

**CENTER FOR MARINE RESOURCES AND ENVIRONMENTAL  
TECHNOLOGY and SEABED TECHNOLOGY REASERCH CENTER  
UNIVERSITY OF MISSISSIPPI**

Activities Report for Cruise GOM1-08-MC118 aboard the *R/V Pelican*  
Sampling and Deployment Cruise  
Mississippi Canyon Federal Lease Block 118  
Northern Gulf of Mexico  
April 22-28, 2008  
Compiled By  
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**CRUISE OBJECTIVES**

- 1) Collect sediment samples using Gravity and Box Coring devices, and
- 2) Deploy a second Pore Fluid Array

**PARTICIPANTS**

University of Mississippi: Center for Marine Resources and Environmental Technology (CMRET) and Seabed Technology Research Center (STRC):

Bob Woolsey, Carol Lutken, and Ken Sleeper, scientific staff  
Brian Noakes, Jeremy Dew and Andy Gosset, technical staff.

Florida State University

Laura Lapham, Rachel Wilson and Jim Nelson, scientific staff

University of Georgia

Marshall Bowles, scientific staff

University of North Carolina, Chapel Hill

Karen Lloyd and Wes Ingram, scientific staff

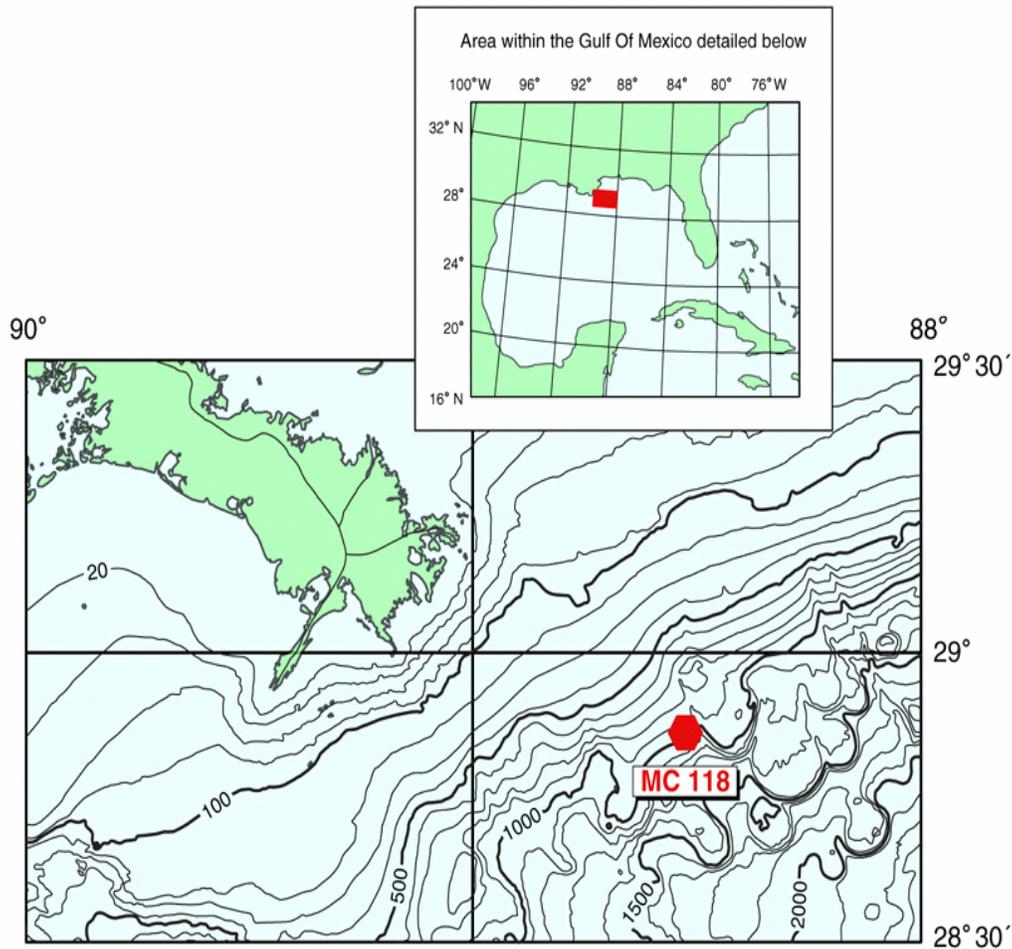
*R/V Pelican* Crew:

Craig LeBoeuf, Captain; Joe Thomas, First Mate; Jack Pennington, Chief Engineer;  
Sam LeBouef, Vessel Technician; Jordan Westmoreland, Marine Technician; Mike  
Borelon, Deck Hand; and Randy Hughes, Cook.

## INTRODUCTION

During the Gulf of Mexico Hydrates Research Consortium winter meeting (February, 2008) it was agreed upon that a cruise was needed to collect sediment and microbial samples from the Sea Floor Observatory site in the northern Gulf of Mexico. Laura Lapham of FSU and Carol Lutken of CMRET coordinated the sampling needs and protocols of a eleven different investigators. Gravity and box coring were the tools chosen for sample collection and a cruise was contracted with and conducted on the R/V *Pelican* from April 22-28, 2008 at the Observatory site at Mississippi Canyon Federal Lease Block 118 (Fig. 1). A second, but equally important, objective was to deploy a new Pore Fluid Array.

This document summarizes activities of the cruise and includes sub-reports by each of the primary participants as appendices. General core descriptions are provided in Table 3 of Appendix 1; field logs and photographs of the cores are available upon request.



Location of Mississippi Canyon Block 118 in the Gulf of Mexico

**Figure 1.** Location map of Mississippi Canyon Federal Lease Block 118.

## CORING ACTIVITIES

A regimen of sediment sampling activities was conducted during the cruise using gravity and box coring devices. The gravity corer was equipped with a 3000 pound weight connected to the top of a 10m long steel barrel. The barrel was lined with 3inch OD, schedule 40, PVC pipe with three, 3m (10ft) sections glued together for sample retention with an additional 87cm (~3ft) waste section on top. Upon recovery the depth of penetration and depth to the top of the sample were measured. The PVC tubing was then cut into 1.5m (5 ft) sections, labeled and capped with plastic plugs and black electrical tape. Cores were then either stored in an up right position inside the air-conditioned lab for later transport to land-based labs for analysis or were split open at sea. The bottom most section of each core was opened first and, if the geochemists deemed the core interesting, half of the core was taken inside the lab for geochemical and microbial sampling. The other half of the core was cleaned, described, photographed and sampled in a shaded area of the back deck.

Box coring activities utilized a GOMEX type stainless steel box corer with a 0.1m<sup>2</sup> capacity. Sediment and biota samples were collected directly from the box corer after excess water was decanted or, in select cases, following the release of sediment from the corer and subsequent sifting through the recovered material on the deck.

Cores and sediment samples were collected for 11 different investigators as summarized in Table 1, on the following page. Previous surveys of the site (including manned, video, seismic, chirp, and multibeam surveys) and previous coring activities helped direct the current coring activities to areas of highest interest. Sampling these areas of interest was greatly enhanced by recent advancements made by the Consortium.

The Consortium has developed a highly accurate system for collecting sediment samples from targeted areas. The procedure combines high resolution bathymetry data (acquired by an AUV multibeam survey of the site in 2005) with high accuracy Ultra Short Baseline (USBL) acoustic transponders and differential GPS. Figures two and three show the USBL transponder mounted to the top of the core barrel and figure four shows the Hypac navigational system guiding the sampler to the target site. Figures five and six show some of the results of being able to sample specific sites of interest.



Figure 2. USBL transponder (dark gray pressure vessel in blue mount) connected to the weight of the gravity corer.



Figure 3. 10m core barrel on deck with USBL transponder connected to top of corer.

<b>Investigator - PI</b>	<b>Institute</b>	<b>Samples</b>	<b>Analysis</b>	<b>Appendix with Sampling Details/Procedures</b>
Jennifer Biddle - Andreas Teske	UNC-Chapel Hill	Sediment samples collected from box cores (0408-BC02,-BC03 and -BC05)	Stable isotope analysis of bacterial mats	Appendix 3
Marshall Bowles – Samantha Joye	UG-Athens	Sediment samples from box core 0408-BC02	Biogeochemical analysis	Appendix 4
Charlotte Brunner	USM-Stennis	Sediment samples from core 0408-08, box core 0408-BC04 (top 30cm), several core catchers and whole core 0408-09	Micro fossil analysis for biostratigraphy	Table 3, Appendix 1
Wes Ingram - Chris Martens	UNC-Chapel Hill	Whole and partial, split core collection	X-ray fluorescence core scanner	Appendix 1 and 3
Laura Lapham - Jeff Chanton	FSU	Sediment and gas samples from 8 cores	Geochemical Analysis	Appendix 3
Karen Lloyd - Andreas Teske	UNC-Chapel Hill	Sediment samples from 7 cores and one box core (0408-03)	Microbial Analysis	Appendix 3
Melissa Lobegeier	MTSU	Sediment samples from core 0408-01 and whole core 0408-05	Micro fossil analysis	Table 3, Appendix 1
Yuliya Luzinova – Boris Mizaikoff	GIT	Sediment samples from 8 cores	Infrared analysis of carbonate minerals	Appendix 3
Rudy Rogers	MSU	Sediment samples from 3 cores and several core catchers	Microbes and microbial surfactant analysis	Table 3, Appendix 1.
Roger Sassen	TAMU	One gas sample of decomposed hydrate from core 0408-03	Gas content and isotopic analysis	Table 3, Appendix 1
Chuanlun Zhang	UG-Savannah River	Sediment samples fm two cores near a gas vent and samples from one core distant from the mound	Microbial analysis	Table 3, Appendix 1

**Table 1:** Sampling protocols for GOM01-08-MC118 cruise.

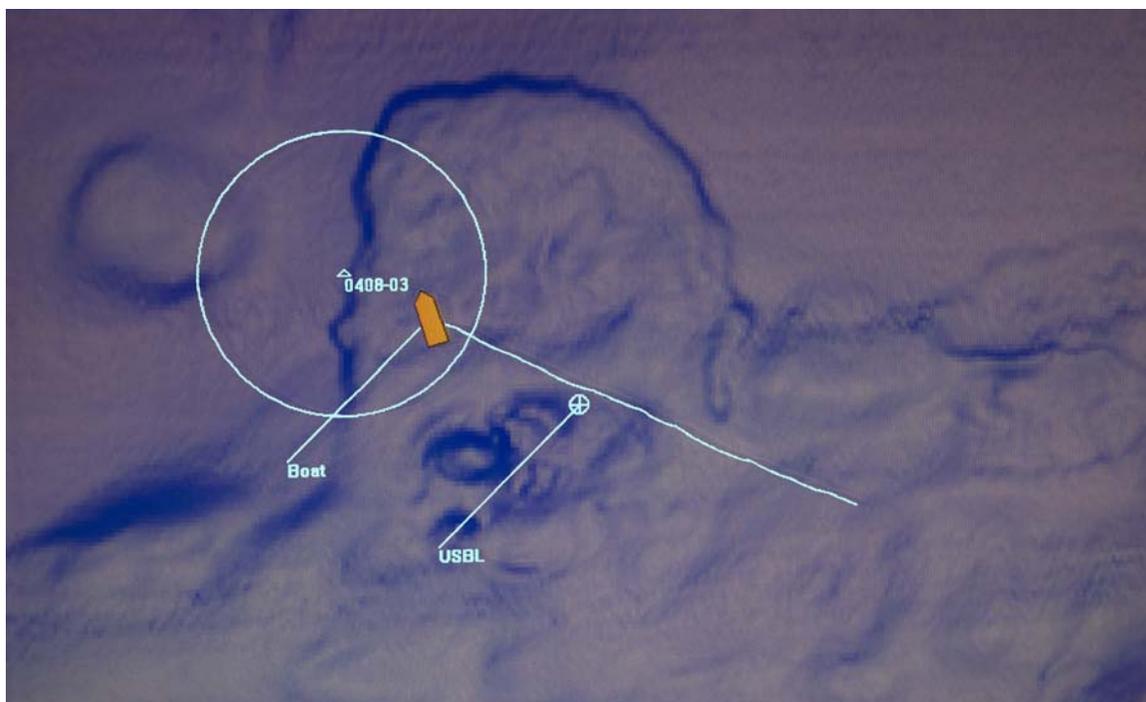


Figure 4. Screen shot of Hypac navigational software with bathymetric image background overlain with real-time DGPS and USBL information. Plotted are the target site (in the center of a 100m radius watch circle labeled 0408-03), the ship (yellow-orange polygon) with its recent track/drift line, and the USBL transponder on the core barrel. The Captain will negotiate the core barrel over the target site for the final drop from about 30m above the sea floor.

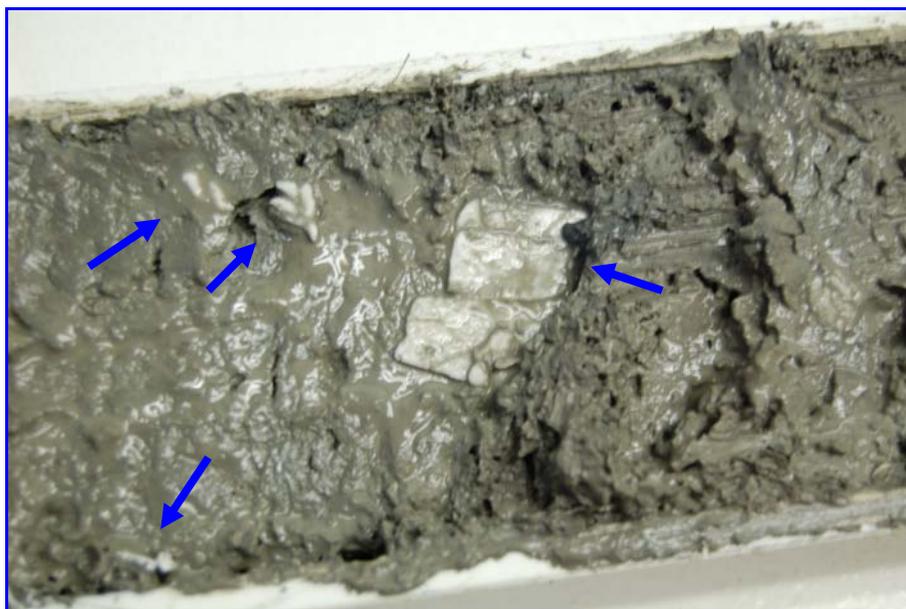


Figure 5. Thin tablets of hydrates in core 0408-03 (blue arrows). The width of the core tube is 3 inches (7.6 cm).



Figure 6. Box core with shell, tubes and bebbiotoa bacterial mats (0408-BC03)

A total of 17 gravity cores and seven box cores were attempted during the cruise. Fifteen of the gravity cores and all of the box cores had significant recovery. Core locations are presented in Figure 7, below, and in Appendix 1. A summary of the cores and sampling protocols is also presented in Appendix 1.

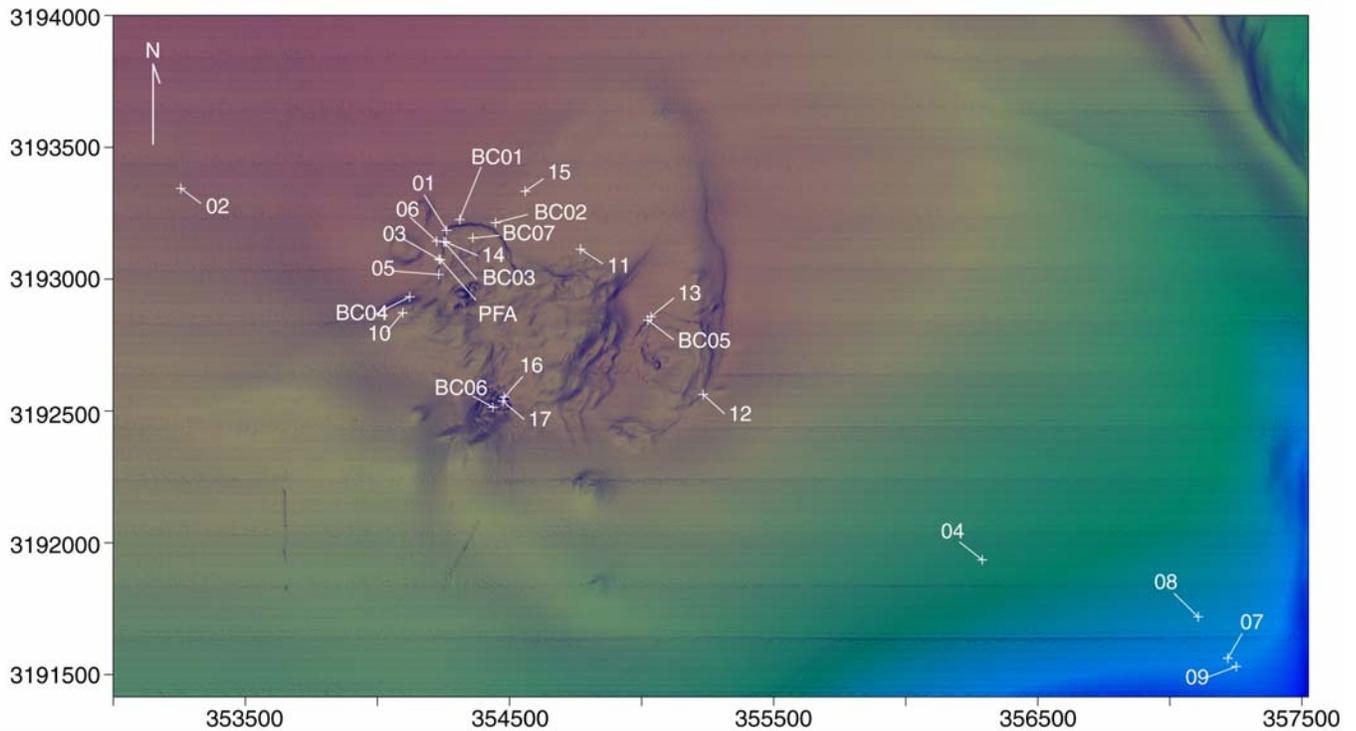


Figure 7. Gravity and box core locations. Actual core numbers are preceded with 0408 to differentiate them from previous coring activities. Box cores are preceded with BC and PFA = Pour Fluid Array. The image is bordered by UTM tick marks in meters. Higher resolution images available upon request.

## **PORE FLUID ARRAY (PFA)**

A second PFA was deployed during the cruise. It will utilize four osmo-pumps to recover sediment pore-fluid samples from selected depths along a twenty-foot probe (Figure 8) that penetrates the shallow subseafloor. Laura Lapham, co-PI on the project, directed early coring activities in an effort to identify a suitable location for the array. The site where gas hydrates were recovered (core 0408-03) was chosen and using a USBL transponder, the array was deployed within 4m (13ft) of the site. The deployment site is in a fracture zone north of the northwest crater complex (Fig. 7 and Table 2 in Appendix 1).



Figure 8. Laura Lapham holding on to the ROV docking bar of the Pore Fluid Array. The green box contains four osmo-samplers and rests on the 2000 pound drive weight.

## **CONCLUSIONS**

Weather conditions and equipment operations were conducive for a productive cruise. A multitude of samples was collected for a broad range of investigations. New coring technologies combining high resolution bathymetry with acoustic transponders provided unprecedented accuracy in targeting areas of high interest. Deployment of the new Pore Fluid Array also benefited significantly from this new technology. Areas of suspected hydrate formation were accurately cored, the presence of hydrates in the fine-grained sediments at the observatory site confirmed, and the array was placed accurately in an optimal location. Future coring operations are planned to test a hypothesis that the presence of hydrates may correlate to specific seismic anomalies.

## **BACKGROUND and ACKNOWLEDGEMENTS**

In 1999, the Center for Marine Resources and Environmental Technology facilitated the establishment of the Gulf of Mexico Hydrates Research Consortium and continues, with the assistance of the Seabed Technology Research Center, to manage the Consortium. The Consortium has as its primary objective the emplacement of a seafloor station designed to monitor the activities of gas hydrates on the seafloor and in the shallow sub-seafloor. In 2004, the consortium chose Mississippi Canyon Federal Lease Block 118 as the preferred site for the station. The Minerals Management Services of the Department of the Interior subsequently set aside a portion of the block for the exclusive use of the Consortium's research effort.

Funding for the research efforts of the Consortium are supported by Minerals Management Services, Dept. of Interior, National Energy and Technology Laboratory, Dept. of Energy, and NOAA's National Institute for Undersea Science and Technology, Dept. of Commerce. Funding for ship time for the cruise was provided by Minerals Management Services and NOAA's National Institute for Undersea Science and Technology.

## **APPENDICES:**

- APPENDIX 1, Summary of Core Locations and Sampling Protocols
- APPENDIX 2, Chief Scientist's Preliminary Cruise Report
- APPENDIX 3, FSU and UNC's Biogeochemical Group's Cruise Report
- APPENDIX 4, University of Georgia's Cruise Report

## Appendix 1, Summary of Core Locations and Sampling Protocols

<b>Core Number</b> <sup>a, b</sup>	<b>Latitude (WGS 84)</b>	<b>Longitude (WGS 84)</b>	<b>Depth (m)</b>
0408-01	28 51 30.6155	88 29 37.1635	875
0408-02	28 51 34.0250	88 30 16.1855	869
0408-03	28 51 25.6876	88 29 39.9823	873
0408-04	28 50 49.4794	88 28 23.5641	940
0408-05	28 51 23.8051	88 29 39.9778	875
0408-06	28 51 27.8996	88 29 40.3390	879
0408-07	28 50 37.7493	88 27 49.0943	981
0408-08	28 50 42.7943	88 27 53.3108	971
0408-09	28 50 36.7399	88 27 47.8980	985
0408-10	28 51 19.0349	88 29 44.9651	878
0408-11	28 51 27.1475	88 29 20.2134	882
0408-12	28 51 09.4427	88 29 02.8576	879
0408-13	28 51 18.9357	88 29 10.2699	870
0408-14	28 51 27.8290	88 29 39.0916	875
0408-15	28 50 59.6006	88 29 37.4566	897
0408-16	28 51 08.8196	88 29 30.6096	873
0408-17	28 51 08.2878	88 29 30.8124	875
0408-BC01	28 51 30.6154	88 29 37.1634	884
0408-BC02	28 51 30.2766	88 29 32.1549	881
0408-BC03	28 51 27.8615	88 29 39.3454	879
0408-BC04	28 51 21.0140	88 29 44.0146	879
0408-BC05	28 51 18.5104	88 29 10.7028	878
0408-BC06	28 51 07.5251	88 29 32.1993	880
0408-BC07	28 51 28.3709	88 29 35.2994	880
0408-PFA	28 51 25.6225	88 29 39.7775	873

**Table 2.** Coring location for gravity and box cores collected during the GOM1-08-MC118 cruise. <sup>a</sup> BC indicates box core, <sup>b</sup> PFA = Pore Fluid Array.

## Appendix 1, Summary of Core Locations and Sampling Protocols

Core #	Penetration (cm)	Recovery (cm)	Location (descriptive)	Sampling Protocol (see notes)	Comments
0408-01	Full (915)	593	NW edge of NW crater complex	GeoChem, Micro, ML, RR, CC-CB	Strong hydrogen sulfide smell with tiny bubbles forming in core minutes after opening
0408-02	653	458	1km NW of mound complex	Capped for transporting	Core collected for Wes Ingram, UNC-Chapel Hill
0408-03	565	964	W edge of NW complex	GeoChem, Micro, Gas, CZ, CC-CB and -RR	Hydrates and abundant gas expansion pockets. Core bubbled profusely during recovery and sediment was forced out the top, around the check ball.
0408-04	654	417	1.25km SE of mound complex	Capped for transporting	Core collected for Wes Ingram, UNC-Chapel Hill
0408-05	930	513	W edge of NW complex	Capped for transporting, CC-CB and -RR	Core collected for Melissa Lobegeier, Middle Tennessee State University
0408-06	Full (915)	598	W edge of NW complex	GeoChem, Micro, WI, CZ, CC-CB and -RR,	Gassy core, moderate to mild hydrogen sulfide smell
0408-07	1030	638	2.25km SE of mound complex	Capped for transporting	Core Collected for Wes Ingram, UNC-Chapel Hill
0408-08	1059	729	2 km SE of mound complex	RR, CB, CZ, WI, CC-CB and -RR	No hydrogen sulfide smell, red zone encountered at 190cm
0408-09	1034	543	2.25km SE of mound complex	Capped for transporting, CC-CB and -RR	Core Collected for Charlotte Brunner, University of Southern Mississippi-Stennis
0408-10	639	131	Depression area 100m W of NW crater comp.	GeoChem, Micro, CC-CB and -RR	Strong hydrogen sulfide smell, carbonate nodules and layers.
0408-11	Full (915)	734	North central edge of mound complex	GeoChem, Micro, WI, CC-CB and -RR,	Bubbling form barrel on recovery, bubbling sediments in open core with faint fracture pattern. Abundant gas pockets. Small flakes of hydrates in bottom most section.
0408-12	603	230	SE corner of SE crater complex	GeoChem, Micro, CC-CB and -RR	Hydrogen sulfide smell, voids (gas pockets), and carbonate nodules and rocks
0408-13	Full (915)	615	NE corner of SE crater complex	GeoChem, Micro, CC-CB and -RR	No odor. Mottled, interlayered light brown or gray and olive with black flecks.

### Notes to Sampling Protocols:

GeoChem = sampling protocol followed for geochemical analysis as described in detail in Appendix 3.

Micro = sampling protocol followed for microbial analysis as described in Appendix 3 and Appendix 4.

Gas = Gas sample of decomposed hydrate collected for Roger Sassen, TAMU. Sample collected by submerging hydrate in 5gallon bucket of water and collecting emanations in submerged mason jar held above degassing sample.

CB = Charlotte Brunner, SMU-Stennis, sediment sample recipient for ~1cm sample every 20cm from core 08 and bagged samples from several core catchers. Sediment sample collected from top 30cm of box core 04.

CC = core catcher bagged sediment sample; CC-CB = Core catcher sample for Charlotte Brunner, CC-RR = Core catcher sample for Rudy Rogers.

CZ = Chuanlun Zhang, UG-Savannah, sediment sample recipient for ~1cm sample every 10cm (5cm for core 08) for top meter and every meter thereafter for the length of the core.

ML = Melissa Lobegeier, MTSU, sediment sample recipient of ~1cm wide samples collected every 5cm for the first (top) 1 meter and then once every meter thereafter.

RR = Rudy Rogers, MSU, sediment sample recipient of ~2-3cm wide samples collected at least once every 3m from three cores and bagged samples from several core catchers.

WI = Wes Ingram, UNC-CH, one or more sections of split-core half saran wrapped and transported to UNC for analysis (see Appendix 3 for details on specific sections).

## Appendix 1, Summary of Core Locations and Sampling Protocols

Core #	Penetration (cm)	Recovery (cm)	Location (descriptive)	Sampling Protocol (see notes)	Comments
0408-14	1011	677	W edge of NW complex, N of PFA	GeoChem, CC-CB and RR	Well laminated upper section, burrowed middle section and mottled to layered lower section.
0408-15	Full (915)	510	Fracture zone SW of SW crater complex	Section 1-3 capped for transporting, CC-CB and RR	Core Collected for Wes Ingram, UNC-Chapel Hill, Bottom, 53cm, section split open and described
0408-16	Null	Null	Sleeping Dragon SW crater	N/A	Minor shell and rock with oily smell in shoe
0408-17	Null	Null	Sleeping Dragon SW crater	N/A	Minor rocky material in shoe
0408-BC01	Box core		N of NW vent complex	GeoChem and Micro	Lost core on deck by heavy handed operator. Black smelly mud with some gastropods, mussels and worms.
0408-BC02	Box core		NNE of NW vent complex	GeoChem and Micro	Target is original PFA. Strong hydrogen sulfide smell with gastropods, mussels and worms.
0408-BC03	Box core		W edge of NW complex, N of PFA/ core 14	GeoChem and Micro	Spectacular box core of apparent tube worms and healthy patch of <i>Beggiatoa</i> bacterial mat.
0408-BC04	Box core		Depression area 100m W of NW crater comp./ core 10	GeoChem, Micro, CB	Collected two grab samples for Charlotte Brunner, lots of forams (?), very gritty with Pogonophoram worms.
0408-BC05	Box Core		NE corner of SE crater complex / Core 13	N/A	Numerous clam shells and soft sided tube worms (parchment paper worms). No smell
0408-BC06	Box core		SW crater complex	Micro	Dead clam shells at surface, but intact, one live polychaete, one rock, little bits of oil leaving a sheen on the water surface. No visible mat but did collect a grab sample collected for mat analysis.
0408-BC07	Box core		Central area of NW vent complex	N/A	

**Table 3.** Summary of core logs and sampling protocols for GOM1-08-MC118.

### Notes to Sampling Protocols:

GeoChem = sampling protocol followed for geochemical analysis as described in detail in Appendix 3.

Micro = sampling protocol followed for microbial analysis as described in Appendix 3 and Appendix 4.

Gas = Gas sample of decomposed hydrate collected for Roger Sassen, TAMU. Sample collected by submerging hydrate in 5gallon bucket of water and collecting emanations in submerged mason jar held above degassing sample.

CB = Charlotte Brunner, SMU-Stennis, sediment sample recipient for ~1cm sample every 20cm from core 08 and bagged samples from several core catchers. Sediment sample collected from top 30cm of box core 04.

CC = core catcher bagged sediment sample; CC-CB = Core catcher sample for Charlotte Brunner, CC-RR = Core catcher sample for Rudy Rogers.

CZ = Chuanlun Zhang, UG-Savannah, sediment sample recipient for ~1cm sample every 10cm (5cm for core 08) for top meter and every meter thereafter for the length of the core.

ML = Melissa Lobegeier, MTSU, sediment sample recipient of ~1cm wide samples collected every 5cm for the first (top) 1 meter and then once every meter thereafter.

RR = Rudy Rogers, MSU, sediment sample recipient of ~2-3cm wide samples collected at least once every 3m from three cores and bagged samples from several core catchers.

WI = Wes Ingram, UNC-CH, one or more sections of split-core half saran wrapped and transported to UNC for analysis (see Appendix 3 for details on specific sections).

## Appendix 2, Chief Scientist's Preliminary Cruise Report

During the period from April 22 through 27, we conducted a Coring and PFA deployment cruise.

On this occasion we attempted 17 cores with our 10m, 1.5 ton, Gravity Corer, fitted with USBL. Of the 17, 15 had significant recoveries. Two coring attempts (at East Crater, Southwest Crater Complex) conducted under high current conditions (1.5 kts) before termination of coring activity retrieved only carbonate fragments in the core catcher (however, oil saturated). Of the successful cores most had penetrations ranging from 6 to 10m with recoveries up to 8m. About half had a hydrogen sulfide smell; one contained considerable hydrates and one had minor hydrates. All were in fine-grain sediments (mainly silt with some clay). The hydrate rich core was taken at a fracture zone located on the west flank of the Northwest Crater margin. It penetrated 5.65m and contained blades and thin tablets of hydrate which were vertically oriented and subparallel (polygonal/tensional micro-fracture control?). The hydrates were mainly concentrated in the lower core section, gas having blown out the upper core material, but the lower 1.5 to 2m of material retained, contained intact hydrates. To what depth the hydrates extend is an interesting unknown. The gas boiling up past the ball valve of the Gravity Corer as it approached the surface was so violent you could not make out the large drive weight--very impressive! We will review the core site with our new enhanced geophysical data once processed.

We had used the USBL with the box corer previously, but this was a first, using it with our 10m Gravity Corer. Our resulting accuracy was rather impressive. We typically lower to within 30m of the bottom, monitor the rate of drift, then when we are within range of the target, with final adjustments from the helm, we execute the drop. We also had a good and successful deployment for Laura's new improved PFA, (also using the USBL fitted with floats, equating to 250 lbs. positive flotation, to prevent impact with the bottom). We detached our cable rig using acoustic releases. The placement was almost directly on top of the site where we recovered the hydrate-bearing core (4m to the west).

The hydrate in the other core (0408-11) was also very interesting, if not in volume, at least in its mode of occurrence. The hydrates were unlike anything we had ever seen before in that they appeared as small irregular grains estimated to be 2 to 4mm in dia. (rapidly dissociated on exposure), disseminated throughout a short section of core, (5 to 20 cm). Having so observed, we can relate this mode of occurrence to other cores taken previously at the mound site (noted as gas blips).

J. R. Woolsey, Chief Scientist

## Appendix 3, FSU and UNC's Biogeochemical Group's Cruise Report

Cruise Report of coring efforts aboard the R/V/ Pelican

MC 118, April 22-April 28, 2008

Participants: Laura Lapham, FSU; Karen Lloyd, UNC; Wes Ingram, UNC; Marshall Bowles, UGa; Jimmy Nelson, FSU; and Rachel Young, FSU.

The goals of this cruise were to 1) collect gravity cores for biogeochemical and microbial analysis, 2) deploy the second Pore-Fluid Array and 3) collect box cores for targeted geochemical and microbial analysis under bacterial mats.

**1. Coring:** Eight gravity cores ( core # 1, 3, 6, 10, 11, 12, 13, and 14) were sampled by splitting them lengthwise and half was visually described and half was sub-sampled for geochemical and microbial analyses.

**Geochemical constituents to be measured:** Methane and other light hydrocarbon concentration and isotope analysis, porosity, DIC concentrations and isotopes, and SO<sub>4</sub>, Cl, Br, I, Na, K, Mg, Ca, Li, Sr, Ba, Mn, and B concentrations.

**Sub-sampling procedure:** The gravity cores were sub-sampled every 25 cm. Using a 30-mL cut-off syringe, sediment was collected and 10 mL placed into a pre-weighed glass serum vial. These vials were immediately capped and frozen upside down with a 5-mL water. The remaining sediment in the cut-off syringe was then used to fill a 50-mL centrifuge tube. Tubes were then centrifuged at 5000rpm for 15 minutes to separate out the pore-fluids from the sediments. Once separated, the supernatant was drawn into a 10-mL plastic syringe. Aliquots of these fluids were then taken for pore-fluids major ion analysis, DIC concentration and isotopes, and sulfides. Ion sub-samples were acidified with 200uL of 10% HNO<sub>3</sub> acid to eliminate reoxidation of sulfide to sulfate.

The remaining sediments in centrifuge tubes were sub-sampled for Yulia Luzinova to conduct IR analysis. This analysis determines the composition of the carbonate (calcite, dolomite, etc) and indicates whether or not the carbonate is authigenic (produced in situ) or allogenic (transported from sedimentation).

**Shipboard analysis:** Sulfate and chloride concentrations were measured at sea on a Dionex 2010i ion chromatograph. There were no anomalous chloride concentrations measured. However, the sulfate profiles varied widely in spatial scale and we found several different depths of sulfate depletion:

<u>Core #</u>	<u>Depth of sulfate depletion</u>
Core 1	50cm
Core 3	50cm
Core 6	125cm
Core 11	200cm

## Appendix 3, FSU and UNC's Biogeochemical Group's Cruise Report

**Gas hydrate gas and expansion void gas:** Six gas samples were collected from expansion voids in degassing cores and one sample of hydrate bound gas. These samples will be measured for hydrocarbon concentrations and isotopes.

**Microbial analysis:** Samples were immediately frozen for microbiological analysis from selected depths of gravity cores 01, 03, 06, 10, 11, 12, and 13. Samples from the same sections were fixed with 3% formaldehyde in preparation for fluorescence in situ hybridization work. RNA will be extracted from frozen sections, reverse transcribed, and PCR-amplified, and sequenced, in order to obtain a description of the active population of microbes present. Since all other microbiological work at MC118 has been within the top 30 cm, these gravity cores will provide important clues to how the deep biosphere may be affecting these surface communities. Previous geochemical analysis has suggested that methanogen activity contributes to the otherwise thermogenic methane flux in these sediments. Now we have the opportunity to characterize these hypothesized communities directly.

**Whole core collections:** The following cores recovered by the *RV Pelican* in April 2008 are stored at the University of North Carolina, Department of Geological Sciences Core Lab: (1) **Core 0408-02**, (2) **Core 0408-04**, (3) **Core 0408-07**, and (4) **Core 0408-15**. The above cores were not split and described by the shipboard scientists. The following cores were split and described by the shipboard scientists and are also stored at the UNC core lab: (1) **one-half of Core 0408-08, all sections**, (2) **one-half of Core 0408-06 Section D**, and (3) **one-half of Core 0408-11 Section 1**. The core lab facility is equipped with a cold storage room that maintains a temperature of ~38° F adequate for long-term storage of sediment samples.

**Analyses:** All non-destructive measurements will be performed prior to any destructive sampling of the above listed cores. After completion of these analyses the above material will be open to sampling by the investigator and other collaborators. It is estimated that non-destructive analyses can be completed before the end of 2008.

**Non-Destructive Analyses:** We will quantify solid-phase bulk geochemical variability in the cores using an Avaatech XRF-scanner at the core lab facility at UNC-Chapel Hill. The XRF-scanning method provides continuous, non-destructive measurements of elemental geochemistry up to millimeter-scale resolution. We will focus our attention on the following elemental profiles. Ba concentration and Ba/Al, a proxy for methane hydrate eruption events, Ca/Al and Si/Al, to quantify biogenic carbonate and silica sedimentation (e.g. Aluminum, titanium and iron, primarily derived from a detrital source), and redox sensitive trace metals vanadium, chromium and manganese. Lithostratigraphy will also be carried out on all recovered cores aided by microlithostratigraphy and preparation of sediment smear slides. These data will be provided to the MC-118 gas hydrate consortium upon completion.

**Destructive Analyses:** A minute amount of sediment will be collected in each core at a 10 cm resolution for nannofossil and lithostratigraphic smear slides. Select samples will be taken for ICP emission spectrometry at various depths to calibrate X-ray fluorescence counts. Samples will be taken from each region of the core that exhibit lithostratigraphic change to account for the full variability of bulk sedimentation. Additional samples will be taken and prepared where needed for foraminiferal

## Appendix 3, FSU and UNC's Biogeochemical Group's Cruise Report

biostratigraphy. These samples will also be used for stable carbon and oxygen isotopic measurements of select planktonic foraminifera. After these destructive analyses, the above cores will be open for sample request. Collaborators will be given priority for sample request and Charlotte Brunner has priority for sampling for Biostratigraphy.

**2. Deployment of the Pore-Fluid Array:** The PFA was successfully deployed on the western rim of the northwestern complex at MC 118. It was positioned within 4 meters of core 3 which contained gas hydrate. This location is ideal to answer the question of the time-scale of hydrate formation and decomposition events. By collecting pore-fluids over time with the PFA and analyzing these fluids for chloride and methane concentrations, we can determine if hydrate formation and decomposition events. The PFA was outfitted with 4 pumps, collecting at overlying water, and 30, 284, and 533 cm below the cement weight. Pumps contained copper coil long enough for a year deployment. We will gain visual confirmation of the success of this PFA deployment when the SSD (with cameras) visits the site on the May/June 2008 cruise.

**3. Collection of box cores:** Box core samples that contained visible surficial bacterial mat (box cores 02, 03, and 05) were scooped into 1-gallon containers, refrigerated, and transferred to Chapel Hill for analysis by Jennifer Biddle. She plans to investigate the stable carbon and nitrogen isotopes of *Beggiatoa sp.* present in these samples. Anoxic sediment from box core 01 that spilled out on the ship's deck was scooped up and transferred to Drew Steen in Chapel Hill in order to develop methods to analyze carbohydrates in anoxic porewater.

Anoxic sediment from box core 03 was slurried with overlying water shipboard, distributed into Hungate tubes, gassed with N<sub>2</sub>, and sealed in N<sub>2</sub>-gassed paint cans for anoxic incubation at in situ temperature. Karen Lloyd will then attempt to correlate microbiological changes to the changing geochemistry as the incubation progresses.

Box core 0408-BC02 was sampled by Marshall Bowles, University of Georgia, for geochemical and microbial analysis. Sampling and analytical details for these samples are provided in Appendix 3, University of Georgia's Cruise Report.

## APPENDIX 4, University of Georgia's Cruise Report

### Mississippi Canyon lease block 118 April 2008 Cruise Report

#### Biogeochemistry summary by Dr. Samantha Joye and Marshall Bowles

##### *Introduction*

During the April 2008 cruise our goal was to investigate the microbial activity within vacuolated sulfide oxidizing bacteria (VSOB) (e.g. *Beggiatoa sp.*) containing sediment. We collected sediment from a box core (BC03) at the northwest vent site (28° 51' 27.8615 N and 88° 29' 39.3454 W) that successfully retrieved *Beggiatoa sp.* (see Figure 1a). The sediment core contained what appeared to be authigenic carbonates with thick mat material interspersed at the seawater-sediment interface. The sediment was black at the surface, indicative of iron sulfide, and at depth became light gray (see Figure 1b).

A.



B.



Figure 1. A) Close up of *Beggiatoa sp.* in box core subsample B) Sediment sample showing color gradient

##### *Methods*

We will perform a geochemical characterization of the box core sediments. Specifically we will measure the environmental concentrations of methane ( $\text{CH}_4$ ), sulfate ( $\text{SO}_4^{2-}$ ), ammonium ( $\text{NH}_4^+$ ), hydrogen ( $\text{H}_2$ ), sulfide ( $\text{H}_2\text{S}$ ), phosphate ( $\text{PO}_4^{3+}$ ), nitrate ( $\text{NO}_3^-$ ), nitrite ( $\text{NO}_2^-$ ), iron ( $\text{Fe}^{2+}$ ), Dissolved Inorganic Carbon (DIC), Dissolved Organic Carbon (DOC), and Volatile Fatty Acids (VFA). During the cruise geochemical samples were collected and preserved in the same manner as Joye and others (2004). Molecular samples were collected for Florescent In Situ Hybridization (FISH) as described by Orcutt and others (2005). Finally, samples for clone libraries were immediately frozen ( $-20^\circ\text{C}$ ).

The rates of sulfate reduction will be determined for 3 cm intervals of sediment using plug flow reactors as described in Pallud and others (2007). Briefly, intact sediment will be transferred into reactors wherein, they will be subjected to a constant flow of artificial porewater (APW), containing sulfate as the sole electron acceptor. A breakthrough curve will be generated using the inert tracer  $\text{Br}^-$ , to assure constant flow. Some fraction of the known

## APPENDIX 4, University of Georgia's Cruise Report

amount of sulfate in the APW will be reduced to hydrogen sulfide, and sulfate concentration on the outflow will be measured to determine the rate of sulfate reduction.

### *Preliminary Findings*

So far sulfate and sulfide concentrations have been determined for the box core sediments. Sulfate concentrations are below seawater concentrations (23 mM) at the first horizon, but fall to 10.7 mM by 21.5 cm (Figure 2). The sulfide concentrations reach approximately 12 mM by 21.5 cm horizon. The low sulfate concentrations and high sulfide concentrations imply an active zone of sulfate reduction at depth.

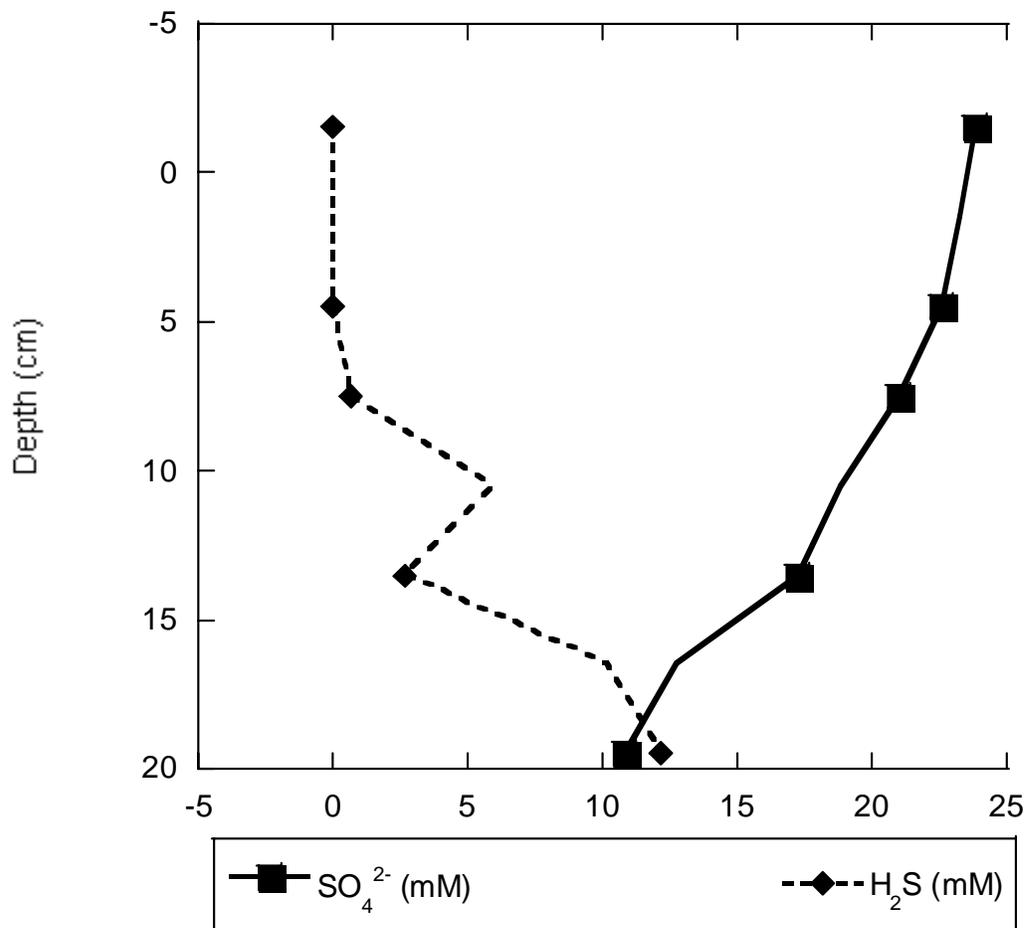


Figure 2. Sulfate and sulfide concentrations versus depth from box core sediments.

## APPENDIX 4, University of Georgia's Cruise Report

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