stage 2 (coring and wireline) operations.

The Operational Plan also provides a timeline for our proposed field program, establishing boundaries for the amount of work that can be completed in the time available and allowing for the most efficient use of that time. To be consistent with the baseline requirements of a Complementary Project Proposal (CPP) for the International Ocean Discovery Program (see section 3.4), we have also prepared this Operational Plan to meet all requirements of that program and in close consultation with the IODP Science Operator at Texas A&M University and the IODP Science Evaluation (SEP) review process. In particular, the Operational Plan preserves the 56-day timeline of a standard IODP scientific expedition, as per SEP recommendation, and identifies alternate sites locations (three) and proposed activities in the event that any of the primary sites (seven) are not completed as planned or as time becomes available. Other specific and unique requirements related to the measurement technologies for achieving our scientific goals (i.e., deployment of large-diameter drilling pipe or ROVs from *D/V JOIDES Resolution*) have been discussed with IODP Science Operator and are included in the Operational Plan to the extent possible at this time. On this basis, the Operational plan also provides a framework to consider the technical and scientific staffing needs and logistical consideration for the proposed expedition.

The preliminary Operational Plan with technical and rig time details at each primary site are provided below. Alternate site locations are discussed in Section 2.2. These will be revised as needed until the actual drilling program.

### Operational Plan

The expedition is designed as a standard 56-day scientific IODP expedition (Tables 4 & 5). The scientific objectives are best achieved with 2 operational stages. Stage 1 will drill four (4) LWD holes at Mad Dog and Orca. Stage 2 will conduct coring and wireline operations at three Terrebonne locations. Coring will feature conventional APC/XCB and RCB coring tools, as many as 30 PBCT runs, wireline logging (including NMR and penetrometer testing), in situ fluid sampling and short-duration formation pressure tests using the MDT straddle packer tool.

This two-stage strategy allows for shipboard analysis of the LWD results prior to Stage 2 and improved time and cost efficiency with mid-expedition personnel and equipment transfers. We estimate preliminary rig times for Stage 1 and 2 activities, including transits, BHA assembly, and drilling, coring, and logging operations (Tables 4 & 5). These estimates are computed using the Coring Time Estimator with input from the JR Science Operator at Texas A&M University.

**Stage 1 Activities**

We will drill four (4) LWD holes at Mad Dog and Orca sites. The LWD program will include: geoVISION (GVR), EcoScope, SonicScope, SonicVision, TeleScope, and ProVision-Plus (NMR) tools. Based on prior experience, operational times are estimated assuming that LWD tools are combined and penetrate at 25 m/hr (average) in a separate lowering at each site.

**Stage 1 Operations**

| Operations                                  | Mob &   | Set Up | Downhole |       | Cum. | Cum. |
|---|---------|--------|----------|-------|------|------|
|   | Transit |        | Coring   | Meas. | Time | Time |
|   | (hr)    | (hr)   | (hr)     | (hr)  | (hr) | Days |
| Mobilization, Galveston TX and transit      | 44.0    |        |          |       | 44.0 | 1.8  |
| <b>MADOG-01A: Drill and LWD to 648 mbsf</b> |         |        |          |       |      |      |
| -LWD rig up                                 |         | 6.0    |          |       | 6.0  | 0.3  |
| -Run pipe to SF                             |         | 4.5    |          |       | 10.5 | 0.4  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |          | 30.9  | 41.4 | 1.7  |
| -Pull pipe                                  |         | 3.7    |          |       | 45.1 | 1.9  |
| Total, incl. transit to MADOG-02A (3.0 nmi) | 3.0     |        |          |       | 48.1 | 2.0  |
| <b>MADOG-02A: Drill and LWD to 607 mbsf</b> |         |        |          |       |      |      |
| -LWD rig up                                 |         | 6.0    |          |       | 6.0  | 0.3  |
| -Run pipe to SF                             |         | 4.6    |          |       | 10.6 | 0.4  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |          | 29.3  | 39.9 | 1.7  |
| -Pull pipe                                  |         | 3.7    |          |       | 43.6 | 1.8  |
| Total, incl. transit to ORCA-03A (61 nmi)   | 5.8     |        |          |       | 49.4 | 2.1  |
| <b>ORCAB-03A: Drill and LWD to 619 mbsf</b> |         |        |          |       |      |      |
| -LWD rig up                                 |         | 6.0    |          |       | 6.0  | 0.3  |
| -Run pipe to SF                             |         | 5.3    |          |       | 11.3 | 0.5  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |          | 29.8  | 41.1 | 1.7  |
| -Pull pipe                                  |         | 4.4    |          |       | 45.5 | 1.9  |
| Total, incl. transit to ORCAB-04A (0.5 nmi) | 0.5     |        |          |       | 46.0 | 1.9  |
| <b>ORCAB-04A: Drill and LWD to 695 mbsf</b> |         |        |          |       |      |      |
| -LWD rig up                                 |         | 4.0    |          |       | 4.0  | 0.2  |
| -Run pipe to SF                             |         | 5.2    |          |       | 9.2  | 0.4  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |          | 32.8  | 42.0 | 1.7  |
| -Pull pipe                                  |         | 4.4    |          |       | 46.4 | 1.9  |
| Total, incl. transit to TBONE-01A (22 nmi)  | 2.1     |        |          |       | 48.5 | 2.0  |

**Table 4: GOM2 CPP Preliminary Operational Plan and Time Estimates for Stage 1 drilling. Times are based on the JRSO Time Estimator and recent hydrate drilling experience. Average rate assumptions: LWD 25 m/hr; APC 9 m/hr; RCB/XCB 4.5 m/hr; PCBT 4 hr/run; penetrometer 2 hr/run; RIH, Pulling pipe 560 m/hr.**

**Stage 2 Operations**

| Operations                                    | Mob &       | Downhole    |              |              | Cum.          | Cum.        |
|---|-------------|-------------|--------------|--------------|---------------|-------------|
|   | Transit     | Set Up      | Coring       | Meas.        | Time          | Time        |
|   | (hr)        | (hr)        | (hr)         | (hr)         | (hr)          | Days        |
| <b>TBONE-01A: Drill and Core to 940 mbsf</b>  |             |             |              |              |               |             |
| -Coring setup                                 |             | 4.0         |              |              | 4.0           | 0.2         |
| -Run pipe to SF                               |             | 5.5         |              |              | 9.5           | 0.4         |
| -Hole A: APC/XCB core to 500mbsf              |             |             | 88.9         |              | 98.4          | 4.1         |
| -Acquire 10 PCTB cores (upper sand & blue)    |             |             | 40.0         |              | 138.4         | 5.8         |
| -Hole B: RCB core 500m to TD                  |             |             | 124.8        |              | 263.2         | 11.0        |
| -Wireline penetrometer at ~8 depths to TD     |             |             |              | 17.6         | 280.8         | 11.7        |
| -Deploy FFF and Re-enter with lg pipe         |             |             | 19.3         |              | 300.1         | 12.5        |
| -GR logging & MDT formation testing           |             |             |              | 44.0         | 344.1         | 14.3        |
| -Pull pipe                                    |             | 5.7         |              |              | 349.8         | 14.6        |
| Total, incl. transit to TBONE-02A (1.0 nmi)   | 1.0         |             |              |              | 350.8         | 14.6        |
| <b>TBONE-02A: Drill and Core to 850 mbsf</b>  |             |             |              |              |               |             |
| -Coring setup                                 |             | 4.0         |              |              | 4.0           | 0.2         |
| -Run pipe to SF                               |             | 5.5         |              |              | 9.5           | 0.4         |
| -Hole A: APC/XCB core to 500mbsf              |             |             | 88.9         |              | 98.4          | 4.1         |
| -Acquire 10 PCTB cores (upper sand & blue)    |             |             | 40.0         |              | 138.4         | 5.8         |
| -Hole B: RCB core 500m to TD                  |             |             | 104.7        |              | 243.1         | 10.1        |
| -Wireline penetrometer at ~8 depths to TD     |             |             |              | 17.6         | 260.7         | 10.9        |
| -Hole C: wash to TD and Re-enter with lg pipe |             |             | 53.1         |              | 313.8         | 13.1        |
| -NMR + GR logging & MDT testing               |             |             |              | 58.0         | 371.8         | 15.5        |
| -Pull pipe                                    |             | 5.6         |              |              | 377.4         | 15.7        |
| Total, incl. transit to TBONE-03A (3.0 nmi)   | 3.0         |             |              |              | 380.4         | 15.8        |
| <b>TBONE-03A: Drill and Core to 940 mbsf</b>  |             |             |              |              |               |             |
| -Coring setup                                 |             | 4.0         |              |              | 4.0           | 0.2         |
| -Run pipe to SF                               |             | 5.6         |              |              | 9.6           | 0.4         |
| -Hole A: APC/XCB core to 500mbsf              |             |             | 88.9         |              | 98.4          | 4.1         |
| -Acquire 10 PCTB cores (upper sand & blue)    |             |             | 40.0         |              | 138.4         | 5.8         |
| -Hole B: RCB core 500m to TD                  |             |             | 124.9        |              | 263.3         | 11.0        |
| -Wireline penetrometer at ~8 depths to TD     |             |             |              | 17.6         | 280.9         | 11.7        |
| -Deploy FFF and Re-enter with lg pipe         |             |             | 19.4         |              | 300.3         | 12.5        |
| -GR logging & MDT formation testing           |             |             |              | 44.0         | 344.3         | 14.3        |
| -Pull pipe                                    |             | 5.7         |              |              | 350.0         | 14.6        |
| Total, incl. transit/demob in Galveston TX    | 25.7        |             |              |              | 375.7         | 15.7        |
| <b>Total Times (hr):</b>                      | <b>85.1</b> | <b>89.8</b> | <b>832.9</b> | <b>288.8</b> | <b>1342.9</b> | <b>56.0</b> |

Table 5: GOM2 CPP Preliminary Operational Plan and Time Estimates for Stage 2 drilling. Times are based on the JRSO Time Estimator and recent hydrate drilling experience. Average rate assumptions: LWD 25 m/hr; APC 9 m/hr; RCB/XCB 4.5 m/hr; PCBT 4 hr/run; penetrometer 2 hr/run; RIH, Pulling pipe 560 m/hr.

**Stage 2 Activities**

Three coring holes will be drilled at Terrebonne. Conventional core will be processed according to standard IODP procedures and interspersed with pressure coring in hydrate bearing intervals. PCTB pressure coring tool will be deployed at approximately 10 different intervals in the Orange and Blue sands. Coring will be punctuated by in situ temperature and pressure measurements made using SETP/T2P wireline penetrometers. Temperature-depth record from the APCT-3 will be recorded.

We will deploy wide-diameter wireline logging tools, including NMR for direct measurement of pore structure and hydrate saturation, MDT to measure open-hole permeability and recover in situ fluid samples, and FMI to collect formation images with twice the borehole coverage. This will require leasing large-diameter drill pipe and using the new handling capability on the JR rig floor. These tools can be deployed in a new hole drilled explicitly for logging or in the RCB hole after coring. In both cases, a free fall funnel is placed at the seafloor for re-entry and the larger-diameter drill pipe is lowered and used as a conduit to the interval to be logged/tested. The MDT will be then be set at selected depths to perform a borehole drawdown test and recover in situ fluid samples (Tables 4 & 5).

## **Appendix B: IODP Complementary Project Proposal for Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope (GOM<sup>2</sup>)**

This appendix contains the Complementary Project Proposal (CPP) submitted by this project October 2015. A Complementary Project Proposal (CPP) is a Full Proposal that has a commitment from a third party source for a substantial amount of financial support. Expeditions arising from such proposals follow the normal IODP rules and the IODP Sample, Data and Obligations Policy that defines the data moratorium, data access, and publication responsibilities. The level of scientific staffing for the entity contributing the CPP funds is negotiated on a case-by-case basis.



## IODP Proposal Cover Sheet

887 - Cpp 2

Gulf of Mexico Methane Hydrate

|            |  |      |                |
|------------|--|------|----------------|
| Title      | Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope  |      |                |
| Proponents | P. Flemings, T. Collett, F. Colwell, A. Cook, D. Divins, D. Goldberg, G. Guerin, A. Malinverno, D. Sawyer, E. Solomon, D. Sawyer, M. Bowles, F. Wang, T. Shanahan, M. Lever, |      |                |
| Keywords   | Methane Hydrates, Gulf of Mexico   | Area | Gulf of Mexico |

## Contact Information

|                 |                                |        |       |
|-----------------|--------------------------------|--------|-------|
| Contact Person: | Peter Flemings                 |        |       |
| Department:     | Jackson School of Geosciences  |        |       |
| Organization:   | University of Texas at Austin  |        |       |
| Address:        | 10100 Burnet Rd., J.J.Pickle R | Austin | 78758 |
| Tel.:           | 512-475-9520                   | Fax:   |       |
| E-mail:         | pflerings@jsg.utexas.edu       |        |       |

## Abstract

We will study the genesis of methane hydrate in coarse-grained sediments through scientific drilling in the northern Gulf of Mexico. Methane hydrates in coarse-grained sediments are poorly understood, though they often have much larger concentrations of hydrate than fine-grained systems. Additionally, these hydrate accumulations will likely react more rapidly to environmental perturbations because of the high permeability of the host sediment. Methane hydrate in coarse-grained sediments may be sourced by local-transport of methane formed by microbial methanogenesis or by long-range aqueous or gaseous transport. Scientific drilling will be used to distinguish the relative importance of these transport mechanisms and their scales. The results will inform multi-phase reaction transport models of methane hydrate formation. More broadly, this integrated research program will improve our understanding of microbial activity, the pathways and rates of microbial methanogenesis, and the cycling of carbon in continental margin sediments.

Our proposed drilling program involves coring and logging at three sites in the northern Gulf of Mexico. In Phase 1 (8 days), we will drill four logging-while-drilling holes at the Mad Dog and Orca Basin sites where seismic mapping shows evidence suggesting hydrate in coarse-grained sediments. In Phase 2 (48 days), we will conduct coring and wireline operations at Terrebonne, a previously drilled location with methane hydrate at high saturation in coarse-grained sediment. The sampling and measurement program at Terrebonne will feature conventional APC/XCB and RCB coring tools, as many as 30 pressure cores, wireline logging (including NMR and penetrometer testing), in situ fluid sampling and short-duration formation pressure tests using the MDT straddle packer tool.

This is a Complementary Project Proposal, and The University of Texas Austin, through support from the US Department of Energy, will provide financing for advanced technological measurements. For example, the MDT has never been deployed on the JOIDES Resolution and will provide an opportunity to directly measure in situ permeability of a methane hydrate accumulation. The extensive pressure coring program will allow us to image and study the physical, geochemical and microbiological properties of coarse-grained methane hydrate accumulations at in situ conditions.

887 - Cpp 2

### Scientific Objectives

Our main scientific objective is to illuminate the processes that lead to the genesis of methane hydrate deposits in coarse-grained systems. We will explore whether the hydrate-forming methane is generated in situ within the hydrate stability zone by microbial processes or whether microbial or thermogenic methane is transported upward from deeper sources. Our microbiology analyses and geochemical measurements will provide key constraints for reaction-transport models that describe the evolution of this system. More broadly, this integrated research program will improve our understanding of microbial activity, the pathways and rates of microbial methanogenesis, and the cycling of carbon in continental margin sediments. We will also investigate the response of coarse-grained hydrate reservoirs to environmental perturbations by collecting relevant core and log measurements and performing experiments in situ and on samples obtained by pressure coring.

Non-standard measurements technology needed to achieve the proposed scientific objectives.

Non-standard measurements including shipboard pressure coring and testing systems, state-of-the-art wireline logging tools, logging- and pressure-while-drilling tools (at previously undrilled sites), and sterile laboratories for microbiological subsampling. For pressure coring, the Aumann PCTB tool, Geolek PCATS system, and systems such as Georgia Tech IPTC/ESC testing chambers will be used. Large-diameter logging tools include NMR, FMI, and MDT borehole packer/fluid sampler; the new Blohm+Voss handling system and 6-5/8" drill pipe will be used.

### Proposed Sites

| Site Name | Position (Lat, Lon) | Water Depth (m) | Penetration (m) |     |       | Brief Site-specific Objectives   |
|-----------|---------------------|-----------------|-----------------|-----|-------|--|
|           |                     |                 | Sed             | Bsm | Total |  |
| TBONE-01A | 26.6628, -91.6762   | 1966            | 940             | 0   | 940   | At TBONE-01A, we propose to twin a pre-existing JIP Leg 2 LWD hole, WR313-H. We plan a complete coring, pressure coring and in situ testing program at this site to obtain and examine methane hydrate in coarse grained layers.   |
| TBONE-02A | 26.6604, -91.6742   | 1940            | 850             | 0   | 850   | At TBONE-02A, we will drill at a new location at the TBONE site, slightly updip and to the southwest of TBONE-01A to target coarse-grained layers and understand methane migration updip of TBONE-01A and TBONE-03A. We propose a conventional coring, pressure coring, wireline logging and in situ testing program at TBONE-02A. |
| TBONE-03A | 26.6632, -91.6839   | 1990            | 990             | 0   | 990   | At TBONE-03A, we propose to twin a pre-existing JIP Leg 2 LWD hole, WR313-G. We plan a complete coring, pressure coring and in situ testing  |

|           |                   |      |     |   |     |  |
|-----------|-------------------|------|-----|---|-----|--|
|           |                   |      |     |   |     | program at this site to obtain and examine methane hydrate in coarse grained layers.   |
| SIGSB-01A | 27.0069, -90.4265 | 2033 | 580 | 0 | 580 | A twin to Hole GC955-H, SIGSB-01A will allow to test our hypothesis regarding the reservoir characteristics that control methane migration and methane hydrate formation and concentration; however, this site lacks the dipping sedimentary sequence with phase reversals that was observed at other sites. For this reason SIGSB-01A is an alternate site that will be drilled if we perceive there to be greater potential to understand methane hydrate systems than coring TBONE-03A and other alternate sites. |
| ORCAB-03A | 26.8555, -91.3312 | 1863 | 619 | 0 | 619 | At ORCAB-03A, we propose LWD logging at a new location to confirm the presence of methane hydrates in coarse grained layers. We propose returning to ORCAB-03A for coring and logging if LWD logging confirms the presence of methane hydrate in coarse grained layers and provides the opportunity to test hypotheses exceeding that of drilling at TBONE-03A and other alternate sites.<br><br>At this site, all coring activities in the following information relate ONLY to the alternate site.                 |
| ORCAB-04A | 26.8518, -91.3355 | 1772 | 695 | 0 | 695 | At ORCAB-04A, we propose LWD logging at a new location to confirm the presence of methane hydrates in coarse grained layers.   |
| MADOG-01A | 27.1714, -90.3366 | 1400 | 648 | 0 | 648 | At MADOG-01A, we propose LWD logging at new location to confirm the presence of gas hydrates in coarse grained layers. We propose returning to MADOG-01A for coring and logging if LWD logging confirms the presence of methane hydrate in coarse grained layers and provides the opportunity to test hypotheses exceeding that of drilling at TBONE-03A and other alternate sites.<br><br>At this site, all coring activities in the following information relate ONLY to the alternate site.                       |
| MADOG-02A | 27.1676, -90.3333 | 1472 | 607 | 0 | 607 | At MADOG-02A, we propose LWD logging at new location to confirm the presence of gas hydrates in coarse grained reservoirs.   |

## Proponent list:

| First name | Last name  | Affiliation   | Country                  | Expertise   | Role           |
|------------|------------|---|--------------------------|---|----------------|
| Peter      | Flemings   | The University of Texas at Austin                       | United States of America | Geoscience - Stratigraphy, Pore Pressure Analysis, Hydrate Systems Modeling                   | Lead Proponent |
| Timothy    | Collett    | U.S. Geological Survey                                  | United States of America | Geoscience - Methane Hydrates   | Lead Proponent |
| Frederick  | Colwell    | Oregon State University                                 | United States of America | Biology - Hydrate Microbiology  | Lead Proponent |
| Ann        | Cook       | Ohio State University                                   | United States of America | Geoscience - Petrophysics   | Lead Proponent |
| David      | Divins     | University of New Hampshire                             | United States of America | Oceanography and Scientific Drilling  | Lead Proponent |
| David      | Goldberg   | Lamont-Doherty Earth Observatory of Columbia University | United States of America | Geoscience - Geophysics, Logging, Hydrates  | Lead Proponent |
| Gilles     | Guerin     | Lamont-Doherty Earth Observatory of Columbia University | United States of America | Geoscience - Logging  | Lead Proponent |
| Alberto    | Malinverno | Lamont-Doherty Earth Observatory of Columbia University | United States of America | Geoscience - Logging, Hydrate Systems Modeling  | Lead Proponent |
| Derek      | Sawyer     | Ohio State University                                   | United States of America | Geoscience - Seismic Interpretation, Geohazards, Slope Stability                              | Lead Proponent |
| Evan       | Solomon    | University of Washington                                | United States of America | Geoscience - Geochemistry   | Lead Proponent |
| Derek      | Sawyer     | Ohio State University                                   | United States of America | Geoscience - Seismic Interpretation, Geohazards, Slope Stability                              | Data Lead      |
| Marshall   | Bowles     | MARUM University of Bremen                              | Germany                  | Marine Biogeochemistry  | Lead Proponent |
| Fengping   | Wang       | Shanghai Jiao Tong University                           | China                    | Microbiology - Deep Biosphere, Microbial mediated biogeochemistry                             | Lead Proponent |
| Timothy    | Shanahan   | The University of Texas                                 | United States of America | Geoscience - sedimentary geology and geochemistry, organic geochemistry, isotope geochemistry | Lead Proponent |
| Mark       | Lever      | ETH Zurich  | Switzerland              | Marine Biology  | Lead Proponent |

# Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope [GOM]<sup>2</sup>

---

## Table of Contents

|   |    |
|---|----|
| 1. Introduction.....  | 1  |
| 1.1. The problem: methane hydrates in coarse-grained sediments .....  | 1  |
| 1.2. Proposal History and Response to Reviews.....  | 2  |
| 1.3. Relationship to the IODP Science Plan.....   | 2  |
| 1.4. Third Party Commitment to the CPP .....  | 3  |
| 2. Background.....  | 5  |
| 2.1. Gas Hydrate Joint Industry Project .....   | 5  |
| 2.2. Methane origin .....   | 7  |
| 2.3. Genesis of Methane hydrate in coarse-grained sediments .....   | 8  |
| 3. Scientific Questions & Testable Hypotheses.....  | 11 |
| 3.1. How is methane transported from the source to the site of hydrate formation in coarse-grained systems? .....   | 11 |
| 4. Research Strategies .....  | 15 |
| 4.1. Strategy 1: Determination of methanogen biomass (Test 2).....  | 15 |
| 4.2. Strategy 2: Determination of methanogen activity (Test 2).....   | 16 |
| 4.3. Strategy 3: Geochemical observations for reaction-transport models of the diagenetic pathways that create and consume methane and regulate the deep biosphere carbon cycle (Test 1, Test 2, Test 9)..... | 17 |
| 4.4. Strategy 4: Geochemical, temperature, and pressure profiles to constrain long-range gas and fluid transport (Test 1, Test 7, Test 8).....  | 18 |
| 4.4.a. Thermogenic vs. Microbial Methane.....   | 18 |

|  |    |
|--|----|
| 4.4.b. Geochemical Profiles.....   | 19 |
| 4.4.c. Pressure and Temperature Profiles .....                                     | 20 |
| 4.5. Strategy 5: Material behavior to constrain transport models (Test 5).....     | 20 |
| 4.5.a. In Situ Testing.....  | 21 |
| 4.5.b. Laboratory Testing: .....   | 23 |
| 5. Integrated Geochemistry, Microbiology, and Geotechnical Sampling Strategy ..... | 23 |
| 5.1. Conventional Coring .....   | 23 |
| 5.2. Pressure Coring .....   | 25 |
| 6. Geologic Setting.....   | 26 |
| 6.1. Terrebonne .....  | 26 |
| 6.2. Mad Dog.....  | 27 |
| 6.3. Orca Basin.....   | 28 |
| 7. Operational Plan.....   | 28 |
| 7.1. Phase 1 Activities .....  | 29 |
| 7.2. Phase 2 Activities.....   | 30 |
| 7.3. Alternate Sites .....   | 31 |
| 8. References.....   | 31 |

**List of Figures**

Figure 1: A.) Region of investigation on the northern Gulf of Mexico (GOM) continental slope. 4

Figure 2: a) Well sites and seismic line at Terrebonne..... 6

Figure 3: Methane transport mechanisms in marine hydrate deposits..... 9

Figure 4: A) Biogeochemical reactions in methane-bearing continental margin sediments. B) In local-transport microbially generated methane is transported by diffusion into sand layers. C) In long-range transport, gas is advected from below into the hydrate stability zone to form methane hydrate..... 10

Figure 5: Different methane migration mechanisms result in different patterns of gas hydrate distribution within sand reservoirs such as those commonly observed in the Gulf of Mexico. ... 12

Figure 6: We will measure material properties through in-situ tests..... 21

Figure 7: Simulated pressure drawdown using MDT tool at Terrebonne. .... 22

Figure 8: This expedition will be sampling at multiple scales..... 24

**List of Tables**

Table 1: Nine Tests that will illuminate the mechanism for hydrate formation in sand reservoirs. In Section 4, we provide research strategies to answer these questions. .... 14

Table 2: Pressure Coring Measurement workflow and sampling plan developed during an international planning workshop attended by ~25 individuals in March 2015. .... 26

Table 3: GOM2 CPP Preliminary Operational Plan and Time Estimates for Phase 1 drilling. ... 29

Table 4: GOM2 CPP Preliminary Operational Plan and Time Estimates for Phase 2 drilling. ... 30

## **1. Introduction**

### **1.1. The problem: methane hydrates in coarse-grained sediments**

Sediments of the world's continental margins contain vast amounts of natural gas hydrates, composed predominantly of methane. Methane hydrates can occur where there is a sufficient methane supply and pressure is high enough and temperature low enough for hydrate to be thermodynamically stable (Buffett, 2000; Collett et al., 2009; Hester and Brewer, 2009; Kvenvolden, 1993; Tréhu et al., 2006).

The amount of carbon stored in methane hydrates is comparable to that stored in other global near-surface reservoirs such as fossil fuels, soil, land plants, etc. (Archer et al., 2008a; Kvenvolden, 1988; Milkov, 2004; Wallmann et al., 2012). Methane in hydrates occupies a unique place in the global carbon cycle, as it is a solid stored within the lithosphere but is nonetheless metastable. Episodes of methane hydrate dissociation have been proposed to explain climate perturbations in the geologic past (Dickens et al., 1995) and have the potential to affect climate in the future (Archer et al., 2008a; Reagan and Moridis, 2008). Methane hydrates may be a geohazard, as high pore pressures caused by their dissociation may trigger submarine mass movements (McIver, 1982). Methane hydrate deposits are also potential energy resources (Boswell, 2009; Collett, 2002).

Much of what we know today about marine methane hydrates has been gleaned from scientific ocean drilling (Collett et al., 2014; Riedel et al., 2006; Ryu et al., 2013; Tréhu et al., 2006). Drilling allows us to obtain samples, make in situ measurements, and validate inferences from geophysical surveys and theoretical models. Most drilling studies have concentrated on methane hydrates within fine-grained, low-permeability sediments, which likely contain the largest fraction of the global hydrate reservoir (Boswell and Collett, 2011). Experiments and observations, however, show that hydrate formation is favored in the large pores of coarse-grained sediments, where hydrates can occupy a large fraction of pore space. Methane hydrates in coarse-grained sediments will react most rapidly to environmental perturbations, because the high concentrations can result in high gas bubble fractions that are mobile within high-permeability sediments. Concentrated methane hydrates in coarse-grained, permeable sediments are also the most attractive reservoirs for energy resources (Kneafsey and Moridis, 2014).



**In this proposed CPP drilling project, we will study the genesis of methane hydrates in coarse-grained sediments in the northern Gulf of Mexico.** We propose a drilling program to investigate methane hydrate systems in coarse-grained sediment at three locations in the Gulf of Mexico: Terrebonne, Mad Dog, and Orca Basin (Figure 1).

## **1.2. Proposal History and Response to Reviews**

This original proposal was submitted in March 2015 and reviewed by SEP in June 2015. We presented a ‘preview’ to the EPSP in Sept. 2015. The SEP review was thoughtful and insightful. We summarize three responses (see also the Proponent Response Letter (PRL)).

- 1) SEP: The main weakness was the lack of specific hypothesis tests for hydrate formation. We describe 9 tests and research strategies to answer these tests.
- 2) SEP: The initial plan was too ambitious. We refocused on the specific problem of understanding how hydrates form in dipping sands, removed two drilling sites, and refined the operational plan with input from the JRSO.
- 3) SEP: Present a more complete plan for the investigation of microbial communities and their role in the carbon cycle and strengthen geochemical experimental plan. We developed a much stronger microbiological and geochemical research strategy and included three new proponents with expertise in this field.

## **1.3. Relationship to the IODP Science Plan**

This project is at the center of several IODP 2011 Science Plan objectives (Illuminating Earth’s Past, Present, and Future: The International Ocean Discovery Program Science Plan for 2013-2023). Foremost among these is Challenge 13: ‘What Properties and Processes Govern the Flow and Storage of Carbon in the Subseafloor’: *‘Scientific drilling is critical for resolving flows of carbon through gas hydrate systems, recovering core material, determining in situ hydrate distribution, and monitoring changing properties and conditions through time.’* The project will also focus on the role of microbial communities and fluid flow in the subseafloor carbon-cycle (Challenges 5 and 14) and on how fluids link subseafloor tectonic, thermal, and biogeochemical processes (Challenge 14).

#### **1.4. Third Party Commitment to the CPP**

This is a Complementary Project Proposal (CPP), and The University of Texas Austin will provide NSF with base funding amount of US\$6,000,000. In addition, we will work with the JRSO to provide the financial support for non-standard science measurements including:

- Pressure Coring System, technical support, mobilization, demobilization including:
- Access to the PCS core system and support equipment and service vans.
- The Geotek PCATS pressure core lab van.
- Pressure Core Characterization Tool (PCCT) system.
- Schlumberger MDT testing program (straddle packer configuration, with fluid analyzer, sampling, and large diameter drill pipe) and LWD measurement tools.
- An extensive mud program (for hole stability) and an ROV to image the well head
- The cost for the use of a work boat for resupply

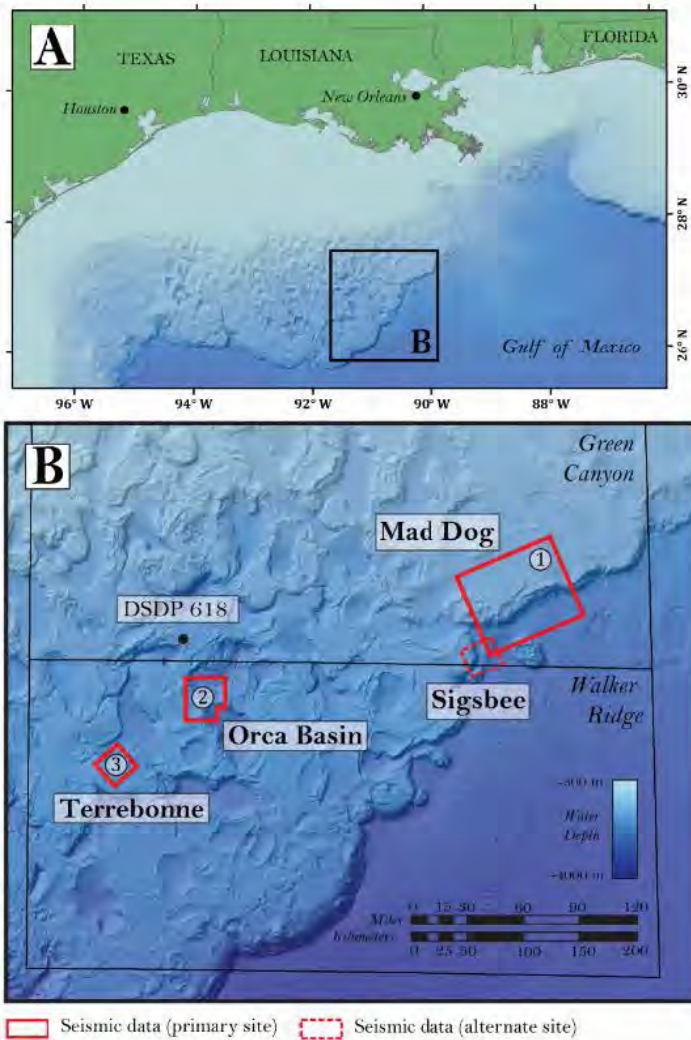


Figure 1: A.) Region of investigation on the northern Gulf of Mexico (GOM) continental slope. B.) Proposed primary sites identified by red boxes: 1) Mad Dog, 2) Orca Basin, and 3) Terrebonne (formerly, JIP Leg 2 WR 313). The dashed red box delineates the alternate site at Sigsbee. The red box is the outline of each 3D seismic data cube over the site. The black circle indicates the location drilled by DSDP Leg 618 (Shipboard Scientific Party, 1986).

## 2. Background

### 2.1. Gas Hydrate Joint Industry Project

In 2009, the Gulf of Mexico Gas Hydrate Joint Industry Project Leg 2 (JIP Leg 2) drilled logging-while-drilling (LWD) holes at several locations in the northern Gulf of Mexico and located natural gas hydrates at high saturation in coarse-grained sediments. In the Terrebonne basin (WR313, 'Terrebonne', Figure 1, (Boswell et al., 2012)) hydrates were found in dipping sand bodies at high saturations between 60-90% (Figure 2). These layers correlate to phase reversals in high-amplitude dipping reflectors on seismic data at the base of the methane hydrate stability zone (Figure 2a). The phase reversals suggest that free gas accumulations occur beneath methane hydrates within sand layers. How methane migrated into and along methane hydrate saturated layers is not well understood (Boswell et al., 2012; Frye et al., 2012). In some sand intervals, methane would have to migrate for several kilometers within the gas hydrate stability zone (GHSZ) if it originated below the GHSZ.

Orca and Mad Dog have similar seismic features as Terrebonne (Figure 1): dipping strata are cross cut by discontinuous bottom simulating reflectors (BSR) with phase reversals (Figure 2e, g). We interpret that the layers intersecting the discontinuous BSR are dipping sand units that contain methane hydrate. We must drill these layers to validate this interpretation.

JIP Leg II advanced our understanding of methane hydrates in coarse-grained sediments, but also raised many questions about the mechanisms causing methane migration in hydrate systems. We need methane hydrate, sediment, and pore water samples to evaluate the mechanisms of hydrate formation in coarse-grained systems. We need measurements of in situ temperature, pressure and salinity to evaluate system thermodynamics and the boundaries of the methane hydrate system. We need to confirm through drilling whether the system at Terrebonne is repeated at other locations that display similar seismic behavior.

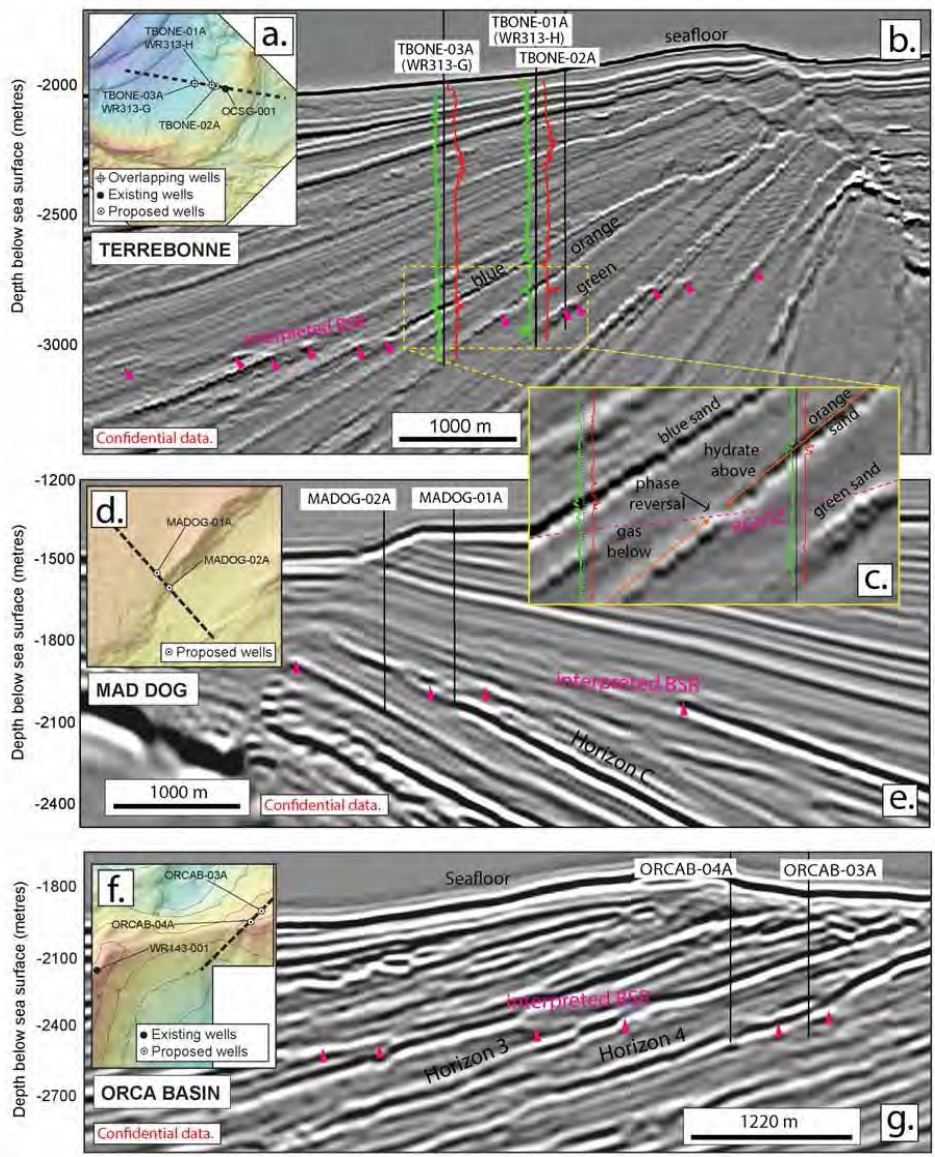


Figure 2: a) Well sites and seismic line at Terrebonne. b) Seismic profile showing existing wells drilled by the Gulf of Mexico Gas Hydrate Joint Industry Project (JIP) and proposed wells at Terrebonne. Dipping sand bodies were drilled by the JIP at WR313-G and WR313-H, and hydrate accumulations were identified with logging-while-drilling geophysical logs. On each former JIP Leg 2 hole, the gamma ray log is shown in green (scale: 0-100 API) and resistivity is shown in red (0.1-100 ohm-m). c) Seismic phase reversal of the Orange Sand unit at the BSR. d) Well sites and seismic line at Mad Dog. e) Seismic profile with proposed wells at Mad Dog. f) Location of well sites and seismic line at Orca Basin. g) Seismic profile showing proposed wells at Orca Basin. No scientific drilling has been performed at Mad Dog or Orca Basin.

## 2.2. Methane origin

Elevated primary organic matter production, the export of terrestrial organic carbon, and high sedimentation rates at continental margins contribute to an increased burial flux of metabolizable organic matter at the seafloor (Gélinas et al., 2001; Hedges and Keil, 1995). Remineralization of this particulate organic carbon (POC) is a key source of energy for the subsurface biosphere (Arndt et al., 2013; Emerson and Hedges, 2003; Jorgensen and Boetius, 2007). Subsurface microbial metabolism causes rapid pore water anoxia and drives high rates of sulfate reduction, promoting microbial methanogenesis in the sediment column (Claypool and Kaplan, 1974; Froelich et al., 1979).

Archaeal and bacterial microbes exist on and adjacent to gas hydrates in diverse marine sediments (Bidle et al., 1999; Boetius et al., 2000; Lanoil et al., 2001; Mikucki et al., 2003; Reed et al., 2002; Valentine, 2002), and have been cultivated under in situ pressures (Parkes et al., 2009). Methanogenic microbes have been detected in hydrate-bearing systems (Mikucki et al., 2003; Yoshioka et al., 2010), although they are rarely reported in high abundance in these sediments. The dominant source of methane documented in shallow marine sediments is microbial methane produced through CO<sub>2</sub>-reduction (Claypool et al., 1985; Kvenvolden and Kastner, 1990; Pohlman et al., 2009; Whiticar et al., 1995). Acetate and other volatile fatty acid fermentation can also be a source of methane (Heuer et al., 2009; Wellsbury et al., 1997). Methane that diffuses or advects back to the seafloor is oxidized at a sulfate-methane transition (SMT) by a consortium of anaerobic methanotrophic archaea and sulfate-reducing bacteria (Knittel and Boetius, 2009), producing HCO<sub>3</sub><sup>-</sup> that forms authigenic carbonates (Claypool et al., 2006; Sun and Turchyn, 2014).

Reaction-transport models are used to estimate profiles of methane and associated metabolic products, and to quantify rates of microbial methanogenesis, methane oxidation, and the resulting gas hydrate accumulations in marine sediments (Archer et al., 2008b; Burwicz et al., 2011; Davie and Buffett, 2001; Hong et al., 2014; Malinverno, 2010; Malinverno and Goldberg, 2015; Wallmann et al., 2006; Wallmann et al., 2012). However, modeling efforts are hampered by the lack of in situ concentration profiles of CH<sub>4</sub>, CO<sub>2</sub>, DIC, alkalinity, and dissolved Cl<sup>-</sup> for

verification of the modeled rates; acquisition of these data require pressure cores throughout the GHSZ.

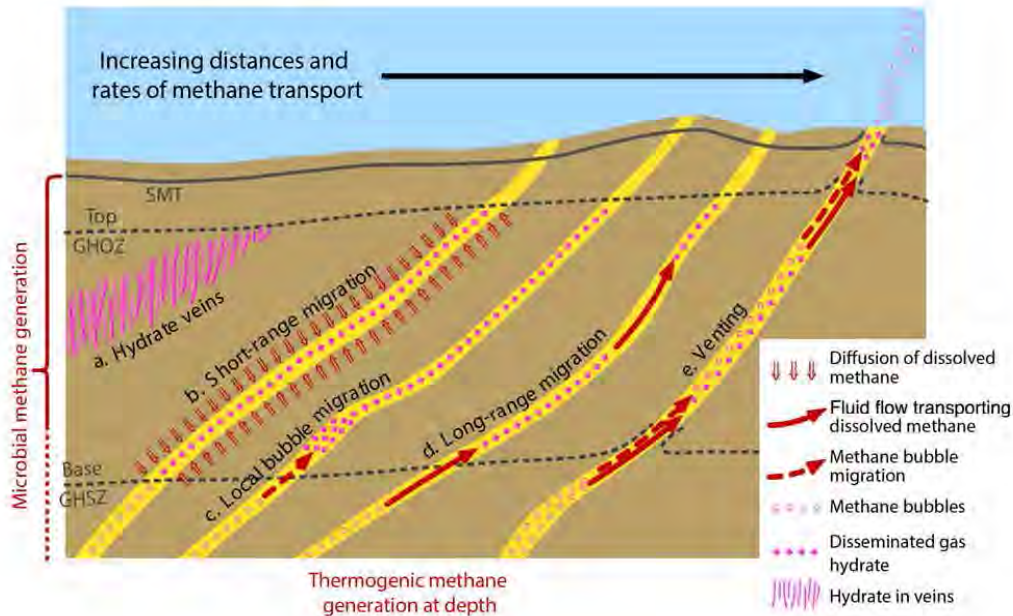
Some of the POC buried below the GHSZ is converted to methane at depth by thermogenic processes that occur at an optimal temperature around 150°C (Tissot and Welte, 1978; Wiese and Kvenvolden, 1993). Most current reaction-transport models do not explicitly consider the transport of microbial methane back into the GHSZ or the long-range transport of thermogenic methane into the GHSZ. As such, these model results may underestimate the size of gas hydrate reservoirs in petroleum basins as well as the global gas hydrate reservoir.

Thermal sources of methane have been observed within hydrate occurrences in the GOM, Caspian Sea, and Black Sea (Collett, 2002). Within the northern GOM slope, recovered hydrates contain both microbial and thermogenic methane (Brooks et al., 1986; Pflaum et al., 1986; Sassen et al., 2004). Thus, the GOM is an ideal location to test the importance of long-range transport of methane in the evolution of a gas hydrate reservoir.

### **2.3. Genesis of Methane hydrate in coarse-grained sediments**

Particulate organic matter is essentially absent from coarse-grained marine sediments. Therefore, to form concentrated hydrate deposits, methane is transported to coarse-grained sediment layers.

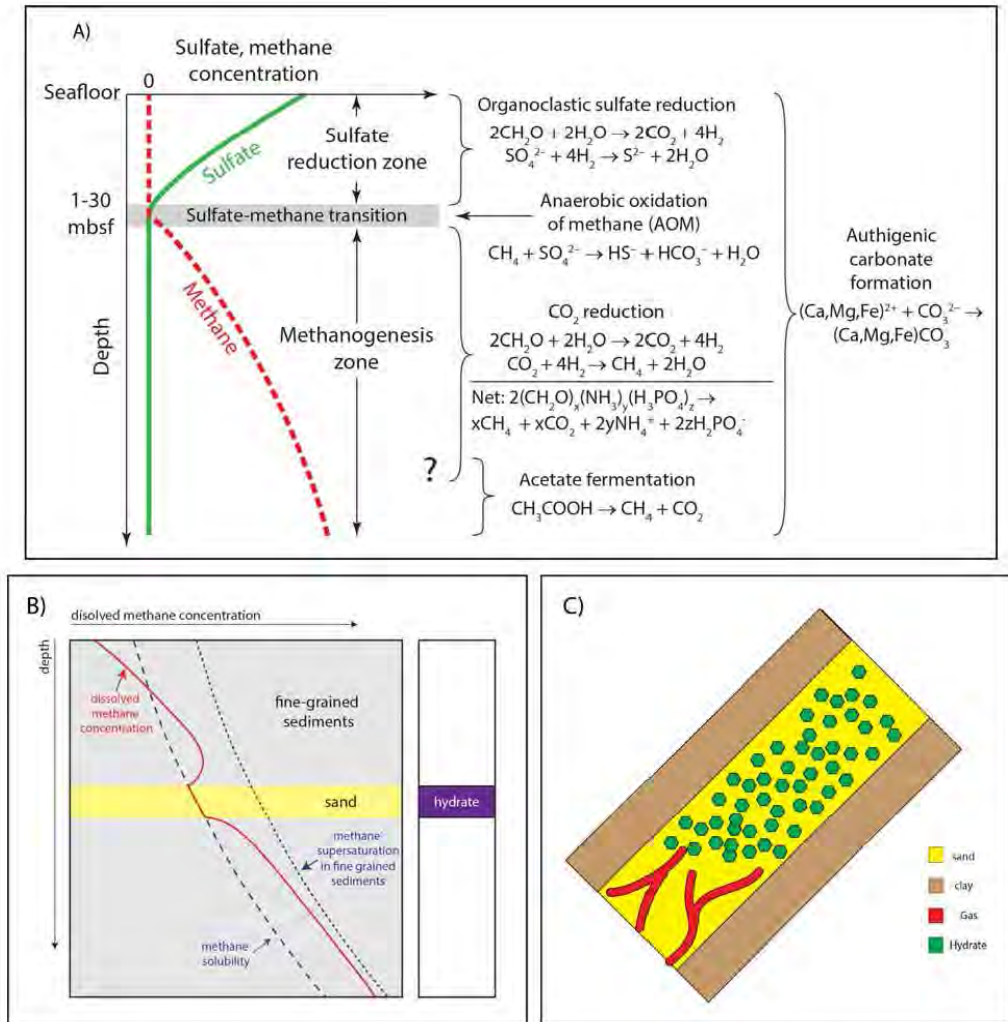
In short-range transport, microbial methane is transported from bounding mudstones to adjacent coarse-grained layers by diffusion to form gas hydrates within the pore space (Figure 3b,(Malinverno, 2010; Rempel, 2011). Over longer distances, gas dissociate from hydrate buried below the GHSZ by continuing sedimentation can migrate upward along coarse-grained, permeable layers and reform gas hydrate near the base of the GHSZ (Figure 3A.c). In long-range transport, fluid flow can transport methane (microbial or thermogenic), either as a dissolved phase or as a free gas phase, over very large distances into the hydrate stability zone. This is commonly proposed as the primary migration process for gas hydrate formation in permeable sand layers (Figure 3A.d)(Boswell et al., 2012; Liu and Flemings, 2006; Uchida et al., 2009).



**Figure 3: Methane transport mechanisms in marine hydrate deposits. A) Sediments are dominantly fine-grained (brown) and contain coarse-grained layers (yellow). Methane can be generated microbially in shallow sediments below the sulfate-methane transition (SMT), or by thermogenic processes deep in the sediment column. Gas hydrate can form between the seafloor and the base of the gas hydrate stability zone (GHSZ). Typically, gas hydrates occur in a gas hydrate occurrence zone (GHOZ) whose top is below the seafloor and is controlled by the depth where methane concentration in pore water reaches solubility. The settings marked 'a' through 'e' illustrate processes that take places as the overall rate of methane transport increases (Malinverno and Goldberg, 2015).**

In settings a-d (Figure 3), anaerobic oxidation typically consumes all dissolved methane between seafloor and the SMT interface (Borowski et al., 1996). Methane concentrations increase with depth below the SMT through an interval where gas hydrates are not present because methane concentration remains below solubility (Tréhu et al., 2006). In contrast, massive gas hydrate deposits can form at the seafloor over localized methane vents (Haeckel et al., 2004; Sassen et al., 2001). When a thick gas interval is present below the GHSZ, or the rates of fluid flow and methane transport are especially high, migrating gas bubbles can rise well into or through the GHSZ and be expelled by venting into the water column (setting e). The survival of free gas in the GHSZ has been attributed to locally high salinity levels due to salt exclusion driven by gas hydrate formation along the bubble pathway (Haeckel et al., 2004; Liu and Flemings, 2007; Smith et al., 2014).





**Figure 4: A) Biogeochemical reactions in methane-bearing continental margin sediments. B) In local-transport microbially generated methane is transported by diffusion into sand layers. C) In long-range transport, gas is advected from below into the hydrate stability zone to form methane hydrate.**

To understand the processes of methane hydrate formation in coarse-grained deposits, we must understand the relative importance of different transport mechanisms and their scales. We

describe below how observations made through scientific drilling can distinguish these processes. To understand the dynamic behavior of these systems, we need to build a model that simulates the genesis of these deposits. Observations made in scientific drilling are critical to parameterize these models.

### 3. Scientific Questions & Testable Hypotheses

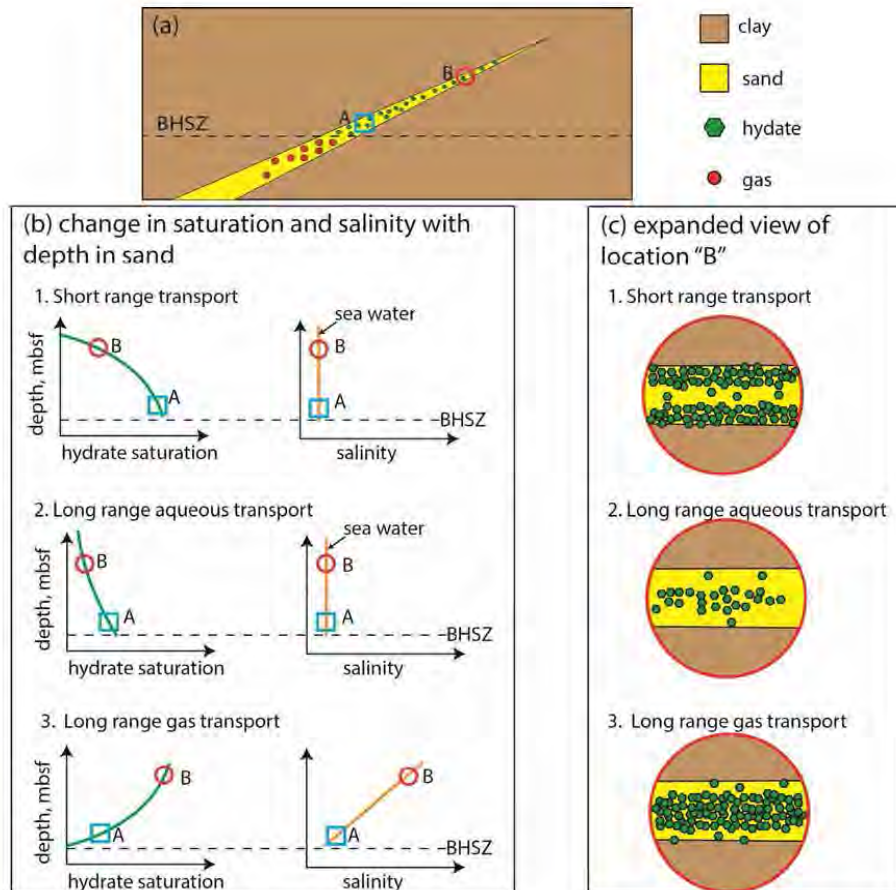
#### 3.1. How is methane transported from the source to the site of hydrate formation in coarse-grained systems?

In short-range transport, microbial methane is generated in fine-grained sediment that contain organic matter. Hydrate formation is inhibited in the small pores of these sediments, which results in a methane concentration gradient that drives a diffusive flux of methane into coarse-grained sand layers, forming concentrated hydrate deposits (Cook and Malinverno, 2013; Malinverno, 2010; Malinverno and Goldberg, 2015). In short range migration, the distribution of gas hydrate in the sand layers should be controlled by the local microbial methane generation, and can be modeled on the basis of the local organic matter content and reaction rates. The largest predicted hydrate concentrations are typically near the base of the GHSZ (Figure 5b.1). Moreover, the hydrate-bearing coarse-grained layers should be surrounded by fine-grained intervals that are hydrate-free (Cook and Malinverno, 2013) and there should be hydrate "spikes" at the coarse-grained bed boundaries (Rempel, 2011) (Figure 5c.1).

In long-range aqueous transport, water with dissolved gas is advected upward from beneath the GHSZ. As the solubility of methane declines upward, hydrate is precipitated, and in this model, hydrate concentration decreases upward (Xu and Ruppel, 1999) (Figure 5b.2). Because the solubility of methane in water is low, a large volume of water must be advected from depth to form a significant concentration of methane hydrate. As the hydrate formation rate is slow for reasonable advection rates, the salinity is not elevated above background values (Figure 5b.2).

In long-range free gas transport, gas bubbles move upward. If gas supply is sufficiently high, salinity rises as hydrate forms and the system is driven toward three-phase equilibrium with methane simultaneously present in hydrate, as free gas, and in solution (Liu and Flemings, 2007; Smith et al., 2014). In this case, there will be a systematic increase in gas hydrate saturation and pore water salinity upward from the base of the GHSZ (Figure 5b.3). At a given depth, hydrate

concentration will most likely be uniform in the sand. However, the concentration of methane will decline away from the sand into the surrounding fine-grained sediments.



**Figure 5: Different methane migration mechanisms result in different patterns of gas hydrate distribution within sand reservoirs such as those commonly observed in the Gulf of Mexico. To test these mechanisms we will drill multiple holes (blue rectangle and red circle) that are at different structural levels within one hydrate bearing sand (discussion in the text).**

We describe how ocean drilling can illuminate which, if any, of these hypothesized methane transport models drives hydrate formation in sand reservoirs. The scenario we envision in Figure 5 reproduces the overall geometry of sand layers in our primary locations. Multiple penetrations

within a single sand body at different structural levels are necessary. Hypothesis tests are as follows and summarized in Table 1:

**TEST 1:** Is the gas of thermogenic origin? If so, then short-range transport did not form the deposit. If the gas is microbial, transport could have occurred by any of the models.

**TEST 2:** Are sufficient methanogens present to produce the observed gas hydrate? For in situ production to be the dominant source of methane for gas hydrate as required in short-range transport, enough methanogenic microbes must be present.

**TEST 3:** Are the methanogens sufficiently active to produce the observed gas hydrate amounts? For in situ microbial methane production to be the dominant gas source, geochemical and thermodynamic conditions must be appropriate for them to be metabolically active. To be active, the cells need to have sufficient energetic substrates (e.g., H<sub>2</sub>, formate, acetate) and low enough methane concentrations such that they are not thermodynamically restricted.

**TEST 4:** What is the spatial variation in hydrate saturation within a dipping sand layer (Figure 5b)? Hydrate saturation that increases upward from the base of the GHSZ favors long-range gas transport. If hydrate saturation is higher near the base of the GHSZ, transport could be by short-range diffusion or by long-range aqueous flow.

**TEST 5:** Is the permeability of the hydrate-bearing sand sufficient to allow long-range methane transport? In long-range transport, there must be significant permeability in the hydrate-bearing sand to allow methane transport from the base of the GHSZ.

**TEST 6:** What is the saturation distribution within a hydrate-bearing sand bed? At the bed scale, if methane diffuses from the surrounding fine-grained sediment into the sand by short range transport, the highest saturations are expected at the interface between the sand bed and the fine-grained mud (Figure 5c.1). In long-range migration (aqueous or gas flow), hydrate saturations are not expected to be highest at the bed boundary, but rather should be highest at the center of the bed (where flow velocities are the highest) or be controlled by local permeability heterogeneities.

**TEST 7:** What is the dissolved methane concentration above and below hydrate-bearing sand beds? For diffusive transport of microbial methane to be significant as in short-range transport, the methane concentration measured above or below the sand layer should be near the solubility.

In contrast, if long-range transport dominates, the dissolved methane concentration in fine-grained sediments surrounding the sand bed is below the methane solubility.

**TEST 8: What is the thermodynamic state of the hydrate reservoir?** In long-range gas transport, three-phase equilibrium is present during hydrate formation, so that temperature, pressure, and salinity should be at the thermodynamic phase boundary. In contrast, long-range aqueous transport or short-range transport should not result in a salinity anomaly and three-phase equilibrium will only be present at the base of the GHSZ.

**TEST 9: Is the pore water derived from depth or sourced locally?** Long-range transport implies that the pore water originates at depths below the GHSZ, whereas this deep origin is not required in short-range transport.

| Test   | Short-range migration  | Long-range migration, aqueous flow                                 | Long-range migration, gas flow  |
|--|--|--|---|
| 1 Origin of methane  | Microbial  | Microbial or thermogenic   | Microbial or thermogenic  |
| 2 Sufficient methanogen biomass  | Required   | Not required   | Not required  |
| 3 Methanogen activity  | Required   | Not required   | Not required  |
| 4 Variation in hydrate saturation within a dipping sand layer            | Matches predictions of a reaction-transport model of in situ generation of microbial methane | Highest near the base of the GHSZ, decreases upward                | Lowest near the base of the GHSZ, increases upward  |
| 5 Permeability of hydrate-bearing sand layer                             | Can be small   | Must be high enough to allow significant water flow                | Must be high enough to allow significant gas flow   |
| 6 Hydrate saturation within a sand bed                                   | Increases toward bed boundary  | Constant or increases toward center                                | Constant or increases toward center   |
| 7 Dissolved methane concentration above/below a hydrate-bearing sand bed | Near methane solubility  | Can be less than methane solubility                                | Can be less than methane solubility   |
| 8 Thermodynamic state (P, T) and pore water salinity                     | 3-phase stability only at the base of the GHSZ   | 3-phase stability only at the base of the GHSZ                     | 3-phase stability within the reservoir driven by elevated salinity due to hydrate formation |
| 9 Source of pore water   | No geochemical indication of migration from depth  | Geochemical profiles indicate migration from depths below the GHSZ | Geochemical profiles indicate migration from depths below the GHSZ                          |

**Table 1: Nine Tests that will illuminate the mechanism for hydrate formation in sand reservoirs. In Section 4, we provide research strategies to answer these questions.**

## 4. Research Strategies

### 4.1. Strategy 1: Determination of methanogen biomass (Test 2)

The microbiological techniques to be used require sterile anaerobic sediment and pressure core subsampling including the use of contaminant tracers where possible (Smith et al., 2000). To determine presence and biomass of methanogens responsible for making methane we will conduct quantitative polymerase chain reaction (qPCR), and/or droplet digital PCR (ddPCR) to target methanogen-specific functional genes (e.g., methyl co-M reductase or *mcrA*; c.f., (Lever, 2013)). Compared to qPCR, ddPCR has greater sensitivity towards target genes and reduced interference from PCR inhibitors (e.g., organic compounds and metals) (Hindson et al., 2011; Kim et al., 2014a, b). Further evidence of the presence and relative abundance of methanogens will be gained from microbial community diversity characterization using Illumina sequencing of the 16S rRNA genes in DNA extracted from the samples. Metagenome sequencing (to sequence and identify key functional genes and link these to microbial taxa) will be conducted on a subset of samples from cores that are minimally altered as a result of drilling and that are deemed representative of clay or sand communities at different depths based on other microbial and chemical analyses.

We will also measure intact polar lipids (IPLs) (Kaneko and Poulson, 2013; Strapoć et al., 2008), which provide a unique marker for extant microbial activity, complementary to the genetic approaches described above. Because most IPLs are thought to degrade quickly after cell death by loss of their covalently bonded polar head groups (Harvey et al., 1986; White et al., 1979), their presence in the deep subsurface provides strong support for the presence of viable communities of organisms (Biddle et al., 2006; Lipp et al., 2008). Thus, IPLs separate fossilized and extant methanogen biomass preserved in the sediment record. A variety of IPL-based biomarkers can be employed to estimate variations in methanogenic biomass, such as the archaeal tetraether and diether lipids with varying polar head groups (Lipp and Hinrichs, 2009). Additional information on the sources of these lipids can be gained from comparisons with the biomarkers of non-methanogenic organisms (Lim et al., 2012) and by compound specific stable isotope ( $\delta^{13}\text{C}$ ,  $\delta\text{D}$ ) analysis (Biddle et al., 2006; Kaneko and Poulson, 2013). Furthermore, the compositional makeup of IPLs can provide additional information on microbial community makeup (Fang et al., 2000; Rütters et al., 2002; Sturt et al., 2004) and the environmental conditions under which these

organisms grew (Shimada et al., 2008; Van Mooy et al., 2006), particularly when used in conjunction with genetic surveys (Gibson et al., 2013).

Direct microscopic counts of microbial abundance (Lunau et al., 2005), fluorescence in situ hybridization (FISH) abundance measurements (Boetius et al., 2000), and cultivation of novel methanogens (Chen et al., 2014; Imachi et al., 2011; Mikucki et al., 2003) will also help to address the question of methanogen distribution and biomass.

#### **4.2. Strategy 2: Determination of methanogen activity (Test 2)**

These analyses will constrain models to determine whether methane undergoes local vs. long-range transport in these complex systems. They will also shed light on the microbial processes anticipated to produce methane, the conditions under which these processes occur, and when and where in the system they are likely/unlikely to occur.

To assess microbial methanogenic activity we will extract microbial RNA from the samples and measure the RNA:DNA ratio for selected microbes or selected functional genes (e.g., *mcrA*) (Freitag et al., 2010). Extracted RNA will also be submitted to metatranscriptomic analysis to determine the primary genes being transcribed by the communities in the sediments (Mills et al., 2012). Two additional methods will be explored. We will attempt to collect samples that can be preserved and subsequently analyzed to determine the metabolome of deep sediment communities (Kimes et al., 2013). This rapidly developing technology has promise to explain metabolic pathways used by microbes to produce byproducts (e.g., methane or VFAs), consume energy, and survive in situ. We will also attempt to measure the rates of activities of methanogens under in situ (i.e., high pressure, low temperature) conditions, in sand, clay, and at the interface samples that have not been decompressed (Bowles et al., 2011). Investigation of community metabolomes and rates on pressure-preserved deep sediments have not yet been accomplished. Thus, these would be demanding and uncertain analyses but have the prospective benefit of providing unprecedented insight into the biogeochemistry of these systems.

### **4.3. Strategy 3: Geochemical observations for reaction-transport models of the diagenetic pathways that create and consume methane and regulate the deep biosphere carbon cycle (Test 1, Test 2, Test 9).**

High-resolution geochemical profiles of metabolic substrates and products including methane, ammonium, alkalinity, CO<sub>2</sub>, DIC, volatile fatty acids, and dissolved H<sub>2</sub> from conventional cores and pressure cores within the GHSZ are essential for informing models of microbial methanogenesis. The geochemistry and microbiology program will provide comprehensive profiles of metabolites and products for well-constrained models to test whether the methane generated in situ is sufficient enough to account for the observed gas hydrate distribution and concentrations or whether a significant component of methane from long-range transport is required.

The first-order parameter needed for models of the seafloor carbon cycle is the concentration and age of particulate organic carbon, and the concentration of POC with depth. To date, reliable H<sub>2</sub> data in marine sediments is rare, but is very important for informing both the reaction-transport models and the microbiology analyses. Thermodynamic calculations of in situ metabolic reactions require knowledge of H<sub>2</sub> concentrations with depth. Thus, headspace gas samples will be collected for shipboard measurement of H<sub>2</sub> concentrations using refined methods based on those described in D'Hondt et al. (2009) and Lin et al. (2012). We will also measure other metabolites associated with organic matter diagenesis such as the VFAs, DOC, CO<sub>2</sub>/DIC, and carbon isotopes in all these species. The evolution of the carbon isotopic composition of these species with depth coupled with the C and H isotopic evolution of methane will be essential for determining diagenetic pathways and for modeling methane production rates.

Furthermore, the activity of acetogens will be examined in select corresponding samples using phylogenetic analyses based on the *fhs* gene that encodes the FTHS enzyme used by all acetogens in the reductive acetyl-CoA pathway (Lever et al., 2010). These analyses along with the C and H isotope composition of methane will be important for characterizing the role of methyl-type fermentation in methane production.

Carbon cycling at and above the SMTZ will also be constrained through reaction transport modeling, informed by the high-resolution profiles of SO<sub>4</sub>, Ca, Mg, DIC, alkalinity, NH<sub>4</sub>, CH<sub>4</sub>



(immediately below SMTZ, before gas saturation is reached), dissolved sulfide,  $\delta^{13}\text{C-CH}_4$ , and  $\delta^{13}\text{C-DIC}$ .

The evolution of metabolic products with depth will constrain models of microbial methanogenesis within the gas hydrate stability zone. Direct tracking of in situ methane concentrations with depth will be critical for validating these models. Conventional cores, however, only provide reliable methane concentrations in the uppermost portion of the sediment column (typically a few meters below the SMTZ) due to degassing during core recovery. Thus, to date, there is only sparse data on in situ methane concentrations from pressure cores in marine sediments, and, as such, models of microbial methanogenesis are poorly constrained. This project provides an opportunity to advance these models and our understanding of the depth distribution of methane production rates by collecting pressure cores throughout the gas hydrate stability zone. Pressure cores will provide in situ methane concentrations and C isotope ratios of  $\text{CH}_4$  and  $\text{CO}_2$ , and the H isotope ratio of  $\text{CH}_4$ .

The pressure cores will be collected at select depths downhole at each site, however samples from conventional cores will be collected at high-resolution through the entire cored interval, filling in gaps in the pressure core record. Depth profiles of  $\text{NH}_4$ , DIC, alkalinity, pH, Br,  $\delta^{13}\text{C-DIC}$ , and  $\delta^{13}\text{C-CH}_4$  will be essential for placing additional constraints on metabolic rates and carbon cycling (Solomon et al., 2014; Wallmann et al., 2006), and will guide microbiology sampling to assess methanogen activity. Furthermore, analysis of authigenic carbonate  $\delta^{13}\text{C}$  and composition will help close the carbon budget and will provide additional information on carbon and methane cycling below the SMTZ i.e. (Solomon et al., 2014; Teichert et al., 2014).

#### **4.4. Strategy 4: Geochemical, temperature, and pressure profiles to constrain long-range gas and fluid transport (Test 1, Test 7, Test 8)**

Our formation models have specific predictions regarding the thermodynamic state of the reservoir and the origin of both the water and the methane (Figure 5b). Geochemical, temperature, and pressure will inform and test these models.

##### **4.4.a. Thermogenic vs. Microbial Methane**

We will distinguish whether methane is generated by microbial or thermogenic processes through a combination of gas composition (relative abundances of methane, ethane, propane, butane,

etc.), stable carbon isotopes of methane and CO<sub>2</sub>, and stable hydrogen isotopes of methane (Bernard et al., 1978; Claypool and Kaplan, 1974; Schoell, 1980; Whiticar et al., 1986; Whiticar, 1999). In addition, measurements of multiply substituted (clumped) isotopologues in methane samples will place strong constraints on the temperature at the source, and thus the origin of methane (Stolper et al., 2014a; Stolper et al., 2014b; Wang et al., 2015).

#### 4.4.b. Geochemical Profiles

The pore water composition also places constraints on the long-range transport of fluids into the gas hydrate stability zone. Pore water composition is sensitive to fluid-rock reactions and temperatures at the fluid source, and provides an independent assessment of fluid/gas sources and fluid flow paths. In the shallow sediment column, the pore water composition is primarily controlled by low-temperature diagenetic reactions such as early organic matter diagenesis, silicate weathering, adsorption/desorption, and ion exchange (Kastner et al., 2014; Solomon et al., 2014; Wallmann et al., 2008). The pore water within the sediment pore space becomes more exotic with depth (temperature) and increasing control of fluid-rock reactions on the pore water composition. Thus, migration of deeper-sourced fluids along permeable sand horizons in the gas hydrate stability zone is manifested as anomalies/excursions in many solute and isotope ratio profiles.

One of the most important of these early, higher-temperature diagenetic reactions in the Gulf of Mexico is the smectite to illite transformation, which involves dehydration producing low chloride fluids. This reaction is most intense between ~80-150 °C (Pytte and Reynolds, 1989; Saffer and Tobin, 2011), coinciding with the temperature window for optimal production of thermogenic methane. The smectite-illite transition causes an inverse relation between pore water  $\delta D$  and  $\delta^{18}O$ , and is useful for estimating the temperature at the source of the fluid (Capuano, 1992; Hensen et al., 2004; Sheppard and Gilg, 1996; Yeh, 1980). The alkali metals are highly reactive due in part to their large atomic radii and low ionization energies. Potassium is consumed during the smectite to illite transformation, but all other alkali metals and B are preferentially released from sediments to the fluid at temperatures >50-60 °C with each having a specific threshold for fluid release (Kastner and Solomon, 2012; Kastner et al., 2014; Vanneste et al., 2011; Wei, 2007; Wei et al., 2010; You et al., 1996). Furthermore, the pore water Li concentrations and Li isotope ratios are a useful geothermometer in marine sedimentary systems

(Chan and Kastner, 2000; Kastner and Solomon, 2012), and the pore water Sr isotopic composition is sensitive to specific fluid-rock reactions and fluid mixing with deeper-sourced fluids often readily observable in the pore water Sr isotope profiles.

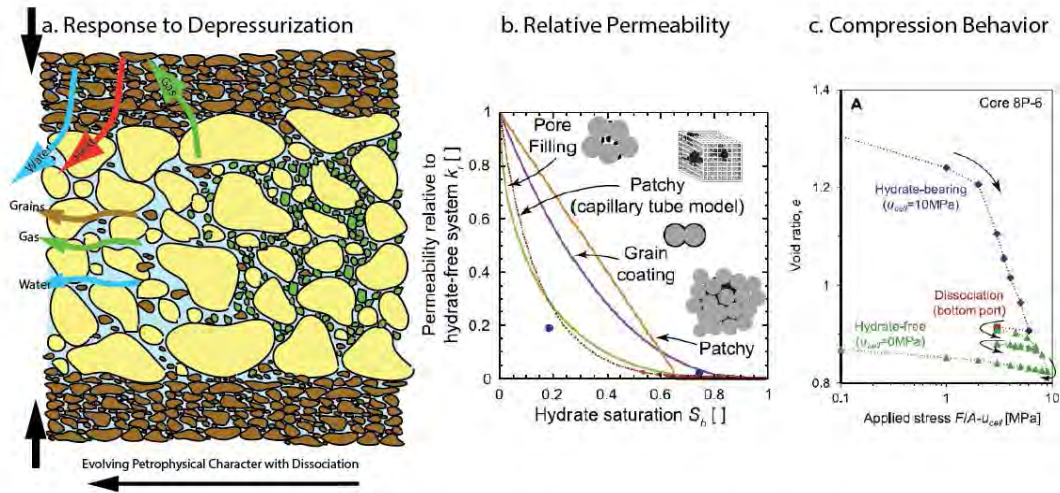
Salinity will be determined from the pore water composition, conductivity, and refractive index. The established approach for this is to determine the salinity using pressure cores. Specifically, cores will be de-gassed and the hydrate saturation calculated. The pore water composition will be sampled after depressurization and then the in situ salinity will be calculated based on the hydrate saturation present (e.g. Milkov et al., 2004). We are also developing methodologies to extract pore fluids at in situ pressure and temperature from pressure cores (Santamarina et al., 2012) to constrain the in situ pore water composition. Finally, we will also use the MDT (modular formation dynamic tester) in probe mode to sample fluids from hydrate bearing sandstones downhole.

#### **4.4.c. Pressure and Temperature Profiles**

Temperature will be measured with the APCT while piston coring. In addition, pressure and temperature will be measured with SETP and/or T2P penetrometer tool (Flemings et al., 2008). The MDT tool will also measure pressure.

#### **4.5. Strategy 5: Material behavior to constrain transport models (Test 5)**

We will further our understanding of the genesis of these hydrate systems through dynamic multi-phase, reaction-transport modeling. To do so, we must constrain material and transport properties as a function of hydrate saturation.



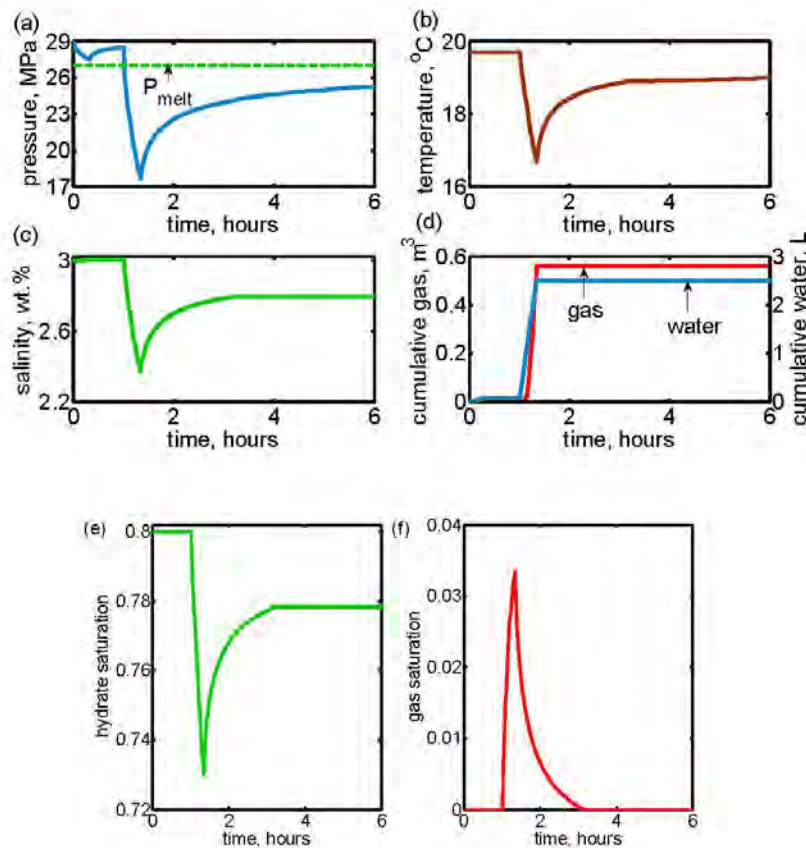
**Figure 6:** We will measure material properties through in-situ tests a.) and through laboratory tests on conventional cores and pressure cores (b and c). b. At Nankai, absolute permeabilities were found to be on the order of 1 Darcy and relative permeabilities strongly depend on hydrate saturation (Santamarina et al., 2015). c.) Compression behavior can also be derived from pressure cores (Santamarina et al., 2015).

#### 4.5.a. In Situ Testing

We will measure formation permeability with Schlumberger's MDT wireline system. Short term depressurization tests using wireline packers were performed on Arctic hydrates at the Imperial Oil Limited Malik 5L-38 in Canada's North West Territories in 2002 and at Mount Elbert on the Alaska North slope in 2007 (Anderson et al., 2011). Both tests include multiple flow and subsequent build-up periods in multiple stratigraphic levels. Initial permeability was measured, the onset of dissociation was imaged, and the volume of the flow was measured.

We will also use Schlumberger's MDT wireline packer system (e.g. Saffer et al., 2013). We illustrate the results that are possible through a 1-dimensional numerical simulation of well testing at the Terrebonne location (Figure 7). We first (0-20 minutes) pump out fluid at a constant rate (Figure 7a). This reduces the pressure in the well to 27.4 MPa, which is above the hydrate dissociation pressure at the initial temperature and salinity (Figure 7a: the dashed green line, 27 MPa). The temperature and the salinity remain at their initial values. We then shut down the pump (20-60 minutes). Pressure builds to 28.5 MPa as water flows through the low permeability hydrate-bearing sand occurs. We then (60-80 minutes) pump out fluid at a constant rate and the pressure reduces to 17.7 MPa at 80 minutes (9.3 MPa lower than the initial hydrate dissociation

pressure (green dashed line, Figure 7a)). Hydrate saturation decreases (Figure 7e), and gas saturation increases in the sediment around the well (Figure 7f). Temperature in the well decreases to 16.7 °C due to the latent heat of hydrate dissociation (Figure 7b), and salinity decreases to 2.4 wt% due to the fresh water released from hydrate (Figure 7c).



**Figure 7: Simulated pressure drawdown using MDT tool at Terrebonne. (a) pressure, (b) temperature, and (c) salinity in the well, (d) the cumulative gas and water volume produced per square meter of the sediment at the pressure of 1 atm and temperature of 15 °C, and (e) the hydrate and (f) gas saturation around the well during the depressurization test. The sediment has an intrinsic permeability of  $10^{-13} \text{ m}^2$  in absence of hydrate. The initial pressure, temperature and salinity in the reservoir are 29 MPa, 19.7 °C and 3 wt.%, respectively as inferred for the hydrate reservoir at the depth of 862 m at WR313-G in Terrebonne (Hutchinson et al., 2009). The initial hydrate saturation is 80% with the remainder of the pore space filled with water and the porosity is 35% (Boswell et al., 2012). The intrinsic permeability is 100 mD ( $10^{-13} \text{ m}^2$ ) in absence of hydrate and this yields a permeability of 0.3 mD ( $10^{-15} \text{ m}^2$ ) when hydrate saturation is 75%, which matches the permeability of the hydrate-filled sediment (75% hydrate saturation) to water in Mount Elbert tests (0.2-0.5 mD) (Anderson et al., 2011). The initial drawdown was at  $0.25 \text{ kg m}^{-2}/\text{hr}$  and the 2<sup>nd</sup> draw down was at  $8 \text{ kg m}^{-2} \text{ hour}^{-1}$ .**

Pressure increases at a much slower rate in the 2<sup>nd</sup> build-up relative to the first build-up (Figure 6a). Hydrate reforms in the sediment around the well: hydrate saturation increases (Figure 6e), and gas saturation decreases (Figure 6f), which leads to increasing temperature and salinity (Figure 6b, c). The gas released from the hydrate around the well is re-fixed into hydrate at 188 minutes. Hydrate saturation and salinity are constant from 3 to 6 hours. No free gas can be drawn from the sediment from 0 to 66 minutes because the effective gas saturation is below the residual value (0.02) (Figure 6d). There is totally about 2.55 L liquid, and 0.56 m<sup>3</sup> methane gas produced per square meter of the sediment at 360 minutes at 1 atm and 15 °C (Figure 6d).

From these results, we can derive: (1) in situ water permeability (first build-up in Figure 6a); (2) water permeability in presence of gas or water relative permeability (second build-up in Figure 6a). From the temperature and salinity curves, we can estimate the amount of hydrate dissociated during flow periods and the amount of hydrate reformed during build-up. We will also use the MDT tool in its 'probe' mode. In this mode, the tool presses against the side of the borehole and performs a local fluid withdrawal. We will use the tool in this manner both to measure local permeability and to take fluid samples from the hydrate bearing bed.

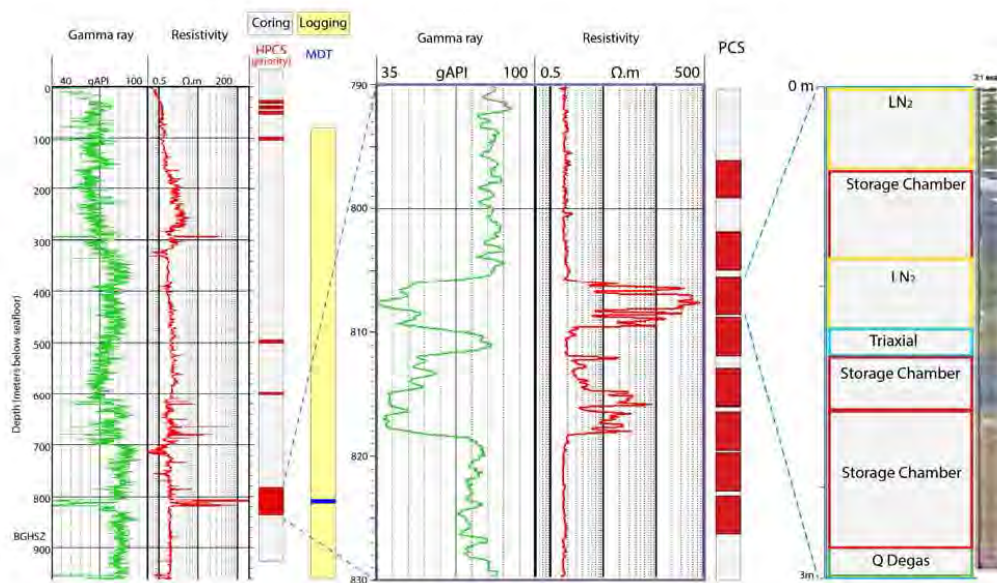
#### **4.5.b. Laboratory Testing:**

We will perform constant head tests and constant-rate of strain tests to determine permeability on conventional whole core (Reece et al., 2012). We will perform tests on pressurized cores to determine the permeability of stable methane hydrate systems and the evolution of permeability as hydrate dissociates (Santamarina et al., 2015). Undrained strength can be studied through direct analysis of pressure cores (Santamarina et al., 2015). We will also study the bulk compressibility of the material at various hydrate saturations (e.g., Figure 7c).

## **5. Integrated Geochemistry, Microbiology, and Geotechnical Sampling Strategy**

### **5.1. Conventional Coring**

We will systematically sample in multiple boreholes that penetrate a hydrate bearing sand at different structural levels (e.g. Figure 1a, 5a) at multiple scales (Figure 8).



**Figure 8:** This expedition will be sampling at multiple scales. A) At the scale of the total hole, we will be performing logging measurements and on whole cores we will be continuously sampling at approximately the meter scale for pore water, moisture and density (MAD), and other traditional shipboard measurements. In addition, we will be measuring pressure and temperature down hole. Finally, we will be taking multiple pressure cores down the entire well profile to take pore water samples under pressure. B) At the 10 meter scale, we will increase the intensity of our sampling. We will take multiple pressure cores both with the sand and in the bounding mudstone above and below. C) Finally at the m scale of a single pressure core, we will first cat-scan the pressurized core. Finally, a sampling plan for each pressure core will be defined according to the cat-scan results. This example cat-scan image is taken from Yamamoto et al. (2012). Note pressure core samples are denoted in red in the HPCS column.

At the scale of the entire borehole (Figure 8a), we will perform high-resolution pore water sampling (1 sample every 0.5 m) in one borehole to characterize biogeochemical cycling through the SMTZ. At the other coring holes, one whole-round per section will be collected to 20 m below the SMTZ. The sample resolution will decrease to one every 3 m to the first occurrence of gas hydrate (defined by LWD). Subsequently, samples will be collected every 5 m to TD. Pore water will be extracted from conventional cores through a combination of whole round squeezing and rhizon sampling guided by infrared scans of the cores on the catwalk. Microbiology whole rounds will be collected adjacent to the pore water samples. Whole round samples will be taken for geotechnical sampling. Discrete physical properties and sediment samples (e.g. biomarkers, TOC/IC, MAD, XRD) and sediment plugs for headspace analyses of gases will also be collected immediately adjacent to the pore water whole rounds (e.g. Vannucchi et al., 2012).

At the scale of the hydrate reservoir (Figure 8b, 8c), high-resolution pore water and microbiology sampling across the transitions from mudstone to sand will constrain variations in microbial communities, metabolites, metabolic products, and microbial activity, with an emphasis on sampling at the interface. We anticipate increasing the sample resolution to one every 50 cm through these transitions. These profiles will also be useful for defining anomalies and gradients associated with deeper-sourced fluid and gas migration along the sands.

## 5.2. Pressure Coring

We will pressure core with the PCTB (Pressure Core Tool with Ball). Versions of this tool were deployed in offshore Japan (Yamamoto et al., 2012), South Korea (Kim et al., 2011), and China (Zhang et al., 2014). Shipboard, the Pressure Core Analysis and Transfer System (PCATS) will be used to manipulate, analyze, and subsample pressure cores without being depressurized (Schultheiss et al., 2011). Subsamples of the pressure cores will be processed at research institutions around the globe with capability to manipulate and analyze pressure cores. The Pressure Core Characterization Tool (PCCT) is one such system now housed by the USGS at Woods Hole (Santamarina et al., 2012; Santamarina et al., 2015).

Pressure cores collected over the entire hole will better define the methane concentration profile, inform models of microbial methanogenesis, and address the methane transport mechanisms. The exact target depth of these background pressure cores will vary from hole to hole. High-resolution pressure core sampling will occur through the transitions from mudstone to gas hydrate-bearing sands. These pressure cores will quantify gas hydrate saturations, and characterize background pore water profiles (Figure 8). Each pressure core will have a detailed sub-sampling program guided by cat-scans of the entire 1.5 m core upon recovery (Figure 8c).

We outline a broad experimental plan for ship-based and shore based pressure core research (Table 2). Limited testing will occur at sea due to time limitations. A sub-section will be degassed shipboard for quantitative analysis of total methane concentration and gas hydrate saturation, and another sub-sample will be dedicated to shipboard triaxial analyses. Sub-samples will be stored in liquid nitrogen for shore-based characterization. Sub-samples for physical properties and pore water geochemical analyses will be stored under pressure in storage containers, and then processed on shore. Sub-samples for shore-based microbiology experiments and analyses



including measuring the rates and activities of methanogens will be stored under in situ conditions – these samples will span all pressure coring depths and will particularly focus on the interface between mudstone and sand.

|     |                                   |   |
|-----|-----------------------------------|---|
| 1)  | Routine Coring Measurements       | Velocity, bulk density, and linear X-rays will be done to all pressure cores immediately as they are recovered on deck.   |
| 2)  | Temporary Core Storage            | Next, 3 m pressure cores that are acquired will be placed in a storage chamber. Any pressure cores recovered at the site will be stored until the conclusion of drilling the site.  |
| 3)  | Complete Sampling Plan            | After the Site is finished, a sampling plan will be developed that will subdivide plans for Shipboard Analysis and those for Post-Cruise Analysis:  |
| 3a) | Shipboard Sampling and Analysis   | Shipboard measurements will be used to confirm hydrate presence and concentration. We will cut 1 or 2 samples from each pressure core (~10 cm) and put these into a cell. We will measure permeability, measure pore fluid chemistry, measure hydrate concentration and perform degassing.<br>On select pressure cores, pore water will be extracted under pressure to sample true background composition. Once the samples are degassed they will be analyzed for standard properties in the shipboard core flow. This will either be through a syringe type device or through displacement of fluid during a permeability test. |
| 3b) | Sampling for Shore Based Analysis | <u>Storage under pressure:</u> Cores will be subsampled and placed in pressure containers for delivery to individual research institutions. These samples could be later further subsampled on shore.<br><u>Liquid Nitrogen:</u> Some samples will be stored at atmospheric pressure in liquid nitrogen. The samples will be rapidly depressurized and plunged into liquid nitrogen. Plans are underway to develop an approach to freeze the samples under pressure and then depressurize these samples and plunge into nitrogen.   |
| 4)  | Shore-Based Analysis              | Shore based analysis will include analyses now done with the PCCT such as permeability, mechanical properties, hydrate saturation and the taking of biologic samples. We hope to develop the ability to perform Micro CT on pressure core samples.  |

Table 2: Pressure Coring Measurement workflow and sampling plan developed during an international planning workshop attended by ~25 individuals in March 2015.

## 6. Geologic Setting

### 6.1. Terrebonne

The Terrebonne (TBONE) location (Figure 1) is on the southeastern side of the salt-bounded Terrebonne mini-basin. The shallow sedimentary column is composed of turbidite sequences dipping about 10 degrees (Frye et al., 2012) (Figure 2). The base of the GHSZ is quite deep at

880 m, due to the 2000 m water column and possibly to a low geothermal gradient. In 2009, JIP Leg 2 holes, WR313-H and WR313-G, were drilled using LWD (Figure 2). The logs confirmed high saturation of methane hydrate ( $S_h > 80\%$ ) in multiple coarse-grained layers (Collett et al., 2012). The upper section of each well contained an interval of marine muds several hundred meters thick with methane hydrate in near-vertical fractures.

The Blue Sand was penetrated just above the base of the GHSZ in WR313-G (Figure 2). At this location, methane hydrate rich sand layers are interbedded with marine muds. 20 m of hydrate-bearing sands range in saturation from 40-90%. The Blue Sand was also intersected updip in WR313-H near 670 mbsf (Figure 2; Figure 8). In this location, the sand has thinned significantly to a total of only ~3 m of sand but still contained methane hydrate at high saturations ( $S_h > 60\%$ ) (Frye et al., 2012). In WR313-H, the Blue Sand is several hundred meters vertically above the base of the GHSZ and is over a kilometer along the Blue Sand from the base of the GHSZ.

In WR313-H, the 9 m-thick Orange Sand underlies the Blue Sand near 800 mbsf (Figure 2; Figure 8). In this location, the Orange Sand is more massive in character than the Blue Sand and consists of two lobes with high hydrate saturation, nearly 90% (Collett et al., 2012).

We propose three sites at TBONE: a twin of WR313-H (TBONE-01A), a hole updip of WR313-H (TBONE-02A), and a twin of WR313-G (TBONE-03A) (Figure 2).

We will characterize, sample and test methane hydrate accumulations in two morphologically different coarse-grained units, the Blue and Orange Sands, at both down-dip and up-dip locations at Terrebonne.

## 6.2. Mad Dog

The Mad Dog location straddles the edge of the Sigsbee Escarpment (Figure 1) near the western end of the deepwater Mississippi fan-fold belt (Hall, 2002). This location is close to the “Mad Dog” oil field.

Like the Terrebonne location, Mad Dog has dipping seismic reflectors, termed Horizon 3 and Horizon 4 that change phase at the base of the GHSZ (Figure 2). We hypothesize that these reflectors with phase reversals are coarse-grained intervals containing methane hydrate within the GHSZ and water and gas-bearing coarse-grained layers below the GHSZ. We will drill two LWD

holes at Mad Dog, MADOG-01A and MADOG-02A, slightly updip of the horizons with phase reversals (Figure 2), to document the presence of methane hydrate and the sediment type. If hydrates are present, revisiting Mad Dog to core and measure in situ properties is proposed as an alternate to site TBONE-03A.

### 6.3. Orca Basin

The Orca Basin is a large minibasin within the Walker Ridge and Green Canyon protraction areas (Pilcher and Blumstein, 2007). We focus on the southern edge of the minibasin, in water depths ~1900 m (Figure 1). Four industry wells were drilled within the Orca Basin, ~6 km southeast to east of our proposed sites. Data from these wells shows that the basin is infilled by a thick supra-salt sequence of late Miocene to Pleistocene muddy sediments, with minor interbedded sands (McCormack et al., 2013; Murphy et al., 2011). Several of these wells show evidence of methane hydrate in marine muds, but not in coarse-grained reservoirs.

We believe the two primary sites at Orca Basin, ORCAB-03A and ORCAB-04A will be drilled with LWD to confirm the presence of methane hydrate in coarse-grained units (Figure 2). Having mapped these faults at the Orca Basin site, we feel confident that the reflectors of interest reverse polarity at the BSR, indicating the presence of methane hydrate in coarse-grained layers. If methane hydrate is present, Orca Basin offers an opportunity to test our methane transport mechanism hypothesis and revisiting ORCAB-03 to core and measure in situ properties is proposed as an alternate to site TBONE-03A.

## 7. Operational Plan

The expedition is designed as a standard 56-day scientific IODP expedition (Tables 3&4). The scientific objectives are best achieved with 2 operational phases. Phase 1 will drill four (4) LWD holes at Mad Dog and Orca. Phase 2 will conduct coring and wireline operations at three Terrebonne locations. Coring will feature conventional APC/XCB and RCB coring tools, as many as 30 PBCT runs, wireline logging (including NMR and penetrometer testing), in situ fluid sampling and short-duration formation pressure tests using the MDT straddle packer tool.

This two-phase strategy allows for shipboard analysis of the LWD results prior to Phase 2 and improved time and cost efficiency with mid-expedition personnel and equipment transfers. We estimate preliminary rig times for Phase 1 and 2 activities, including transits, BHA assembly, and

drilling, coring, and logging operations (Tables 3&4). These estimates are computed using the Coring Time Estimator with input from the JR Science Operator at Texas A&M University.

**7.1. Phase 1 Activities**

We will drill four (4) LWD holes at Mad Dog and Orca sites. The LWD program will include: geoVISION (GVR), EcoScope, SonicScope, SonicVison, TeleScope, and ProVision-Plus (NMR) tools. Based on prior experience, operational times are estimated assuming that LWD tools are combined and penetrate at 25 m/hr (average) in a separate lowering at each site.

| A. PHASE 1 OPERATIONS                       |         |        |        |                |      |      |
|---|---------|--------|--------|----------------|------|------|
| Operations                                  | Mob &   | Set Up | Coring | Downhole Meas. | Cum. | Cum. |
|   | Transit |        |        |                | Time | Time |
|   | (hr)    | (hr)   | (hr)   | (hr)           | (hr) | Days |
| Mobilization, Galveston TX and transit      | 44.0    |        |        |                | 44.0 | 1.8  |
| <b>MADOG-01A: Drill and LWD to 648 mbsf</b> |         |        |        |                |      |      |
| -LWD rig up                                 |         | 6.0    |        |                | 6.0  | 0.3  |
| -Run pipe to SF                             |         | 4.5    |        |                | 10.5 | 0.4  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |        | 30.9           | 41.4 | 1.7  |
| -Pull pipe                                  |         | 3.7    |        |                | 45.1 | 1.9  |
| Total, incl. transit to MADOG-02A (3.0 nmi) | 3.0     |        |        |                | 48.1 | 2.0  |
| <b>MADOG-02A: Drill and LWD to 607 mbsf</b> |         |        |        |                |      |      |
| -LWD rig up                                 |         | 6.0    |        |                | 6.0  | 0.3  |
| -Run pipe to SF                             |         | 4.6    |        |                | 10.6 | 0.4  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |        | 29.3           | 39.9 | 1.7  |
| -Pull pipe                                  |         | 3.7    |        |                | 43.6 | 1.8  |
| Total, incl. transit to ORCA-03A (61 nmi)   | 5.8     |        |        |                | 49.4 | 2.1  |
| <b>ORCAB-03A: Drill and LWD to 619 mbsf</b> |         |        |        |                |      |      |
| -LWD rig up                                 |         | 6.0    |        |                | 6.0  | 0.3  |
| -Run pipe to SF                             |         | 5.3    |        |                | 11.3 | 0.5  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |        | 29.8           | 41.1 | 1.7  |
| -Pull pipe                                  |         | 4.4    |        |                | 45.5 | 1.9  |
| Total, incl. transit to ORCAB-04A (0.5 nmi) | 0.5     |        |        |                | 46.0 | 1.9  |
| <b>ORCAB-04A: Drill and LWD to 695 mbsf</b> |         |        |        |                |      |      |
| -LWD rig up                                 |         | 4.0    |        |                | 4.0  | 0.2  |
| -Run pipe to SF                             |         | 5.2    |        |                | 9.2  | 0.4  |
| -Hole A: drill with LWD at 25 m/hr to TD    |         |        |        | 32.8           | 42.0 | 1.7  |
| -Pull pipe                                  |         | 4.4    |        |                | 46.4 | 1.9  |
| Total, incl. transit to TBONE-01A (22 nmi)  | 2.1     |        |        |                | 48.5 | 2.0  |

Table 3: GOM2 CPP Preliminary Operational Plan and Time Estimates for Phase 1 drilling. Times are based on the JRSO Time Estimator and recent hydrate drilling experience. Average rate assumptions: LWD 25 m/hr; APC 9 m/hr; RCB/XCB 4.5 m/hr; PCBT 4 hr/run; penetrometer 2 hr/run; RHH, Pulling pipe 560 m/hr.

**B. PHASE 2 OPERATIONS**

| Operations                                    | Mob &       |             | Downhole     |              | Cum.          | Cum.        |
|---|-------------|-------------|--------------|--------------|---------------|-------------|
|   | Transit     | Set Up      | Coring       | Meas.        | Time          | Time        |
|   | (hr)        | (hr)        | (hr)         | (hr)         | (hr)          | Days        |
| <b>TBONE-01A: Drill and Core to 940 mbsf</b>  |             |             |              |              |               |             |
| -Coring setup                                 |             | 4.0         |              |              | 4.0           | 0.2         |
| -Run pipe to SF                               |             | 5.5         |              |              | 9.5           | 0.4         |
| -Hole A: APC/XCB core to 500mbsf              |             |             | 88.9         |              | 98.4          | 4.1         |
| -Acquire 10 PCTB cores (upper sand & blue)    |             |             | 40.0         |              | 138.4         | 5.8         |
| -Hole B: RCB core 500m to TD                  |             |             | 124.8        |              | 263.2         | 11.0        |
| -Wireline penetrometer at ~8 depths to TD     |             |             |              | 17.6         | 280.8         | 11.7        |
| -Deploy FFF and Re-enter with lg pipe         |             |             | 19.3         |              | 300.1         | 12.5        |
| -GR logging & MDT formation testing           |             |             |              | 44.0         | 344.1         | 14.3        |
| -Pull pipe                                    |             | 5.7         |              |              | 349.8         | 14.6        |
| Total, incl. transit to TBONE-02A (1.0 nmi)   | 1.0         |             |              |              | 350.8         | 14.6        |
| <b>TBONE-02A: Drill and Core to 850 mbsf</b>  |             |             |              |              |               |             |
| -Coring setup                                 |             | 4.0         |              |              | 4.0           | 0.2         |
| -Run pipe to SF                               |             | 5.5         |              |              | 9.5           | 0.4         |
| -Hole A: APC/XCB core to 500mbsf              |             |             | 88.9         |              | 98.4          | 4.1         |
| -Acquire 10 PCTB cores (upper sand & blue)    |             |             | 40.0         |              | 138.4         | 5.8         |
| -Hole B: RCB core 500m to TD                  |             |             | 104.7        |              | 243.1         | 10.1        |
| -Wireline penetrometer at ~8 depths to TD     |             |             |              | 17.6         | 260.7         | 10.9        |
| -Hole C: wash to TD and Re-enter with lg pipe |             |             | 53.1         |              | 313.8         | 13.1        |
| -NMR + GR logging & MDT testing               |             |             |              | 58.0         | 371.8         | 15.5        |
| -Pull pipe                                    |             | 5.6         |              |              | 377.4         | 15.7        |
| Total, incl. transit to TBONE-03A (3.0 nmi)   | 3.0         |             |              |              | 380.4         | 15.8        |
| <b>TBONE-03A: Drill and Core to 940 mbsf</b>  |             |             |              |              |               |             |
| -Coring setup                                 |             | 4.0         |              |              | 4.0           | 0.2         |
| -Run pipe to SF                               |             | 5.6         |              |              | 9.6           | 0.4         |
| -Hole A: APC/XCB core to 500mbsf              |             |             | 88.9         |              | 98.4          | 4.1         |
| -Acquire 10 PCTB cores (upper sand & blue)    |             |             | 40.0         |              | 138.4         | 5.8         |
| -Hole B: RCB core 500m to TD                  |             |             | 124.9        |              | 263.3         | 11.0        |
| -Wireline penetrometer at ~8 depths to TD     |             |             |              | 17.6         | 280.9         | 11.7        |
| -Deploy FFF and Re-enter with lg pipe         |             |             | 19.4         |              | 300.3         | 12.5        |
| -GR logging & MDT formation testing           |             |             |              | 44.0         | 344.3         | 14.3        |
| -Pull pipe                                    |             | 5.7         |              |              | 350.0         | 14.6        |
| Total, incl. transit/demob in Galveston TX    | 25.7        |             |              |              | 375.7         | 15.7        |
| <b>Total Times (hr):</b>                      | <b>85.1</b> | <b>89.8</b> | <b>832.9</b> | <b>288.8</b> | <b>1342.9</b> | <b>56.0</b> |

Table 4: GOM2 CPP Preliminary Operational Plan and Time Estimates for Phase 2 drilling. Times are based on the JRSO Time Estimator and recent hydrate drilling experience. Average rate assumptions: LWD 25 m/hr; APC 9 m/hr; RCB/XCB 4.5 m/hr; PCBT 4 hr/run; penetrometer 2 hr/run; RIH, Pulling pipe 560 m/hr.

**7.2. Phase 2 Activities**

Three coring holes will be drilled at Terrebonne. Conventional core will be processed according to standard IODP procedures and interspersed with pressure coring in hydrate bearing intervals.

PCBT pressure coring tool will be deployed at approximately 10 different intervals in the Orange and Blue sands. Coring will be punctuated by in situ temperature and pressure measurements made using SETP/T2P wireline penetrometers. Temperature-depth record from the APCT-3 will be recorded.

We will deploy wide-diameter wireline logging tools, including NMR for direct measurement of pore structure and hydrate saturation, MDT to measure open-hole permeability and recover in situ fluid samples, and FMI to collect formation images with twice the borehole coverage. This will require leasing large-diameter drill pipe and using the new handling capability on the JR rig floor. These tools can be deployed in a new hole drilled explicitly for logging or in the RCB hole after coring. In both cases, a free fall funnel is placed at the seafloor for re-entry and the larger-diameter drill pipe is lowered and used as a conduit to the interval to be logged/tested. The MDT will be then be set at selected depths to perform a borehole drawdown test and recover in situ fluid samples (Tables 3&4).

### 7.3. Alternate Sites

There are three alternate sites: MADOG-01A, ORCAB-03A, and SIGSB-01A. After Phase 1, we may select one of the LWD sites at Mad Dog or Orca for conventional coring, pressure coring, and in situ measurements, if that site has higher potential to address GOM<sup>2</sup> objectives than the primary TBONE-03A hole. Another potential alternate site is SIGSB-01A; this site was drilled using LWD tools during JIP Leg 2 and penetrated a sand unit (>30 m thick) interbedded with thin hydrate-filled mud layers.

## 8. References

- Anderson, B. J., Hancock, S., Wilson, S., Enger, C., Collett, T., Boswell, R., and Hunter, R., 2011, Formation pressure testing at the Mount Elbert Gas Hydrate Stratigraphic Test Well, Alaska North Slope: Operational summary, history matching, and interpretations: *Marine and Petroleum Geology*, v. 28, no. 2, p. 478-492, doi:10.1016/j.marpetgeo.2010.02.012.
- Archer, D., Buffett, B. A., and Brovkin, V., 2008a, Ocean methane hydrates as a slow tipping point in the global carbon cycle: *Proc. Nat. Acad. Sci.*, v. 106, p. 20596-20601, doi: 20510.21073/pnas.0800885105.

- Archer, D., Buffett, B. A., and Brovkin, V., 2008b, Ocean methane hydrates as a slow tipping point in the global carbon cycle: *Proceedings of the National Academy of Sciences*, v. 106, no. 49, p. 20596-20601, doi:10.1073/pnas.0800885105.
- Arndt, S., Jørgensen, B. B., LaRowe, D. E., Middelburg, J. J., Pancost, R. D., and Regnier, P., 2013, Quantifying the degradation of organic matter in marine sediments: A review and synthesis: *Earth-Science Reviews*, v. 123, p. 53-86, <http://dx.doi.org/10.1016/j.earscirev.2013.02.008>.
- Bernard, B. B., Brooks, J. M., and Sackett, W. M., 1978, Light hydrocarbons in recent Texas continental shelf and slope sediments: *Journal of Geophysical Research: Oceans*, v. 83, no. C8, p. 4053-4061, 10.1029/JC083iC08p04053.
- Biddle, J. F., Lipp, J. S., Lever, M. A., Lloyd, K. G., Sørensen, K. B., Anderson, R., Fredricks, H. F., Elvert, M., Kelly, T. J., Schrag, D. P., Sogin, M. L., Brenchley, J. E., Teske, A., House, C. H., and Hinrichs, K.-U., 2006, Heterotrophic Archaea dominate sedimentary subsurface ecosystems off Peru: *Proceedings of the National Academy of Sciences of the United States of America*, v. 103, no. 10, p. 3846-3851, doi:10.1073/pnas.0600035103.
- Bidle, K. A., Kastner, M., and Bartlett, D. H., 1999, A phylogenetic analysis of microbial communities associated with methane hydrate containing marine fluids and sediments in the Cascadia margin (ODP site 892B): *FEMS Microbiol. Lett.*, v. 177, p. 101-108.
- Boetius, A., Ravensschlag, K., Schubert, C. J., Rickert, D., Widdel, F., Gieseke, A., Amann, R., Jørgensen, B. B., Witte, U., and Pfannkuche, O., 2000, A marine microbial consortium apparently mediating anaerobic oxidation of methane: *Nature*, v. 407, p. 623-626.
- Borowski, W. S., Paull, C. K., and Ussler, W. I., 1996, Marine pore-water sulfate profiles indicate in situ methane flux from underlying gas hydrate: *Geology*, v. 24, no. 7, p. 655-658.
- Boswell, R., 2009, Is gas hydrate energy within reach?: *Science*, v. 325, no. 5943, p. 957-958, doi:10.1126/science.1175074.
- Boswell, R., and Collett, T. S., 2011, Current perspectives on gas hydrate resources: *Energy & Environmental Science*, v. 4, no. 4, p. 1206-1215, doi:10.1039/C0EE00203H.
- Boswell, R., Collett, T. S., Frye, M., Shedd, W., McConnell, D. R., and Shelander, D., 2012, Subsurface gas hydrates in the northern Gulf of Mexico: *Marine and Petroleum Geology*, v. 34, no. 1, p. 4-30, doi:10.1016/j.marpetgeo.2011.10.003.
- Bowles, M. W., Samarkin, V. A., and Joye, S. B., 2011, Improved measurement of microbial activity in deep-sea sediments at in situ pressure and methane concentration: *Limnology and Oceanography: Methods*, v. 9, no. 10, p. 499-506, doi:10.4319/lom.2011.9.499.
- Brooks, J. M., Cox, H. B., Bryant, W. R., Kennicutt II, M. C., Mann, R. G., and McDonald, T. J., 1986, Association of gas hydrates and oil seepage in the Gulf of Mexico: *Organic Geochemistry*, v. 10, no. 1-3, p. 221-234, doi:10.1016/0146-6380(86)90025-2.
- Buffett, B. A., 2000, Clathrate hydrates: *Annual Review of Earth and Planetary Sciences*, v. 28, p. 477-507, doi:10.1146/annurev.earth.28.1.477.
- Burwicz, E. B., Rüpke, L. H., and Wallmann, K., 2011, Estimation of the global amount of submarine gas hydrates formed via microbial methane formation based on numerical reaction-transport modeling and a novel parameterization of Holocene sedimentation: *Geochimica et Cosmochimica Acta*, v. 75, no. 16, p. 4562-4576, doi:10.1016/j.gca.2011.05.029.
- Capuano, R. M., 1992, The temperature dependence of hydrogen isotope fractionation between clay minerals and water: Evidence from a geopressured system: *Geochimica et Cosmochimica Acta*, v. 56, no. 6, p. 2547-2554, doi:10.1016/0016-7037(92)90208-Z.

- Chan, L.-H., and Kastner, M., 2000, Lithium isotopic compositions of pore fluids and sediments in the Costa Rica subduction zone: implications for fluid processes and sediment contribution to the arc volcanoes: *Earth and Planetary Science Letters*, v. 183, no. 1–2, p. 275-290, doi:10.1016/S0012-821X(00)00275-2.
- Chen, Y., Li, Y. L., Zhou, G. T., Li, H., Lin, Y. T., Xiao, X., and Wang, F. P., 2014, Biomineralization mediated by anaerobic methane-consuming cell consortia: *Sci Rep*, v. 4, p. 5696, doi:10.1038/srep05696.
- Claypool, G. E., and Kaplan, I. R., 1974, The Origin and Distribution of Methane in Marine Sediments, *in* Kaplan, I., ed., *Natural Gases in Marine Sediments*, Volume 3, Springer US, p. 99-139.
- Claypool, G. E., Threlkeld, C. N., Mankiewicz, P. N., Arthur, M. A., and Anderson, T. F., 1985, Isotopic composition of interstitial fluids and origin of methane in slope sediment of the Middle America Trench, Deep-Sea Drilling Project Leg-84: *Init. Rep. DSDP 84*, p. 683-691.
- Claypool, G. E., Milikov, A. V., Lee, Y. J., Torres, M. E., Borowski, W. S., and Tomaru, H., 2006, Microbial methane generation and gas transport in shallow sediments of an accretionary complex, southern Hydrate Ridge (ODP Leg 204), offshore Oregon USA: *Proc. ODP, Scientific Results, Ocean Drilling Program*.
- Collett, T. S., 2002, Energy resource potential of natural gas hydrates: *Am. Assoc. Petr. Geol. Bull.*, v. 86, p. 1971-1992.
- Collett, T. S., Johnson, A. H., Knapp, C. C., and Boswell, R., 2009, Natural gas hydrates: A review, *in* Collett, T. S., Johnson, A., Knapp, C., and Boswell, R., eds., *Natural gas hydrates—Energy resource potential and associated geologic hazards*, AAPG Memoir 89: Tulsa, Oklahoma, The American Association of Petroleum Geologists, p. 146-219.
- Collett, T. S., Lee, M. W., Zyrianova, M. V., Mrozewski, S. A., Guerin, G., Cook, A. E., and Goldberg, D. S., 2012, Gulf of Mexico Gas Hydrate Joint Industry Project Leg II logging-while-drilling data acquisition and analysis: *Marine and Petroleum Geology*, v. 34, no. 1, p. 41-61, doi:10.1016/j.marpetgeo.2011.08.003.
- Collett, T. S., Boswell, R., Cochran, J. R., Kumar, P., Lall, M., Mazumdar, A., Ramana, M. V., Ramprasad, T., Riedel, M., Sain, K., Sathe, A. V., and Vishwanath, K., 2014, Geologic implications of gas hydrates in the offshore of India: Results of the National Gas Hydrate Program Expedition 01: *Marine and Petroleum Geology*, v. 58, p. 3-28, doi:10.1016/j.marpetgeo.2014.07.021.
- Cook, A. E., and Malinverno, A., 2013, Short migration of methane into a gas hydrate-bearing sand layer at Walker Ridge, Gulf of Mexico: *Geochemistry, Geophysics, Geosystems*, v. 14, no. 2, p. 283-291, doi:10.1002/ggge.20040.
- D'Hondt, S., Spivack, A. J., Pockalny, R., Ferdelman, T. G., Fischer, J. P., Kallmeyer, J., Abrams, L. J., Smith, D. C., Graham, D., Hasiuk, F., Schrum, H., and Stancin, A. M., 2009, Subseafloor sedimentary life in the South Pacific Gyre: *Proceedings of the National Academy of Sciences*, v. 106, no. 28, p. 11651-11656, doi:10.1073/pnas.0811793106.
- Davie, M. K., and Buffett, B. A., 2001, A numerical model for the formation of gas hydrate below the sea floor: *Journal of Geophysical Research*, v. 106, no. B1, p. 497-514.
- Dickens, G. R., O'Neil, J. R., Rea, D. K., and Owen, R. M., 1995, Dissociation of oceanic methane hydrate as a cause of the carbon isotope excursion at the end of the Paleocene: *Paleoceanogr.*, v. 10, p. 965-971.



- Emerson, S., and Hedges, J., 2003, 6.11 - Sediment Diagenesis and Benthic Flux, *in* Turekian, H. D. H. K., ed., *Treatise on Geochemistry*, v. <http://dx.doi.org/10.1016/B0-08-043751-6/06112-0>; Oxford, Pergamon, p. 293-319.
- Fang, J., Barcelona, M. J., and Alvarez, P. J. J., 2000, A direct comparison between fatty acid analysis and intact phospholipid profiling for microbial identification: *Organic Geochemistry*, v. 31, no. 9, p. 881-887, doi:10.1016/S0146-6380(00)00053-X.
- Flemings, P. B., Long, H., Dugan, B., Germaine, J. T., John, C. M., Behrmann, J. H., Sawyer, D., and IODP Expedition 308 Scientists, 2008, Pore pressure penetrometers document high overpressure near the seafloor where multiple submarine landslides have occurred on the continental slope, offshore Louisiana, Gulf of Mexico: *Earth and Planetary Science Letters*, v. 269, no. 3-4, p. 309-325, doi:10.1016/j.epsl.2007.12.005.
- Freitag, T. E., Toet, S., Ineson, P., and Prosser, J. I., 2010, Links between methane flux and transcriptional activities of methanogens and methane oxidizers in a blanket peat bog: *FEMS Microbiology Ecology*, v. 73, no. 1, p. 157-165, doi:10.1111/j.1574-6941.2010.00871.x.
- Froelich, P. N., Klinkhammer, G. P., Bender, M. L., Luedtke, N. A., Heath, G. R., Cullen, D., Dauphin, P., Hammond, D., Hartman, B., and Maynard, V., 1979, Early oxidation of organic matter in pelagic sediments of the eastern equatorial Atlantic: suboxic diagenesis: *Geochimica et Cosmochimica Acta*, v. 43, no. 7, p. 1075-1090, doi:10.1016/0016-7037(79)90095-4.
- Frye, M., Shedd, W., and Boswell, R., 2012, Gas hydrate resource potential in the Terrebonne Basin, Northern Gulf of Mexico: *Marine and Petroleum Geology*, v. 34, no. 1, p. 150-168, doi:10.1016/j.marpetgeo.2011.08.001.
- Gélinas, Y., Baldock, J. A., and Hedges, J. I., 2001, Organic Carbon Composition of Marine Sediments: Effect of Oxygen Exposure on Oil Generation Potential: *Science*, v. 294, no. 5540, p. 145-148, <http://www.sciencemag.org/content/294/5540/145.abstractN2>.
- Gibson, R. A., van der Meer, M. T. J., Hopmans, E. C., Reysenbach, A. L., Schouten, S., and Sinninghe Damsté, J. S., 2013, Comparison of intact polar lipid with microbial community composition of vent deposits of the Rainbow and Lucky Strike hydrothermal fields: *Geobiology*, v. 11, no. 1, p. 72-85, doi:10.1111/gbi.12017.
- Haeckel, M., Suess, E., Wallmann, K., and Rickert, D., 2004, Rising methane gas bubbles form massive hydrate layers at the seafloor: *Geochimica et Cosmochimica Acta*, v. 68, no. 21, p. 4335-4345, doi:10.1016/j.gca.2004.01.018.
- Hall, S. H., 2002, The role of autochthonous salt inflation and deflation in the northern Gulf of Mexico: *Marine and Petroleum Geology*, v. 19, no. 6, p. 649-682, doi:10.1016/s0264-8172(02)00025-9.
- Harvey, H. R., Fallon, R. D., and Patton, J. S., 1986, The effect of organic matter and oxygen on the degradation of bacterial membrane lipids in marine sediments: *Geochimica et Cosmochimica Acta*, v. 50, no. 5, p. 795-804, doi:10.1016/0016-7037(86)90355-8.
- Hedges, J. I., and Keil, R. G., 1995, Sedimentary organic matter preservation: an assessment and speculative synthesis: *Marine Chemistry*, v. 49, no. 2-3, p. 81-115, doi:10.1016/0304-4203(95)00008-F.
- Hensen, C., Wallmann, K., Schmidt, M., Ranero, C. R., and Suess, E., 2004, Fluid expulsion related to mud extrusion off Costa Rica—A window to the subducting slab: *Geology*, v. 32, no. 3, p. 201, doi:10.1130/g20119.1.
- Hester, K. C., and Brewer, P. G., 2009, Clathrate hydrates in nature: *Ann. Rev. Mar. Sci.*, v. 2009.1, p. 303-327, doi:10.1146/annurev.marine.010908.163824.

- Heuer, V. B., Pohlman, J. W., Torres, M. E., Elvert, M., and Hinrichs, K.-U., 2009, The stable carbon isotope biogeochemistry of acetate and other dissolved carbon species in deep seafloor sediments at the northern Cascadia Margin: *Geochimica et Cosmochimica Acta*, v. 73, no. 11, p. 3323-3336, doi:10.1016/j.gca.2009.03.001.
- Hindson, B. J., Ness, K. D., Masquelier, D. A., Belgrader, P., Heredia, N. J., Makarewicz, A. J., Bright, I. J., Lucero, M. Y., Hiddessen, A. L., Legler, T. C., Kitano, T. K., Hodel, M. R., Petersen, J. F., Wyatt, P. W., Steenblock, E. R., Shah, P. H., Bousse, L. J., Troup, C. B., Mellen, J. C., Wittmann, D. K., Erndt, N. G., Cauley, T. H., Koehler, R. T., So, A. P., Dube, S., Rose, K. A., Montesclaros, L., Wang, S., Stumbo, D. P., Hodges, S. P., Romine, S., Milanovich, F. P., White, H. E., Regan, J. F., Karlin-Neumann, G. A., Hindson, C. M., Saxonov, S., and Colston, B. W., 2011, High-Throughput Droplet Digital PCR System for Absolute Quantitation of DNA Copy Number: *Analytical Chemistry*, v. 83, no. 22, p. 8604-8610, doi:10.1021/ac202028g.
- Hong, J., Feng, H., Wang, F., Ranjan, A., Chen, J., Jiang, J., Ghirlando, R., Xiao, T. S., Wu, C., and Bai, Y., 2014, The Catalytic Subunit of the SWRI Remodeler Is a Histone Chaperone for the H2A.Z-H2B Dimer: *Molecular Cell*, v. 53, no. 3, p. 498-505, doi:10.1016/j.molcel.2014.01.010.
- Hutchinson, D., Boswell, R., Collett, T., Dai, J. C., Dugan, B., Frye, M., Jones, E., McConnell, D., Rose, K., Ruppel, C., Shedd, W., Shelander, D., and Wood, W., 2009, Gulf of Mexico Gas Hydrate Joint Industry Project Leg II — Walker Ridge 313 site selection.
- Imachi, H., Aoi, K., Tasumi, E., Saito, Y., Yamanaka, Y., Saito, Y., Yamaguchi, T., Tomaru, H., Takeuchi, R., Morono, Y., Inagaki, F., and Takai, K., 2011, Cultivation of methanogenic community from seafloor sediments using a continuous-flow bioreactor: *ISME J*, v. 5, no. 12, p. 1913-1925, doi:10.1038/ismej.2011.64.
- Jorgensen, B. B., and Boetius, A., 2007, Feast and famine [mdash] microbial life in the deep-sea bed: *Nat Rev Micro*, v. 5, no. 10, p. 770-781, <http://dx.doi.org/10.1038/nrmicro1745>.
- Kaneko, M., and Poulson, S. R., 2013, The rate of oxygen isotope exchange between nitrate and water: *Geochimica et Cosmochimica Acta*, v. 118, p. 148-156, doi:10.1016/j.gca.2013.05.010.
- Kastner, M., and Solomon, E. A., 2012, Experimental and field studies of Li mobility in subduction zones: *Mineralogical Magazine*, v. 76, no. 6, p. 1920, <http://minmag.geoscienceworld.org/content/76/6/1912.short>.
- Kastner, M., Solomon, E. A., Harris, R. N., and Torres, M. E., 2014, Fluid origins, thermal regimes, and fluid and solute fluxes in the forearc of subduction zones, in Stein, R., Blackman, D. K., Inagaki, F., and Larsen, H.-C., eds., *Developments in Marine Geology*, Volume Volume 7, Elsevier, p. 671-733.
- Kim, G. Y., Yi, B. Y., Yoo, D. G., Ryu, B. J., and Riedel, M., 2011, Evidence of gas hydrate from downhole logging data in the Ulleung Basin, East Sea: *Marine and Petroleum Geology*, v. 28, no. 10, p. 1979-1985, doi:10.1016/j.marpetgeo.2011.01.011.
- Kim, T., Jeong, S.-Y., and Cho, K.-S., 2014a, Comparison of droplet digital PCR and quantitative real-time PCR for examining population dynamics of bacteria in soil: *Applied Microbiology and Biotechnology*, v. 98, no. 13, p. 6105-6113, doi:10.1007/s00253-014-5794-4.
- Kim, T., Jeong, S.-Y., and Cho, K.-S., 2014b, Comparison of droplet digital PCR and quantitative real-time PCR in *merA*-based methanogen community analysis: *Biotechnology Reports*, v. 4, p. 1-4, doi:10.1016/j.btre.2014.06.010.

- Kimes, N. E., Callaghan, A. V., Aktas, D. F., Smith, W. L., Sunner, J., Golding, B., Drozdowska, M., Hazen, T. C., Sufliata, J. M., and Morris, P. J., 2013, Metagenomic analysis and metabolite profiling of deep-sea sediments from the Gulf of Mexico following the Deepwater Horizon oil spill: *Frontiers in Microbiology*, v. 4, p. 50, doi:10.3389/fmicb.2013.00050.
- Kneafsey, T. J., and Moridis, G. J., 2014, X-Ray computed tomography examination and comparison of gas hydrate dissociation in NGHP-01 expedition (India) and Mount Elbert (Alaska) sediment cores: Experimental observations and numerical modeling: *Marine and Petroleum Geology*, v. 58, p. 526-539, doi:10.1016/j.marpetgeo.2014.06.016.
- Knittel, K., and Boetius, A., 2009, Anaerobic oxidation of methane: progress with an unknown process: *Annu Rev Microbiol*, v. 63, p. 311-334, doi:10.1146/annurev.micro.61.080706.093130.
- Kvenvolden, K. A., 1988, Methane Hydrate: A Major Reservoir of Carbon in the Shallow Geosphere?: *Chemical Geology*, v. 71, p. 11.
- Kvenvolden, K. A., and Kastner, M., 1990, Gas hydrates of the Peruvian outer continental margin: *Proceedings ODP*, v. Scientific Results 112, p. 517-526.
- Kvenvolden, K. A., 1993, Gas hydrates-geological perspective and global change: *Reviews of Geophysics*, v. 31, p. 173-187.
- Lanoil, B. D., Sassen, R., La Duc, M. T., Sweet, S. T., and Nealson, K. H., 2001, Bacteria and Archaea physically associated with Gulf of Mexico gas hydrates: *Appl Environ Microbiol*, v. 67, no. 11, p. 5143-5153, doi:10.1128/AEM.67.11.5143-5153.2001.
- Lever, M. A., Heuer, V. B., Morono, Y., Masui, N., Schmidt, F., Alperin, M. J., Inagaki, F., Hinrichs, K.-U., and Teske, A., 2010, Acetogenesis in Deep Subseafloor Sediments of The Juan de Fuca Ridge Flank: A Synthesis of Geochemical, Thermodynamic, and Gene-based Evidence: *Geomicrobiology Journal*, v. 27, no. 2, p. 183-211, doi:10.1080/01490450903456681.
- Lever, M. A., 2013, Functional gene surveys from ocean drilling expeditions – a review and perspective: *FEMS Microbiology Ecology*, v. 84, no. 1, p. 1-23, doi:10.1111/1574-6941.12051.
- Lim, K. L. H., Pancost, R. D., Hornibrook, E. R. C., Maxfield, P. J., and Evershed, R. P., 2012, Archaeol: An Indicator of Methanogenesis in Water-Saturated Soils: *Archaea*, v. 2012, p. 9, doi:10.1155/2012/896727.
- Lin, Y.-S., Heuer, V. B., Goldhammer, T., Kellermann, M. Y., Zabel, M., and Hinrichs, K.-U., 2012, Towards constraining H<sub>2</sub> concentration in subseafloor sediment: A proposal for combined analysis by two distinct approaches: *Geochimica et Cosmochimica Acta*, v. 77, p. 186-201, doi:10.1016/j.gca.2011.11.008.
- Lipp, J. S., Morono, Y., Inagaki, F., and Hinrichs, K.-U., 2008, Significant contribution of Archaea to extant biomass in marine subsurface sediments: *Nature*, v. 454, no. 7207, p. 991-994, doi:10.1038/nature07174.
- Lipp, J. S., and Hinrichs, K.-U., 2009, Structural diversity and fate of intact polar lipids in marine sediments: *Geochimica et Cosmochimica Acta*, v. 73, no. 22, p. 6816-6833, doi:10.1016/j.gca.2009.08.003.
- Liu, X., and Flemings, P. B., 2006, Passing gas through the hydrate stability zone at southern Hydrate Ridge, offshore Oregon: *Earth and Planetary Science Letters*, v. 241, no. 1-2, p. 211-226, doi:10.1016/j.epsl.2005.10.026.

- Liu, X., and Flemings, P. B., 2007, Dynamic multiphase flow model of hydrate formation in marine sediments: *Journal of Geophysical Research*, v. 112, no. B3, doi:10.1029/2005jb004227.
- Lunau, M., Lemke, A., Walther, K., Martens-Habbenha, W., and Simon, M., 2005, An improved method for counting bacteria from sediments and turbid environments by epifluorescence microscopy: *Environ Microbiol*, v. 7, no. 7, p. 961-968, doi:10.1111/j.1462-2920.2005.00767.x.
- Malinverno, A., 2010, Marine gas hydrates in thin sand layers that soak up microbial methane: *Earth Planet. Sci. Lett.*, v. 292, p. 399-408, doi:10.1016/j.epsl.2010.02.008.
- Malinverno, A., and Goldberg, D. S., 2015, Testing short-range migration of microbial methane as a hydrate formation mechanism: Results from Andaman Sea and Kumano Basin drill sites and global implications: *Earth and Planetary Science Letters*, v. 422, p. 105-114, doi:10.1016/j.epsl.2015.04.019.
- McCormack, P., Guchereau, H., Zumwalt, C., Gales, S., Draganov, M., Miller, D., Shirley, R., and Sanders, A., 2013, Well Summary, Union Oil Company of California, Coronado #2 OCS-G 21841 002 ST00BP00, Walker Ridge Block 98: Houston, Sperry Drilling Services, Halliburton.
- McIver, R. D., 1982, Role of naturally occurring gas hydrates in sediment transport: *Am. Assoc. Petr. Geol. Bull.*, v. 66, p. 789-792.
- Mikucki, J. A., Liu, Y., Delwiche, M., Colwell, F. S., and Boone, D. R., 2003, Isolation of a Methanogen from Deep Marine Sediments That Contain Methane Hydrates, and Description of *Methanoculleus submarinus* sp. nov.: *Applied and Environmental Microbiology*, v. 69, no. 6, p. 3311-3316, doi:10.1128/aem.69.6.3311-3316.2003.
- Milkov, A. V., 2004, Global estimates of hydrate-bound gas in marine sediments: how much is really out there?: *Earth-Science Reviews*, v. 66, no. 3-4, p. 183-197, doi:10.1016/j.earscirev.2003.11.002.
- Milkov, A. V., Y.-J. Lee, Borowski, W. S., Torres, M. E., Xu, W., Tomaru, H., Trehu, A. M., Schultheiss, P., Dickens, G. R., and Claypool, G. E., 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*, v. 222, p. 829-843.
- Mills, H. J., Kiel Reese, B., Shepard, A., Dowd, S. E., Riedinger, N., Morono, Y., and Inagaki, F., 2012, Characterization of metabolically active bacterial populations in subseafloor Nankai Trough sediments above, within and below the sulfate-methane transition zone: *Frontiers in Microbiology*, v. 3, doi:10.3389/fmicb.2012.00113.
- Murphy, M., Guchereau, H., Chevis, M., O'Donnell, S., Ervin, P., and Porche, R., 2011, Well Summary, Chevron USA Inc. Coronado, OCS-G 21849 001 ST00BP00, Walker Ridge Block 134: Houston, Sperry Drilling Services, Halliburton.
- Parkes, R. J., Sellek, G., Webster, G., Martin, D., Anders, E., Weightman, A. J., and Sass, H., 2009, Culturable prokaryotic diversity of deep, gas hydrate sediments: first use of a continuous high-pressure, anaerobic, enrichment and isolation system for subseafloor sediments (DeepIsoBUG): *Environ Microbiol*, v. 11, no. 12, p. 3140-3153, doi:10.1111/j.1462-2920.2009.02018.x.
- Pflaum, R. C., Brooks, J. M., Cox, H. B., Kennicutt II, M. C., and Sheu, D.-D., 1986, Molecular and isotopic analysis of core gases and gas hydrates, Deep Sea Drilling Project Leg 96, *in* Bouma, A. H., Coleman, J. M., Meyer, A. W., and others, eds., *Initial Reports of the Deep Sea Drilling Project*, vol. 96: Washington, D. C., U.S. Govt. Printing Office, p. 781-784.

- Pilcher, R. S., and Blumstein, R. D., 2007, Brine volume and salt dissolution rates in Orca Basin, northeast Gulf of Mexico: *AAPG Bulletin*, v. 91, no. 6, p. 823-833, doi:10.1306/12180606049.
- Pohlman, J. W., Kaneko, M., Heuer, V. B., Coffin, R. B., and Whiticar, M., 2009, Methane sources and production in the northern Cascadia margin gas hydrate system: *Earth and Planetary Science Letters*, v. 287, no. 3–4, p. 504-512, doi:10.1016/j.epsl.2009.08.037.
- Pytte, A. M., and Reynolds, R. C., 1989, The Thermal Transformation of Smectite to Illite, *in* Naeser, N., and McCulloh, T., eds., *Thermal History of Sedimentary Basins*, v. doi:10.1007/978-1-4612-3492-0\_8, Springer New York, p. 133-140.
- Reagan, M. T., and Moridis, G. J., 2008, Dynamic response of oceanic hydrate deposits to ocean temperature change: *Journal of Geophysical Research: Oceans*, v. 113, no. C12, p. C12023, doi:10.1029/2008JC004938.
- Reece, J. S., Flemings, P. B., Dugan, B., Long, H., and Germaine, J. T., 2012, Permeability-porosity relationships of shallow mudstones in the Ursa Basin, northern deepwater Gulf of Mexico: *Journal of Geophysical Research: Solid Earth*, v. 117, no. B12, p. n/a-n/a, doi:10.1029/2012jb009438.
- Reed, D. W., Fujita, Y., Delwiche, M. E., Blackwelder, D. B., Sheridan, P. P., Uchida, T., and Colwell, F. S., 2002, Microbial Communities from Methane Hydrate-Bearing Deep Marine Sediments in a Forearc Basin: *Applied and Environmental Microbiology*, v. 68, no. 8, p. 3759-3770, doi:10.1128/aem.68.8.3759-3770.2002.
- Rempel, A. W., 2011, A model for the diffusive growth of hydrate saturation anomalies in layered sediments: *J. Geophys. Res.*, v. 116, p. B10105, doi:10.1029/2011JB008484.
- Riedel, M., Collett, T. S., Malone, M. J., and Expedition 311 Scientists, 2006, *Proc. IODP, 311: Washington, DC (Integrated Ocean Drilling Program Management International, Inc.)*, v. doi:10.2204/iodp.proc.311.2006, doi:10.2204/iodp.proc.311.2006.
- Rütters, H., Sass, H., Cypionka, H., and Rullkötter, J., 2002, Phospholipid analysis as a tool to study complex microbial communities in marine sediments: *Journal of Microbiological Methods*, v. 48, no. 2–3, p. 149-160, doi:10.1016/S0167-7012(01)00319-0.
- Ryu, B.-J., Collett, T. S., Riedel, M., Kim, G. Y., Chun, J.-H., Bahk, J.-J., Lee, J. Y., Kim, J.-H., and Yoo, D.-G., 2013, Scientific results of the Second Gas Hydrate Drilling Expedition in the Ulleung Basin (UBGH2): *Marine and Petroleum Geology*, v. 47, no. 0, p. 1-20, doi:10.1016/j.marpetgeo.2013.07.007.
- Saffer, D. M., and Tobin, H. J., 2011, Hydrogeology and Mechanics of Subduction Zone Forearcs: Fluid Flow and Pore Pressure: *Annual Review of Earth and Planetary Sciences*, v. 39, no. 1, p. 157-186, doi:10.1146/annurev-earth-040610-133408.
- Saffer, D. M., Flemings, P. B., Boutt, D., Doan, M. L., Ito, T., McNeill, L., Byrne, T., Conin, M., Lin, W., Kano, Y., Araki, E., Eguchi, N., and Toczko, S., 2013, In situ stress and pore pressure in the Kumano Forearc Basin, offshore SW Honshu from downhole measurements during riser drilling: *Geochemistry, Geophysics, Geosystems*, v. 14, no. 5, p. 1454-1470, doi:10.1002/ggge.20051.
- Santamarina, J. C., Dai, S., Jang, J., and Terzariol, M., 2012, Pressure Core Characterization Tools for Hydrate-Bearing Sediment: *Scientific Drilling*, v. 14, p. 44-48.
- Santamarina, J. C., Dai, S., Terzariol, M., Jang, J., Waite, W. F., Winters, W. J., Nagao, J., Yoneda, J., Konno, Y., Fujii, T., and Suzuki, K., 2015, Hydro-bio-geomechanical properties of hydrate-bearing sediments from Nankai Trough: *Marine and Petroleum Geology*, v. xxx, p. 1-17, doi:10.1016/j.marpetgeo.2015.02.033.

- Sassen, R., Sweet, S. T., Milkov, A. V., and Kennicutt II, M. C., 2001, Thermogenic vent gas and gas hydrate in the Gulf of Mexico slope: Is gas hydrate decomposition significant? *Geology*, v. 29, p. 107-110.
- Sassen, R., Roberts, H. H., Carney, R., Milkov, A. V., DeFreitas, D. A., Lanoil, B., and Zhang, C., 2004, Free hydrocarbon gas, gas hydrate, and authigenic minerals in chemosynthetic communities of the northern Gulf of Mexico continental slope: relation to microbial processes: *Chem. Geol.*, v. 205, p. 195-217, doi:10.1016/j.chemgeo.2003.1012.1032.
- Schoell, M., 1980, The hydrogen and carbon isotopic composition of methane from natural gases of various origins: *Geochimica et Cosmochimica Acta*, v. 44, no. 5, p. 649-661, doi:10.1016/0016-7037(80)90155-6.
- Schultheiss, P. J., Holland, M., Roberts, J., Huggett, Q., Druce, M., and Fox, P., PCATS: Pressure Core Analysis and Transfer System, *in* Proceedings 7th International Conference on Gas Hydrates, Edinburgh, Scotland, United Kingdom, July 17- 21, 2011 2011, p. 10.
- Sheppard, S. M. F., and Gilg, H. A., 1996, Stable isotope geochemistry of clay minerals: *Clay Minerals*, v. 31, p. 1-24.
- Shimada, H., Nemoto, N., Shida, Y., Oshima, T., and Yamagishi, A., 2008, Effects of pH and Temperature on the Composition of Polar Lipids in *Thermoplasma acidophilum* HO-62: *Journal of Bacteriology*, v. 190, no. 15, p. 5404-5411, doi:10.1128/JB.00415-08.
- Shipboard Scientific Party, 1986, Site 618, *in* Bouma, A. H., Coleman, J. M., and Meyer, A. W., eds., *Init. Repts. DSDP, Volume 96*.
- Smith, A. J., Flemings, P. B., Liu, X., and Darnell, K., 2014, The evolution of methane vents that pierce the hydrate stability zone in the world's oceans: *Journal of Geophysical Research: Solid Earth*, v. doi:10.1002/2013JB010686, p. 2013JB010686, doi:10.1002/2013JB010686.
- Smith, D. C., Spivak, A. J., Fisk, M. R., Haveman, S. A., and Staudigel, H., 2000, Tracer-based estimates of drilling-induced microbial contamination of deep sea crust: *Geomicrobiology*, v. 17, p. 2017-2219.
- Solomon, E. A., Spivack, A. J., Kastner, M., Torres, M. E., and Robertson, G., 2014, Gas hydrate distribution and carbon sequestration through coupled microbial methanogenesis and silicate weathering in the Krishna–Godavari Basin, offshore India: *Marine and Petroleum Geology*, v. 58, Part A, no. 0, p. 233-253, doi:10.1016/j.marpetgeo.2014.08.020.
- Stolper, D. A., Lawson, M., Davis, C. L., Ferreira, A. A., Neto, E. V. S., Ellis, G. S., Lewan, M. D., Martini, A. M., Tang, Y., Schoell, M., Sessions, A. L., and Eiler, J. M., 2014a, Formation temperatures of thermogenic and biogenic methane: *Science*, v. 344, no. 6191, p. 1500-1503, <http://www.sciencemag.org/content/344/6191/1500.abstract>.
- Stolper, D. A., Sessions, A. L., Ferreira, A. A., Santos Neto, E. V., Schimmelmann, A., Shusta, S. S., Valentine, D. L., and Eiler, J. M., 2014b, Combined  $^{13}\text{C}$ - $\text{D}$  and  $\text{D}$ - $\text{D}$  clumping in methane: Methods and preliminary results: *Geochimica et Cosmochimica Acta*, v. 126, no. 0, p. 169-191, doi:10.1016/j.gca.2013.10.045.
- Strapoć, D., Picardal, F. W., Turich, C., Schaperdoth, I., Macalady, J. L., Lipp, J. S., Lin, Y.-S., Ertel, T. F., Schubotz, F., Hinrichs, K.-U., Mastalerz, M., and Schimmelmann, A., 2008, Methane-Producing Microbial Community in a Coal Bed of the Illinois Basin: *Applied and Environmental Microbiology*, v. 74, no. 8, p. 2424-2432, doi:10.1128/AEM.02341-07.
- Sturt, H. F., Summons, R. E., Smith, K., Elvert, M., and Hinrichs, K.-U., 2004, Intact polar membrane lipids in prokaryotes and sediments deciphered by high-performance liquid chromatography/electrospray ionization multistage mass spectrometry—new biomarkers

- for biogeochemistry and microbial ecology: *Rapid Communications in Mass Spectrometry*, v. 18, no. 6, p. 617-628, doi:10.1002/rem.1378.
- Sun, X., and Turchyn, A. V., 2014, Significant contribution of authigenic carbonate to marine carbon burial: *Nature Geosci.*, v. 7, no. 3, p. 201-204, doi:10.1038/ngeo2070.
- Teichert, B. M. A., Johnson, J. E., Solomon, E. A., Giosan, L., Rose, K., Kocherla, M., Connolly, E. C., and Torres, M. E., 2014, Composition and origin of authigenic carbonates in the Krishna-Godavari and Mahanadi Basins, eastern continental margin of India: *Marine and Petroleum Geology*, v. 58, Part A, p. 438-460, doi:10.1016/j.marpetgeo.2014.08.023.
- Tissot, B. P., and Welte, D. H., 1978, *Petroleum formation and occurrence*, New York, Springer-Verlag.
- Tréhu, A. M., Ruppel, C., Holland, M., Dickens, G. R., Torres, M. E., Collett, T. S., Goldberg, D. S., Riedel, M., and Schultheiss, P., 2006, Gas hydrates in marine sediments: Lessons from scientific ocean drilling: *Oceanography*, v. 19, p. 124-142.
- Uchida, T., Waseda, A., and Namikawa, T., 2009, Methane accumulation and high concentration of gas hydrate in marine and terrestrial sandy sediments, *in* Collett, T. S., Johnson, A., Knapp, C., and Boswell, R., eds., *Natural gas hydrates—Energy resource potential and associated geologic hazards*, AAPG Memoir 89: Tulsa, Oklahoma, The American Association of Petroleum Geologists, p. 401-413.
- Valentine, D. L., 2002, Biogeochemistry and microbial ecology of methane oxidation in anoxic environments: a review: *Antonie van Leeuwenhoek*, v. 81, p. 271-282.
- Van Mooy, B. A. S., Roco, G., Fredricks, H. F., Evans, C. T., and Devol, A. H., 2006, Sulfolipids dramatically decrease phosphorus demand by picocyanobacteria in oligotrophic marine environments: *Proceedings of the National Academy of Sciences*, v. 103, no. 23, p. 8607-8612, doi:10.1073/pnas.0600540103.
- Vanneste, H., Kelly-Gerreyn, B. A., Connelly, D. P., James, R. H., Haeckel, M., Fisher, R. E., Heeschen, K., and Mills, R. A., 2011, Spatial variation in fluid flow and geochemical fluxes across the sediment-seawater interface at the Carlos Ribeiro mud volcano (Gulf of Cadiz): *Geochimica et Cosmochimica Acta*, v. 75, no. 4, p. 1124-1144, doi:10.1016/j.gca.2010.11.017.
- Vannucchi, P., Sage, F., Phipps Morgan, J., Remitti, F., and Collot, J.-Y., 2012, Toward a dynamic concept of the subduction channel at erosive convergent margins with implications for interplate material transfer: *Geochemistry, Geophysics, Geosystems*, v. 13, no. 2, p. n/a-n/a, 10.1029/2011GC003846.
- Wallmann, K., Aloisi, G., Haeckel, M., Obzhirov, A., Pavlova, G., and Tishchenko, P., 2006, Kinetics of organic matter degradation, microbial methane generation, and gas hydrate formation in anoxic marine sediments: *Geochimica et Cosmochimica Acta*, v. 70, no. 15, p. 3905-3927, doi:10.1016/j.gca.2006.06.003.
- Wallmann, K., Aloisi, G., Haeckel, M., Tishchenko, P., Pavlova, G., Greinert, J., Kutterolf, S., and Eisenhauer, A., 2008, Silicate weathering in anoxic marine sediments: *Geochimica et Cosmochimica Acta*, v. 72, no. 12, p. 2895-2918, doi:10.1016/j.gca.2008.03.026.
- Wallmann, K., Piñero, E., Burwicz, E., Haeckel, M., Hensen, C., Dale, A., and Ruepke, L., 2012, The global inventory of methane hydrate in marine sediments: A theoretical approach: *Energies*, v. 5, p. 2449-2498, doi:2410.3390/en5072449.
- Wang, D. T., Gruen, D. S., Sherwood, L. B., Hinrichs, K.-U., Stewart, L. C., Holden, J. F., Hristov, A. N., Pohlman, J. W., Morrill, P. L., Könneke, M., Delwiche, K. B., Reeves, E. P., Sutcliffe, C. N., Ritter, D. J., Seewald, J. S., McIntosh, J. C., Hemond, H. F., Kubo,

- M., Cardace, D., Hoehler, T. M., and Ono, S., 2015, Nonequilibrium clumped isotope signals in microbial methane: *Science*, v. 10.1126/science.aaa4326.
- Wei, W., 2007, Fluid origins, paths, and fluid-rock reactions at convergent margins, using halogens, Cl stable isotopes, and alkali metals as geochemical tracers, <http://www.escholarship.org/uc/item/7xj422dq>.
- Wei, W., Kastner, M., Rosenbauer, R., Chan, L. H., and Weinstein, Y., 2010, Alkali elements as geothermometers for ridge flanks and subduction zones: Proceedings of the 13th International Conference on Water-Rock Interaction WRI-13, CRC Press, Taylor & Francis Group, London, UK, p. 223-227.
- Wellsbury, P., Goodman, K., Barth, T., Cragg, B. A., Barnes, S. P., and Parkes, R. J., 1997, Deep marine biosphere fuelled by increasing organic matter availability during burial and heating: *Nature*, v. 388, no. 6642, p. 573-576.
- White, D. C., Bobbie, R. J., King, J. D., Nickels, J. S., and Amoe, P., 1979, Lipid Analysis of Sediments for Microbial Biomass and Community Structure, in Lichtfeld, C. D., and Seyfried, P. L., eds., *Methodology for Biomass Determination and Microbial Activities in Sediments*, v. doi:10.1520/STP38143S: Philadelphia, ASTM Special Technical Publication 673, p. 87-103.
- Whiticar, M. J., Faber, E., and Schoell, M., 1986, Biogenic methane formation in marine and freshwater environments: CO<sub>2</sub> reduction vs. acetate fermentation—Isotope evidence: *Geochimica et Cosmochimica Acta*, v. 50, no. 5, p. 693-709, [http://dx.doi.org/10.1016/0016-7037\(86\)90346-7](http://dx.doi.org/10.1016/0016-7037(86)90346-7).
- Whiticar, M. J., Hovland, M., Kastner, M., and Sample, J. C., 1995, Organic geochemistry of gases, fluids, and hydrates at the Cascadia accretionary margin: Proceedings ODP, Scientific Results, p. 385-397.
- Whiticar, M. J., 1999, Carbon and hydrogen isotope systematics of bacterial formation and oxidation of methane: *Chemical Geology*, v. 161, no. 1-3, p. 291-314, doi:10.1016/S0009-2541(99)00092-3.
- Wiese, K., and Kvenvolden, K. A., 1993, Introduction to microbial and thermal methane, in Howell, D. G., ed., *The Future of Energy Gases*, U.S.G.S. Professional Paper 1570: Denver, Colorado, U.S. Geological Survey, p. 13-20.
- Xu, W., and Ruppel, C., 1999, Predicting the occurrence, distribution, and evolution of methane gas hydrate in porous marine sediments: *Journal of Geophysical Research*, v. 104, no. 3, p. 5081-5095.
- Yamamoto, K., Inada, N., Kubo, S., Fujii, T., Suzuki, K., and Konno, Y., 2012, Pressure core sampling in the Eastern Nankai Trough: DOE/NETL Fire in the Ice Newsletter, v. 12, no. 2.
- Yeh, H.-W., 1980, DH Ratios and late-stage dehydration of shales during burial: *Geochimica et Cosmochimica Acta*, v. 44, no. 2, p. 341-352, doi:10.1016/0016-7037(80)90142-8.
- Yoshioka, H., Maruyama, A., Nakamura, T., Higashi, Y., Fuse, H., Sakata, S., and Bartlett, D. H., 2010, Activities and distribution of methanogenic and methane-oxidizing microbes in marine sediments from the Cascadia Margin: *Geobiology*, v. 8, no. 3, p. 223-233, doi:10.1111/j.1472-4669.2009.00231.x.
- You, C. F., Castillo, P. R., Gieskes, J. M., Chan, L. H., and Spivack, A. J., 1996, Trace element behavior in hydrothermal experiments: Implications for fluid processes at shallow depths in subduction zones: *Earth and Planetary Science Letters*, v. 140, p. 41-52, doi:10.1016/0012-821x(96)00049-0.



Zhang, G., Yang, S., Zhang, M., Liang, J., Lu, J., Holland, M., Schultheiss, P., and GMGS2 Science Team, 2014, GMGS2 Expedition Investigates Rich and Complex Gas Hydrate Environment in the South China Sea: Fire in the ice, Methane hydrate newsletter, US Department of Energy, Office of Fossil Energy, National Energy Technology Laboratory., v. 14, no. 1, p. 1-5.

## **Appendix C: Methane Hydrate Pressure Coring and Analysis: Gulf of Mexico Scientific Planning Workshop Report**

### **1. Executive Summary**

On March 9th and 10th, 2015, 27 scientists and engineers from around the world held a Scientific Planning workshop to prioritize research goals, identify gaps, and plan for the analysis of pressure cores acquired during UT's DOE-supported hydrate drilling program entitled 'Deepwater Methane Hydrate Characterization and Scientific Assessment'. The specific goals of the workshop were to:

1. Determine the critical information needed to be gained from pressure coring
2. Determine what types of analyses needed to be done to achieve the critical information.
3. Take this information to develop an overall scientific plan for drilling, logging and coring that addresses these goals and that was technically feasible.
4. Develop a collaborative team composed of scientists and institutions enthused with participating in the research program

During the first day, there was an overview of the existing technical capabilities. This was followed by a discussion of previous experimental results and proposed future experimental results. The second day was spent in breakout groups developing a strategy for the experimental plan.

A Science Plan for DOE Drilling was developed that looked at both on ship and shore-based sampling and analysis. Over sixty-five measurements were identified as either critical or useful for meeting the three Scientific Objectives of the project: understanding the characteristic physical properties of methane hydrate reservoirs in sand; identifying the source gas and migration mechanism for methane hydrate systems in sand-rich marine reservoirs; and gaining a better understanding of the production potential of methane hydrate reservoirs in sand sediment.

### **Day 1 Presentations**

#### ***Goal 1. Review of scientific, technical and logistical goals of the DOE drilling experiment***

Drilling for Methane Hydrates in the Gulf of Mexico [P. Flemings]: Peter Flemings opened the workshop by reviewing the agenda, reviewing the goals of the workshop, and presenting an overview of the proposal submitted to the International Ocean Discovery Program (IODP) entitled 'Genesis of Methane Hydrate in Coarse-Grained Systems: Northern Gulf of Mexico Slope'. The workshop goals represent one component of the main project goals which are to drill, core, and perform in-situ testing of sand-rich marine hydrate reservoirs in the Gulf of Mexico. The workshop focus was to outline a process and a scientific plan for the successful analysis of pressure cores.

Specific workshop goals were reviewed for each day including how the breakout sessions would work on the 2<sup>nd</sup> day of the workshop. The UT project will characterize methane hydrate morphology, concentration, formation permeability, geochemistry, and in situ thermodynamic conditions in marine sand reservoirs in the Gulf of Mexico. The project will acquire new data never acquired in U.S.; strengthen the understanding of methane hydrate morphology, concentration, physical properties, geochemistry, and geological characteristics; and provide a foundation to model and predict production behavior of these reservoirs. In the longer term the project will impact our understanding of energy security in the U.S. by characterizing methane hydrates in marine sands as a first step toward demonstrating that production is feasible. Decades of DOE investment in basic research in unconventional resources (shale gas/tight gas/tight oil) transformed US energy supply and methane hydrate resources are of a similar scale.

Flemings reviewed the three phases of the UT project: Phase 1- will analyze and identify drilling locations, submit drilling proposal, to develop the drilling & experimental program, and to submit an International Ocean Discovery Program (IODP) Phase 1 Report

Complementary Program Proposal; Phase 2 (Tasks 5-11) will strengthen the project by nurturing a Complementary Project Proposal (CPP), securing research vessel access, refining the science and operational plans, contracting pressure coring teams, completing National Environmental Policy Act (NEPA) requirements, and securing a Pressure Coring and Core Analysis System; and Phase 3 (through Task 16) will finalize drilling & operational plans, complete drilling program, and perform post-cruise science.

The top scientific objectives the project intends to address were reviewed as well as the some of the specific knowledge to be gained through the IODP.

- What are the characteristic physical properties of methane hydrate reservoirs in sand?
  - What is the response of methane hydrate deposits in coarse-grained systems to natural and induced perturbations?
  - What controls the formation of methane hydrates in coarse-grained sedimentary systems?
- What is the source gas and migration mechanism for methane hydrate systems in sand-rich marine reservoirs?
- What is the production potential of methane hydrate reservoirs in sand sediment?

### ***Review of current pressurized coring capabilities and recent scientific achievements in pressure coring analysis***

Pressure Core Handling & Processing and PCATS [P. Schultheiss]: Peter Schultheiss of Geotek Coring presented a review of pressure core handling and analysis showing the history of pressure coring development and data starting with the Pressure Core Barrel (PCB) developed in the early 70's. Schultheiss also reviewed three wireline pressure coring tools; the Pressure Core Barrel (PCB), the Pressure Core Sampler (PCS), and the Pressure Temperature Core Sampler (PTCS) developed for JOGMEC. He then described the transition from stand-alone coring tools (that had no significant analysis capability) to the development and details of integrated systems that included wire-line coring tools that are compatible with pressure core analysis capabilities. These included the coring tools developed during the European Hydrate Autoclave Coring Equipment (HYACE and HYACINTH) programs as well as the successors to earlier PCS tools including the current PCTB (Pressure Coring Tool - with Ball valve) as is intended to be used in this project.

Schultheiss provided a detailed review of the Pressure Core Analysis and Transfer System (PCATS), which included schematics and descriptions of core transfer, cutting, storage, and analysis. Analysis capability of the PCATS includes non-destructive techniques of X-ray imaging, p-wave velocity, and gamma density measurements; and a depressurization chamber with gas collection. He then discussed subsampling for longer term storage, third-party analysis, and the PCATS Tri-axial test apparatus. Third parties measurements included P&S wave velocities, resistivity, and penetration strength. The tri-axial system is capable of measuring tri-axial stress/strain properties, elastic properties from resonance data, and direct-flow permeability.

Schultheiss concluded his review with a discussion about GOM2 emphasizing that the program should help maximize information and improvements from the various expeditions outlined including: tool deployment and procedures; core recovery and quality; and core handling, testing, sub-sampling.

Expected Performance, Predicted Core Quality [T. Pettigrew]: Tom Pettigrew of Pettigrew Engineering then presented on pressure coring tools detailing the steps of coring starting with getting the corer down the hole. Pettigrew discussed the different PCTB Bit configurations and provided detail schematics on running the hole, landing the corer, coring, latching the core, and pulling the core. Pettigrew then provided a list of initial issues resulting from the on shore testing of the PCTB (under the prior DOE-Chevron JIP project) that have been resolved and discussed future testing plans.

Pressure Core Characterization Tools - Overview, components, and possibilities [S. Dai]: Sheng Dai, of DOE's National Energy Technology Laboratory (professor, Georgia Institute of Technology), together with Bill Waite of the United States Geological Survey (USGS) gave a review of the Pressure Core Characterization Tools (PCCT) outlining the different testing chambers used

at Georgia Tech. Dai discussed the manipulation of pressure cores showing many photos of the PCCT manipulation and analysis tools. The analysis tools reviewed included the Instrumented Pressure Testing Chamber (IPTC), Bio Chamber, Effective Stress Chamber, Direct Shear Chamber, and Controlled Depressurization Chamber. The IPTC was first deployed in Gulf of Mexico for JIP drilling in 2005, but has also been used for hydrate drilling programs in India (NGHP-01), Korea (UBGG-1), and Japan. It has eight ports for direct contact sensors like the Bender Element to measure S-wave velocity ( $V_s$ ), the Pinducer to measure Compressional Wave Velocity ( $V_p$ ), the Strength Cone to measure  $S_u$ , and the Electrical probe to measure  $\sigma_{ele}$ ; among others. Waite followed with a discussion of the PCCT deployment in Japan for the Japanese-led Nankai Trough project. Waite's description of the key conclusions about properties and behavior of hydrate-bearing sands is included in: (Santamarina et al, 2012)

Gas Hydrate Bearing Sediment Core Sample Characterization & Re-Formation/Dissociation Investigations [C Koh]: Carolyn A. Koh of the Colorado School of Mines, explored the structure and composition of hydrates using MAS-NMR, Raman, and Gas Chromatography of Liquid-Nitrogen preserved and re-Pressurized Hydrate-Bearing Sediments (HBS) Core Samples. Koh also discuss the centers experience in Mineralogy, Multi-scale imaging, Physical Properties, and Acoustic resonance at the center and with their partners at the USGS and Lawrence-Berkeley National Labs. (Koh et al., 2011)

Nankai-Trough 2012/2013 Drilling/Coring/Production Test [K. Kiyofumi]: Kiyofumi Suzuki reviewed JOGMEC's experience with pressure coring, including some discussion of their drilling procedure and some resulting drilling-induced structures found in their cores from the Nankai Trough 2012/2013 Drilling/Coring/Production Test. He also described the geological setting of the Nankai Trough Test site and briefly touched on results from their gas production test.

Methane Hydrate Pressure Coring and Analysis: Gulf of Mexico Scientific Planning Workshop [B Anderson]: Brian Anderson, of West Virginia University, reviewed production tests within hydrate reservoirs and efforts to simulate production of methane hydrates. Industry tests were pursued in the 1970's. However the rates were low and the issue was not pursued. In 1998 and 2002 tests were performed at Mallik (NW Canada). In 2007 BP-DOE-USGS performed the "Mt. Elbert" (MPU) field test. In 2007 & 2008 Mallik (NW Terr. Canada) Depressurization. In 2011/2012 the Ignik Sikumi Test (PBU) was performed. This was a scientific field trial of chemical exchange that was followed by a period of depressurization. Finally, in 2013 JOGMEC/METI/JAPEX performed a depressurization test in the Nankai Trough, offshore Japan. Anderson then reviewed results from the 'International Methane Hydrate Reservoir Simulator Code Comparison Project' (Anderson et al, 2011). Anderson then discussed efforts to simulate the production of the Orange Sand at the Walker Ridge 313 Block in the Gulf of Mexico (Myshakin et al., 2012). Anderson pointed out that we have not yet demonstrated how to produce hydrates in complex and sensitive settings. We have not demonstrated the ability to obtain and maintain required flow rates to be economic. There is the possibility that there will be high water production. In addition, the sands are unconsolidated. We must understand the implications for sand production, subsidence, and well stability.

Development of the Pressure-core Nondestructive Analysis Tools (PNATs) for Methane Hydrate Sedimentary Cores– Part 1 (Tool Development, Permeability) [Y. Konno]: Yoshihiro Konno and Jun Yoneda of National Institute of Advanced Industrial Science and Technology (AIST) gave a review of their Pressure Core Non-Destructive Analysis Tools (PNATs). PNATs can provide reservoir parameters such as hydrate saturation, X-ray CT image, wave velocity, permeability, mechanical properties and so on, under pressure. The components of PNATS include the Core Storage Cabinet (PNATs-Cabinet), the X-Ray CT Imaging Tool (PNATs-X), the Manipulation and Cutting (PNATs-Manipulator), the Transparent Acrylic Cell Triaxial Testing System (PNATs-TACTT), the Depressurization Testing Chamber (PNATs-AIST IPTC), and Sub Sampling (PNATs-Sub Sampler); some of these components are under construction. Konno then described analysis of pressure cores that were recovered from hydrate-bearing sandy sediments in the Eastern Nankai Trough, June – July 2012. In this operation, PCATs by Geotek was used for Chikyu onboard operation, and the USGS PCCT and PNATs (under development) by AIST were used at the AIST Hokkaido center. Konno compared different measurements of effective permeability for hydrate-bearing sandy sediments in the range of 1–100 md and showed P-wave velocity, gamma density, and X-ray image measurements from PCATs. Konno also discussed

the comparison of compressional wave velocity ( $v_p$ ) and hydrate saturation data to Helgerud (2001) model predictions to understand hydrate morphology (Yamamoto and Ruppel, 2015 ).

Development of the Pressure-core Nondestructive Analysis Tools (PNATs) for Methane Hydrate Sedimentary Cores – Part 2 (Mechanical properties) [J. Yoneda]: Yoneda discussed AIST's investigation of the mechanical properties of hydrate-bearing sediments using PNATs (Yamamoto and Ruppel, 2015). Pressure-core-based tri-axial compression testing was conducted on natural gas hydrate-bearing sediments while maintaining the pore pressure within the hydrate stability zone. Several images of hydrate bearing sediments taken through the observation window of the PNATs-TACTT were shown as well as images through the transparent acrylic cell during compression. Yoneda discussed their process for determining shearing speeds and showed stress and strain curves for the sediments measured. Yoneda demonstrated that sediments containing natural gas hydrate exhibited brittle failure, while hydrate-free sediments exhibited ductile failure; the effective friction angle for the sediments without hydrate was shown to be 30° to 37°; the strengthening of sediments is mainly caused by the cohesion induced by hydrates (Santamarina et al., 2015). Yoneda closed with a discussion of triaxial testing of hydrate samples placed in liquid nitrogen prior to loading in the experimental device.

Laboratory Generation of Methane Hydrates [D. Meyer]: Dylan Meyer, David DiCarlo and Kehua You of the University of Texas Institute for Geophysics (UTIG) together with Tim Kneafsey of Lawrence Berkeley National Laboratory discussed their findings with artificially generated methane hydrates specifically looking at salinity-buffered hydrate formation and hydrate front propagation (You et al., 2015).

Science and Logistics Planning for Pressure Core Analyses (Post-PCATS) [C. Ruppel]: Carolyn Ruppel, Project Chief of the USGS Gas Hydrates Project, opened her presentation by posing the question of how we establish Gulf of Mexico coring analysis program by 2018. Ruppel reviewed the history of coring analysis starting with the ODP PCS system in the mid 90's which could only analyze short depressurized core, touched on the history of Parr Vessels, and re-capped the current analysis capability of the US Portable Pressure Core Analysis System. Ruppel stressed the importance of measuring and using multiple properties from pressure cores as indirect constraints on the corresponding reservoir characteristics. Ruppel reviewed their experience with Georgia Institute of Technology on the integration of PCCT measurements with other datasets. She emphasized the value and difficulty of obtaining pressure cores. She emphasized that unlike conventional cores where extensive analysis is done on every core, pressure cores must be carefully sub-divided and dedicated to specific analyses. Ruppel emphasized the exhaustive pressure core analysis is best done substantially after the cruise (following post-cruise science planning workshops) and provided evidence that the cores would still provide accurate data even after delays as long as they are stored, transported, and tested at pressure and temperature conditions within the hydrate stability zone.

Ruppel proposed that four phases are fundamental to a scientific plan for coring: 1.) what needs to be thought through before the cruise?; 2.) what should be done on the ship?; 3.) what needs to be done after the cruise?; and 4.) how will the data be integrated post-cruise? Ruppel then provided some key logistical considerations for the PCCT Analyses, including the requirements for cold rooms, ventilation, and especially shippable storage chambers. Ruppel then discussed what was needed immediately and in the long term for the project. Short term needs include obtaining US Department of Transportation and American Bureau of Shipping Certification of storage chambers, and altering the PCCT. Long term needs include extending the use of the bio cell for subsampling, refining the core sawing and depressurization process, and possibly creating a portable visualization cell, which would be a community responsibility. A framework for the management of the project similar to the Interlab Comparison Team was suggested.

Pore-scale physical property characterization [H. Daigle]: Hugh Daigle of the University of Texas, Petroleum and Geosystems Engineering, presented on his experience with pore-scale physical property characterization stressing the importance of pore-scale characterization in conventional cores to obtain the proper interpretation of pressure core results as pore-scale properties have a strong influence on the distribution of hydrates, even in sands. Daigle reviewed the specific pore-scale properties that influence the location and rates of hydrate formation including: the availability of methane and water; pore

size (magnitude and distribution); grain surface area; and surface charge/cation exchange capacity. Daigle proposed several parameters that should be included in the scientific plan and then discussed some specific considerations for the measurement of permeability, diffusion, pore-size, surface area, and exchangeable cations. (Daigle and Dugan, 2011)

Determination of In-Situ Salinity [K. You]: Kehua You, Dylan Meyer and Kristopher Darnell of UT reviewed the basic approach to depressurization (Milkov, 20114). She then presented a model to describe the mass balance and pressure vs. time behavior during depressurization. Discussion then focused on what approach should be taken for depressurization. Depressurization can be done directly from the coring autoclave. This involves dedicating one of a limited number of autoclaves to depressurization (hence it cannot be used for further coring) and also involves sacrificing the entire pressure core to the analysis. However, it has the advantage that it limits the amount of additional fluid added to the system. Alternatively, depressurization could be done in PCATS, but this is not recommended. Finally, depressurization of a sub-sample could be done in a decompression chamber after sub-sampling with PCATS. There was discussion about the impact of having significant amounts of fluid introduced into the PCATS during the subsampling. It was pointed out that the water added into PCATS could be tagged with a tracer and thus we could use this to correct measurements at the end to the in situ concentration. Kehua next emphasized the importance of developing an approach for extracting pore fluids from hydrate samples under in-situ pressure and temperature conditions. Three techniques were reviewed. 1) Efforts have been made to use a syringe system with the IPTC system. However, the sample is stiff, it is difficult to remove the fluids, and the pressure conditions are changed when using a syringe. 2) A second approach is to displace the fluid from the pressurized core using a non-wetting and high viscosity fluid under high pressure. This technique has been proposed but not yet successfully demonstrated. 3) Finally, one direct approach is to use a downhole wireline tool such as Schlumberger's MDT to directly pull a fluid sample from the formation.

Pressure core based study of GH in the Ulleung Basin, Korea [J.Y. Lee]: Joo Young Lee, of the Korean Institute of Geoscience and Mineral Resources (KIGAM), Gas Hydrate R&D Organization gave a presentation titled "Pressure core based study of GH in the Ulleung Basin, Korea". They successfully recovered twenty-one pressure cores described how the cores were subdivided for gas quantification, production experiments, and 3D CT scans. Lee reviewed some of the data acquired on-board the ship including p-wave, gamma density, X-ray, and 3D CT scans showing a gas hydrate saturation of 0.21 (Lee et al., 2013). Lee also showed some of the data from subsequent on-shore results of degassing and production tests. The depressurization test quantified electrical resistivity, vertical effective stress, void ratio, and hydraulic conductivity as a function of three depressurization steps.

## Day 2 Breakout Sessions

### ***Goal 4. Developing a Science Plan for DOE Drilling: a plan for the successful acquisition and analysis of pressure cores***

The scientific objectives of the project were again reviewed in preparation for the breakout sessions. The scientific objectives were posed as follows:

1. What are the characteristic physical properties of methane hydrate reservoirs in sand?
2. What is the response of methane hydrate deposits in coarse-grained systems to natural and induced perturbations?
3. What controls the formation of methane hydrates in coarse-grained sedimentary systems?
4. What is the source gas and migration mechanism for methane hydrate systems in sand-rich marine reservoirs?
5. What is the production potential of methane hydrate reservoirs in sand sediment?

### Breakout Session 1 & 2:

The first two breakout sessions focused on the following questions. Breakout 1: What are the key parameters we need to achieve our research goals [our scientific objectives above]? And Breakout 2: Measurement logistics. How and where do we measure these desired parameters? In Breakout Session 1, the workshop attendees were divided into two groups. Group A,

led by Tetsuya Fujii and Brian Anderson, focused on the parameters needed for understanding Reservoir Genesis. Group B, led by Hugh Daigle, focused on the parameters associated with Reservoir Perturbation. In Breakout Session 2 the workshop attendees were again divided into two groups. Group A led by Hugh Daigle and Peter Schultheiss, discussed what parameters could be measured on the ship while the second group, Group B led by Sheng Dai & Bill Waite, discussed what could be measured on shore, post-cruise, again assuming no limitations on shipping pressurized cores to different laboratories. Between both groups over sixty-five different measurements were identified as being critical or useful for meeting the scientific objectives as shown in Table 7. A spreadsheet with the full list of parameters and their corresponding scientific objective and possible measurement location (ship or shore) is available and a view the contents can be found in the Table 6.

**Table 6: Output of Breakout Sessions 1 & 2**

| Measurement   | Shipboard | Land | Both | Pressure core (under pressure) | Conv. Or degassed Core | Whole core? | Tool                   | Potential Contact person | Status of Development |
|---|-----------|------|------|--------------------------------|------------------------|-------------|------------------------|--------------------------|-----------------------|
| <b>Gas chemistry</b>                                  |           |      |      |                                |                        |             |                        |                          |                       |
| Headspace: Hydrocarbons, CO2 and Fixed Gases (N2, O2) | x         |      |      | N                              | Y                      | Y           | GC                     |                          |                       |
| Void Gas: Hydrocarbons, CO2 and Fixed Gases (N2, O2)  | x         |      |      | N                              | Y                      | Y           | GC                     |                          |                       |
| Pressure core degassing (gas & fluid analysis)        |           |      | x    | Y                              | N                      | Y           | GC                     |                          |                       |
| Pure hydrate degassing (gas & fluid analysis)         |           |      | x    | Y                              | Y                      | Y           | GC                     |                          |                       |
| Stable carbon isotopes (C1, C2, CO2)                  |           |      | x    | Y                              | Y                      | Y           | Mass spec/spectrometer |                          |                       |
| Stable hydrogen isotopes (C1)                         |           |      | x    | Y                              | Y                      | Y           | Mass spec              |                          |                       |
| d13C-DIC  |           | x    |      | Y                              | Y                      | Y           | Mass spec              |                          |                       |
| Location of dissociating gas hydrate (infrared)       | x         |      |      | N                              | Y                      | Y           | IR track               |                          |                       |



| Measurement                         | Shipboard | Land | Both | Pressure core (under pressure) | Conv. Or degassed Core | Whole core? | Tool                  | Potential Contact person | Status of Development |
|-------------------------------------|-----------|------|------|--------------------------------|------------------------|-------------|-----------------------|--------------------------|-----------------------|
| <b>Fluid chemistry</b>              |           |      |      |                                |                        |             |                       |                          |                       |
| Chloride                            | x         |      |      | Y                              | Y                      | Y           | Titration             |                          |                       |
| Salinity                            | x         |      |      | Y                              | Y                      | Y           | Refractometer         |                          |                       |
| pH                                  | x         |      |      | Y                              | Y                      | Y           | Titration             |                          |                       |
| SO4, Br (if not measured shipboard) | x         |      |      | Y                              | Y                      | Y           | IC                    |                          |                       |
| Alkalinity                          | x         |      |      | Y                              | Y                      | Y           | Titration             |                          |                       |
| Other fluid chemistry               |           | x    |      | Y                              | Y                      | Y           | Other (ICP, IC, etc.) |                          |                       |
| Dissolved gas concentrations        | x         |      |      | Y                              | N                      | Y           | ??                    | USGS / NETL              |                       |
|                                     |           |      |      |                                |                        |             |                       |                          |                       |
| <b>Solid chemistry</b>              |           |      |      |                                |                        |             |                       |                          |                       |
| Total Carbon (TC)                   |           | x    |      | N                              | Y                      | N           | CHN analyzer          |                          |                       |
| Bulk Carbonate                      |           | x    |      | N                              | Y                      | N           | CHN analyzer          |                          |                       |
| d14C-DIC                            |           | x    |      | N                              | Y                      | N           | Mass spec             |                          |                       |
| C and N stable isotopes             |           | x    |      | N                              | Y                      | N           | Mass spec             |                          |                       |
| Rock eval/pyrolysis                 |           | x    |      | N                              | Y                      | N           | Rock eval             |                          |                       |
| Cation exchange capacity            |           | x    |      | N                              | Y                      | N           | Titration             |                          |                       |
| Mineralogy/elemental                |           | x    |      | N                              | Y                      | N           | XRD/XRF               | CKOH                     |                       |
| Hydrate structure                   |           | x    |      | Y                              | N                      | Y           | Raman/MAS-NMR         | CKOH                     |                       |
| Cage occupancy                      |           | x    |      | Y                              | N                      | Y           | Raman                 | CKOH                     |                       |

| Measurement  | Shipboard | Land | Both | Pressure core (under pressure) | Conv. Or degassed Core | Whole core? | Tool                           | Potential Contact person | Status of Development |
|--|-----------|------|------|--------------------------------|------------------------|-------------|--------------------------------|--------------------------|-----------------------|
| <b>Basic index properties (can be done on disturbed samples)</b> |           |      |      |                                |                        |             |                                |                          |                       |
| Liquid and Plastic Limit   |           |      | x    | N                              | Y                      | N           | Atterberg test                 | TEST                     |                       |
| Specific Surface   |           |      | x    | N                              | Y                      | N           | Methylene blue                 | USGS / CSM / LBNL        |                       |
| Grain Size distribution  |           |      | x    | N                              | Y                      | N           | Sedigraph/Settling column/LPSA | USGS / LBNL / NETL / CSM |                       |
| Fine grain distribution  |           | x    |      | N                              | Y                      | N           | Sedigraph/Settling column/LPSA | USGS                     |                       |
| Grain Shape/texture (optical, cryo-SEM)                          |           |      | x    | N                              | Y                      | N           | optical microscope, SEM        | USGS / +OTHERS           |                       |
| Hydrate grain size/shape   |           | x    |      | N                              | Y                      | N           | Cryo SEM                       | LAURA STERN, USGS        |                       |
| Grain density  |           |      | x    | N                              | Y                      | N           | Helium pycnometer              | USGS / NETL / LBNL       |                       |

| Measurement   | Shipboard | Land | Both | Pressure core (under pressure) | Conv. Or degassed Core | Whole core? | Tool                                   | Potential Contact person                      | Status of Development |
|---|-----------|------|------|--------------------------------|------------------------|-------------|--|---|-----------------------|
| <b>Physical properties (require minimally disturbed samples)</b>            |           |      |      |                                |                        |             |  |   |                       |
| In situ pressure, temperature, & stress                                     | x         |      |      | N                              | N                      | N           | LWD/penetrometer                       |   |                       |
| Thermal conductivity  |           |      | x    | Y                              | Y                      | Y           | PCCT/Needle probe                      |   |                       |
| thermodynamics of hydrates at T,P,gas composition, water chemistry/salinity |           |      | x    | Y                              | N                      | Y           | PCCT                                   | SCHULTHEISS / BOSWELL / ALL WITH P CORE       |                       |
| Hydrate metastability during dissociation                                   |           | x    |      | Y                              | N                      | Y           | PCCT                                   | SCHULTHEISS / BOSWELL / ALL WITH P CORE / CSM |                       |
| Paleomag  |           |      | x    | N                              | Y                      | N           | Magnetometer                           | HUGH  |                       |
| Hydrate saturation  |           |      | x    | Y                              | Y                      | Y           | PCATS/PCCT/Chlorinity/resistivity etc. | ALL   |                       |
| Continuous density from gamma ray   |           |      | x    | Y                              | Y                      | Y           | MSCL/PCATS/PCCT                        |   |                       |
| P-wave velocities   |           |      | x    | Y                              | Y                      | Y           | MSCL/PCATS                             | BOSWELL                                       |                       |
| Electrical resistance   |           |      | x    | Y                              | Y                      | Y           | MSCL/PCCT                              | USGS  |                       |
| Natural gamma radiation   |           |      | x    | N                              | Y                      | Y           | MSCL                                   |   |                       |
| Magnetic susceptibility   |           |      | x    | N                              | Y                      | Y           | MSCL                                   |   |                       |
| Moisture and Density (MAD) (porosity)                                       |           |      | x    | N                              | Y                      | N           | Helium pycnometer                      | USGS / LBNL                                   |                       |
| Wettability   |           | x    |      | Y                              | Y                      | Y           | Porous plate/PCATS/PCCT                | CSM   |                       |

| Measurement  | Ship board | Land | Both | Pressure core (under pressure) | Conv. Or degassed Core | Whole core? | Tool                               | Potential Contact person | Status of Development |
|--|------------|------|------|--------------------------------|------------------------|-------------|------------------------------------|--------------------------|-----------------------|
| <b><i>Physical properties (require minimally disturbed samples)</i></b>  |            |      |      |                                |                        |             |                                    |                          |                       |
| Gas saturation below base of stability   | x          |      |      | Y                              | N                      | Y           | PCATS                              |                          |                       |
| Archie parameters  |            | x    |      | Y                              | Y                      | Y           | Resistivity probe                  | HUGH                     |                       |
| Routine sedimentological description   | x          |      |      | Y                              | Y                      | both        | X-ray, visual description          | USGS / UT AUSTIN         |                       |
| Heat capacity  |            |      | x    | N                              | N                      | N           | Calculation                        |                          |                       |
| Capillary pressure & residual water saturation   |            | x    |      | Y                              | Y                      | Y           | Porous plate/PCATS/PCCT            | HUGH                     |                       |
| X-Ray Imagery/CT imagery/micro-CT imagery/synchrotron  |            |      | x    | Y                              | Y                      | Y           | X-ray/micro-CT/CT                  | HUGH                     |                       |
| Permeability/relative perm/anisotropy  |            |      | x    | Y                              | Y                      | Y           | PCATS/PCCT/CRS/Triax               | BOSWELL                  |                       |
| Pore scale salinity distribution and hydrate distribution during production - using a micromodel with a tracer/florescent; micro-CT with BaCl2 exclusion |            | x    |      | Y                              | N                      | Y           | CT/micro-CT                        | NETL / LBNL / CSM        |                       |
| Diffusion coefficient  |            | x    |      | Y                              | Y                      | Y           | NMR                                | HUGH / MELANIE           |                       |
| Pore size  |            | x    |      | Y                              | Y                      | Y           | micro-CT/porous plate/MICP/SEM/NMR | NETL / LBNL / CSM / HUGH |                       |
| Fines mobility   |            | x    |      | Y                              | Y                      | Y           | PCATS/PCCT                         |                          |                       |
| Shear velocity   |            |      | x    | Y                              | N                      | Y           | PCCT/PCATS                         | BOSWELL                  |                       |

| Measurement  | Shipboard | Land | Both | Pressure core (under pressure) | Conv. Or degassed core | Whole core? | Tool       | Potential Contact Person | Status of Development |
|--|-----------|------|------|--------------------------------|------------------------|-------------|------------|--------------------------|-----------------------|
| <b><i>Mechanical properties</i></b>  |           |      |      |                                |                        |             |            |                          |                       |
| Shear Strength   |           |      | x    | Y                              | Y                      | Y           | PCCT/PCATS | BOSWELL                  |                       |
| Compressibility/overconsolidation ratio/settling due to dissociation           |           | x    |      | Y                              | Y                      | Y           | PCCT/PCATS | BOSWELL                  |                       |
| Attenuation/damping  |           | x    |      | Y                              | Y                      | Y           | PCCT/PCATS |                          |                       |
| Stress-strain constitutive properties  |           | x    |      | Y                              | Y                      | Y           | PCCT/PCATS | BOSWELL                  |                       |
| K0   |           | x    |      | Y                              | Y                      | Y           | PCCT/PCATS | BOSWELL                  |                       |
| Friction angle   |           | x    |      | Y                              | Y                      | Y           | PCCT/PCATS | BOSWELL                  |                       |
|  |           |      |      |                                |                        |             |            |                          |                       |
| <b><i>Microbiology</i></b>   |           |      |      |                                |                        |             |            |                          |                       |
| Biomass (cell numbers, DNA quantity)   |           | x    |      | Y                              | N                      | Y           | PCCT       | COLWELL                  |                       |
| Microbial diversity (16S rDNA community characterization, intact polar lipids) |           | x    |      | Y                              | N                      | Y           | PCCT       | COLWELL                  |                       |
| Microbial activity (14-C-labeled substrate turnover, metatranscriptomics)      |           | x    |      | Y                              | N                      | Y           | PCCT       | COLWELL                  |                       |
| Functional capabilities (metagenomes, targeted functional genes)               |           | x    |      | Y                              | N                      | Y           | PCCT       | COLWELL                  |                       |
| Cultivations, growth studies   |           | x    |      | Y                              | N                      | Y           | PCCT       | COLWELL                  |                       |

**Table 7: Identified ship-board measurements contributing toward scientific goals 1-3.**

|    | Analysis Category<br>1. Can be done on disturbed samples<br>2. Require minimally disturbed samples | Measurement:<br>Measurements in black text were collected to start the discussion, Measurements in light text were added during the discussion. | What are the characteristics of Hydrate Reservoirs? | What is the Source Gas and Migration Mechanism? | What is the Production Potential? | Ship | Land | Both |
|----|--|---|---|---|-----------------------------------|------|------|------|
| 1  | Gas chemistry  | Headspace: Hydrocarbons, CO2 and Fixed Gases (N2, O2)   |   | 2   |                                   | x    |      |      |
| 2  | Gas chemistry  | Void Gas: Hydrocarbons, CO2 and Fixed Gases (N2, O2)  |   | 2   |                                   | x    |      |      |
| 3  | Gas chemistry  | Pressure core degassing (gas & fluid analysis)  | 1   | 2   |                                   | x    | x    | x    |
| 4  | Gas chemistry  | Pure hydrate degassing (gas & fluid analysis)   | 1   | 2   |                                   | x    | x    | x    |
| 5  | Gas chemistry  | Stable carbon isotopes (C1, C2, CO2)  |   | 2   |                                   | x    | x    | x    |
| 6  | Gas chemistry  | Stable hydrogen isotopes (C1)   |   | 2   |                                   | x    | x    | x    |
| 7  | Gas chemistry  | d13C-DIC  |   | 2   |                                   |      | x    |      |
| 8  | Gas chemistry  | Location of dissociating gas hydrate (infrared)   | 1   |   |                                   | x    |      |      |
| 9  | Fluid chemistry  | Chloride  | 1   |   |                                   | x    |      |      |
| 10 | Fluid chemistry  | Salinity  | 1   |   |                                   | x    |      |      |
| 11 | Fluid chemistry  | pH  | 1   | 2   |                                   | x    |      |      |
| 12 | Fluid chemistry  | SO4, Br (if not measured shipboard)   |   | 2   |                                   | x    |      |      |
| 13 | Fluid chemistry  | Alkalinity  | 1   | 2   |                                   | x    |      |      |
| 14 | Fluid chemistry  | Other fluid chemistry   |   |   |                                   |      | x    |      |
| 15 | Fluid chemistry  | Dissolved gas concentrations  |   |   |                                   | x    |      |      |
| 16 | Solid chemistry  | Total Carbon (TC)   |   | 2   |                                   |      | x    |      |

|    | Analysis Category<br>1. Can be done on disturbed samples<br>2. Require minimally disturbed samples | Measurement:<br>Measurements in black text were collected to start the discussion, Measurements in light text were added during the discussion. | What are the characteristics of Hydrate Reservoirs? | What is the Source Gas and Migration Mechanism? | What is the Production Potential? | Ship | Land | Both |
|----|--|---|---|---|-----------------------------------|------|------|------|
| 17 | Solid chemistry  | Bulk Carbonate  |   | 2   |                                   |      | x    |      |
| 18 | Solid chemistry  | d14C-DIC  | 1   | 2   |                                   |      | x    |      |
| 19 | Solid chemistry  | C and N stable isotopes   |   | 2   |                                   |      | x    |      |
| 20 | Solid chemistry  | Rock eval/pyrolysis   | 1   | 2   |                                   |      | x    |      |
| 21 | Solid chemistry  | Cation exchange capacity  | 1   |   |                                   |      | x    |      |
| 22 | Solid chemistry  | Mineralogy/elemental  | 1   |   |                                   |      | x    |      |
| 23 | Solid chemistry  | Hydrate structure   | 1   | 2   | 3                                 |      | x    |      |
| 24 | Solid chemistry  | Cage occupancy  | 1   | 2   | 3                                 |      | x    |      |
| 25 | Basic index properties <sup>1</sup>  | Liquid and Plastic Limit  | 1   |   |                                   | x    | x    | x    |
| 26 | Basic index properties <sup>1</sup>  | Specific Surface  | 1   |   |                                   | x    | x    | x    |
| 27 | Basic index properties <sup>1</sup>  | Grain Size distribution   | 1   |   |                                   | x    | x    | x    |
| 28 | Basic index properties <sup>1</sup>  | Fine grain distribution   |   |   |                                   |      | x    |      |
| 29 | Basic index properties <sup>1</sup>  | Grain Shape/texture (optical, cryo-SEM)   | 1   |   |                                   | x    | x    | x    |
| 30 | Basic index properties <sup>1</sup>  | Hydrate grain size/shape  |   |   |                                   |      | x    |      |
| 31 | Basic index properties <sup>1</sup>  | Grain density   | 1   |   |                                   | x    | x    | x    |
| 32 | Physical properties  | In situ pressure, temperature, & stress   | 1   | 2   | 3                                 | x    |      |      |
| 33 | Physical properties <sup>2</sup>   | Thermal conductivity  | 1   |   | 3                                 | x    | x    | x    |
| 34 | Physical properties <sup>2</sup>   | Thermodynamics of hydrates at T,P,gas composition, water chemistry/salinity   | 1   |   |                                   | x    | x    | x    |
| 35 | Physical properties <sup>2</sup>   | Hydrate metastability during dissociation   | 1   |   |                                   |      | x    |      |
| 36 | Physical properties <sup>2</sup>   | Paleomag  | 1   |   |                                   | x    | x    | x    |
| 37 | Physical properties <sup>2</sup>   | Hydrate saturation  | 1   | 2   | 3                                 | x    | x    | x    |
| 38 | Physical properties <sup>2</sup>   | Continuous density from gamma ray   | 1   |   |                                   | x    | x    | x    |

|    | Analysis Category<br>1. Can be done on disturbed samples<br>2. Require minimally disturbed samples | Measurement:<br>Measurements in black text were collected to start the discussion, Measurements in light text were added during the discussion.                      | What are the characteristics of Hydrate Reservoirs? | What is the Source Gas and Migration Mechanism ? | What is the Production Potential? | Ship | Land | Both |
|----|--|--|---|--|-----------------------------------|------|------|------|
| 39 | Physical properties <sup>2</sup>   | P-wave velocities  | 1   |  |                                   | x    | x    | x    |
| 40 | Physical properties <sup>2</sup>   | Electrical resistance  | 1   |  |                                   | x    | x    | x    |
| 41 | Physical properties <sup>2</sup>   | Natural gamma radiation  | 1   |  |                                   | x    | x    | x    |
| 42 | Physical properties <sup>2</sup>   | Magnetic susceptibility  | 1   |  |                                   | x    | x    | x    |
| 43 | Physical properties <sup>2</sup>   | Moisture and Density (MAD) (porosity)  | 1   |  |                                   | x    | x    | x    |
| 44 | Physical properties <sup>2</sup>   | Wettability  | 1   |  | 3                                 |      | x    |      |
| 45 | Physical properties <sup>2</sup>   | Gas saturation below base of stability   | 1   | 2  | 3                                 | x    |      |      |
| 46 | Physical properties <sup>2</sup>   | Archie parameters  | 1   |  |                                   |      | x    |      |
| 47 | Physical properties <sup>2</sup>   | Routine sedimentological description   | 1   | 2  | 3                                 | x    |      |      |
| 48 | Physical properties <sup>2</sup>   | Heat capacity  | 1   |  | 3                                 | x    | x    | x    |
| 49 | Physical properties <sup>2</sup>   | Capillary pressure & residual water saturation   | 1   |  | 3                                 |      | x    |      |
| 50 | Physical properties <sup>2</sup>   | X-Ray Imagery/CT imagery/micro-CT imagery/synchrotron  | 1   |  |                                   | x    | x    | x    |
| 51 | Physical properties <sup>2</sup>   | Permeability/relative perm/anisotropy  | 1   | 2  | 3                                 | x    | x    | x    |
| 52 | Physical properties <sup>2</sup>   | Pore scale salinity distribution and hydrate distribution during production - using a micromodel with a tracer/florescent; micro-CT with BaCl <sub>2</sub> exclusion | 1   |  |                                   |      | x    |      |
| 53 | Physical properties <sup>2</sup>   | Diffusion coefficient  | 1   | 2  |                                   |      | x    |      |
| 54 | Physical properties <sup>2</sup>   | Pore size  | 1   | 2  |                                   |      | x    |      |
| 55 | Physical properties <sup>2</sup>   | Fines mobility   | 1   |  | 3                                 |      | x    |      |
| 56 | Physical properties <sup>2</sup>   | Shear velocity   | 1   |  |                                   | x    | x    | x    |
| 57 | Mechanical properties  | Shear Strength   | 1   |  |                                   | x    | x    | x    |
| 58 | Mechanical properties  | Compressibility/over consolidation ratio/settling due to dissociation  | 1   |  | 3                                 |      | x    |      |



|    | Analysis Category<br>1. Can be done on disturbed samples<br>2. Require minimally disturbed samples | Measurement:<br>Measurements in black text were collected to start the discussion, Measurements in light text were added during the discussion. | What are the characteristics of Hydrate Reservoirs? | What is the Source Gas and Migration Mechanism? | What is the Production Potential? | Ship | Land | Both |
|----|--|---|---|---|-----------------------------------|------|------|------|
| 59 | Mechanical properties  | Attenuation/damping   | 1   |   |                                   |      | x    |      |
| 60 | Mechanical properties  | Stress-strain constitutive properties   | 1   |   | 3                                 |      | x    |      |
| 61 | Mechanical properties  | K0  | 1   |   | 3                                 |      | x    |      |
| 62 | Mechanical properties  | Friction angle  | 1   |   | 3                                 |      | x    |      |
| 63 | Microbiology   | Biomass (cell numbers, DNA quantity)  | 1   | 2   |                                   |      | x    |      |
| 64 | Microbiology   | Microbial diversity (16S rDNA community characterization, intact polar lipids)  | 1   | 2   |                                   |      | x    |      |
| 65 | Microbiology   | Microbial activity (14-C-labeled substrate turnover, metatranscriptomics)   | 1   | 2   |                                   |      | x    |      |
| 66 | Microbiology   | Functional capabilities (metagenomes, targeted functional genes)  | 1   | 2   |                                   |      | x    |      |
| 67 | Microbiology   | Cultivations, growth studies  | 1   | 2   |                                   |      | x    |      |

The parameters identified as critical for answering the question “What are the characteristic physical properties of methane hydrate reservoirs in sand?”, scientific objective 1, that need to be collected shipboard were: gas and liquid analysis of from degassed pressure cores, gas and liquid analysis from degassed pure hydrate, identification of the location of dissociating gas hydrate using infrared analysis, liquid analysis of salinity, pH, and alkalinity. The gas and liquid analysis of degasses core could and should also be done on shore. Many indices of physical and basic properties of rocks should also be measured on the ship and on shore.

In addition to the parameters that should be measured on ship and shore, several other parameters were discussed that did not need to be or could not be measured on the ship and so should be measured on shore. These include identification of the solid chemistry, wettability, pore size, permeability, and various indications of microbiology.

The parameters identified as critical for answering the question “What is the source gas and migration mechanism for methane hydrate systems in sand-rich marine reservoirs?”, scientific objective 2, that need to be collected shipboard in addition to what was already identified for Objective 1 were: measurements of headspace and void gas chemistry, measurements of gas stable carbon and hydrogen isotopes, and sulphate and bromide fluid chemistry.

The parameters identified as critical for answering the question “What is the production potential of methane hydrate reservoirs in sand sediment?”, scientific objective 3, that need to be collected shipboard and on shore all fall into what was already identified for Objective 1.

### Breakout Session 3:

In Breakout Session 3, our goal was to develop a scientific plan for a marine hydrate drilling program. In order to facilitate the discussion, a scenario based on previous drilling at Walker Ridge 313 was outlined with text and figures by Flemings. The scenario was summarized as follows:

1. The location has previously been drilled and logged with LWD (logging while drilling). Two important hydrate-bearing intervals in sands are present.
2. The proposed plan is to acquire a full suite of wireline logs during the experiment.
3. One MDT (Modular Dynamics Test) will be performed
4. Up to 10 PCS cores may be acquired.
5. Pressurized cores can be shipped to specific land-based laboratories.
6. You have 10 to 15 days of ship time

David Divins led the discussion in Breakout Session 3. The broad goal was to outline a broad PCS experimental plan that will be applied to sites where we pressure core. The discussion was divided into two parts: 1) what is the plan with respect to pressure cores; and 2) What are some of the other non-pressure core measurements that need to be made.

### Pressure Core Analyses:

We outline a broad experimental plan for ship-based and shore based pressure core research as shown in Table 8 and Figure 3.

Subsamples of the pressure cores will be processed at research institutions around the globe with capability to manipulate and analyze pressure cores. The Pressure Core Characterization Tool (PCCT) is one such system now housed by the USGS at Woods Hole (Santamarina et al., 2012; Santamarina et al., 2015).

### *Core Sampling:*

High-resolution pressure core sampling will occur through the transitions from mudstone to gas hydrate-bearing sands. These pressure cores will quantify gas hydrate saturations, and characterize background pore water profiles. Limited testing will occur at sea due to time constraints, but each pressure core should have a detailed sub-sampling program guided by pressure velocity, density, and cat-scans of the entire core taken immediately upon recovery. It seemed obvious to the group that PCATS, or a PCATS like system, should be used on the ship for this and to execute the sub-sampling plan once established.

Exceptions to running every core through velocity, density and cat-scans might be to ensure minimal contamination from the surrounding drilling fluid/sea water through rapid degassing followed by dunking in Liquid Nitrogen (this might also be done on cores where the pressure is not holding to preserve as much hydrate as possible). Another exception might be slow depressurization of the core straight from the Autoclave while on the ship. This, while ensuring minimal contamination of the cores from the surrounding drilling fluid/sea water, holds up the autoclave for maybe too long of a period and is risky since there is no PCATS data to suggest the presence of hydrate. Also, unlike depressurization in the PCCT, there is no way to penetrate the liner to ensure the lithology will be preserved nor there be a way to measure the internal core temperature. There was some consideration for storing the PCATS samples in immiscible fluid or gas instead of sea water, but nothing came out of the discussion.

*Ship-board Analysis:*

After the site is finished and the detailed sub-sampling plan determined. One to two sub-sections (~10 cm) from each core will be cut and degassed shipboard for quantitative analysis of total methane concentration and gas hydrate saturation, and other sub-samples will be dedicated to shipboard triaxial analyses. Any sample degassed on ship and not kept in liquid nitrogen should receive all of the shipboard measurement routinely done on conventional cores and/or be shipped for additional shore based analysis as conventional cores.

*Sub-Sampling and Shore-based Analyses:*

Sub-samples for physical properties and pore water geochemical analyses will be stored under pressure in storage containers, and then processed on shore.  $\leq 1.2$  meter is the envisioned standard length and they should be stored at 4-6 degrees. Sub-samples for shore-based microbiology experiments and analyses including measuring the rates and activities of methanogens will be stored under in situ conditions; these samples will span all pressure coring depths and will particularly focus on the interface between mudstone and sand.

Some sub-samples for shore-based characterization should be stored in liquid nitrogen after very rapidly depressurization. The group discussed that it might be possible to cryo-freeze under full pressure, then depressurize, then put in nitrogen, then ship to shore. Thus reducing ice crystal formation and damage to the sample.

Once on-shore, further sub-sampling of pressure cores will be done. Pressurized flow experiments will be planned to look at capillary pressure, measuring permeability at different hydrate saturations over a long term pressure deactivation in some type of tri-axial vessel. Conventional CT or Micro CT will be run as well as run controlled depressurization experiments looking at the thermodynamics of hydrates with temperature, pressure, gas composition, water chemistry/salinity, and geochemistry. Microbiology measurements that don't depend upon pressure but might depend on selecting samples of different grain size could be done on degassed sub-samples if the lithology is known. The group also touched on the possibility of using Cryo-SEM to image hydrates which, if possible, would be very significant.

**Table 8: Board Experimental Plan for Ship and Shore Based Analyses**

|     |                                   |  |
|-----|-----------------------------------|--|
| 1)  | Routine Coring Measurements       | Velocity, bulk density, and linear X-rays will be done to all pressure cores immediately as they are recovered on deck.  |
| 2)  | Temporary Core Storage            | Next, 3 m pressure cores that are acquired will be placed in a storage chamber. Any pressure cores recovered at the site will be stored until the conclusion of drilling the site.   |
| 3)  | Complete Sampling Plan            | After the Site is finished, a sampling plan will be developed that will subdivide plans for Shipboard Analysis and those for Post-Cruise Analysis:   |
| 3a) | Shipboard Sampling and Analysis   | <p>Shipboard measurements will be used to confirm hydrate presence and concentration. We will cut 1 or 2 samples from each pressure core (~10 cm) and put these into a cell. We will measure permeability, measure pore fluid chemistry, measure hydrate concentration and perform degassing.</p> <p>On select pressure cores, pore water will be extracted under pressure to sample true background composition. Once the samples are degassed they will be analyzed for standard properties in the shipboard core flow. This will either be through a syringe type device or through displacement of fluid during a permeability test.</p> |
| 3b) | Sampling for Shore Based Analysis | <p><u>Storage under pressure:</u> Cores will be subsampled and placed in pressure containers for delivery to individual research institutions. These samples could be later further subsampled on shore.</p> <p><u>Liquid Nitrogen:</u> Some samples will be stored at atmospheric pressure in liquid nitrogen. The samples will be rapidly depressurized and plunged into liquid nitrogen. Plans are underway to develop an approach to freeze the samples under pressure and then depressurize these samples and plunge into nitrogen.</p>   |
| 4)  | Shore-Based Analysis              | Shore based analysis will include analyses now done with the PCCT such as permeability, mechanical properties, hydrate saturation and the taking of biologic samples. We hope to develop the ability to perform Micro CT on pressure core samples.   |

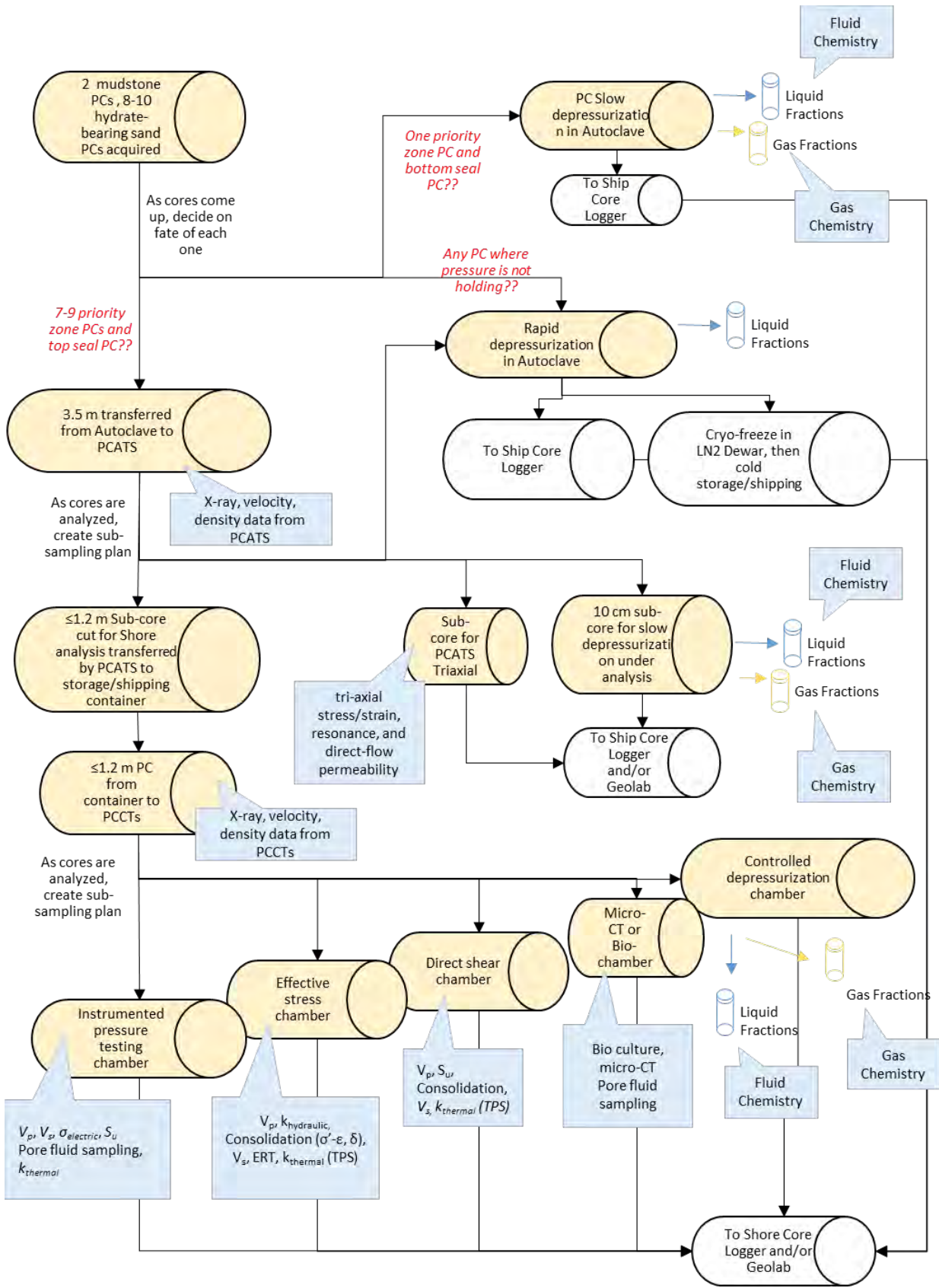


Figure 3: Board Experimental Plan for Ship and Shore Based Analyses

**Additional Important Measurements:**

Beyond pressure coring, the following was recognized to be of critical importance. Whole round conventional cores from the mudstone need to be taken to look at pore water composition and material properties in the absence of hydrate. In the well, in-situ temperature (penetrometer, or APC), and in-situ pressure (penetrometer, XPT) should be taken. A Single probe drawdown and sample (XPT) might be done. The group discussed packing off an interval. If you pack off an interval, the borehole volume overwhelms the measurement in low permeability material. A drill-stem across a packed interval might be appropriate. Some type of borehole monitoring or borehole experiment could be done such as monitoring the long term temperature and pressure as the system equilibrates by leaving the thermal and acoustic fiber in the hole. The application of the Simple Cabled Instrument for Measuring Parameters In-Situ (SCIMPI) from the IODP should be explored as well as a possible tracer injection.

**The Project Team*****Goal 5. Developing a project team composed of scientists and institutions enthused with participating in research program***

Workshop attendees from around the globe freely contributed their knowledge in pressure coring to the workshop. When, and if, pressure coring occurs in the DOE-sponsored Gulf of Mexico Methane Hydrate drilling program, it is envisioned that workshop participants will be given the opportunity to participate in the experimental analysis of the pressure cores. This may occur through shipment of cores to particular institutions or through participation at facilities with particular areas of expertise.

**Summary**

On March 9th and 10th, 2015, 27 scientists and engineers from around the world held a Scientific Planning workshop to prioritize research goals, identify gaps, and plan for the analysis of pressure cores acquired during UT's DOE-supported hydrate drilling program entitled 'Deepwater Methane Hydrate Characterization and Scientific Assessment'. During the first day, there was an overview of the existing technical capabilities. This was followed by a discussion of previous experimental results and proposed future experimental results. The second day was spent in breakout groups strategizing components of the experimental plan.

Scientific, technical and logistical goals of the DOE drilling experiment were reviewed by the group including a discussion of the impact of the project to the energy security in the US and abroad as predicted based on the new knowledge that will be gained through this project and on the history of similar projects in unconventional reservoirs which have transformed the US energy supply.

A Science Plan for DOE Drilling was developed that looked at both on ship and shore-based sampling and analysis. Over sixty-five measurements were identified as either critical or useful for meeting the three Scientific Objectives of the project: understanding the characteristic physical properties of methane hydrate reservoirs in sand; identifying the source gas and migration mechanism for methane hydrate systems in sand-rich marine reservoirs; and gaining a better understanding of the production potential of methane hydrate reservoirs in sand sediment.

A project team composed of scientists and institutions enthused with participating in research program was identified.

**Table 9: Methane Hydrate Pressure Coring and Analysis: Gulf of Mexico Scientific Planning Workshop Agenda****Monday, March 9<sup>th</sup>****Overview and Technical Review**

- 8:00 am Research Program and Workshop Goals [P. Flemings]
- 8:30 am Pressure Core Handling & Processing and PCATS [P. Schultheiss]
- 9:00 am Discussion
- 9:15 am PCS - Expected Performance, Predicted Core Quality [T. Pettigrew]
- 9:45 am Discussion
- 10:00 am Break
- 10:15 am Pressure Core Characterization Tools (PCCT) [S. Dai]
- 10:45 am Discussion
- 11:00 am Applications of PCCT [W. Waite]
- 11:30 am Discussion
- 12:00 pm Lunch

**Experience and Approach**

## Previous Experimental Results/Field Experiences

- 1:00 pm Laboratory-Scale Experimental and Modeling Investigations of Hydrate Bearing Sediments [C. Koh]
- 1:20 pm Nankai-Trough 2012/2013 Drilling/Coring/Production Test [K. Suzuki]
- 1:40 pm Reservoir Modeling - Coring Program Input Parameters [B. Anderson]
- 2:00 pm Development of the Pressure-core Nondestructive Analysis Tools (PNATs) for Methane Hydrate Sedimentary Cores – Part 1 (Tool Development, Permeability) [Y. Konno]
- 2:20 pm Development of the Pressure-core Nondestructive Analysis Tools (PNATs) for Methane Hydrate Sedimentary Cores – Part 2 (Mechanical Properties) [J. Yoneda]
- 2:40 pm Laboratory Generation of Methane Hydrates [D. Meyer, T. Kneafsey, D. DiCarlo, K. You]

**Proposed Experimental Program to Achieve Project Goals**

- 3:00 pm Proposed Pressure Core Analyses - Science Planning, Workflow, & Logistics [C. Ruppel]
- 3:40 pm Pore-scale Physical Property Characterization [H. Daigle]
- 4:00 pm Determination of In-Situ Salinity [K. You, D. Meyer, K. Darnell]
- 4:20 pm Pressure Core Study of Gas Hydrates in Ulleung Basin [J. Lee]
- 4:20 pm Review of Key Parameters for Science Goals
- 7:00 pm Dinner at the Flemings' Home

**Tuesday, March 10<sup>th</sup>****Scientific Plan Development**

- 8:00 am Review of Goals for Day [P. Flemings]
- 8:30 am Breakout 1: What are the key parameters we need to achieve our research goals?
- 10:00 am Breakout 2: Measurement logistics: how and where do we measure the desired parameters?
- 11:30 am Brief Working Group Reports
- 12:00 pm Lunch

**Experimental Program and Next Steps**

- 1:00 pm Breakout 3: Reconvene to define experimental program
- 4:00 pm Define near term action items to move forward

**Table 10: Attendees and Contact Information**

| Last Name | First Name | Email                       | Organization                          |
|-----------|------------|-----------------------------|---------------------------------------|
| Anderson  | Brian      | brian.anderson@mail.wvu.edu | West Virginia University              |
| Baker     | Rick       | richard.baker@netl.doe.gov  | National Energy Technology Laboratory |

|             |                |                                      |  |
|-------------|----------------|--------------------------------------|--|
| Dai         | Sheng          | dais@netl.doe.gov                    | National Energy Technology Laboratory                            |
| Daigle      | Hugh           | daigle@austin.utexas.edu             | The University of Texas at Austin                                |
| Darnell     | Kris           | kdarnell@utexas.edu                  | The University of Texas at Austin                                |
| DiCarlo     | David          | dicarlo@mail.utexas.edu              | The University of Texas at Austin                                |
| Divins      | David          | david.divins@unh.edu                 | University of New Hampshire                                      |
| Espinoza    | Nicolas        | espinoza@austin.utexas.edu           | The University of Texas at Austin                                |
| Flemings    | Peter          | pfflemings@jsg.utexas.edu            | The University of Texas at Austin                                |
| Fujii       | Tetsuya        | fujii-tetsuya@jogmec.go.jp           | Japan Oil, Gas and Metals National Corporation                   |
| Holland     | Melanie        | melanie@geotek.co.uk                 | Geotek Coring  |
| Kneafsey    | Tim            | tjkneafsey@lbl.gov                   | Lawrence Berkeley Lab  |
| Koh         | Carolyn        | cannkoh01@gmail.com                  | Colorado School of Mines   |
| Konno       | Yoshihiro      | yoshihiro-konno@aist.go.jp           | National Institute of Advanced Industrial Science and Technology |
| Lee         | Joo Young      | jyl@kigam.re.kr                      | Korean Institute of Geoscience and Mineral Resources             |
| Meyer       | Dylan          | meyerdw3@gmail.com                   | The University of Texas at Austin                                |
| Nagao       | Jiro           | jiro.nagao@aist.go.jp                | National Institute of Advanced Industrial Science and Technology |
| Pettigrew   | Tom            | pettigrew.engineering@windstream.net | Pettigrew Engineering  |
| Polito      | Peter          | peter.polito@jsg.utexas.edu          | The University of Texas at Austin                                |
| Ruppel      | Carolyn        | cruppel@usgs.gov                     | United States Geological Survey                                  |
| Schultheiss | Peter          | peter@geotek.co.uk                   | Geotek Coring  |
| Seol        | Yongkoo        | yongkoo.seol@netl.doe.gov            | National Energy Technology Laboratory                            |
| Suzuki      | Kiyofumi       | suzuki-kiyofumi@jogmec.go.jp         | Japan Oil, Gas and Metals National Corporation                   |
| Tachau      | Anna           | anna.tachau@austin.utexas.edu        | The University of Texas at Austin                                |
| Waite       | William (Bill) | wwaite@usgs.gov                      | United States Geological Survey                                  |
| Yoneda      | Jun            | jun.yoneda@aist.go.jp                | National Institute of Advanced Industrial Science and Technology |
| You         | Kehua          | khyouml@gmail.com                    | The University of Texas at Austin                                |

## References

- Anderson, B. J., Hancock, S., Wilson, S., Enger, C., Collett, T., Boswell, R., and Hunter, R., 2011, Formation pressure testing at the Mount Elbert Gas Hydrate Stratigraphic Test Well, Alaska North Slope: Operational summary, history matching, and interpretations: *Marine and Petroleum Geology*, v. 28, no. 2, p. 478-492.
- Daigle, H., and Dugan, B., 2011, Capillary controls on methane hydrate distribution and fracturing in advective systems: *Geochemistry, Geophysics, Geosystems*, v. 12, no. 1, p. n/a-n/a.
- Helgerud, M.B., 2001, Wave speeds in gas hydrate and sediments containing gas hydrate: A laboratory and modeling study: Palo Alto, California, Stanford University, Ph.D. dissertation, 248 p., illust.
- Koh, C. A., Sloan, E. D., Sum, A. K., and Wu, D. T., 2011, Fundamentals and Applications of Gas Hydrates: *Annual Review of Chemical and Biomolecular Engineering*, v. 2, no. 1, p. 237-257.



- Lee, J. Y., Jung, J. W., Lee, M. H., Bahk, J. J., Choi, J., Ryu, B. J., and Schultheiss, P., 2013, Pressure core based study of gas hydrates in the Ulleung Basin and implication for geomechanical controls on gas hydrate occurrence: *Marine and Petroleum Geology*, v. 47, p. 85-98.
- Milkov, A. V., 2004, Global estimates of hydrate-bound gas in marine sediments: how much is really out there?: *Earth-Science Reviews*, v. 66, no. 3–4, p. 183-197.
- Myshakin, E. M., Gaddipati, M., Rose, K., and Anderson, B. J., 2012, Numerical simulations of depressurization-induced gas production from gas hydrate reservoirs at the Walker Ridge 313 site, northern Gulf of Mexico: *Marine and Petroleum Geology*, v. 34, no. 1, p. 169-185.
- Santamarina, J. C., Dai, S., Jang, J., and Terzariol, M., 2012, Pressure Core Characterization Tools for Hydrate-Bearing Sediment: *Scientific Drilling*, v. 14, p. 44-48.
- Santamarina, J. C., Dai, S., Terzariol, M., Jang, J., Waite, W. F., Winters, W. J., Nagao, J., Yoneda, J., Konno, Y., Fujii, T., and Suzuki, K., 2015, Hydro-bio-geomechanical properties of hydrate-bearing sediments from Nankai Trough: *Marine and Petroleum Geology*, v. xxx, p. 1-17.
- Yamamoto, K., and Ruppel, C., 2015, Gas hydrate drilling in Eastern Nankai, *Marine and Petroleum Geology*, Volume 66, p. 295-496.
- You, K., Kneafsey, T. J., Flemings, P. B., Polito, P., and Bryant, S. L., 2015, Salinity-buffered methane hydrate formation and dissociation in gas-rich systems: *Journal of Geophysical Research: Solid Earth*, v. 120, no. 2, p. 643-661.

## **Appendix D: Hybrid Pressure Coring Tool with Ball Valve (PCTB) 2015 Laboratory Test Program**



## HYBRID PRESSURE CORING TOOL WITH BALL VALVE (PCTB) 2015 LABORATORY TEST PROGRAM

GEOTEK LTD DOCUMENT NO. UT1-2015 (R2)

PREPARED FOR:

UNIVERSITY OF TEXAS

PREPARED BY:

GEOTEK CORING INC  
2698 South Redwood Rd, Suite N,  
West Valley City,  
Utah 84119,  
United States

T: +1 801 631 2874  
F: +1 801 886 9040  
E: [info@geotekcoring.com](mailto:info@geotekcoring.com)  
W: [www.geotekcoring.com](http://www.geotekcoring.com)

| ISSUE | REPORT STATUS | PREPARED | APPROVED | DATE     |
|-------|---------------|----------|----------|----------|
| R2    | Final Report  | JA/JR    | JA/PS    | 29/01/16 |



# TABLE OF CONTENTS

EXECUTIVE SUMMARY ..... 1

1. INTRODUCTION ..... 2

1.1. TRT ANALYSIS AND RECOMMENDATIONS ..... 2

1.2. TRT REMEDIAL ACTION ..... 3

*Figure 1. Flow slots added to the Ball Valve Release Collet (a) and to the Ball Follower (b)..... 3*

*Figure 2. Full Function Pressure Test setup..... 3*

1.3. ADDITIONAL IMPROVEMENTS ..... 4

2. TEST PROGRAM ..... 5

2.1. FULL FUNCTION PRESSURE TEST ..... 5

2.1.1. FULL FUNCTION PRESSURE TEST PLAN ..... 5

2.1.2. FULL FUNCTION PRESSURE TEST PROCEDURE ..... 6

2.1.3. FULL FUNCTION PRESSURE TEST RESULTS ..... 7

2.2. INNER TUBE AND CORE LINER IMPLOSION TESTS ..... 8

*Figure 3. Internally balanced test rod assembly keeps axial compression forces from affecting core liner. .... 9*

2.2.1. INNER CORE TUBE AND CORE LINER IMPLOSION TEST PLAN ..... 9

2.2.2. INNER TUBE AND CORE LINER IMPLOSION TEST RESULTS ..... 10

APPENDICES ..... 11

1. INNER TUBE IMPLOSION CALCULATIONS ..... 11

2. INNER TUBE AND CORE LINER IMPLOSION TEST ASSEMBLY DRAWING ..... 12

3. PCTB005A, FULL FUNCTION PRESSURE TEST PROCEDURE ..... 13

4. FULL FUNCTION PRESSURE TEST CHARTS 1-17 ..... 22

5. IMPLOSION TEST PRESSURE CHARTS ..... 36

PCTB 2015 Lab Test Program Final Report



Document No. UT1-2015 (R2)

ii

Geotek Coring Inc



## EXECUTIVE SUMMARY

### Full Function Pressure Testing

A modified Hybrid Pressure Coring Tool with Ball Valve (PCTB) was developed under a DOE funded contract between Chevron Development Co. and Aumann & Associates, Inc. (AAI). New tools were manufactured to the modified design during 2013-2014. Final Acceptance Tests (FAT) were conducted on the tools and they were field tested at the Catoosa Test Facility (CTF) in Oklahoma in November, 2013. Performance was not satisfactory because pressure was not retained in the tool correctly. A Technical Review Team (TRT) was convened in an attempt to determine the cause and recommend solutions and a recommended test plan to verify acceptable performance before any future field test or operation was undertaken.

The TRT recommended: a) that an engineering study be undertaken to review all aspects of the autoclave and pressure control section functions with regard to pressure retention and b) that an additional full function hydrostatic lab tests to be carried out to determine the root cause of the pressure retention failure.

Geotek Coring subsequently reviewed the design of the autoclave and pressure section and identified possible causes for the failure to reliably trap pressure in the autoclave. Modifications to some components were undertaken prior to performing 'Full Function Pressure Tests' in the laboratory which practically simulate the down-hole operation for pressure retention.

Several initial Full Function Pressure Tests were conducted that proved the ability of the PCTB to hold full pressure reliably with little or no leakage when the pressure control section operated properly. The results of these tests indicated that the primary problems associated with pressure retention had been found and resolved. It should be noted that these provisional TRT tests were conducted with water and not drilling mud.

With the subsequent detailed Full Function Pressure Tests (March 25 through April 10, 2015) a 10.4 lb/gal water base mud was used during 14 tests that all proved successful in demonstrating the correct functioning of the tool.

### Implosion Testing

In addition to the full function pressure tests a series of inner tube and core liner implosion tests were run to characterize the performance of these components. Implosion tests were carried out with and without the core liners with both the original thin wall stainless steel inner tube and with the new thick wall alloy steel tubes. The test conditions closely simulated downhole conditions with the exception that a hydrostatic pressure was used instead of a pressure created by the flow. The tests were conducted using a PCTB II autoclave as a pressure vessel with the inner tube and liner in the normal coring position.

Implosion pressures were obtained from the downhole recorder data and also observed on the digital pressure gages attached to the manifolds. It was concluded that the heavy wall inner tube may increase core liner implosion pressure by a small amount but the main benefit to using the heavy wall steel inner tube is that the tube was not damaged when the plastic core liner imploded. The heavy wall steel inner tube by itself did not implode even at 4000 psi. The results agreed reasonably well with the calculated value for inner tube implosion strength.



## 1. INTRODUCTION

A modified Hybrid Pressure Coring Tool with Ball Valve (PCTB) was developed under a DoE funded contract between Chevron Development Co. and Aumann & Associates, Inc. (AAI). New tools were manufactured to the modified design during 2013-2014. Final Acceptance Tests (FAT) were conducted on the tools and they were field tested at the Catoosa Test Facility (CTF) in Oklahoma in November, 2013. Performance was not satisfactory. A Technical Review Team (TRT) was convened in an attempt to determine the cause and recommend solutions and a recommended test plan to verify acceptable performance before any future field test or operation was undertaken.

One of the main issues identified was the failure of the autoclave to hold pressure and an apparent failure of the pressure control section to boost the autoclave pressure as it was designed to do. Pressure was not retained during the dimensional tests or coring runs even though the ball valve was closed and appeared to operate properly on several runs. At a minimum, a hydrostatic pressure of from 333 psi (0.45 psi per foot x 740 ft) to 522 psi (0.45 psi per foot x 11600 ft) should have been recovered even with no pressure boost from the pressure control section. The maximum pressure recovered was around 100 psi.

The pressure boost from the pressure control section also did not occur and this was verified by the fish pill recorder data. Note that this would result if the ball valve closure was delayed and did not close immediately or if there was a leak somewhere else. It did not mean the pressure section did not function.

The return spring jumped coils and jammed on at least one dimensional test preventing the ball from fully closing. This return spring was also made assembly difficult at times and also was observed to jam during pre-run testing.

### 1.1. TRT ANALYSIS AND RECOMMENDATIONS

The TRT reviewed participant's notes and the drilling record regarding the PCTB pressure retention failures. Also, several horizontal Full Function Pressure Tests were conducted by AAI at their facilities. The horizontal Full Function Pressure Test configuration encases the autoclave and pressure control section in chambers that can be used to simulate actual bottom hole pressures. Pressures above, below and inside the autoclave can be controlled and monitored while manipulating the position of the inner PCTB components, simulating wireline operations. Downhole recorders were used to record autoclave and annular pressures. The tests showed a pressure draw down of over 180 psi due to the increase in chamber volume as the inner tube plug continues to move up after ball closure. The tests proved that the pressure draw down effect is lessened, but not eliminated, by the addition of the new inner tube check valve. (Note that previous tests showed that without the new check valve, pressure draw down could be up to 600 psi.) This could have explained the capture of 170 psi instead of the expected 350 psi in the dimensional tests carried out during the field test at CTF.

The TRT recommended;

- Initiate an engineering study reviewing all aspects of the autoclave and pressure control section functions with regard to pressure retention.
- Carry out additional full function hydrostatic lab tests to determine the root cause of the pressure retention failure.



**1.2. TRT REMEDIAL ACTION**

- AAI reviewed the design of the autoclave and pressure section trying to identify possible causes for the failure to reliably trap pressure in the autoclave.
- The review indicated that two chambers in the autoclave can become pressure traps if operations are done with viscous drilling mud instead of seawater. These pressure trapped chambers can result in very slow ball rotation. The slow rotation would make it easier to jam the ball in a partially open position and could also allow the nitrogen charged fluid from the pressure control section to escape before the ball fully closed.
- Parts were modified to provide flow slots to provide paths for fluid escape to speed up ball rotation even when used with viscous drilling fluids (see Figure 1).



Figure 1. Flow slots added to the Ball Valve Release Collet (a) and to the Ball Follower (b).

- Ball valve closure tests were conducted at AAI using thick grease to simulate a viscous drilling mud. The grease appears to be more viscous than typical drilling mud. Ball closure was timed with and without grease pumped into the chambers. Assemblies with parts that were modified with flow slots were also tested in the same way. Closure times were measured by counting video frames. The tests showed significant reduction in closure time with the added flow slots especially with more viscous fluids.
- Several Full Function Pressure Tests were conducted as part of the TRT investigation. A Full Function Pressure Test is a pressure test carried out horizontally in the service shop, see the test setup in Figure 2.

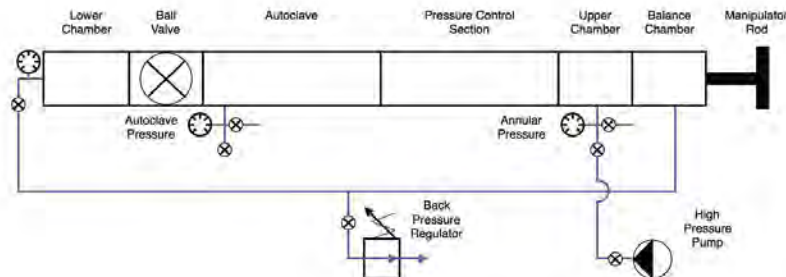


Figure 2. Full Function Pressure Test setup.





## PCTB 2015 Lab Test Program Final Report

The full function pressure test consists of an autoclave and pressure control section assembled together as in a normal coring run. This assembly is surrounded by chambers simulating the annular area of the drill pipe above and below and around the inner barrel assembly. At the top of this assembly is a pressure balanced manipulator rod that is used to simulate the pull by the wireline at the conclusion of coring to actuate the coring tool, close the ball valve, etc. The test does not simulate cutting the core. The pressure control section is pre-charged with nitrogen and the regulator set as in a normal coring operation.

The chambers are filled with water and the test chambers and autoclave are pressurized to the desired simulated static bottom hole pressure using an air over water hydrostatic pump. Then the manipulator rod is pulled to close the ball and open the sleeve valve just as in an actual coring run. During the test, the position of the manipulator rod can be measured to ascertain the position of the internal parts of the autoclave and pressure control section. The chamber and autoclave pressures are monitored real time. Fish pill pressure downhole pressure recorders are also used to make a permanent record of the test pressures inside the autoclave and annular area.

AAI used this test system to simulate the field conditions encountered during the CTF field test and to also to test improvement modifications that were suggested by the engineering study or TRT recommendations.

The full function pressure test indicated that if the pressure control section does not supply additional pressurized fluid, there is significant pressure drawdown in the autoclave due to volumetric changes while coming out of the hole. The pressure reduction can be up to 350 psi from this effect. That means a surface pressure of 150 psi may be full pressure recovery with a 500 psi hydrostatic pressure or, little or no pressure when recovering pressures lower than 350 psi. This is not the result of a leak or malfunction of the autoclave but is an unfortunate feature of the design of the tool where axial motion is required after ball valve closure. Note that at higher bottom hole pressures, this pressure drop effect is not necessarily amplified significantly. In other words, with a 2000 psi bottom hole pressure one might expect to see 1650 psi (2000 psi – 350 psi) recovered at the surface which does not appear to be as severe a pressure loss and may not be perceived as a failed run even without pressure boost from the pressure control section.

Several Full Function Pressure Tests proved the ability of the PTCB to hold full pressure reliably with little or no leakage when the pressure control section operated properly. Note that the TRT tests were conducted with water and not drilling mud.

Although the lab test results indicated that the primary problems may have been discovered and resolved, the TRT recommended additional lab testing before more field tests are attempted. This is the reason for the current test program. Subsequent to the TRT report, the DoE GoM project was contracted to the University of Texas. In early 2015 Aumann & Associates, Inc. and Geotek, LLC jointly formed a new company Geotek Coring, Inc. (GCI). GCI inherited the PCTB product line from AAI and the Pressure Core Analysis and Transfer System (PCATS) from Geotek, LLC. This enables GCI to provide a turnkey pressure coring service including core analysis. GCI contracted with the University of Texas to conduct the additional lab tests including additional Full Function Pressure Tests and the Core Liner and Inner Tube Implosion Tests.

### 1.3. ADDITIONAL IMPROVEMENTS

Two changes were made following the TRT Full Function Tests.

- A manufacturing error was discovered shortly after the conclusion of the TRT Full Function Tests. A critical hole leading to the Inner Tube Plug fill valve had not been



drilled by the supplier. Without this hole, the full benefit of the new inner tube plug fill valve design could not be realized. This error was corrected prior to running this series of Tests.

- The thin wall stainless steel inner tubes were replaced by thicker wall alloy steel tubes to provide the maximum possible resistance to collapse. In addition, it is believed that a heavier tube might also provide additional resistance to inner tube rotation and this might result in higher core recovery and quality. These tubes were manufactured prior to running this series of Full Function Pressure Tests. Both styles of inner tubes were tested during the Implosion Tests.

## 2. TEST PROGRAM

### 2.1. FULL FUNCTION PRESSURE TEST

Based on the TRT recommendations, additional Full Function Pressure Tests were conducted at the Geotek Coring, Inc. facilities from March 25 through April 10, 2015. A copy of the Full Function Test Procedure, PCTB005, is provided in the Appendix. The DoE Service Van located at GCI, was used to stage the tests. Tests were to be done with both water and drilling mud. Tom Pettigrew prepared the following test plan below. The test plan called for each series of two tests to be completed successfully back to back before moving to the next series. GCI personnel conducted the tests.

#### 2.1.1. FULL FUNCTION PRESSURE TEST PLAN

- 1 Test Objective
  - 1.1 To obtain a high degree of confidence in overall PCTB operation with focus on pressure retention.
- 2 Overall Test Procedure
  - 2.1 Carry out full function lab test using bench test apparatus, recording PCTB internal pressures using fish pills, and recording retained pressure using pressure gauges or pressure transducers.
  - 2.2 Repeat function test until a high degree of confidence in overall PCTB operation, with focus on pressure retention, is obtained.
- 3 Test Measurements
  - 3.1 PCTB internal pressures will be recorded electronically using fish pill recorders and archived.
  - 3.2 PCTB retained pressures will be determined using analog pressure gauges, and/or pressure transducers, and archived for review.
  - 3.3 Notes, and photographic evidence where applicable, regarding all failures will be archived for review.
- 4 Test Analysis
  - 4.1 The recorded fish pill pressure data will be reviewed to determine proper actuation and pressure boost has occurred.
  - 4.2 The retained pressure data will be compared to pre-test PCTB pressure boost/regulator settings to determine percentage of captured pressure is retained.
- 5 Pre-Test Preparation
  - 5.1 Identify and mark each PCTB subassembly as indicated below:
    - 5.1.1 3 each PCTB Pressure Sections – mark as #1, #2, and #3.
    - 5.1.2 4 each PCTB Autoclaves – mark as #1, #2, #3, and #4.



PCTB 2015 Lab Test Program Final Report

Note: PCTB subassemblies are to be identified as for example 1.1 where Pressure Section #1 and Autoclave #1 make up the subassembly.

**2.1.2. FULL FUNCTION PRESSURE TEST PROCEDURE**

1. Full Function Test: Medium = water; Simulated Borehole Pressure = 500 psi
  - 1.1. Full function test PCTB subassembly 1.1.
  - 1.2. Repeat full function test of PCTB 1.1 until 2 consecutive successful tests are achieved.
2. Full Function Test: Medium = water; Simulated Borehole Pressure = 1,500 psi
  - 2.1. Full function test PCTB subassembly 1.1.
  - 2.2. Repeat full function test of PCTB 1.1 until 2 consecutive successful tests are achieved.
3. Full Function Test: Medium = 10 ppg drilling mud; Simulated Borehole Pressure = 500 psi
  - 3.1. Full function test PCTB subassembly 1.1.
  - 3.2. Repeat full function test of PCTB 1.1 until 2 consecutive successful tests are achieved.
4. Full Function Test: Medium = 10 ppg drilling mud; Simulated Borehole Pressure = 1,500 psi
  - 4.1. Full function test PCTB subassembly 1.1.
  - 4.2. Repeat full function test of PCTB 1.1 until 2 consecutive successful tests are achieved.

\*\*As time allows, perform the following full function bench tests.

5. Full Function Test: Medium = water; Simulated Borehole Pressure = 1,500 psi
  - 5.1. Full function test PCTB subassembly 2.2.
  - 5.2. Repeat full function test of PCTB 2.2 until 2 consecutive successful tests are achieved.
6. Full Function Test: Medium = water; Simulated Borehole Pressure = 1,500 psi
  - 6.1. Full function test PCTB subassembly 2.3.
  - 6.2. Repeat full function test of PCTB 2.3 until 2 consecutive successful tests are achieved.
7. Full Function Test: Medium = water; Simulated Borehole Pressure = 1,500 psi
  - 7.1. Full function test PCTB subassembly 3.4.
  - 7.2. Repeat full function test of PCTB 3.4 until 2 consecutive successful tests are achieved.

The Full Function Test Plan Matrix based on the above plan and procedure is provided below.

| PCTB FULL FUNCTION BENCH TEST MATRIX                           |                  |                   |                |                 |       |
|--|------------------|-------------------|----------------|-----------------|-------|
| FIRST DIGIT = PRESSURE SECTION #<br>SECOND DIGIT = AUTOCLAVE # | 500 PSI<br>WATER | 1500 PSI<br>WATER | 500 PSI<br>MUD | 1500 PSI<br>MUD | TOTAL |
| TOOL No.   |                  |                   |                |                 |       |
| 1.1  | 2                | 2                 | 2              | 2               | 8     |
| 2.2  |                  | 2                 |                |                 | 2     |
| 2.3  |                  | 2                 |                |                 | 2     |
| 3.4  |                  | 2                 |                |                 | 2     |
| Total  | 2                | 8                 | 2              | 2               | 14    |



### **2.1.3. FULL FUNCTION PRESSURE TEST RESULTS**

- Fifteen gallons of a 10.4 lb/gal water base mud was obtained from TerraTek that they had previously mixed for one of their tests.
- All fourteen tests were completed successfully.
- The pressure control system applied the desired pressure boost in every test.
- The only tests that required to be rerun were due to operator error or test equipment malfunction.
- There were problems initially with the relief valve that is part of the Full Function Pressure Test fixture. The failure of the relief valve could cause the annulus pressure to go high if it stuck closed or low if it stuck open and had poor pressure control. It was replaced with a more accurate back pressure regulator.
- There was also some problem encountered resulting from the nature of the horizontal test due to gravitational effects causing occasional binding of the parts during pulling. It was discovered these forces could be compensated by adjusting support stands and then continuing the test.
- The use of drilling mud had no detectable affect. Pressure was properly boosted in every case and full pressure retained in the autoclave in each test with no visible or measurable leakage.
- A summary of the test results is provided in the table below.
- The test charts created from the data downloaded from the pressure recorder installed in the autoclaves and annulus are presented in the Appendix. They clearly show the proper function of the tools.



PCTB 2015 Lab Test Program Final Report

| Test Attempt | Test No. | Assy No. | M=Mud W=Water | N2 Pressure | Set Pressure | BHP Before Closure | BHP After Closure | BHP Change | Autoclave Pressure After Closure | Autoclave Pressure Boost | Autoclave Pressure after POOH | Autoclave Pressure Change after POOH | Comment  |
|--------------|----------|----------|---------------|-------------|--------------|--------------------|-------------------|------------|----------------------------------|--------------------------|-------------------------------|--------------------------------------|--|
|              |          |          |               | psi         | psi          | psi                | psi               | psi        | psi                              | psi                      | psi                           | psi                                  |  |
| 1            | 1.1      | 1.1      | W             | 1388        | 693          |                    |                   |            |                                  |                          |                               |                                      | Test failed because incorrect release sleeve was used in autoclave assembly.   |
| 2            | 1.1      | 1.1      | W             | 1419        | 742          | 510                | 723               | -213       | 701                              | 191                      | 699                           | -2                                   | Test fixture relief valve failed to open. This caused the annulus pressure to increase with the boost pressure but still a good test.          |
| 3            | 1.2      | 1.1      | W             | 1402        | 729          | 501                | 232               | -269       | 712                              | 211                      | 712                           | 0                                    | Test fixture relief valve popped open and bled too much annular pressure. This was a test equipment problem and not a tool failure. Good test. |
| 4            | 2.1      | 1.1      | W             | 3436        | 1676         | 1503               | 1351              | -152       | 1742                             | 239                      | 1717                          | -25                                  | Good test.   |
| 5            | 2.2      | 1.1      | W             | 3367        | 1690         | 1518               |                   |            |                                  |                          |                               | 0                                    | Test failed due to operator error. A valve in the test apparatus was left closed and caused a hydraulic lock. Could not pull.                  |
| 6            | 2.2      | 1.1      | W             | 3367        | 1690         | 1438               | 1430              | -8         | 1750                             | 312                      | 1722                          | -28                                  | Replaced the test fixture relief valve with a back pressure regulator. Much better control of annular pressure. Good test.                     |
| 7            | 3.1      | 1.1      | W             | 1413        | 701          |                    |                   |            |                                  |                          |                               |                                      | Test stopped due to restriction during pull.   |
| 8            | 3.1      | 1.1      | M             | 1413        | 701          | 458                | 467               | 9          | 703                              | 245                      | 697                           | -6                                   | Jammed during pull. Adjusted stands by elevating top end by 1" and finished pulling okay. Good test.   |
| 9            | 3.2      | 1.1      | M             | 1400        | 700          | 518                | 476               | -42        | 671                              | 153                      | 663                           | -8                                   |  |
| 10           | 4.1      | 1.1      | M             | 3415        | 1702         | 1497               | 1503              | 6          | 1693                             | 196                      | 1663                          | -30                                  | Tool initially hung up due to horizontal misalignment. Freed by adjusting stands and pulling again. Good test.                                 |
| 11           | 4.2      | 1.1      | M             | 3415        | 1687         | 1491               | 1600              | 109        | 1707                             | 216                      | 1676                          | -31                                  | Good test.   |
| 12           | 5.1      | 2.2      | W             | 3400        | 1710         | 1509               | 1445              | -64        | 1676                             | 167                      | 1639                          | -37                                  | Good test.   |
| 13           | 5.2      | 2.2      | W             | 3448        | 1676         | 1484               | 1410              | -74        | 1671                             | 187                      | 1644                          | -27                                  | Fish pill 7067 cracked open during test so no annulus recorded data. But still a good test.  |
| 14           | 6.1      | 2.3      | W             | 3460        | 1727         | 1520               | 1480              | -40        | 1658                             | 138                      | 1635                          | -23                                  | Good test.   |
| 15           | 6.2      | 2.3      | W             | 3400        | 1705         | 1520               | 1522              | 2          | 1702                             | 182                      | 1677                          | -25                                  | Good test.   |
| 16           | 7.1      | 3.4      | W             | 3429        | 1698         | 1535               | 1507              | -28        | 1673                             | 138                      | 1641                          | -32                                  | Good test.   |
| 17           | 7.2      | 3.4      | W             | 3400        | 1698         | 1527               | 1464              | -63        | 1707                             | 180                      | 1678                          | -29                                  | Good test.   |

2.2. INNER TUBE AND CORE LINER IMPLOSION TESTS

Implosion tests were carried out with and without the core liners with both the original thin wall stainless steel inner tube and with the new thick wall alloy steel tubes. Tom Pettigrew prepared the Inner Core Tube and Core Liner Implosion Test Plan provided below. GCI designed and manufactured the special inner tube and core liner plugs and rods that provided pressure seal on each end of the tube or assembly being tested. A rigid bar between the test plugs prevents any plug related pressure generated axial forces from



applying buckling loads to the tube being tested. (See Figure 3). This closely simulates downhole conditions with the exception that a hydrostatic pressure was used instead of a pressure created by the flow. The tests were conducted using a PCTB II autoclave as a pressure vessel with the inner tube and liner in the normal coring position. The upper end was sealed using the Full Function Pressure Test components. The tests were carried out in the DoE Service Van using the existing hydrostatic test pump, high pressure manifolds and digital pressure gages. "Fish Pill" downhole pressure recorders were placed in the autoclave to provide a recorded record of the pressures. Implosion pressures were obtained from the downhole recorder data and also observed on the digital pressure gages attached to the manifolds.

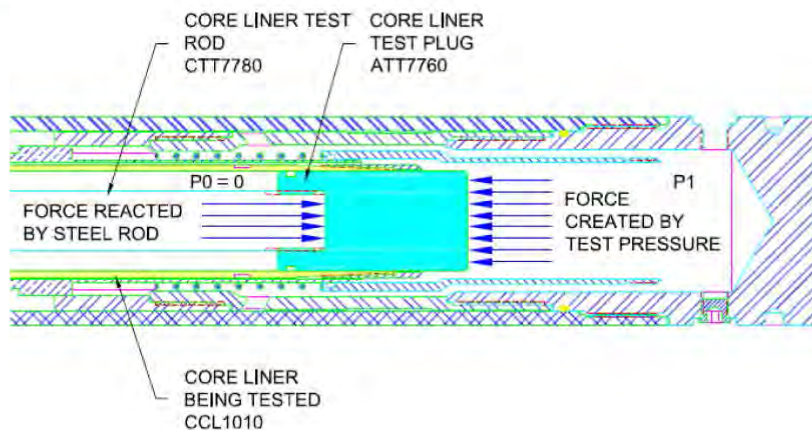


Figure 3. Internally balanced test rod assembly keeps axial compression forces from affecting core liner.

### 2.2.1. INNER CORE TUBE AND CORE LINER IMPLOSION TEST PLAN

1. Test Objectives
  - 1.1. To determine actual pressure required to collapse inner core tube.
2. Overall Test Procedures
  - 2.1. Set up inner core tube so as to apply external pressure to it and apply increasing pressure to failure.
3. Test Measurements
  - 3.1. An electronic applied collapse pressure record will be created using a pressure transducer and will be archived for review.
4. Test Analysis
  - 4.1. The recorded inner core tube collapse pressure will be compared to calculated data as well as noted in the operations manual for future reference.
5. Configurations to be Tested
  - 5.1. The following inner core tube configurations shall be tested.
    - 5.1.1. Thin wall inner tube alone (seal on the end of the steel inner tube).
    - 5.1.2. Thin wall inner tube with liner sealed (seal on the liner and not the steel inner tube).
    - 5.1.3. Thick wall inner tube alone (seal on the end of the steel inner tube).



5.1.4. Thick wall inner tube with liner sealed (seal on the liner and not the steel inner tube).

**2.2.2. INNER TUBE AND CORE LINER IMPLOSION TEST RESULTS**

The above plan was followed. Calculations of the implosion pressures for the thin wall stainless steel inner tube and the thick wall alloy steel inner tubes are provided in Appendix A1. The following table provides the results of the calculations and implosion test results.

- All four implosion tests were completed. Results are shown in the table below.
- The heavy wall inner tube may increase core liner implosion pressure by a small amount.
- The main benefit to using the heavy wall steel inner tube is that the tube was not damaged when the plastic core liner imploded.
- The heavy wall steel inner tube by itself did not implode even at 4000 psi.
- The results agreed reasonably well with the calculated value for inner tube implosion strength. The calculation sheets are provided in Appendix A1. Test values of the thin wall stainless steel inner tube may be slightly lower than the calculated values because of irregularities in the thin wall tube such as minor egging or dents which are not accounted for in the calculations.
- The core liner did not collapse where it was supported inside the heavy wall steel tube. Core liner collapse only occurred below this tube.
- The test charts created from the downhole pressure recorder installed in the tools are presented in Appendix B1.

| TEST | DESCRIPTION  | CALCULATED IMPLOSION PRESSURE (PSI) | IMPLOSION OR MAXIMUM PRESSURE (PSI) |
|------|--|-------------------------------------|-------------------------------------|
| 1    | Thin wall stainless steel inner tube alone               | 372                                 | 337                                 |
| 2    | Thin wall inner tube with core liner sealed              |                                     | 292                                 |
| 3    | Thick wall alloy steel inner tube alone                  | 16,991                              | 4000 - No Implosion                 |
| 4    | Thick wall alloy steel inner tube with core liner sealed |                                     | 326                                 |

The downhole pressure recorder data will be maintained on file at Geotek Coring Inc.



PCTB 2015 Lab Test Program Final Report

## APPENDICES

### 1. INNER TUBE IMPLOSION CALCULATIONS

Given:

$$\mu = 0.3 \quad (\text{Poisson's Ratio})$$

$$\text{Collapse Equation} \quad P_c = \frac{2 \cdot E}{(1 - \mu^2) \cdot \left(\frac{D_o}{t}\right)^3}$$

For Thin Wall Stainless Steel Inner Tube

$$E = 28 \cdot 10^6 \text{ psi} \quad (\text{Modulus of Elasticity for Stainless Steel})$$

$$\text{Outer Diameter} \quad D_o = 2.497 \text{ in}$$

$$\text{Inner Diameter} \quad D_i = 2.406 \text{ in}$$

$$\text{Wall Thickness} \quad t = \frac{D_o - D_i}{2} \quad t = 0.045 \text{ in}$$

$$\text{Collapse Pressure} \quad P_c = 372 \text{ psi}$$

For Thick Wall Alloy Steel Inner Tube

$$E = 30 \cdot 10^6 \text{ psi} \quad (\text{Modulus of Elasticity for Alloy Steel})$$

$$\text{Outer Diameter} \quad D_o = 2.750 \text{ in}$$

$$\text{Inner Diameter} \quad D_i = 2.400 \text{ in}$$

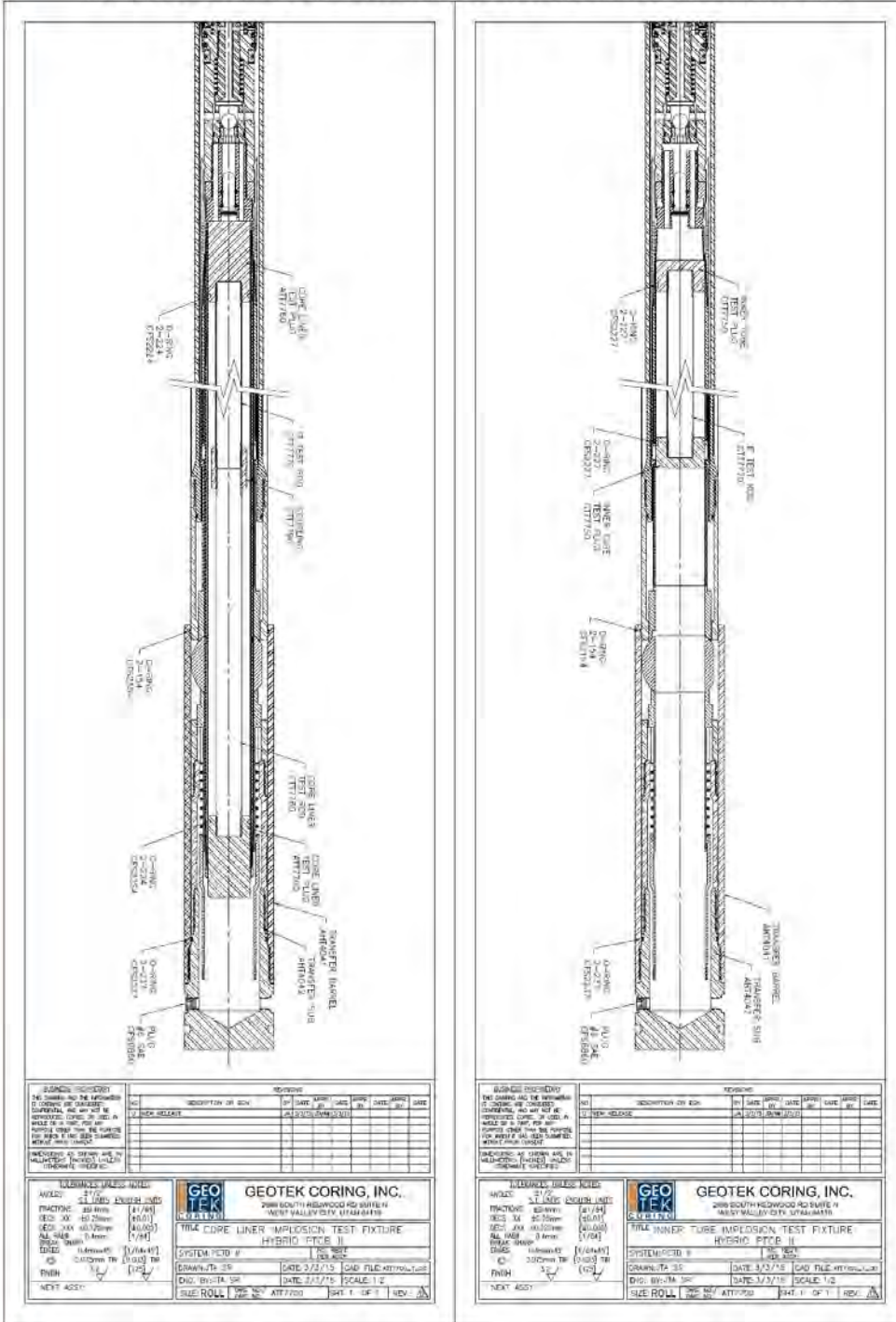
$$\text{Wall Thickness} \quad t = \frac{D_o - D_i}{2} \quad t = 0.175 \text{ in}$$

$$\text{Collapse Pressure} \quad P_c = 16991 \text{ psi}$$






2. INNER TUBE AND CORE LINER IMPLOSION TEST ASSEMBLY DRAWING



PCTB 2015 Lab Test Program Final Report



**3. PCTB005A, FULL FUNCTION PRESSURE TEST PROCEDURE**



2698 S Redwood Rd  
Suite N  
West Valley City, Utah 84119  
(801) 631-2874  
Facsimile (801) 886-9040  
Email: [Jim.Aumann@GeotekCoring.com](mailto:Jim.Aumann@GeotekCoring.com)

**Hybrid PCTB**

**Full Function Pressure Test Procedure**

| REV | DATE      | DESCRIPTION | ORIG    | CHK     | APPR |
|-----|-----------|-------------|---------|---------|------|
| A   | 1/04/2012 | New Release | JTA Sr. | JTA Sr. |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |
|     |           |             |         |         |      |

Document No.: PCTB 005



**1) INTRODUCTION**

The Hybrid Pressure Coring System with Ball Valve (PCTB) was developed as a wireline retrievable pressure retaining coring system by Aumann & Associates, Inc. Aumann & Associates, Inc. subsequently joined Geotek, LLC and formed a new company Geotek Coring, Inc. to provide a turnkey pressure coring and pressure core analysis service.

This document provides the laboratory test procedure for the Full Function Pressure Test. This test is used to verify satisfactory operation and pressure retaining function of the ball valve, seal sub, bullet valves and pressure control system. This test includes both the autoclave and pressure control section assembled normally but with test barrels added that allow the pressure above and below the autoclave to be pressurized to fully simulate ball valve and pressure control section operation during simulated coring runs. This procedure was developed to try to determine the cause and the solution for the failure of the pressure control section to provide the pressure boost and failure of the autoclave to hold pressure reliably during field tests. It can also be used as a final acceptance test for new and refurbished tools.

This Full Function Pressure Test (FFPT) was developed to simulate actual downhole conditions as closely as possible. This test includes both the autoclave and pressure control section assembled with test barrels that allow the pressure above and below the Autoclave/Pressure Control Section assembly to be controlled to fully simulate hydrostatic pressure. This includes full pressure surrounding the inner components during ball closure, top seal engagement and sleeve valve opening. The test is carried out horizontally. The pressure control section is preloaded with nitrogen and the regulator set as in a normal pressure coring run. Adapters screwed into the ends of the autoclave and pressure control section create chambers that simulate the annular space above and below the inner barrel assembly. A pressure balanced rod is used in place of the latches, wireline and wireline pulling tool. Using this test configuration, simulated hydrostatic bottom-hole pressure can be applied to the autoclave, the pressure control section and the annular space above and below the assembly. After the hydrostatic pressure is applied, the pressure balanced rod is physically pulled to engage the upper seals, close the ball and trip the sleeve valve. Pressure recorders inserted in the autoclave and annular chamber are used to record the autoclave and annular pressures.

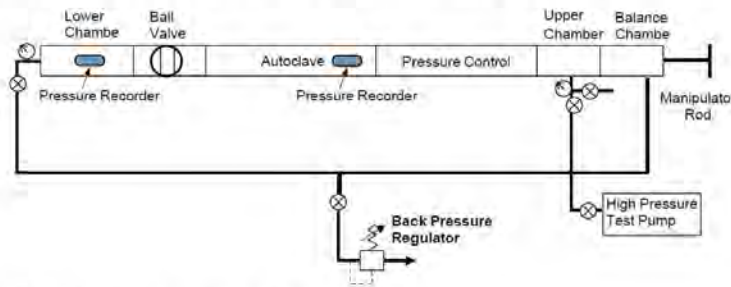


Figure 1, Full Function Pressure Test Configuration



PCTB 2015 Lab Test Program Final Report

Geotek Coring, Inc.

PTCB Autoclave Full Function Test Procedure  
PTCB005 Rev A

**2) EQUIPMENT TO BE TESTED**

PTCB autoclave with top seal ball valve.  
PTCB pressure control assembly

**3) TEST FACILITY**

The tests are normally conducted at Geotek Coring, Inc. (GCI) facilities in Salt Lake City, Utah, U.S.A. The GCI facilities are heated, lighted and equipped with a tool vise, work benches, storage racks, compressed air and water. Available equipment includes a hydraulic test console capable of supplying up to 10,500 psi pressure, pressure manifolds containing calibrated digital pressure gages and high pressure hoses. The facility is stocked with necessary hand tools, wrenches, spinning buggies, lubricants, tool racks and fork lift truck. Other instrumentation such as a load cell and come-a-longs are also available. AAI has used this equipment on many occasions for laboratory testing of other coring systems. If desired, this test can also be conducted in a service van or other site with similar equipment if the client desires.

**4) REFERENCE DOCUMENTS**

The following reference documents should be on hand for easy reference during the tests.

| <u>Item</u> | <u>Document</u>                              |
|-------------|--|
| 1.          | PCTB Inner Barrel Assembly Drawing           |
| 2.          | PCTB Maintenance Manual                      |
| 3.          | PCTB Pressure Test Assembly Drawing          |
| 4.          | PCTB Full Function Pressure Test Data Sheets |

**5) EQUIPMENT**

| <u>Qty</u> | <u>Description</u>  |
|------------|---|
| 1          | Tool vise   |
| 3          | Pipe stands   |
| 1          | Set hand tools including strap wrenches, hex wrenches, 24 and 36 inch pipe wrenches |
| 1          | 5,000 psi hydraulic test console with high pressure hose                            |
| 2          | 5,000 or 10,000 psi digital pressure gages  |
| 1          | 5,000 or 10,000 psi pressure transducer   |
| 2          | Pressure transducer read out boxes, Model NK  |
| 2          | Transducer cable  |
| 1          | Autoclave assembly, PTCB  |
| 1          | Pressure Control Section, PTCB  |
| 1          | Ported Drive Sub  |
| 2          | 10,000 psi test manifold  |
| 1          | 5,000 psi air over water Hydrostatic Test Pump                                      |
|            | PCTB autoclave service tools including the following:                               |
| 1          | CHT4061, Ball Valve Resetting Cap   |
| 1          | CHT4062, Ball Valve Resetting Spacer  |
| 1          | CHT4031, Transfer Barrel  |
| 1          | CHT4032, Transfer Sub   |
| 1          | CHT4033, Upper Test Barrel  |



Geotek Coring, Inc.

PCTB Autoclave Full Function Test Procedure  
PTCB005 Rev A

1 CES7537, Middle Test Barrel

**5) SAFETY**

1. Steel-toed footwear is required to be worn at all times when working with heavy parts or lifts.
2. A hard hat must be worn outside of the service unit on the rig site or during any overhead lifts.
3. Safety glasses must be worn anytime a pressurized chamber is being accessed or if a grinder or other equipment that could produce airborne particles is being used.
4. Use caution around high pressure fluids and especially around gasses that may be present during pressure testing or in the recovered core.
5. Use a locked loop when lifting with a lift strap whenever possible.
6. Use care in balancing parts from an open loop lift strap or when using the spinning buggy.
7. Never unscrew the bullet valves more than the prescribed amount or force them past the retaining ring or remove the retaining ring while there is pressure in the system. They could become projectiles and cause injury or death.
8. Work quickly but don't rush. Being in a hurry can cause an accident.
9. Attend safety meetings at the job site and follow all safety requirements.

**6) TEST PROCEDURES**

This test is intended to fully simulate the downhole operation of the autoclave and pressure control section including the following:

1. Inner Tube Plug seal engagement with the Seal Sub
2. Closure of the ball
3. Movement of the Sleeve Valve to the open position
4. Regulated pressure from the pressure control section applied to the autoclave
5. Seal integrity of the ball valve and inner tube plug seals.

**6.1 Pressure Testing**

- 6.1.1 Pressure testing shall be completed in a safe environment including, as needed, the use of blast protection barriers, a suitable test bay, a cordoned evacuated test area, or other suitable methods to protect personnel in the event of a safety incident.
- 6.1.2
- 6.1.3 Non-authorized personnel shall be restricted from the test area by use of suitable barriers or other adequate means to ensure controlled access to the immediate test area. Hydrostatic pressure test holding time shall be ten (10) minutes. The test medium shall be potable (tap) water. Additives (rust inhibitors or other) are not required to be added to the test medium.
- 6.1.4 Hydrostatic test medium and atmospheric temperatures shall be ambient but shall not exceed 120°F (48°C), to minimize the risk of brittle fracture. The test pressure shall not be applied until the autoclave and its contents are at about the same temperature.
- 6.1.5 Devices used to measure test pressure (pressure transducers, pressure read out boxes, dial gauges, or other) shall be calibrated and suitably correlated. Dial gauge range shall be not less than 1.5 times nor greater than four times the test pressure. Gauges shall be

Page 4 of 9



Geotek Coring, Inc.

PTCB Autoclave Full Function Test Procedure  
PTCB005 Rev A

marked with gradations of a minimum of 1PSIG or 1% of full range, whichever is greater. Digital gages are recommended.

- 6.1.6 Examination shall be made of the autoclave under test to verify no leaks under pressure. Pressure test devices and/or gauges will be monitored during the hold period for any leak indication. The test system, exclusive of minor leakage at pump or valve packing/seals, shall show no evidence of leaking.

**6.2 Inspection and Testing Records**

- 6.2.1 Records of any inspections and tests undertaken shall be signed as completed and retained for customer turnover.
- 6.2.2 Quality records will include any physical inspections, material test reports, function tests, or pressure tests. Clients may also witness any tests undertaken at their discretion.

**6.3 General Procedures**

The following general principles apply to all assembly procedures:

- 6.3.1 Parts and tools are to be treated well and not abused.
- 6.3.2 Care should be taken to protect parts and tools from the weather as much as possible.
- 6.3.3 Use thread protectors, if available, until ready to assemble or run in the hole.
- 6.3.4 Unless otherwise instructed, always coat both pin and box threads with a coat of thread dope or "Never Seize" lubricant before assembly.
- 6.3.5 Unless otherwise instructed, always coat seals and seal surfaces with a coat of seal grease before assembly.

**6.4 Pressure Control Section Assembly**

- 6.4.1 Assemble the pressure control section according to normal assembly practices.
- 6.4.2 Charge the regulator with 2000 psi of nitrogen.
- 6.4.3 Set the regulated nitrogen charge to 1000 psi.
- 6.4.4 Remove the assembly from the tool vise and set the assembly aside.

**6.5 Autoclave Assembly**

- 6.5.1 Assemble the autoclave according to normal assembly practices.
- 6.5.2 Pressure test the autoclave assembly to 1000 psi.



Geotek Coring, Inc.

PTCB Autoclave Full Function Test Procedure  
PTCB005 Rev A

- 6.5.3 Install one spring sleeve, the return spring and the cutting shoe sleeve.  
Note: The cutting shoe sleeve does not screw into anything but seats against the transfer plug.
- 6.5.4 Install the low end transfer barrel and sub.
- 6.5.5 Conduct the low differential pressure test.
- 6.5.6 Leave the assembly in the tool vise.

#### 6.6 Final Assembly for Full Function Pressure Tests

- 6.6.1 Screw the Middle Test Barrel onto the seal sub
- 6.6.2 Screw the Lift Sub onto the Middle Test Barrel.
- 6.6.3 Screw the manipulator rod into the end of the male QLS
- 6.6.4 Screw the Upper Test Barrel onto the lift sub.
- 6.6.5 Slide the Transfer Sub over the 1/2 inch rod and screw the Transfer Sub with 1/2 in hole onto the Upper Test Barrel
- 6.6.6 Screw the 5/16 eye onto the end of the 1/2" diameter rod protruding from the Transfer Sub.

#### 6.7 Full Function Pressure Test

The purposes of these tests are simulate actual down hole conditions as closely as possible. This includes full pressure surrounding the inner components during ball closure, top seal engagement and sleeve valve opening. The pressure inside the autoclave and the position of the inner components can also be measured. The test should be able to determine if there is a pressure drawdown during operation and should verify the timing of the operational sequence of ball valve closure, top seal engagement and tripping of the sleeve valve.

- 6.7.1 Unscrew and remove the transfer plug.
- 6.7.2 Screw a manifold with calibrated digital gage onto a port on each transfer sub. Connect a supply hose from the hydraulic console to each manifold using a T.
- 6.7.3 Install a pressure transducer in the 1/8 NPT port in the side of the drive sub.
- 6.7.4 Tilt the test assembly so that ball valve is the highest point in the assembly.
- 6.7.5 Using a hose, fill from the ball valve end until the inner barrel is full.
- 6.7.6 Reinstall the transfer barrel and transfer plug.

Page 6 of 9



Geotek Coring, Inc.

PTCB Autoclave Full Function Test Procedure  
PTCB005 Rev A

- 6.7.7 Remove the top SAE plug and pump with the high pressure pump until all the air is expelled. Reinstall the SAE plug.
- 6.7.8 Lower the tool back to a horizontal position.
- 6.7.9 Start the pump and increase pressure to 1000 psi (or other pressure if desired) using the air supply pressure to adjust the pump pressure.
- 6.7.10 Close the supply valve on the manifold and open the drain valve on the hydraulic console.
- 6.7.11 Monitor and record the pressure in the inner tube once per minute for a period of ten minutes.
- 6.7.12 Increase the pressure to the proof pressure of 5250 psi. (Note: Proof pressure of 1.5 times the rated working pressure meets ASME code requirements.)
- 6.7.13 Monitor and record the pressure once per minute for a period of ten minutes.
- 6.7.14 When the above tests are complete, reduce the pressure inside the test assembly to zero, by opening the dump valve on the hydraulic console.

NOTE: Either proceed with the Low Differential Pressure Test described in the next section beginning at Step 6.7.xx or complete the following steps if the differential test is not going to be carried out.

- 6.7.15 Disconnect the manifold and hose.
- 6.7.16 Open the ball valve using the ball resetting tool, and drain the water from the tool. CAUTION: MAKE SURE PRESSURE INSIDE THE TEST ASSEMBLY IS LESS THAN 10 PSI BEFORE OPENING THE BALL VALVE.
- 6.7.17 Disassemble the test assembly, and use a cloth to wipe any residual water from the test assembly components.

#### 6.8 Low Differential Pressure Test

The purpose of this test is to verify that the ball valve seals when only a slight differential pressure exists across the ball valve seal.

- 6.8.1 Pull out on the inner tuber plug until the seals engage and the pawls lock into the groove in the seal sub.
- 6.8.2 Connect the exposed port at the end of the inner tube plug to the manifold with calibrated digital gage and supply hose from the hydraulic console.
- 6.8.3 Screw the ball valve resetting tool into the housing extension to open the ball valve about half way.

Page 7 of 8



## PCTB 2015 Lab Test Program Final Report



Geotek Coring, Inc.

PTCB Autoclave Full Function Test Procedure  
PTCB005 Rev A

- 
- 6.8.4 Tilt the test assembly so that ball valve is the highest point in the assembly.
  - 6.8.5 Turn on the water supply from the hydraulic console to the test assembly until water begins to come out of the slightly open ball valve or fill from the ball valve end until the inner barrel is full.
  - 6.8.6 Unscrew the ball valve resetting tool to close the ball.
  - 6.8.7 Lower the tool back to a horizontal position.
  - 6.8.8 Use the hydraulic console to increase the pressure in the assembly to 1000 psi, and allow the pressure to stabilize.
  - 6.8.9 Close the valve on the manifold.
  - 6.8.10 Shut off the air supply to the hydraulic console, bleed off the pressure using the needle valve on the console and remove the hydraulic hose from the manifold. NOTE: Do not adjust the pressure on the air pressure regulator on the hydraulic console.
  - 6.8.11 Install an o-ring into the groove on the ball valve housing.
  - 6.8.12 Screw the lower transfer assembly (transfer adapter and transfer sub) onto the housing extension.
  - 6.8.13 Rotate the assembly until one of the ports on the transfer sub is on top.
  - 6.8.14 Install a test manifold into the lower port on the transfer sub.
  - 6.8.15 Connect the supply hose from the hydraulic console to the manifold to the transfer sub.
  - 6.8.16 Pump until water flows out of the top port on the transfer sub.
  - 6.8.17 Install a plug in the top port on the pressure test adapter.
  - 6.8.18 Start the pump and increase pressure to 1,000 psi using the same setting on the pressure regulator that was used to pressurize the autoclave. Allow the pressure to stabilize until there is no change in pressure on either pressure gage over a 2 minute period.
  - 6.8.19 Record the autoclave and annular pressures.
  - 6.8.20 Crack the dump valve on the pump so that the pressure in the chamber below the ball valve decreases at a rate of about 10 psi/min.
  - 6.8.21 Record the pressure shown on the pressure readout boxes at one minute intervals and verify that the pressure in the autoclave does NOT decrease at the same rate as the pressure annular chamber decreases.

Page 8 of 9



Geotek Coring, Inc.

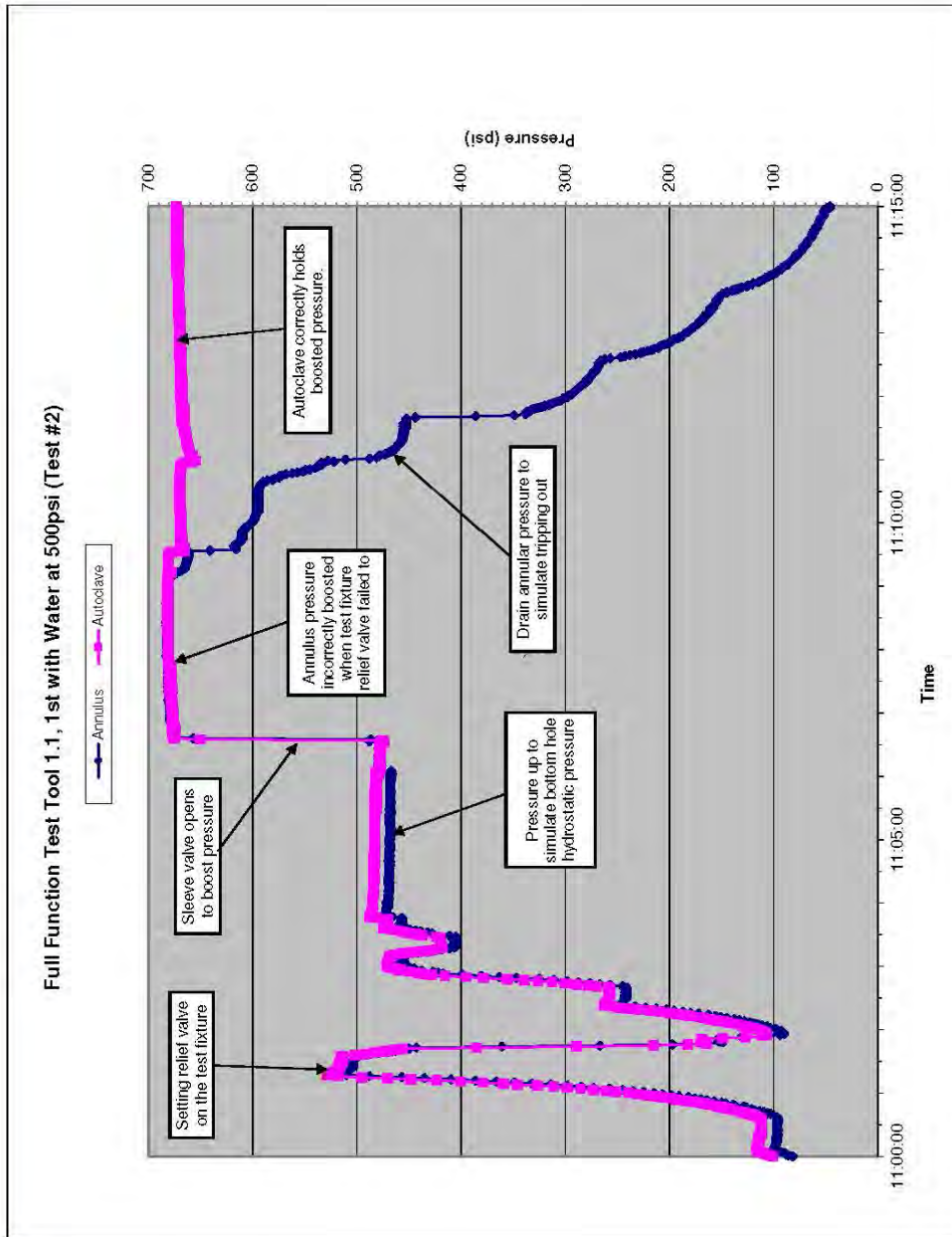
PTCB Autoclave Full Function Test Procedure  
PTCB005 Rev A

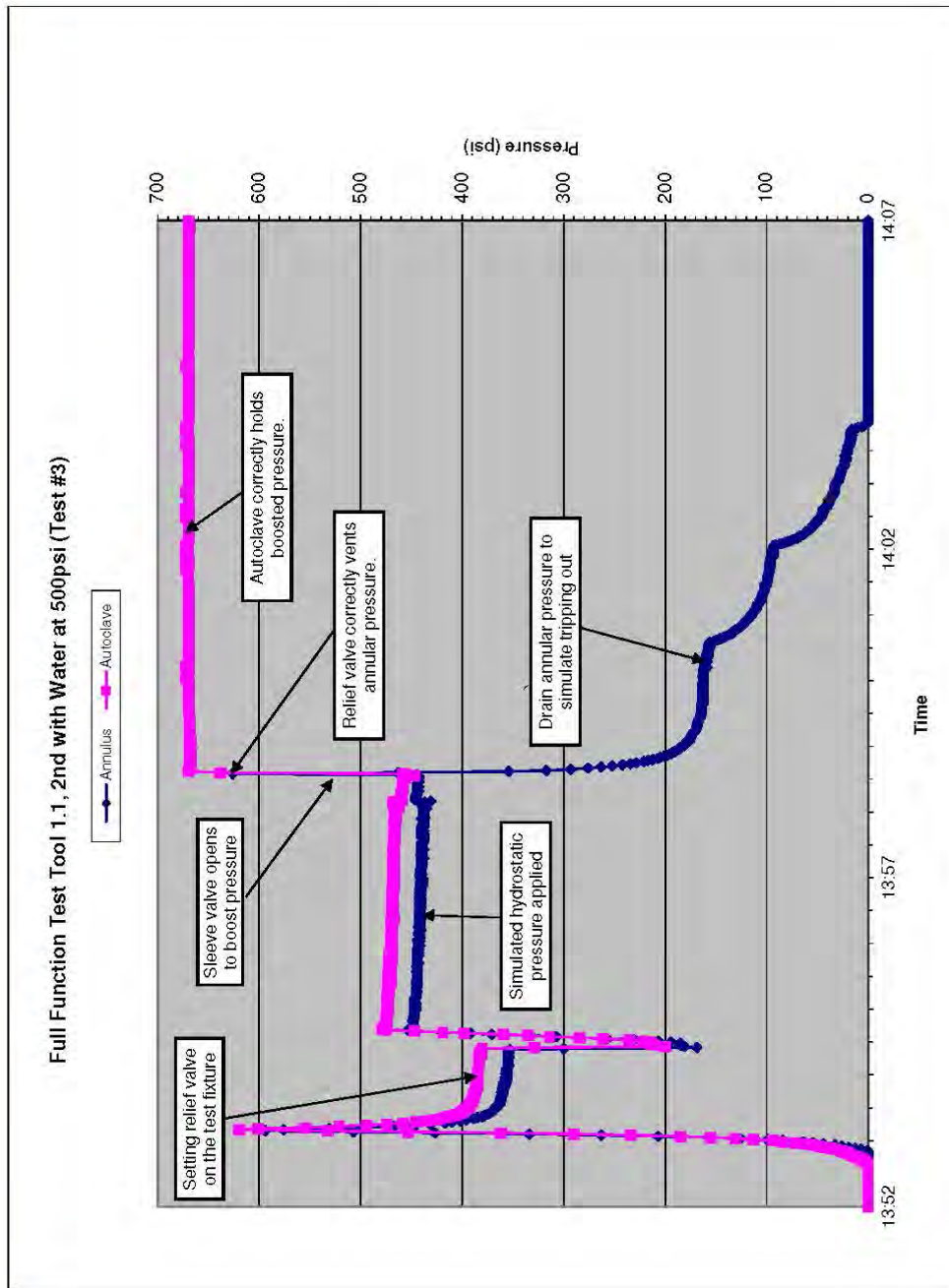
NOTE: The annular pressure may initially decrease slightly as the pressure differential builds and compresses the seals. However, this trend should not continue.

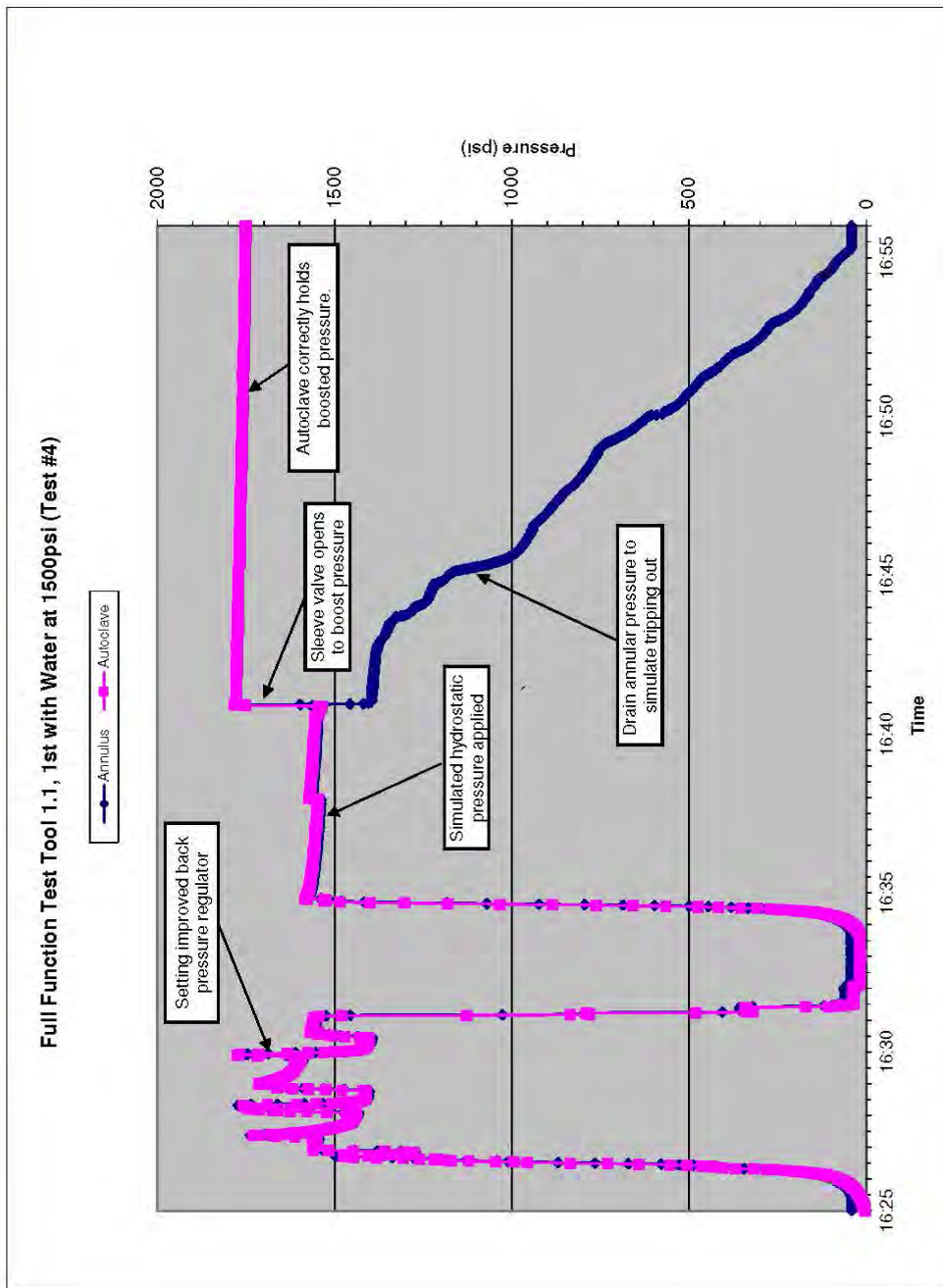
- 6.8.22 Once the annulus pressure is about 100 psi lower than the pressure in the chamber above the ball valve stop bleeding the pressure. Continue to monitor the pressure above the ball valve for at least ten minutes and verify that the pressure does not decrease by more than 2 psi/min at the end of this time period.
- 6.8.23 When the above tests are complete, reduce the pressure inside both test chambers to zero, by opening the dump valve on the hydraulic console or the bleed valve on the test manifold.
- 6.8.24 Open the ball valve using the ball valve resetting tool and drain the water from the tool. CAUTION: MAKE SURE PRESSURE INSIDE THE TEST ASSEMBLY IS LESS THAN 10 PSI BEFORE OPENING THE BALL VALVE.
- 6.8.25 Disassemble the test assembly, and use a cloth to wipe any residual water from the test assembly components.

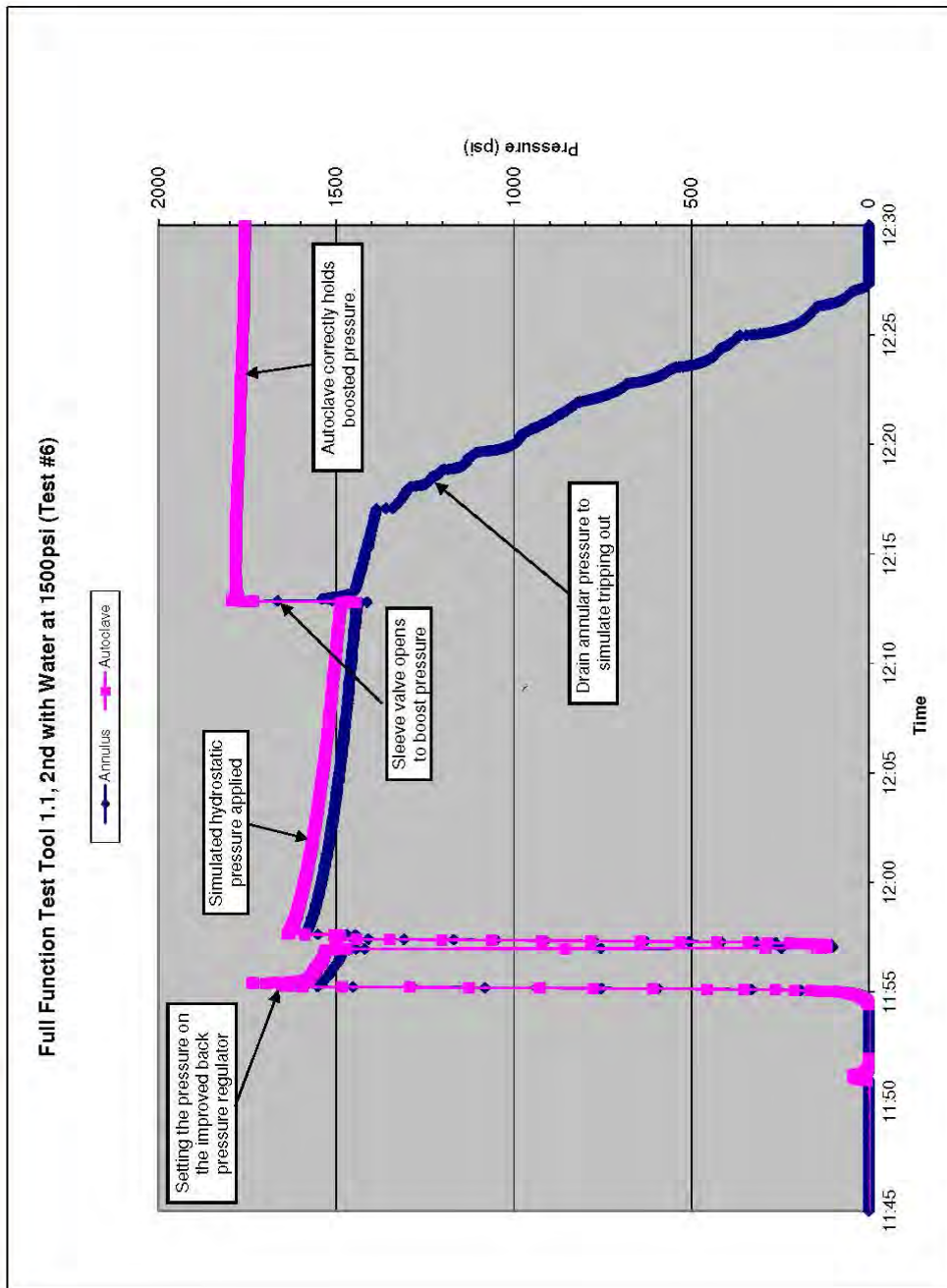


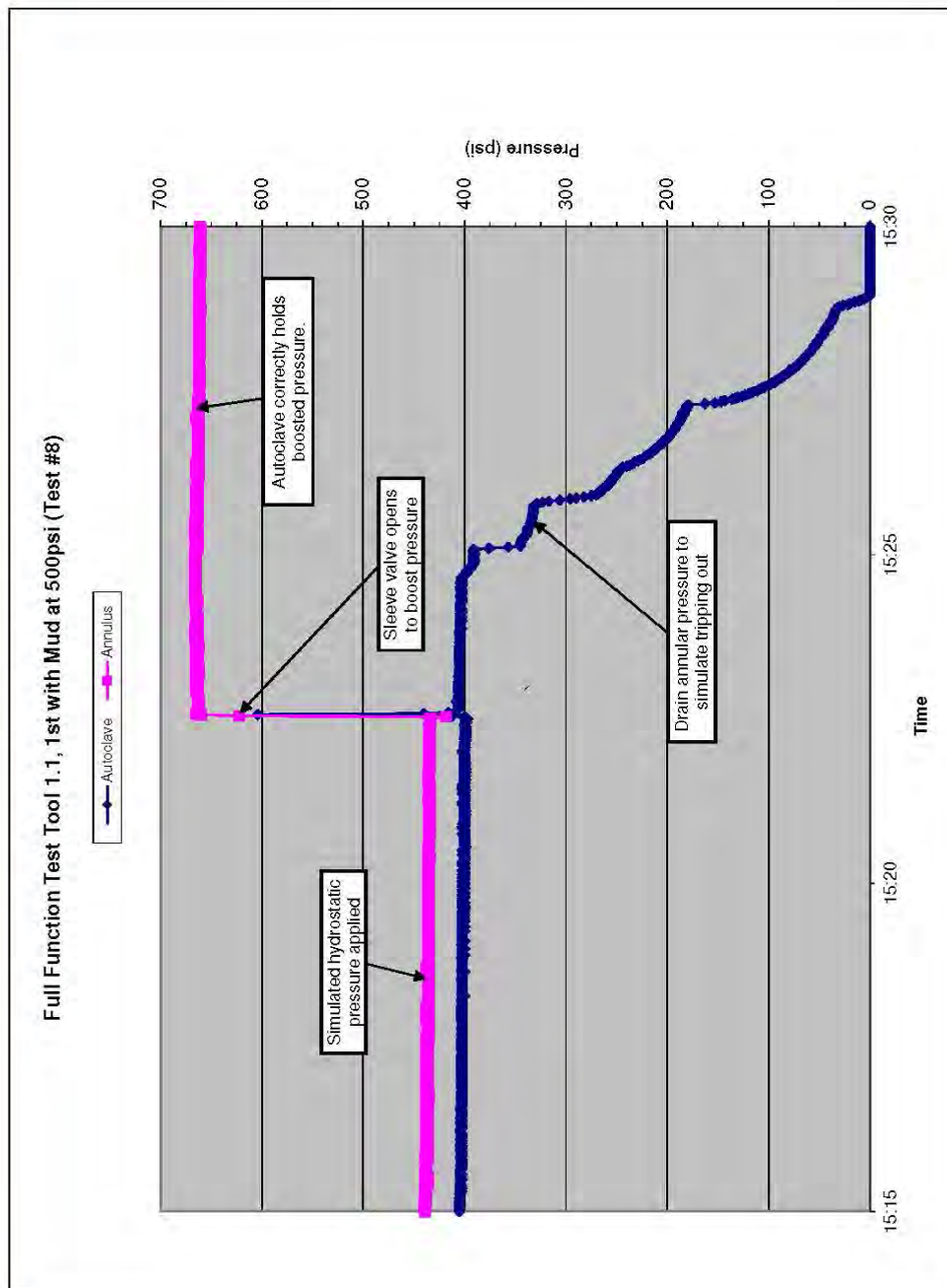
#### 4. FULL FUNCTION PRESSURE TEST CHARTS

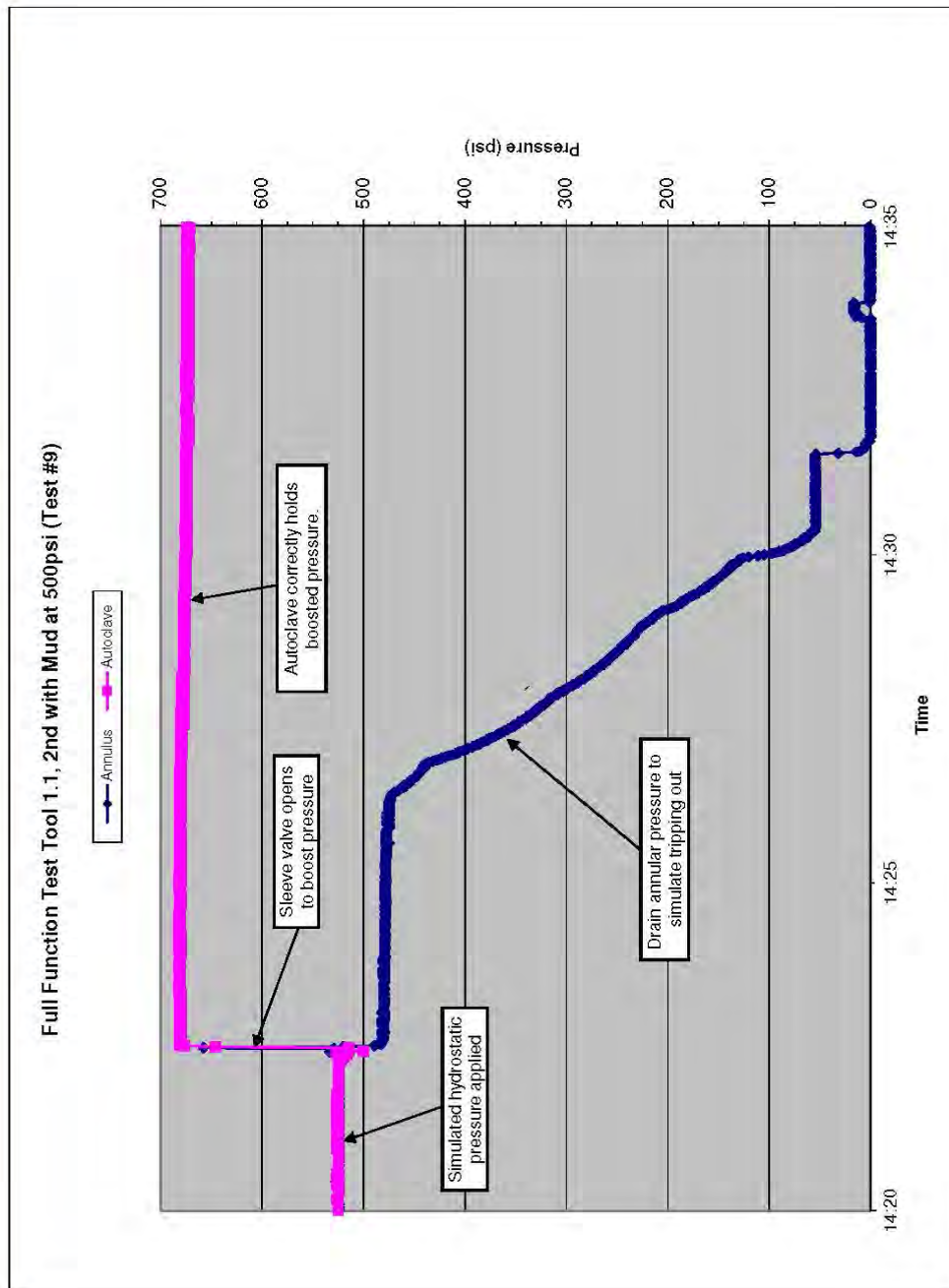




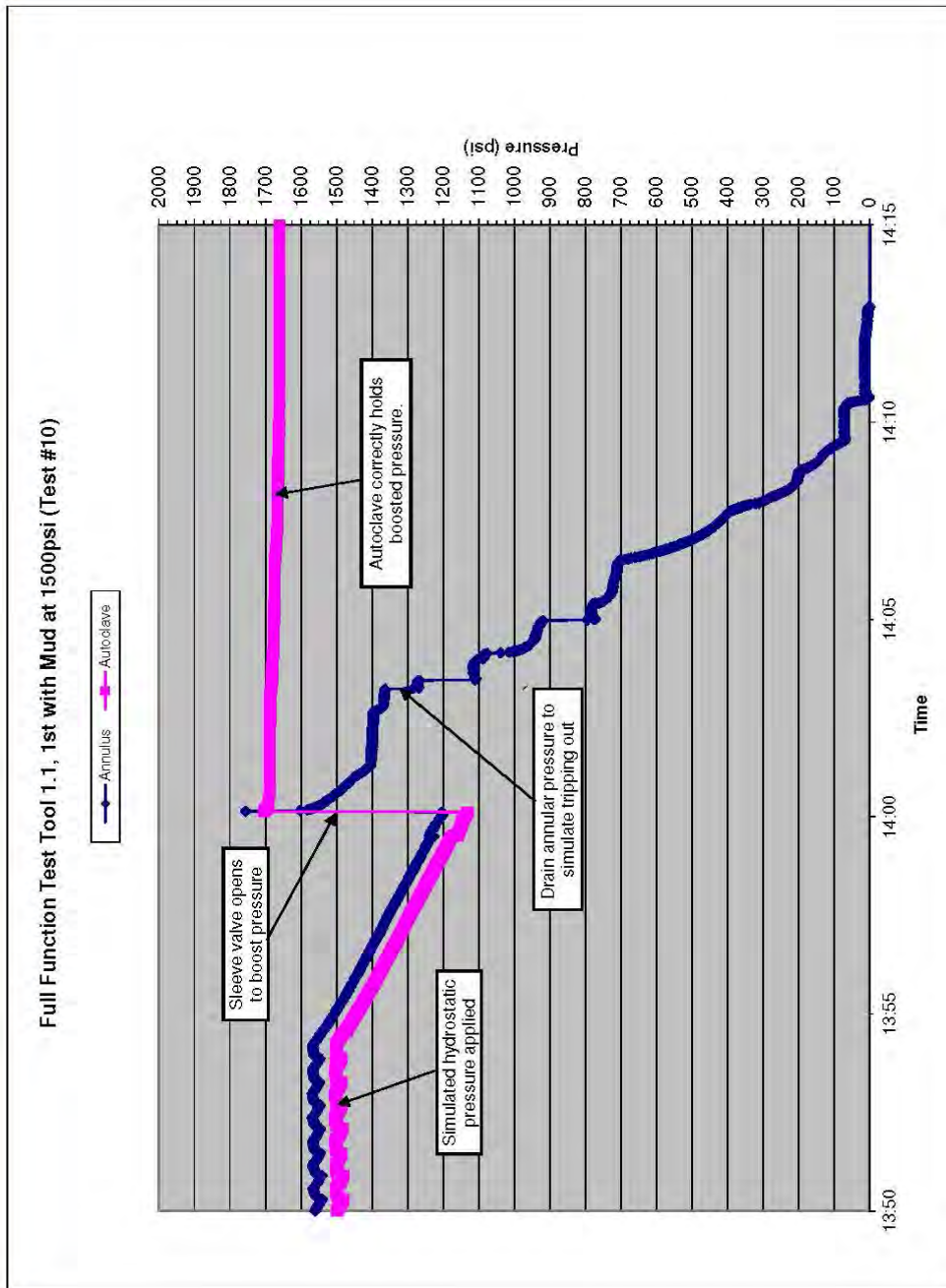


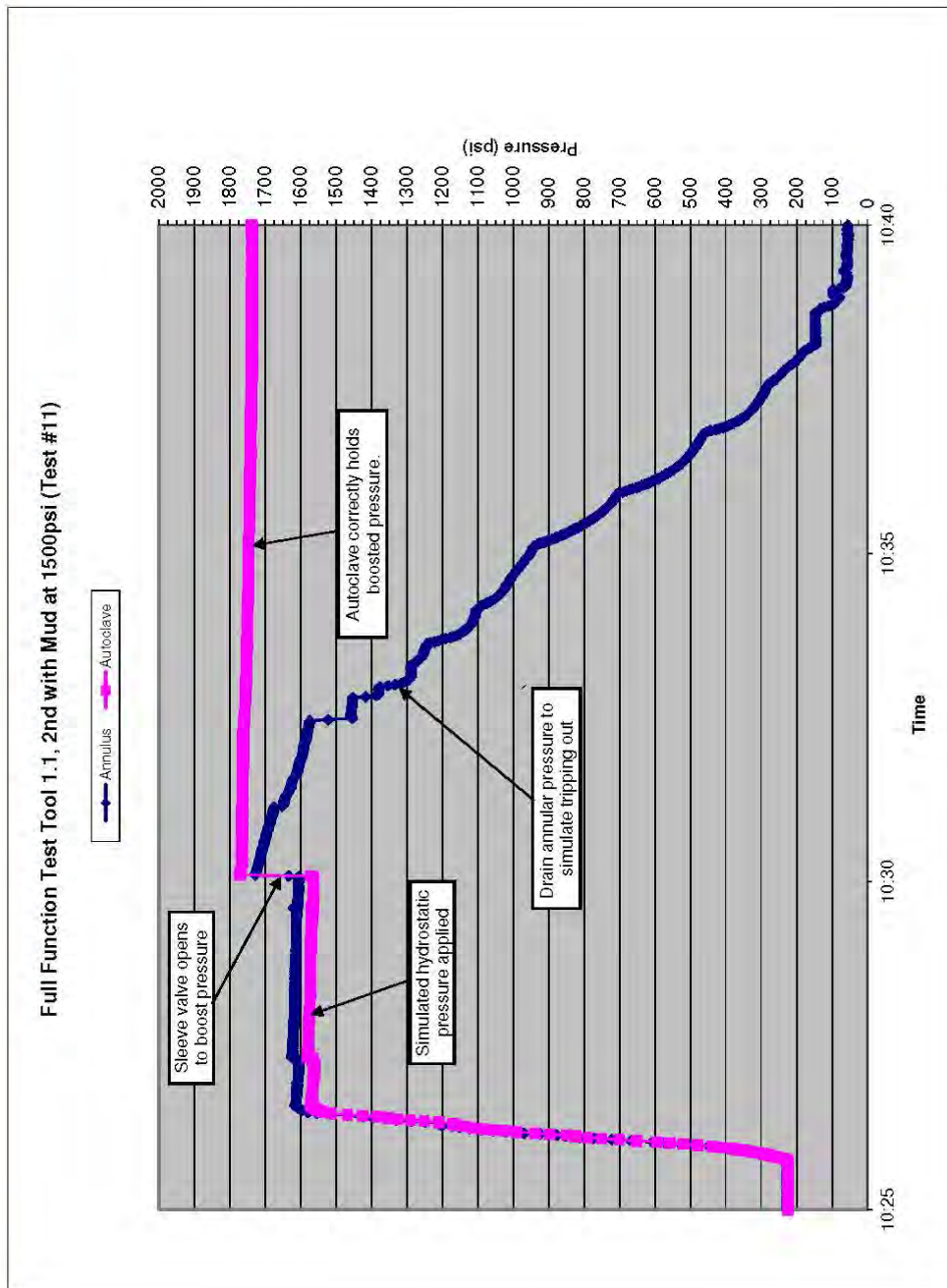


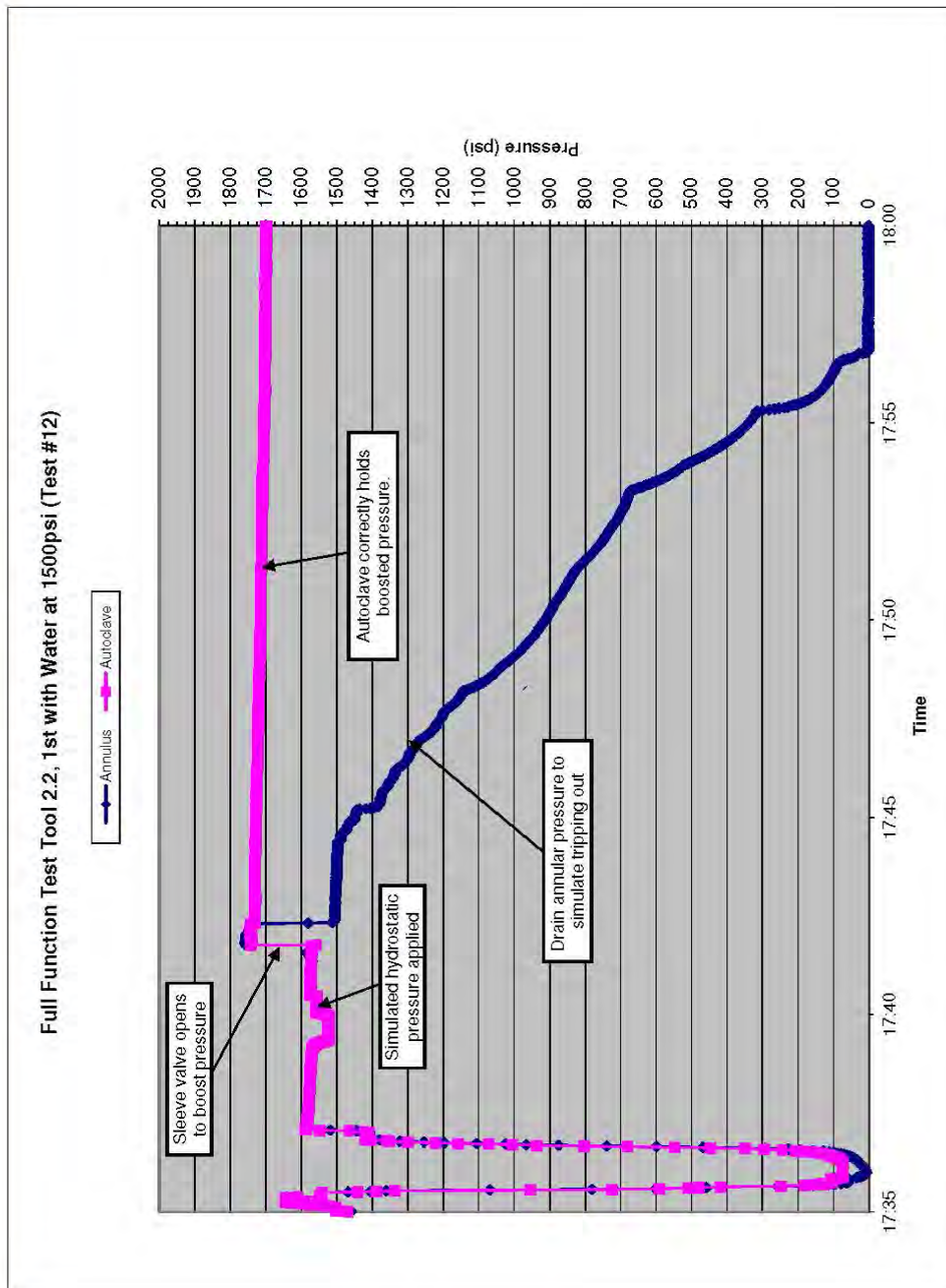


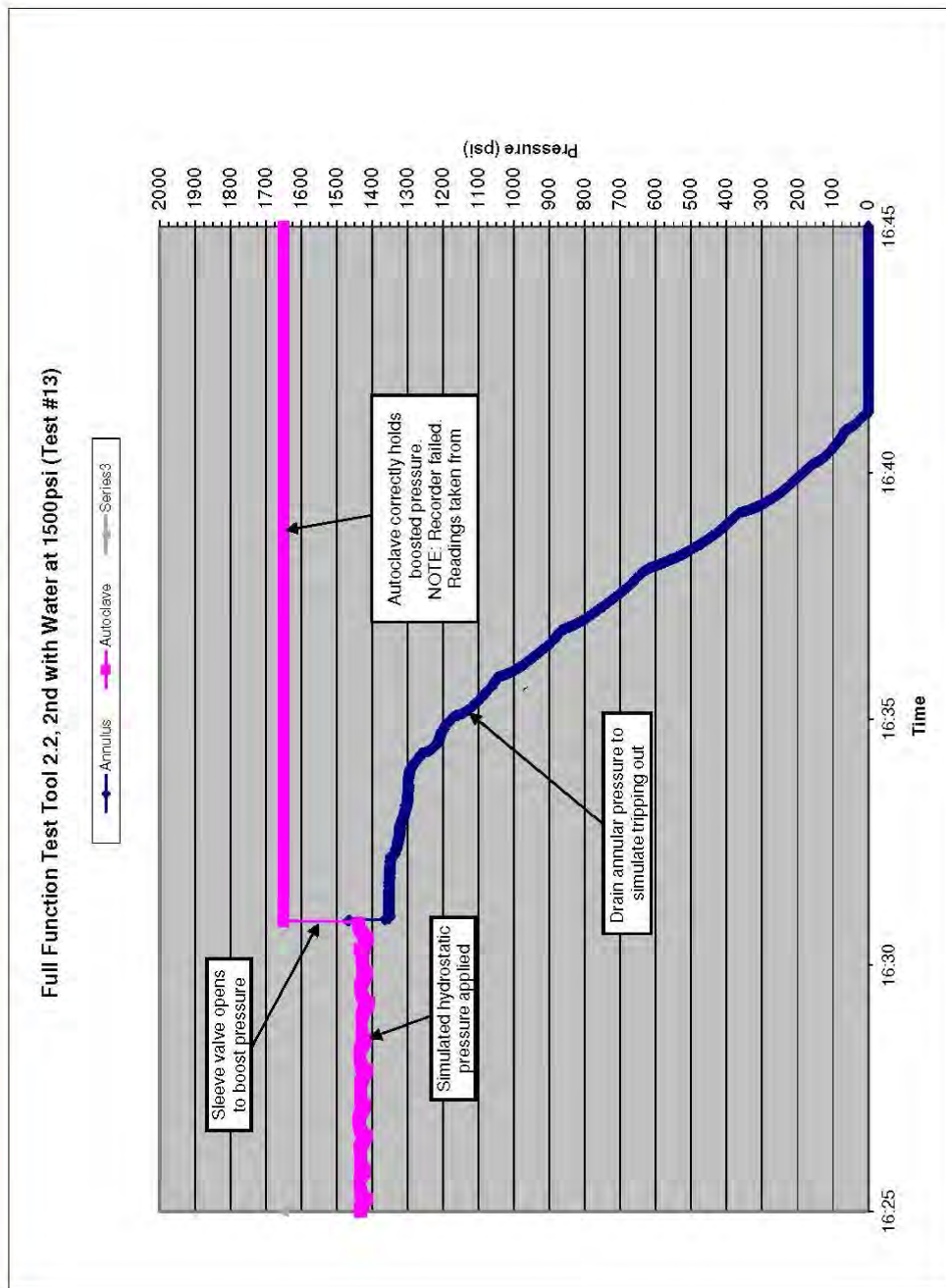


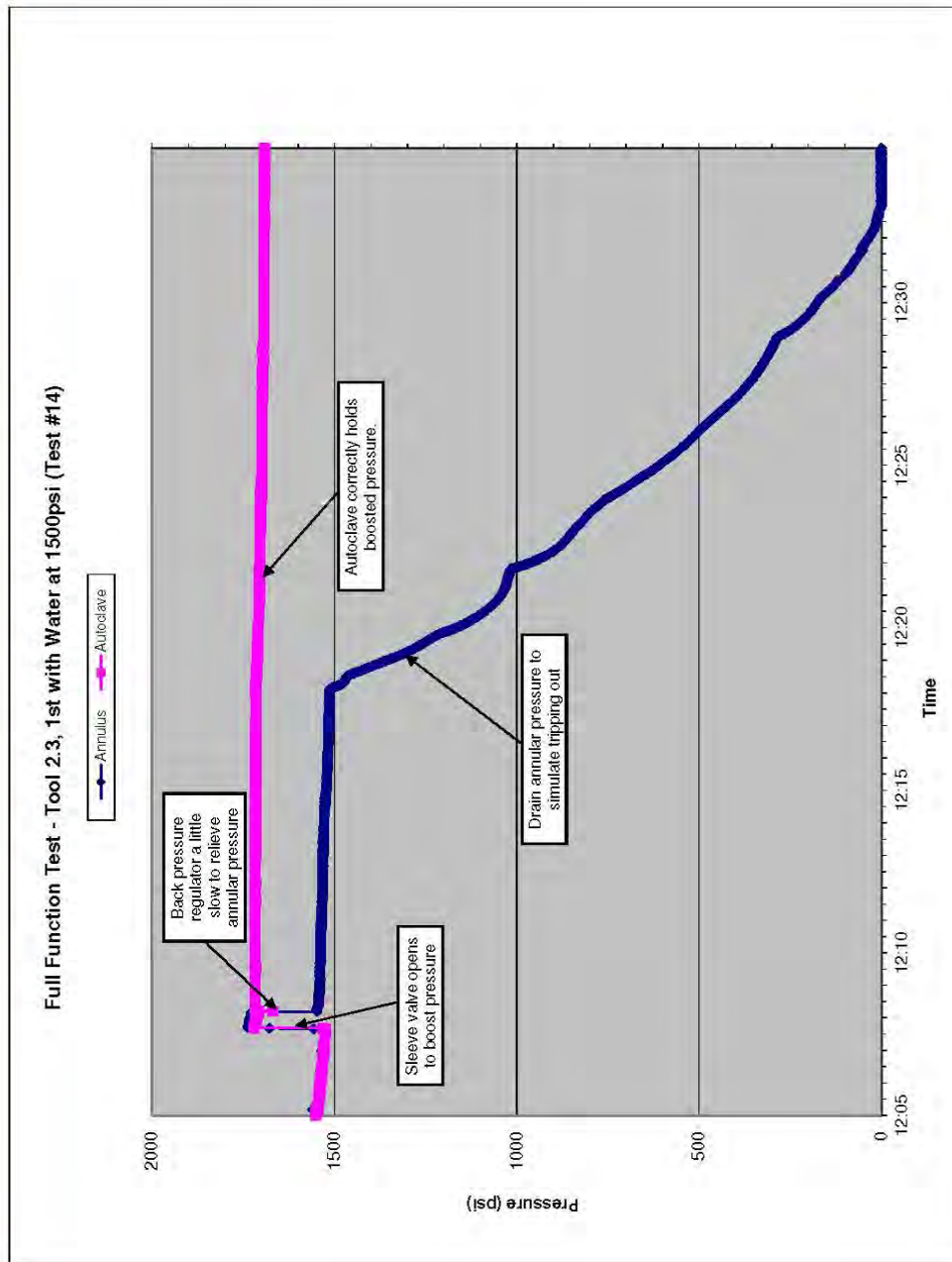


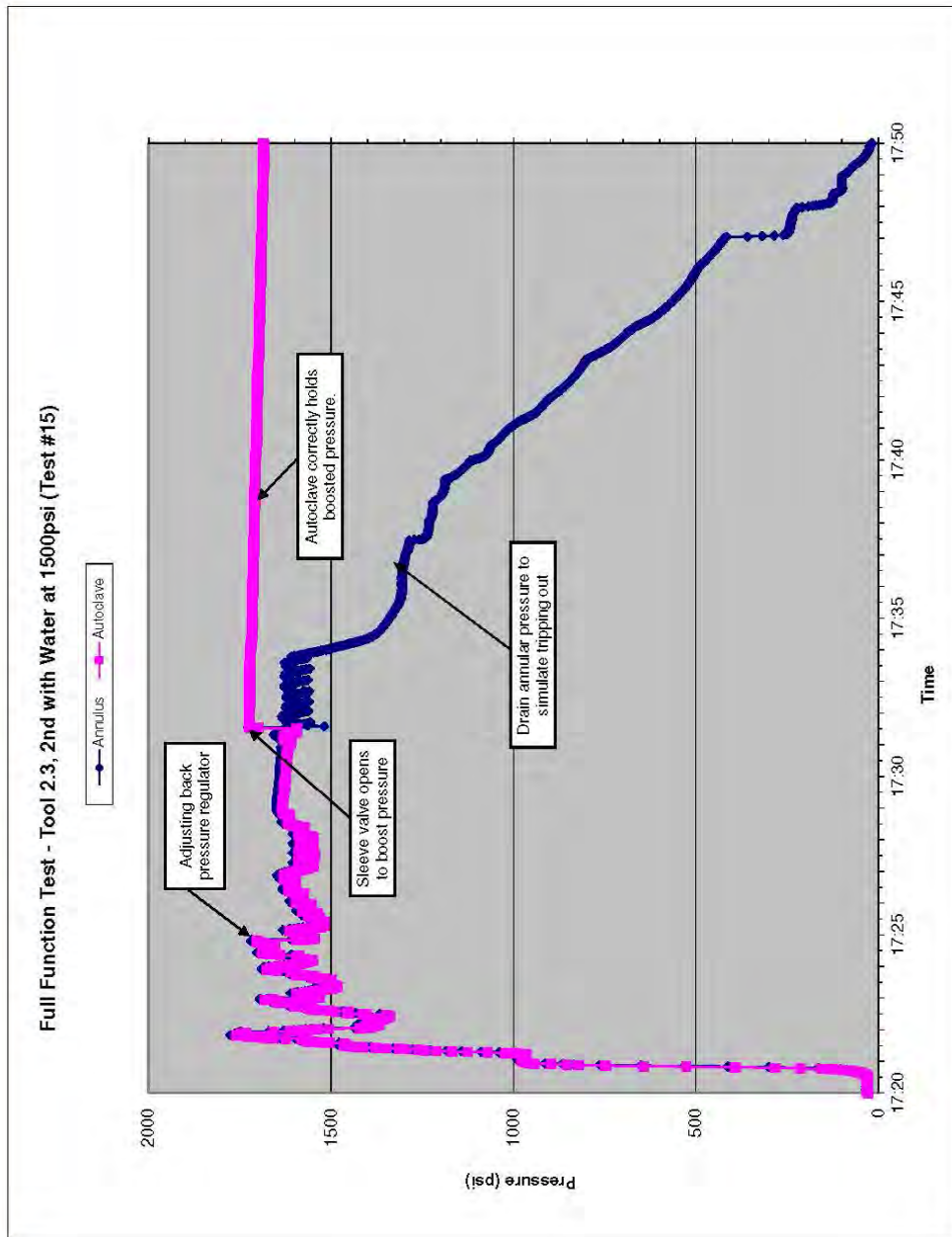






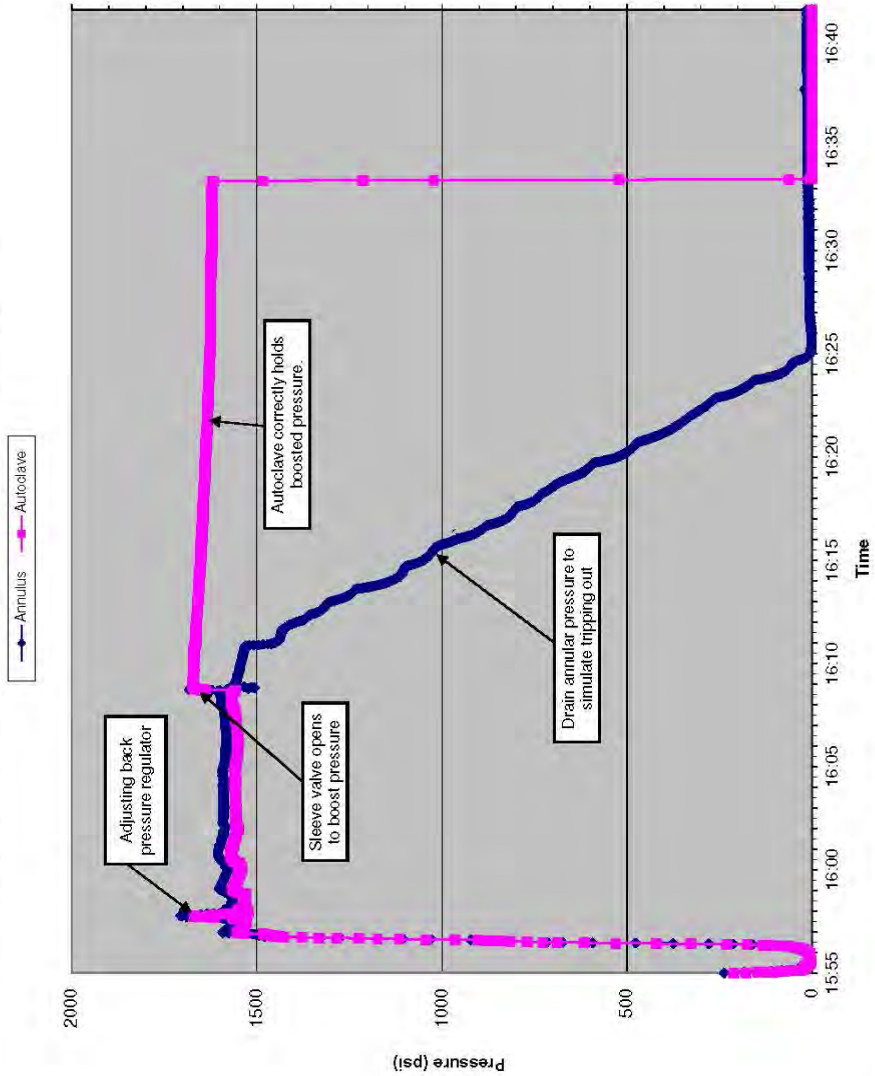






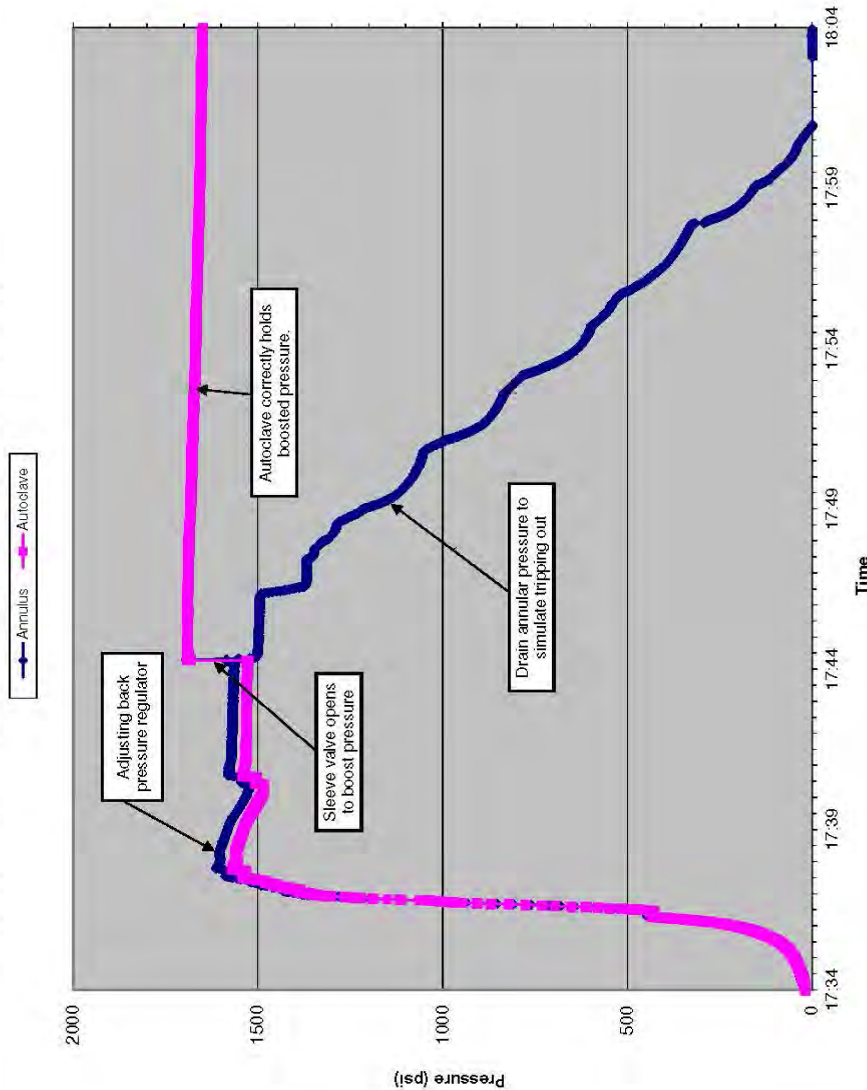


Full Function Test - Tool 3.4, 1st with Water at 1500psi (Test #16)





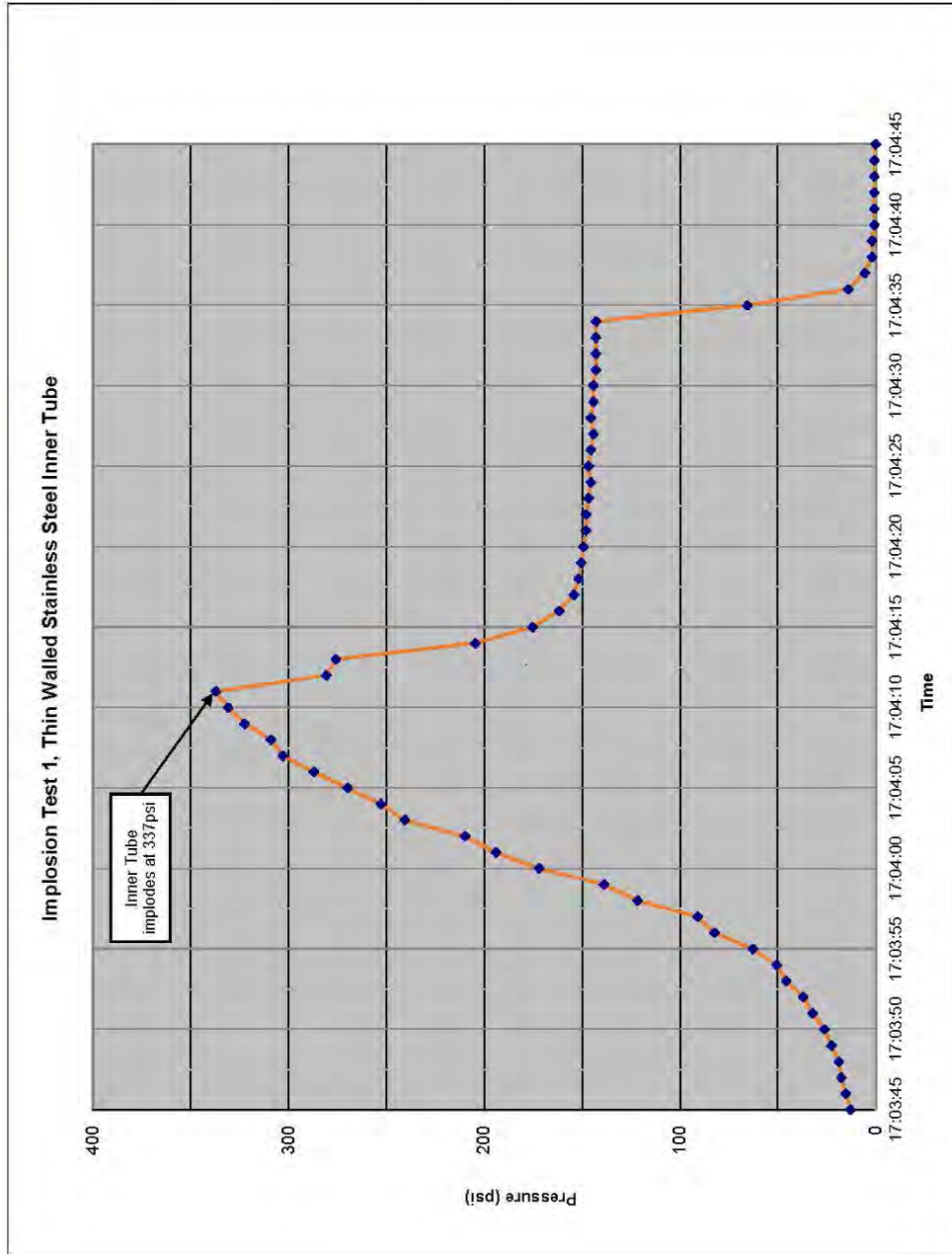
Full Function Test - Tool 3.4, 2nd with Water at 1500psi (Test #17)

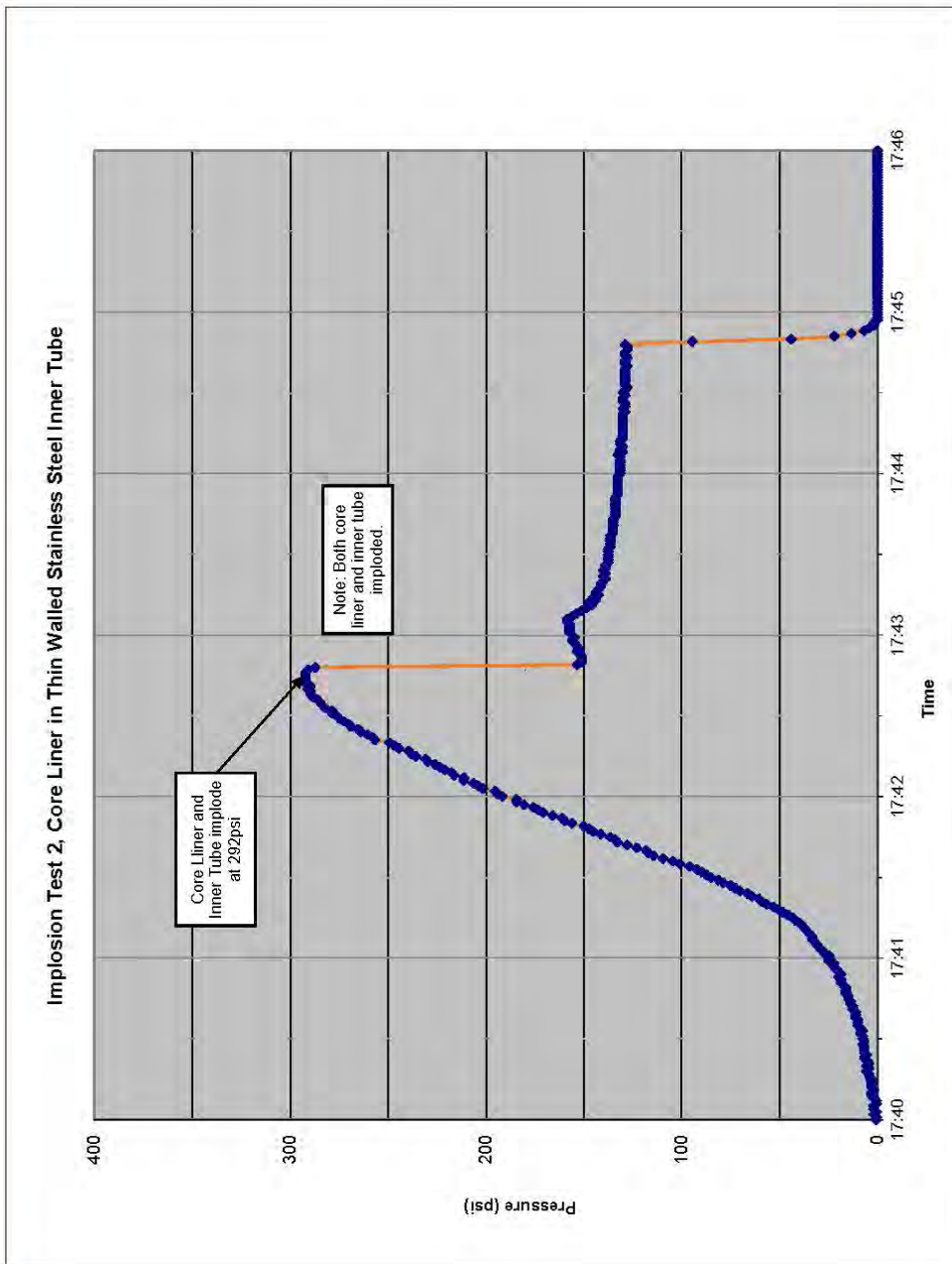


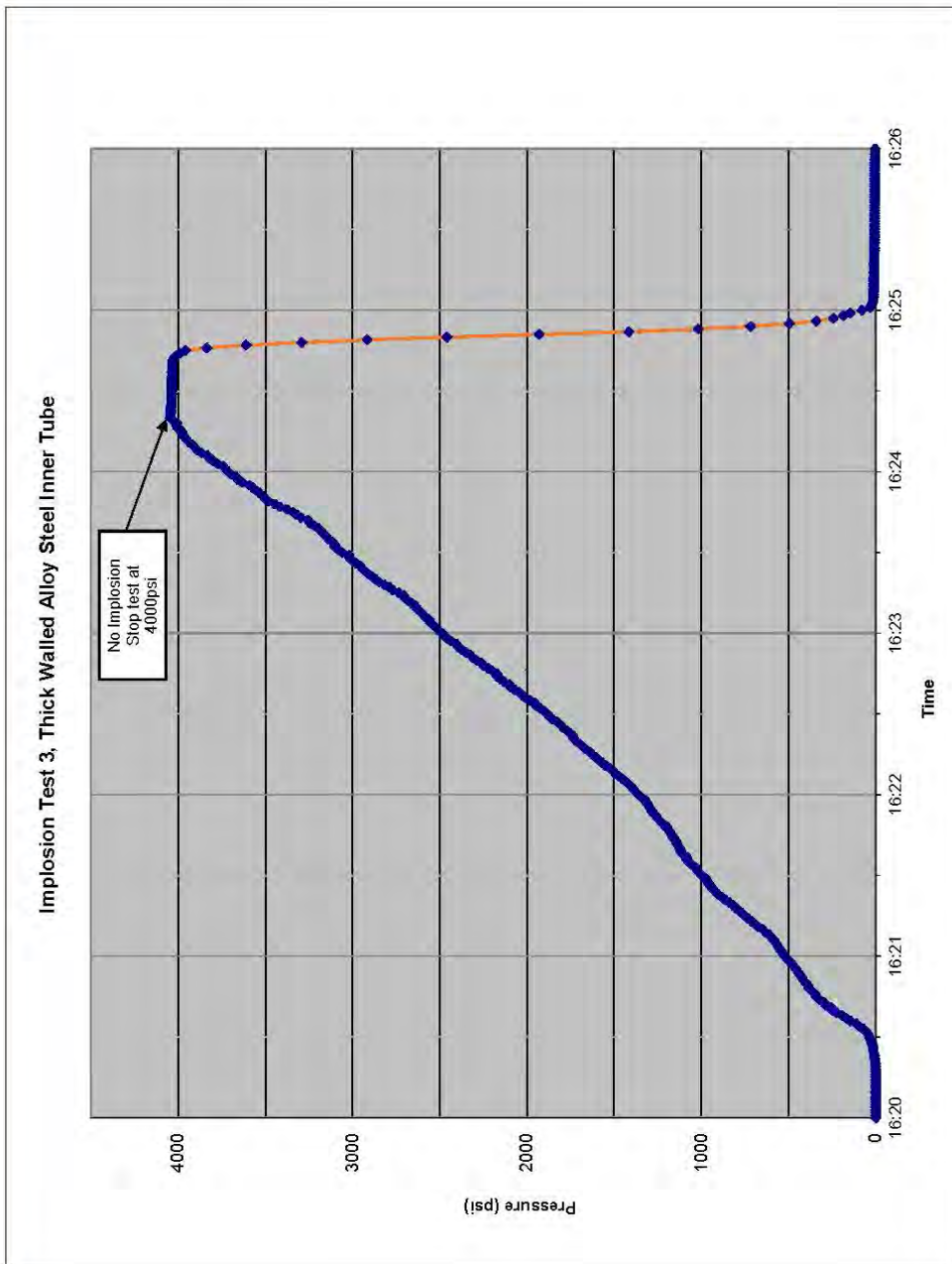


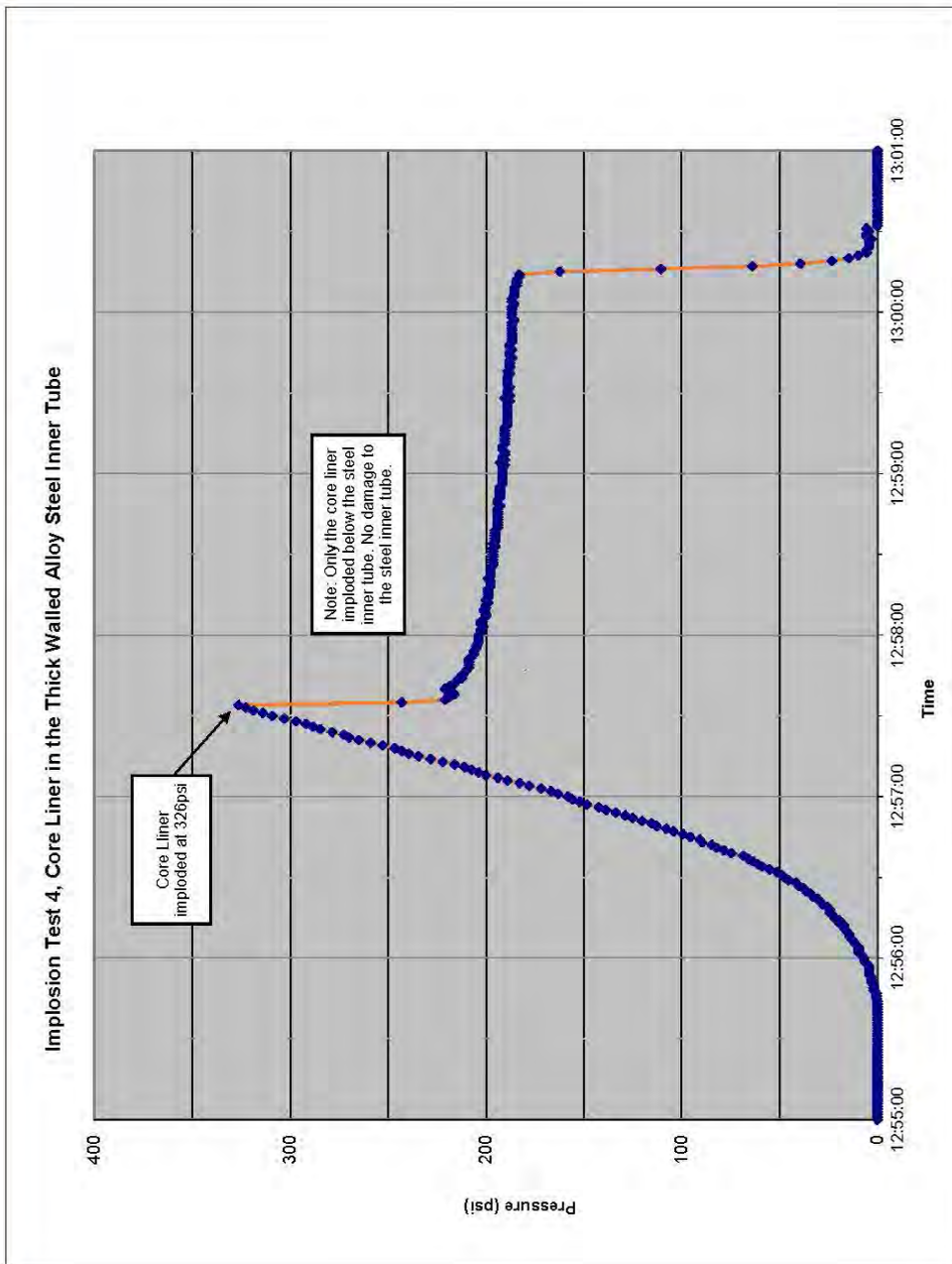


### 5. IMPLOSION TEST PRESSURE CHARTS









## **National Energy Technology Laboratory**

626 Cochrans Mill Road  
P.O. Box 10940  
Pittsburgh, PA 15236-0940

3610 Collins Ferry Road  
P.O. Box 880  
Morgantown, WV 26507-0880

13131 Dairy Ashford Road, Suite 225  
Sugar Land, TX 77478

1450 Queen Avenue SW  
Albany, OR 97321-2198

Arctic Energy Office  
420 L Street, Suite 305  
Anchorage, AK 99501

Visit the NETL website at:  
[www.netl.doe.gov](http://www.netl.doe.gov)

Customer Service Line:  
1-800-553-7681

