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Heuer, V.B.
I.W. Aiello, M. Elvert, N.I. Goldenstein, T. Goldhammer, M. Könneke, X. Liu, S.S. Pape, F.A. Schmidt, J.K. Wendt, G. Zhuang

REPORT AND PRELIMINARY RESULTS OF RV POSEIDON CRUISE POS450

DARCSEAS II

DEEP SUBSEAFLOOR ARCHAEA IN THE WESTERN MEDITERRANEAN SEA: CARBON CYCLE, LIFE STRATEGIES, AND ROLE IN SEDIMENTARY ECOSYSTEMS

BARCELONA (SPAIN) – MALAGA (SPAIN)
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BERICHTE AUS DEM MARUM UND DEM FACHBEREICH GEOWISSENSCHAFTEN
DER UNIVERSITÄT BREMEN

Report and Preliminary Results of *RV POSEIDON* Cruise POS450

DARCSEAS II

Deep subseafloor Archaea in the Western Mediterranean Sea: Carbon Cycle, Life Strategies, and Role in Sedimentary Ecosystems

Barcelona (Spain) – Malaga (Spain)
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2014

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1 Summary/Kurzfassung

RV POSEIDON cruise POS450 “DARCSEAS II” (April 2-13, 2013) was conducted in the Western Mediterranean to study relationships between the riverine input of terrestrial organic matter and marine microbial communities. The expedition targeted the input of two large rivers with contrasting climatic and hydrological regime of their hinterland: the River Rhône (France) and the River Moulouya (Morocco). For geochemical and microbiological investigations, water and sediment samples were taken from the water column and from the seafloor along a series of sites following the trajectory of terrestrial organic matter. Post-cruise, these samples will be used to constrain factors that influence the distribution of benthic archaea in marine sediments. Ultimately, our research aims to better understand the role of benthic archaea in the marine carbon cycle. Four different sampling devices were deployed: gravity corer, multicorer, CTD-rosette, and in-situ pumps. The highest priority of our shipboard work program was the preparation and storage of subsamples for complex geochemical and microbiological experiments and analysis in the home laboratories. Due to time constraints, only few sedimentological and geochemical investigations could be conducted directly on board. Within less than 11 days, the cruise covered 1118 nautical miles (NM), operated 106 hours on site, established 15 new GeoB sites (GeoB17301 – GeoB17315), and yielded >4000 discrete samples for post-cruise research. Site information has been uploaded to the World Data Base Pangaea, metadata of samples have been recorded in the data base ExpeditionDIS, and samples for post-cruise research are stored together with archive halves of the retrieved gravity cores at the MARUM GeoB Core Repository. The recovered number of high quality samples constitutes an excellent basis for the planned scientific studies in the framework of the DARCLIFE project ('*Deep subsurface archaea: carbon cycle, life strategies, and role in sedimentary ecosystems*'; 04/2010 – 3/2015) in which the cruise participants collaborate. DARCLIFE is led by Prof. Kai-Uwe Hinrichs at the University of Bremen and funded through an Advanced Grant of the European Research Council.

FS POSEIDON Fahrt POS450 „DARCSEAS II“ (2.-13. April, 2013) wurde im westlichen Mittelmeer durchgeführt, um die Wechselwirkungen zwischen marinen Mikroorganismen und terrestrischem organischem Material zu erforschen. Dazu untersuchte die Expedition den Einflussbereich von zwei großen Flüssen, die sich in Hinblick auf Klima und hydrographisches Regime ihres Einzugsgebiets unterscheiden: die Mündungen der Rhône in Frankreich und des Moulouya in Marokko. Für geochemische und mikrobiologische Untersuchungen wurden Wassersäule und Sedimente am Meeresboden entlang einer Serie von Stationen beprobt, die dem Eintrag terrestrischen organischen Materials folgen. Nach der Expedition werden diese Proben an Land untersucht, um die Faktoren zu ermitteln, die die Verteilung benthischer Archaeen in marinen Sedimenten beeinflussen. Unsere Forschung zielt letztendlich darauf ab, die Rolle benthischer Archaeen im marinen Kohlenstoffkreislauf zu verstehen. Zur Probenahme wurden vier Geräte eingesetzt: Schwerelot, Multicorer, CTD/rosette und In-situ-Pumpen. Der Schwerpunkt unserer Arbeiten lag auf der

sachgerechten Gewinnung und Konservierung von Proben für die geplanten geochemischen und mikrobiologischen Analysen und Experimente, die an Land mit einer Vielzahl aufwändiger analytischer Methoden durchgeführt werden sollen. Aus Zeitgründen konnten nur wenige sedimentologische und geochemische Untersuchungen direkt an Bord durchgeführt werden. Auf *FS POSEIDON* Fahrt POS450 wurden innerhalb von knapp 11 Tagen 1118 Seemeilen zurückgelegt, 106 Stunden auf Station gearbeitet, 15 neue GeoB Stationen etabliert (GeoB17301 – GeoB17315), und mehr als 4000 einzelne Proben für detaillierte Untersuchungen an Land genommen. Die Stationsdaten wurden in der World Data Base Pangaea hinterlegt. Proben werden zusammen mit Archivhälften der Schwerelotkerne im MARUM GeoB Kernlager aufbewahrt und Informationen zu den Proben wurden in der Datenbank ExpeditionDIS aufgezeichnet. Die gewonnenen hochwertigen Proben bilden eine exzellente Grundlage für die geplanten wissenschaftlichen Untersuchungen im Rahmen des DARCLIFE Projektes ('*Deep subsurface archaea: carbon cycle, life strategies, and role in sedimentary ecosystems*'; April 2010 bis März 2015), in dem die Fahrtteilnehmer an der Universität Bremen zusammenarbeiten. DARCLIFE wird von Prof. Kai-Uwe Hinrichs an der Universität Bremen geleitet und durch ein Advanced Grant des European Research Council gefördert.

2 Participants

Table 1: List of scientific party

Name	Discipline	Institution
Heuer, Verena, Dr.	Chief scientist	GeoB / MARUM
Aiello, Ivano, Dr.	Sedimentology	MLML
Elvert, Marcus, Dr.	Organic geochemistry	GeoB / MARUM
Goldenstein, Nadine, PhD-student	Organic geochemistry	GeoB / MARUM
Goldhammer, Tobias, Dr.	Inorganic geochemistry	GeoB / MARUM
Könneke, Martin, Dr.	Microbiology	GeoB / MARUM
Liu, Xiaolei, Dr.	Organic geochemistry	GeoB / MARUM
Pape, Silvana, technician	Inorganic geochemistry	GeoB / MARUM
Schmidt, Frauke, Dr.	Organic geochemistry	GeoB / MARUM
Wendt, Jenny, technician	Organic geochemistry	GeoB / MARUM
Zhuang, Guangchao, PhD-student	Organic geochemistry	GeoB / MARUM

Participating institutions

GeoB	Dept. of Geosciences, Bremen University Klagenfurter Str., D 28359 Bremen, Germany	www.geo.uni-bremen.de
MARUM	Centre for Marine Environmental Sciences Leobener Str., D 28359 Bremen, Germany	www.marum.de
MLML	Moss Landing Marine Laboratories 8272 Moss Landing Rd, Moss Landing, CA 95039, USA	www.mlml.calstate.edu

Fig. 1: Scientific party of *RV POSEIDON* cruise POS450

3 Research Program

(V. Heuer)

RV POSEIDON cruise POS450 DARCSEAS II (April 2-13, 2013) recovered water samples, suspended particulate matter, and sediment cores from fifteen sites in the Western Mediterranean Sea (Fig. 2) in order to study relationships between marine microbial communities and the riverine input of terrestrial organic matter. The expedition targeted two contrasting river systems, i.e. the River Rhône off France and the River Moulouya off Morocco with temperate and Mediterranean climatic and hydrologic regime, respectively, in their hinterlands. DARCSEAS II is the second expedition of the DARCLIFE project ('*Deep subsurface archaea: carbon cycle, life strategies, and role in sedimentary ecosystems*'; April 2010 – March 2015) in which the cruise participants collaborate. DARCLIFE is led by Prof. Kai-Uwe Hinrichs at the University of Bremen (Department of Geosciences/MARUM Center for Marine Environmental Sciences) and funded through an Advanced Grant of the European Research Council.

Our research program was dedicated to the investigation of Archaea, which constitute a poorly understood domain of life. Archaea have long been considered exotics that only occur in extreme environments like hot springs and salt lakes, but are nowadays increasingly recognized as globally abundant organisms that mediate processes which are important for the emission of greenhouse gases and cycling of nutrients. In sediments below the seafloor, the so-called *benthic archaea* have a cosmopolitan distribution and make up a significant portion of life (e.g. Biddle et al., 2006; Lipp et al., 2008). *Benthic archaea* comprise numerous novel, uncultured phy-

logenetic lineages. The large abundance of archaea in the deep subseafloor might be related to their unique ability to cope with extreme energy starvation (Valentine, 2007), and their presumed ability to degrade complex recalcitrant organic residues highlights their relevance for the carbon cycle and as potential targets for biotechnology. In the DARCLIFE project, a team of about 20 scientists studies *benthic archaea*, their carbon cycle and life strategies in the subseafloor. In particular, we seek to understand the geochemical and (paleo)environmental factors that control the distribution, composition and processes of subseafloor microbial communities. Central to our research strategy is the information encoded in structural and isotopic properties of sedimentary membrane lipids from *benthic archaea*. With additional metagenomic analysis we aim to establish a phylogenetic framework and to gain insights on potential metabolic pathways. With in-depth geochemical examinations of the habitat we seek to elucidate processes that are mediated by *benthic archaea* in situ. In order to develop the full potential of lipids as proxies for studying nearly inaccessible microbial life, we grow model archaea under a set of environmental conditions and examine the impact of environmental parameters on cellular lipid distributions.

Fig. 2: Working area with sampling sites GeoB17301 - GeoB17315 (Map: Google Earth, Data SIO, NOAA, US Navy, NGA, GEBCO, Image Landsat)

Our research approach involves the examination of *benthic archaea* in a global range of diverse sedimentary environments. After investigating strongly contrasting biogeochemical and depositional environments in the Eastern Mediterranean Sea, Marmara Sea, and Black Sea with the first DARCSEAS expedition (*RV Meteor* cruise M84/1, February 9-22, 2011), the research program of DARCSEAS II was tailored to study the relationship between *benthic archaea* and the quality and quantity of the small fraction of organic matter that is transferred into the deep subseafloor. More specifically, we took samples that will help us to better understand how *benthic ar-*

chaea react to the riverine input of terrigenous organic carbon. Approximately 0.38 Gt of organic carbon are transported to the oceans by rivers every year, about 55% in dissolved (DOC) and 45% in particulate form (POC) (Ludwig et al., 1996). Most of the terrestrial organic carbon delivered to the ocean is derived from soil organic matter and a large fraction (~80% or more) of the riverine suspended matter is deposited in continental margin sediments (Hedges et al., 1997; Burdige, 2005). Because riverine organic matter is highly degraded and nitrogen-poor, it might be expected to suffer minimal respiration in the ocean. However, the opposite is true: only a small fraction of the preserved organic matter in sediments is land derived, suggesting that terrestrial organic matter is extensively remineralized at sea (Hedges et al., 1997; Burdige 2005). With DARCSEAS II we aim to explore the role of *benthic archaea* in this process. In our research program, retrieval and preservation of high quality sediment samples had the highest priority, but our approach included intensive probing of the water column as well. The characterization of input is essential and thus we took samples to generate a comprehensive set of geochemical and microbiological data for both the water column and the underlying sediments at each site.

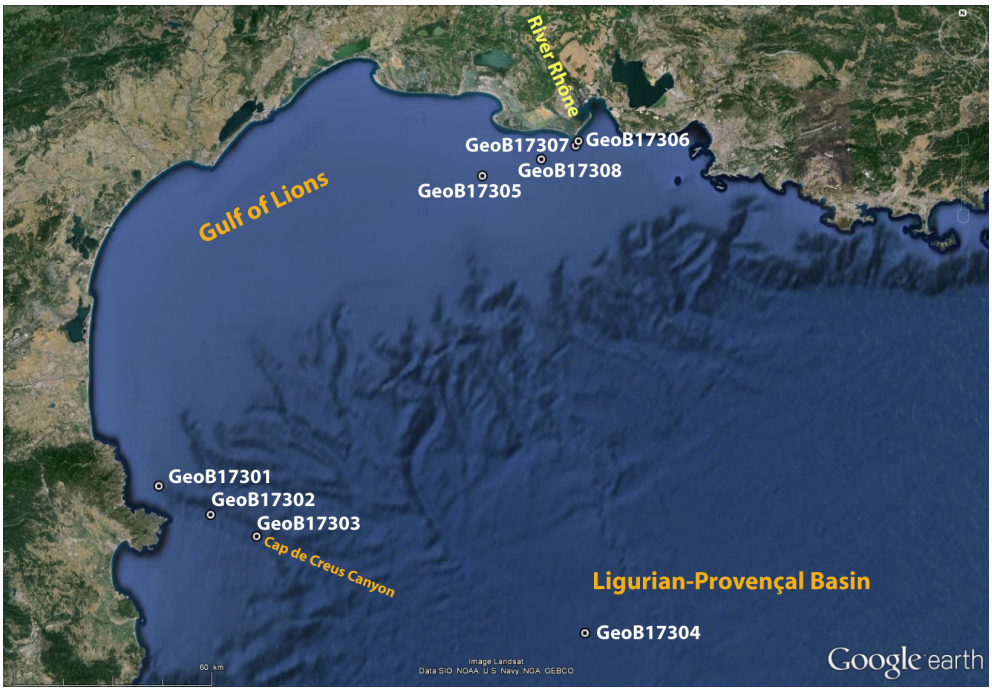

Fig. 3: Sites GeoB17301 - GeoB17308 in the Gulf of Lions. (Map: Google Earth, Data SIO, NOAA, US Navy, NGA, GEBCO, Image Landsat)

In the Gulf of Lions, four sites were sampled to track the terrigenous input of the River Rhône to the shelf (Fig. 3): sites GeoB17306 and GeoB17307 represent the pro-delta of the River Rhône, while sites GeoB17308 and GeoB17305 are located 6.3 NM and 16 NM, respectively, off the river mouth (Fig. 4). Site GeoB17305 is a typical mud-belt site. In addition, four sites were sampled in the Cap de Creus Canyon and Ligurian-Provençal Basin to investigate the transport of sediments from the shelf to the deep sea (Fig. 3): GeoB17301 – GeoB17304. Site GeoB17304 is located on a sedimentary body created the Petit-Rhone deep sea turbiditic channel.

Fig. 4: Mouth of the River Rhône in the Gulf of Lions, Sites GeoB17305 - GeoB17308. (Map: Google Earth, Data SIO, NOAA, US Navy, NGA, GEBCO, Image Landsat)

On the shelf and slope offshore the Moulouya River, we established and investigated GeoB Sites 17309 – 17314 (Fig. 5). During our transit to Malaga, a final site (GeoB17315) was sampled in the Alborán Sea as a marine end-member (Fig. 2).

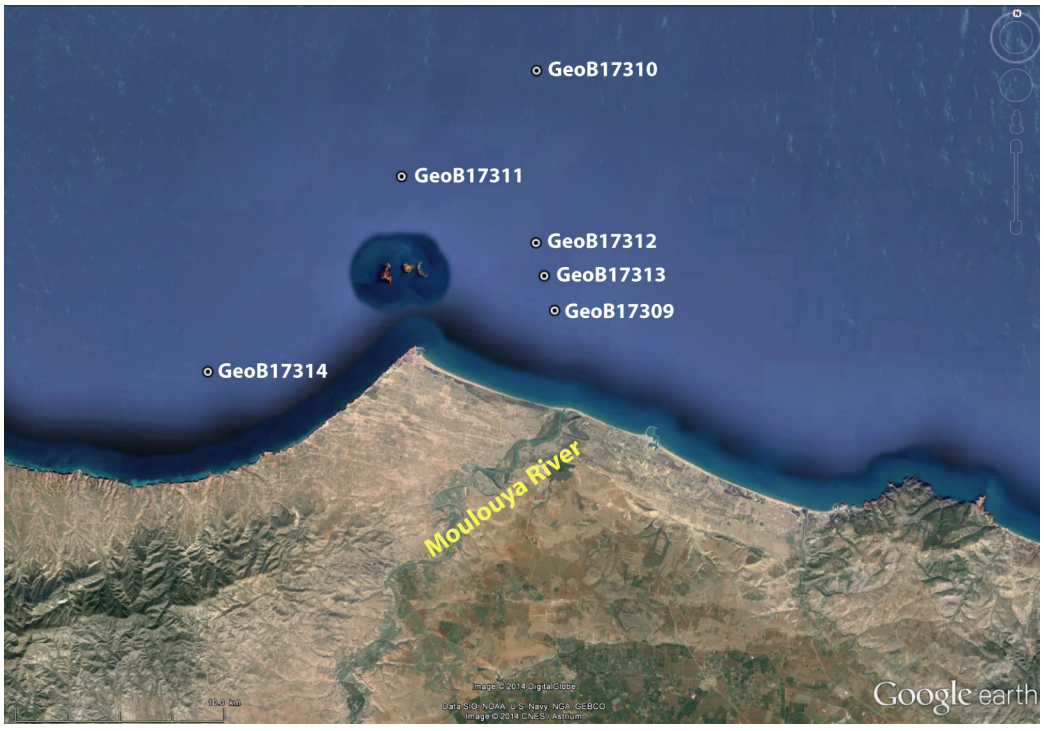

Fig. 5: Mouth of the Moulouya River off Morocco, Sites GeoB17309 - GeoB17314. (Map: Google Earth, Data SIO, NOAA, US Navy, NGA, GEBCO, Image Landsat)

4 Narrative of the Cruise

(V. Heuer)

RV POSEIDON cruise POS450 started as scheduled on April 2, 2013, at 13:30 local time (LT) with leaving the port of Barcelona (Spain) in good weather conditions. At this point, we had not yet received permission from the authorities to work in French waters. For this reason, we changed our cruise track and started operations not in the Rhône Delta (France) but in the Cap de Creus Canyon (Spain). After 11.5 hours of transit, our research program began on April 3, 01:00 LT at 42°24.7'N, 3°17.7'E. In the following three days, we established site GeoB17301 (shelf next to the head of the canyon), GeoB17302 (midslope), GeoB17303 (downslope), and GeoB17304 (deep water site off the Cap de Creus Canyon) (Fig. 3). Because weather conditions turned increasingly difficult, tool deployment from the open work deck became too risky and operations in this area had to be stopped on April 5 at 12:15 LT, before our working program was completed. We could not sample surface sediments by MUC-deployment at Site GeoB17304. In the meantime, we had received permission to work in French waters. Following a 12-hour transit from Site GeoB17304 to the shelf offshore the River Rhône, we started our scientific program in French waters on April 6, 00:00 LT, and investigated site GeoB17305 in the mud belt of the Gulf of Lions, sites GeoB17306 and GeoB17307 close to the river mouth, and site GeoB17308 at an intermediate position between GeoB17307 and GeoB17305 (Fig. 4). On April 7, 15:30 LT, we finished our research program in French waters and began a 69-hour transit to the outlet of the Moulouya River. During transit, all sediment cores were opened and described, sampling plans to address our research questions were carefully discussed and coordinated by the scientific party, and the cores were sampled and processed in the highest possible depth resolution. All samples were carefully preserved for shore-based investigations. We reached Moroccan waters (EEZ) on April 10 at 07:38 LT, entered Moroccan territory at 09:41 LT, and started scientific operations at site GeoB17309 at 12:00 LT. In the following 1.5 days, we investigated five sites on the shelf and slope offshore the Moulouya River and established GeoB Sites 17309 – 17314 (Fig. 5). Since only little information was available about this working area prior to the cruise, investigations were explorative in nature. In some cases, deployments of the multi corer did not yield sediment samples because the seabed was too consolidated. The only successful deployment of the gravity corer penetrated just the upper 74 cm of the seafloor at Site GeoB17309. When two further attempts were undertaken at sites GeoB17310 and GeoB17311, where MUC deployments had successfully recovered sediments, the gravity corer was bent irreversibly and could no longer be deployed. During operations, we added new sites for sediment coring to our program. Due to time constraints, the water column could not be sampled at the last added site GeoB17314, where sediment recovery was most successful. Our shipboard scientific exploration of the riverine input of the Moulouya was finished and the transit to our final site in the Alborán Sea started on April 11, 17:45 LT. We left Moroccan territory on April 12, 01:00 LT and the Moroccan EEZ on April 12, 01:42 LT. On April 12 at 02:24 LT, we reached site GeoB17315 (Fig. 2). Our

scientific program was completed at 35° 43.4' N, 3° 12.3' W on April 12, at 10:00 LT and *RV POSEIDON* cruise POS450 ended in the port of Malaga on April 13, 2013, at 08:30 LT. Within less than 11 days, the cruise covered 1118 NM, operated 106 hours on site, and established 15 new GeoB sites (GeoB17301 – GeoB17315). Our sampling program was very successful in spite of problems with our coring tools. The multicorer suffered from a technical defect and only 40% of the deployments were successful. In addition, we did not have enough spare parts to repair the gravity corer after it had been bent irreversibly. From a total of 60 tool deployments, shipboard scientists retrieved and preserved more than 4000 discrete samples for post-cruise research that were shipped together with 23.9 meters of core to the MARUM GeoB Core Repository at the University of Bremen. Post-cruise, metadata of all samples were recorded in the data base ExpeditionDIS, and site information were uploaded to the World Data Base Pangaea.

5 Sampling Strategy and Shipboard Methods

5.1 Tool Deployment

(V. Heuer)

Our sampling strategy was designed to generate a comprehensive set of geochemical and microbiological data for both the water column and the underlying sediments at each site. The water column was investigated by CTD/rosette deployment. Con ductivity, temperature, pressure, oxygen content and fluorescence were measured continuously during the downcast, and based on this information, up to 12 water samples were collected from selected water depths during the upcast deployment. In addition, suspended particulate matter was sampled from selected corresponding water depths using a tool string equipped with up to four in situ pumps (ISP). Surface sediments were sampled with a multicorer (MUC) that was equipped with 12 cores suited to retrieve the upper meter of sediment with an intact sediment/water interface. Sub-seafloor sediments were sampled with a gravity corer (GC) set for 6.5 m long cores. When several sites were located in very close proximity, like GeoB17306 and GeoB17307 or GeoB17312 and GeoB17313, the water column was only sampled at one location. For safety reasons, GC and MUC had to be deployed during day shift. During night shift we operated CTD/rosette and ISP. Whenever possible, the water column was sampled before sediment cores were taken. In a few cases sediment cores had to be taken first in order to meet the strict time schedule of the crew and cruise. In these cases, the position of the vessel was slightly changed to avoid contamination of water samples with sediments released during core retrieval. An overview on tool deployments is given in Table 2.

Table 2: Overview on sampling strategy and tool deployments during POS450.

Site	Coordinates	Water depth	Description of Site	CTD	ISP	MUC	GC
GeoB 17301	42° 24.75' N, 3° 17.70' E	115 m	Cap de Creus Canyon, shelf area next to canyon head	yes	yes	yes failed	yes recovery: 30 cm
GeoB 17302	42° 20.00' N, 3° 29.00' E	746 m	Cap de Creus Canyon, midslope	yes	yes	yes recovery: 25 cm	yes recovery: 121 cm, 186 cm
GeoB 17303	42° 16.38' N, 3° 39.02' E	1146 m	Cap de Creus Canyon, downslope	yes	yes	no	yes failed
GeoB 17304	41° 59.41' N, 4° 50.13' E	2291 m	Deep water site off the Cap de Creus Canyon in the Petit-Rhône Neofan	yes	yes	no	yes recovery: 234 cm
GeoB 17305	43° 13.80' N, 4° 30.61' E	61 m	Gulf of Lions, marine end member in the mud belt off the River Rhône Delta	yes	yes	yes recovery: 40 cm	yes recovery: 229 cm
GeoB 17306	43° 18.96' N, 4° 52.17' E	30 m	Gulf of Lions, terrestrial end member in prodelta of the River Rhône	yes	yes	yes recovery: 50 cm	yes recovery: 516 cm
GeoB 17307	43° 18.24' N, 4° 51.53' E	52m	Gulf of Lions, prodelta of the River Rhône	yes	yes	yes recovery: 35 cm	yes recovery: 492 cm
GeoB 17308	43° 16.20' N, 4° 43.79' E	62 m	Gulf of Lions, intermediate position between prodelta and mud belt	yes	yes	yes recovery: 35 cm	yes recovery: 405 cm
GeoB 17309	35° 10.00' N, 2° 21.02' W	16 m	Shelf offshore the Moulouya River, shallow water depth, assumed riverine end member	yes	no	no	yes recovery: 74 cm
GeoB 17310	35° 16.23' N, 2° 21.94' W	161 m	Slope offshore the Moulouya River, deep water, assumed marine end member	yes	yes	yes recovery: 20 cm	yes recovery: 139 cm
GeoB 17311	35° 13.31' N, 2° 26.09' W	88 m	Shelf offshore the Moulouya River, at the end of the river plume	yes	yes	yes recovery: 40 cm	yes failed
GeoB 17312	35° 11.74' N, 2° 21.71' W	52 m	Shelf offshore the Moulouya River, in the middle of the river plume	no	no	yes failed	no
GeoB 17313	35° 10.89' N, 2° 21.40' W	28 m	Shelf offshore the Moulouya River, in the middle of the river plume	yes	yes	yes failed	no
GeoB 17314	35° 7.99' N, 2° 31.98' W	66 m	Shelf offshore the Moulouya River, west of Chafarinas Island	no	no	yes recovery: 35 cm	no
GeoB 17315	35° 43.44' N, 3° 12.34' W	1052 m	Alborán Basin	yes	yes	yes recovery: 40 cm	no

yes = deployed, no = not deployed.

5.2 Sampling of the Water Column with CTD/Rosette and In-Situ-Pumps (ISP)

(N. Goldenstein, V. Heuer, M. Könneke, X. Liu, F. Schmidt)

The water column was investigated by CTD-profiles and sampled by niskin bottles and in-situ pumps. The shipboard rosette contained multiple water collectors (rosette with 12 x 10 l Niskin bottles) and was equipped with a SBE-9 Plus-CTD (provided by MARUM), containing sensors for conductivity, temperature, pressure and oxygen. Additionally, a WETLABS fluorometer and a WETLABS turbidity sensor were attached to the CTD. Sensors were calibrated as described by Zonneveld et al (2013) for cruise POS449.

Six different types of samples were taken from the Niskin bottles immediately after recovery:

- **Sample 1**: 0.5 L of water were passed through solid phase extraction (SPE) cartridges on board and stored at +4°C to preserve samples for shore based molecular analysis of dissolved organic matter (DOM) by Fourier Transform Ion Cyclotron Resonance Mass Spectrometry (FT-ICR-MS);
- **Sample 2**: 7 mL of water were filtered through glass fiber filters (0.2 µm) and stored in glass vials at -20°C for shore based quantitative analysis of dissolved organic carbon (DOC);
- **Sample 3**: 35 mL of water were stored in glass vials at -20°C for shore based quantitative and stable carbon isotopic analysis of organic metabolites;
- **Sample 4**: 10 mL of water were filtered (0.2 µm) and stored in plastic vials at +4°C for shore based analysis of inorganic nutrients;
- **Sample 5**: about 500 mL of water were stored in glass bottles at +4°C for shore-based enrichment of planktonic archaea;
- **Sample 6**: 30 mL of water were chemically fixed and stored at -20°C for shore-based determination of microbial cell concentrations.

A tool string equipped with up to four in-situ pumps (ISP) was deployed in the water column for several hours to filter large volumes of water at selected water depths. Deployment of in-situ pumps (ISP) returned samples of particulate matter on glass fiber and membrane filters through which up to 771 liters of sea water had been pumped. In order to separate different size fractions, the following filters were combined: GF/D (Whatman, 142 mm Φ, pore size ~2.7 µm), GF/F (Whatman, 142 mm Φ, pore size 0.7 µm) and Supor-200 (Pall, 142 mm Φ, pore size 0.2 µm). Samples were stored at -20°C and will be used for shore based molecular and carbon isotopic analysis of microbial membrane lipids.

In total, the water column was investigated and sampled at eleven sites. An overview on recorded CTD-data, water samples taken by Niskin bottles, particulate matter samples filtered by ISP deployment and preserved subsamples is given in Table 3.

Table 3: Overview on samples taken from the water column during POS450. For more details see text.

CTD-Cast GeoB	Bottle	Depth (m)	T (°C)	Salinity (PSU)	Fluor-escence (mg/m³)	O_2 (mL/L)	Sample 1	2	3	4	5	6	ISP-Cast GeoB	Pump	Depth (m)	Pumped volume (Liters)
17301-1	4, 5	11	11.6	36.9	1.185	5.77		x	x	x		x	17301-3	Norbert	10	31
17301-1	2, 3	50	12.0	38.0	1.051	4.53	x	x	x	x	x	x	17301-3	Fred	50	583
17301-1	1	96	12.7	38.1	1.038	5.35		x	x	x		x				
17302-1	9, 10	9	13.4	38.2	0.920	5.76		x	x	x		x	17302-2	Norbert	10	70
17302-1	7, 8	50	13.4	38.2	0.950	5.76		x	x	x		x				
17302-1	5, 6	200	13.4	38.4	0.175	5.75		x	x	x		x				
17302-1	3, 4	399	13.3	38.4	0.163	5.77		x	x	x		x				
17302-1	1, 2	592	13.1	38.5	0.083	5.79	x	x	x	x	x	x	17302-2	Fred	600	100
17303-2	10, 11	10	13.4	38.2	2.000	5.76		x	x	x		x	17303-1	Ginger	10	478
17303-2	8, 9	48	13.4	38.4	0.385	5.75		x	x	x		x				
17303-2	6, 7	100	13.3	38.4	0.163	5.76	x	x	x	x	x	x				
17303-2	5 (a)	501	13.1	38.5	0.037	5.79							17303-1	Roger	500	767
17303-2	3, 4	754	13.1	38.5	0.067	5.79		x	x	x		x				
17303-2	1, 2	1054	13.1	38.5	0.083	5.78	x	x	x	x	x	x	17303-1	Fred	1050	666
17304-1	11, 12	9	13.3	38.3	2.242	5.77	x	x	x	x	x	x	17304-2	Ginger	10	419
17304-1	10	50	13.0	38.4	0.163	5.80		x	x	x		x				
17304-1	8, 9	98	13.1	38.4	0.083	5.79		x	x	x		x	17304-2	Roger	100	765
17304-1	7 (a)	500	13.0	38.5	0.052	5.79										
17304-1	5, 6	1000	13.1	38.5	0.052	5.79	x	x	x	x	x	x	17304-2	Norbert	1000	771
17304-1	3, 4	1500	13.1	38.5	0.052	5.78		x	x	x		x				
17304-1	1, 2	1956 (c)	13.2	38.5	0.052	5.77	x	x	x	x	x	x	17304-2	Fred	2200	682
17305-1	3, 4	9	12.4	36.7	1.147	5.93		x	x	x		x				
17305-1	1, 2	50	13.2	38.2	0.337	5.79	x	x	x	x	x	x	17305-2	Ginger	30 (d)	466
17307-7	5, 6	10	13.0	38.0	1.115	5.82		x	x	x		x				
17307-7	3, 4	25	13.1	38.1	0.498	5.79	x	x	x	x	x	x	17307-6	Norbert	25	412
17307-7	1, 2	40	13.1	38.1	0.528	5.79		x	x	x		x				
17308-2	5, 6	10	12.7	37.8	1.115	5.85		x	x	x		x				
17308-2	3, 4	30	13.1	38.0	0.702	5.80	x	x	x	x	x	x	17308-3	Roger	30	537
17308-2	1, 2	50	13.1	38.2	0.401	5.79		x	x	x		x				
17310-3	7, 8	12	15.1	36.9 (b)	1.980	6.65		x	x	x		x	17310-2	Roger	10	8
17310-3	5, 6	70	14.9	37.3 (b)	0.480	6.53	x	x	x	x	x	x	17310-2	Fred	70	478
17310-3	3, 4	80	14.1	37.3 (b)	0.179	6.51		x	x	x		x	17310-2	Norbert	80	505
17310-3	1, 2	141	14.0	37.9 (b)	0.480	6.39		x	x	x		x	17310-2	Ginger	135	459
17311-3	5, 6	12	15.8	36.9	1.067	5.53		x	x	x		x	17311-4	Roger	10	12
17311-3	3, 4	32	15.4	36.9	1.321	5.57	x	x	x	x	x	x	17311-4	Norbert	30	161
17311-3	1, 2	61	14.6	37.1	0.560	5.65		x	x	x		x	17311-4	Ginger	60	418
17313-1	3, 4	11	15.8	36.8	0.700	5.53		x	x	x		x	17313-2	Roger	18	38
17313-1	1, 2	20	15.8	36.8	0.750	5.53	x	x	x	x	x	x	17313-2	Norbert	10	226
17315-1	10, 11, 12	11	15.8	36.7	0.900	5.53	x	x	x	x	x	x	17315-2	Fred	10	558
17315-1	7, 8, 9	100	14.6	37.0	0.163	5.66	x	x	x	x	x	x	17315-2	Roger	100	765
17315-1	4, 5, 6	495	13.1	38.5	0.036	5.78	x	x	x	x	x	x	17315-2	Norbert	500	771
17315-1	1, 2, 3	1002	13.1	38.5	0.052	5.79	x	x	x	x	x	x	17315-2	Ginger	1000	523

x = sample taken, (a) deployment failed; (b) salinity recorded during corresponding CTD-cast GeoB17310-4; (c) communication error during deployment; CTD/rosette and ISP samples were taken from different depths; (d) only one average depth sampled.

5.3 Sampling of Sediments with Gravity Corer and Multi Corer (MUC)

(I. Aiello, M. Elvert, N. Goldenstein, T. Goldhammer, V. Heuer, M. Könneke, X. Liu, S. Pape, F. Schmidt, J. Wendt, G. Zhuang)

Gravity cores were processed in the following way: (1) Immediately after recovery, the cores were cut into one meter long sections, sampled for gas analysis at the freshly cut section ends, sealed with end caps, measured for curatorial purposes, labeled and moved to a refrigerated container (+7°C) for storage or to the laboratory (+18°C) for processing. In general, the initial processing of gravity cores was finished within less than one hour. (2) Next, anaerobic pore-water samples were withdrawn from the closed sections of the gravity cores for analyses of water-soluble inorganic and organic compounds. Samples were taken with rhizon suction samplers and evacuated syringes. The rhizon micro suction samplers (5 cm length, 0.15 µm porous polymer) were inserted into the sediment through small holes which were drilled into the core liner every 20-25 cm, following a pre-defined sampling scheme for all cores. After an initial pore-water sample had been taken for the analysis of oxygen-sensitive ferrous iron, rhizons were allowed to collect more pore-water for up to 1-2 hours. The resulting pore-water samples were split and preserved for the shore-based analysis of nutrients, cations, anions, volatile fatty acids, and alcohols. (3) Following rhizon sampling, small sampling ports were cut into the closed core liners for additional anaerobic probing of fresh sediment. Sampling ports were located adjacent to the rhizon sampling ports, but a distance of ~5 cm was kept in order to avoid artifacts from the pore-water sampling process. For gas analysis, a set of four to five individual samples was collected for (a) shipboard and (b) shore based analysis of dissolved hydrogen, (c) shipboard quantitative and (d) shore based stable carbon isotopic analysis of hydrocarbon gases, and (e) shore-based analysis of methyl substrates. (4) When the gas-sensitive sampling was finished, core sections were cut lengthwise into halves. The working half was used for visual core description and petrographic analysis of smear slides. Based on the lithological core description and shipboard gas analysis, depth intervals were identified and flagged for an intense, closely coordinated geochemical and microbiological sampling program. Particular attention was paid to resolving contrasting geochemical or lithological intervals. (5) The archive half was used for non-destructive extraction of additional pore-water samples for shore-based molecular analysis of DOM by FT-ICR-MS (using rhizon micro suction samplers) and for non-destructive conductivity measurements. (6) The work half was used for solid phase sampling. Samples were taken in high resolution for cell counts, grain size distribution, and elemental and isotopic analysis of bulk sedimentary carbon and nitrogen. Based on lithological description, samples were taken in a lower resolution for metagenomic analysis of microbial diversity, structural and isotopic analysis of intact polar lipids (IPLs), which are the major building blocks of microbial cell membranes, and functional gene analysis. In addition, large volumes of solid phase (~250 mL) were sampled from selected depths and preserved under nitrogen atmosphere at +4°C to +7°C for shore-based incubation experiments. (7) This stand-

ard program was supplemented by additional sampling at selected sites. In particular, solid phase samples were taken at Sites GeoB17306, GeoB17308, and GeoB17314 for shore-based measurement of methanogenesis rates. An overview on the retrieved samples is given in Table 4. In general, all sampling was finished within less than 24 hours after core retrieval. After core description and sampling, both core halves were covered with foil and stored in a refrigerated container (+7°C) until they were shipped to the MARUM GeoB Core Repository at the University of Bremen at -20°C.

Table 4: Overview on samples taken from sediment cores during POS450. For more details see text.

Site & Event	Gear	Gas chemistry	Pore-water chemistry	DOM (FT-ICR-MS)	TOC	Cell counts	Meta-genomics	IPLs	Incubation experiments, enrichment	Rate measure ments
GeoB										
17301-7	GC	28	-	-	3	3	-	6	-	-
17302-3	MUC	8	98	1	14	7	-	9	2 + 1	-
17302-4	GC	8	35	-	4	-	-	1	-	-
17302-5	GC	12	48	-	14	12	3	7	3	-
17304-3	GC	18	180	4	55	43	25	52	4 + 2	-
17305-3	MUC	45	180	-	9	9	3	12	3 + 1	-
17305-5	GC	28	81	-	30	23	-	5	1	-
17306-1	MUC	60	190	-	12	12	3	15	6 + 1	-
17306-2	GC	100	262	8	76	50	25	18	8	160
17307-5	MUC	24	150	-	8	8	3	12	6 + 1	-
17307-8	GC	40	180	5	63	48	5	16	-	-
17308-1	MUC	50	160	-	9	9	2	12	4 + 1	-
17308-4	GC	20	144	3	62	41	11	14	4	144
17309-1	GC	45	27	3	27	23	3	17	1	-
17310-1	MUC	84	117	-	12	-	-	9	2 + 1	-
17310-5	GC	16	45	2	19	14	2	10	6	-
17311-2	MUC	30	170	-	9	9	2	12	2 + 1	-
17314-1	MUC	15	170	-	9	9	5	12	3 + 1	80
17315-3	MUC	45	-	-	9	-	-	21	1	-
Total		766	2237	26	444	320	92	260	66	384

The 12 MUC cores of each deployment were labeled A-M and in general distributed in the following way: Cores A and B were equipped with pre-drilled ports for anaerobic rhizon sampling of pore-waters from intact sediment cores and were used for inorganic pore-water analysis and quantitative and molecular analysis of DOM, respectively. Core C was used for lithological core description. Core D was sampled for gas analysis, determination of microbial cell concentrations (cell counts), metagenomic analysis of microbial diversity, structural and isotopic analysis of sedimentary membrane lipids, and elemental and isotopic analysis of bulk sedimentary carbon and nitrogen. The remaining cores were used to preserve live sediments for shore-based incubation experiments. Pore-water samples for inorganic and organic analysis were rhizoned from the intact sediment cores A and B in the laboratory, where cores were kept upright in the sink. Rhizon micro suction samplers (5 cm length, 0.15 µm porous polymer) were carefully inserted through sampling holes every centimeter in the upper 10 and every 2 to 4 cm down to the bottom. All other

cores were sampled immediately after recovery on the work-deck. Samples were taken in 0.5-5 cm intervals. During processing, air temperatures (around 15-20°C) were only slightly higher than in situ water temperatures (around 10-15°C). An overview on the obtained samples is given in Table 4.

5.4 Sampling of Sediment Cores for Gas Analysis

(M. Elvert, V. Heuer, J. Wendt, G. Zhuang)

5.4.1 Introduction

During *RV POSEIDON* cruise POS450 gas samples were taken from sediment cores for shipboard and shore based analysis with a focus on three types of volatile compounds: (a) CH_4, (b) molecular hydrogen (H_2), and (c) methylated substrates, e.g. methyl amines, methyl sulfides or methanol. In total, 766 samples were taken from sediment cores (Table 4). The water column was not sampled for gas analysis.

Direct ship-board analysis of CH_4 helped to rapidly identify the sulfate-methane transition zone (SMTZ), thus guiding the subsequent sampling strategy for research projects focusing on this important geochemical zone. Shore-based analysis of the stable carbon and hydrogen isotopic composition of CH_4 will help to identify methane sources and sinks (Whiticar, 1999).

Molecular hydrogen is a key metabolite in anoxic environments. It is produced and consumed by a wide variety of microorganisms during the decomposition of organic matter. Concentrations of H_2 in sediments are thought to reflect the predominant terminal electron accepting processes and bioenergetics of the microbial ecosystem *in situ* (e.g., Hoehler et al., 1998).

Methylated carbon substrates are one of the less understood volatile carbon components in marine sediments. They are non-competitive substrates for methanogens in sulfate-rich environments and previous studies demonstrated their presence in seafloor brine pools (Joye et al., 2009) and subseafloor sediments (Yoshioka et al., 2010). Because *in situ* concentrations and production and consumption pathways of methylated compounds remain unclear, we aim to carefully investigate concentrations and, if possible, carbon isotopic compositions in sediment material obtained during POS450. Moreover, sediments with high organic matter contents such as those found in the Rhône Delta will serve for a comprehensive shore-based radiotracer-based study identifying active methyl-to-methane converting reactions.

5.4.2 Sampling Procedures

The multi corer (MUC) cores were processed on deck immediately after core retrieval. The sediment was extruded from the core by measured 0.5-5 cm increments and the freshly exposed sediment surface was sampled. For gas analyses, a set of subsamples of 2-10 mL sediment was collected by cut-off plastic syringes, transferred to gas-tight vials and sealed according to the procedures described below. From gravity cores, syringe samples for gas analysis were first taken at the 1 m cut

ends of the cores on the work deck of *RV POSEIDON*. In order to improve depth resolution, additional samples were taken from the intact segments via window ports right after the core sections were moved into the laboratory. The window sampling ports (ca. 3 cm-wide bands) were cut into the core liner and syringe samples were retrieved from the freshly exposed sediments.

5.4.3 Gas Analysis

CH_4 – Concentrations of dissolved methane were determined on board *RV POSEIDON* according to previously reported protocols (Kvenvolden and McDonald, 1986; D'Hondt et al., 2003): 2-3 mL of wet sediment were enclosed in a gas-tight 22-ml glass vial with a Teflon septum and heated for 20 min at 60°C. After heating, 100-500 µL sub-samples were taken from the headspace gas with a gas-tight syringe and analyzed on board by gas chromatography-flame ionization detection (GC-FID). The GC-FID was calibrated on a daily basis using hydrocarbon gas standards (Scotty). Based on the partial pressure of the gases in the headspace and the headspace volume, the total amount of released hydrocarbon gases can be quantified and normalized to the pore-water volume of the extracted sediment sample, using the volume and porosity of the solid phase sample. The latter was measured on shore. Additional subsamples were stored in gas-tight 22-ml glass vials with 5 mL NaOH at +4°C for shore-based stable carbon and hydrogen isotope analysis of the hydrocarbon gases.

Molecular H_2 – Concentrations of dissolved H_2 were determined using two different protocols, an extraction-based direct method described in Lin et al. (2012), and the headspace equilibration technique published in Hoehler et al. (1998). To prepare samples for the extraction-based method, a sediment sample of 4-6 mL was extruded into a 22-mL headspace vial, which was immediately filled with NaCl solution (35%) to the top, sealed with a thin gray chlorobutyl stopper (VWR International LLC.), and crimp capped. In the vial, a headspace was created by displacing 5-7 mL of the aqueous phase with an equal volume of H_2-free N_2 gas (the bypass gas out of the H_2 analyzer, Peak Performer I). Once the headspace reached the intended volume, the gas-in needle was removed first, and the overpressure in the vial was allowed to escape through the liquid-out needle. The expelled liquid was collected in a syringe and the volume which corresponds to the generated headspace was recorded. The vial was then vortexed, turned upside-down, and allowed to sit for 20 min at room temperature to let H_2 diffuse out of the interstitial water and equilibrate with the headspace. For H_2 analysis, the headspace gas was displaced into a N_2-flushed plastic syringe by injecting into the vial the same volume of NaCl solution. The concentration of H_2 in headspace gases was analyzed on board by gas chromatography with mercury oxide detection using a Peak Performer 1 (Peak Laboratories, LLC, USA). The instrument was calibrated with a 10 ppm H_2 primary standard (Air Liquide, Germany) on a daily basis. To prepare samples for the headspace equilibration technique, a sediment sample of 2-3 mL was extruded into a 12-mL headspace vial, immediately sealed with a thick black butyl stopper, crimp

capped, and flushed with N_2 (purity = 99.999%) for at least 1 min. Samples were stored at 4°C for shore-based incubation and analysis. On shore, samples will be incubated at in situ temperatures and H_2 concentrations in the headspace gas will be analyzed every 1-3 days until a steady-state concentration is reached. To avoid evacuating the headspace by repeated removal of headspace gas, 1 mL of H_2-free N_2 will be injected into the headspace immediately after the removal of headspace gas to ensure a constant gas pressure.

Methylated carbon substrates – For shore-based analysis of methylated carbon substrates, ~10 mL of sediment were transferred into 40 mL glass vials, which were sealed with Teflon septa and immediately stored at -20°C.

5.5 Sampling of Sediment Cores for Pore-Water Analysis

(T. Goldhammer, V. Heuer, F. Schmidt, S. Pape, G. Zhuang)

5.5.1 Introduction

The investigation of sediment pore-waters is essential to better understand how environmental factors affect the distribution, composition and processes of subseafloor microbial communities. Pore-water constituents that serve as nutrients, electron-acceptors, central intermediates or terminal products in metabolic processes reflect biogeochemical processes that drive geosphere-biosphere interactions. The main objectives of the pore-water sampling program were the acquisition of detailed down core profiles of (1) inorganic pore-water constituents to describe the sedimentary chemical environment with respect to redox zonation and mineralization processes, (2) low-molecular-weight organic compounds that act as central intermediates or terminal products in metabolic processes, and (2) molecular composition of DOM to gain information on the preservation and remineralization of organic matter. Each objective required a separate sampling protocol but sampling was closely coordinated in order to generate a comprehensive data set (see *5.3 Sampling of Sediments with Gravity Corer and Multi Corer [MUC]*). In total, 2263 individual samples were taken for pore-water analysis from sediment cores at all sites (Table 4). In addition, water column samples recovered by CTD/rosette were preserved for shore-based analysis of inorganic and organic compounds (Table 3, see *5.2 Sampling of the Water Column with CTD/Rosette and In-Situ-Pumps [ISP]*).

5.5.2 Sampling Procedures

Surface sediments were retrieved by MUC and cores for pore-water sampling were brought to the laboratory and kept upright in the sink. Pore-water samples were taken by rhizon micro suction samplers (5 cm length, 0.15 µm porous polymer) that had been cleaned by flushing with at least 30 mL of dilute hydrochloric acid (pH ~1-2) and subsequent rinsing with at least 30 mL Milli-Q water. In MUC cores, rhizons were carefully inserted through sampling holes every centimeter in the upper 10 cm and every 2 to 4 cm down to the bottom. The gravity cores were cut into sections, which were brought to the laboratory and horizontally secured on the table. Sampling holes

were drilled into the liner every 25 cm and rhizon micro suction samplers were inserted. Three way Luer-lock stopcocks were connected to the adapters of the rhizons for proper closing and simultaneous connection of two syringes. First, a 10 mL syringe was attached, evacuated and kept open with a spacer. After the first 0.5 mL was sampled, it was discarded through the stopcock and vacuum was reapplied. After 1 mL was sampled, the pore-water was transferred into 1 mL disposable syringes using the three-way stopcock, sealed with a cap, and instantly analyzed for dissolved ferrous iron (Fe^{2+}). The syringes were then left connected to the rhizons until about 8 mL of pore-water had been retrieved, which took about 10 – 30 min for multi cores, and up to 2 h for gravity cores.

For DOM analysis 20 to 50 ml of pore-waters were extracted with rhizons from selected sediment depth intervals of MUC and gravity cores. MUC cores were sampled through sampling holes, whereas gravity cores were sampled after core splitting. Pore waters were stored in pre-combusted 50 ml glass vials at +4°C in the dark until further onboard processing. For the DOC concentration measurements subsamples of 1 ml were stored in pre-combusted glass vials at -20°C in the dark.

5.5.3 Inorganic Pore-Water Chemistry

Objectives - The focus of the Inorganic Geochemistry Group was the geochemical characterization of the diverse benthic and sedimentary environments encountered at the different stations. Our aim was to retrieve detailed chemical information on signature compounds that are sensitive to microbial turnover as well as on species that are considered non-reactive and suitable as conservative tracers of transport processes. Our approach embraces nutrient chemistry, (trace) metal chemistry, standard anion and cation analyses.

Onboard analysis of dissolved iron – Ferrous iron (Fe^{2+}) was detected by spectral photometry (Hach Lange DR3000 photometer) at a wavelength of 565 nm. An iron sensitive color complex was formed by adding 1 mL of plain sample to 50 µL of a ferrospectral reagent in disposable polystyrene cuvettes. In case of high iron concentrations, the original sample was diluted with oxygen free pure water to match the respective calibration range.

Onboard sample processing – Besides the aliquots used for onboard Fe^{2+} analysis, we collected the following sample splits for onshore laboratory analyses: 2mL of plain sample for ammonium (NH_4^+) and phosphate (PO_4^{3-}) determination (flow injection conductivity and spectral photometry), 50 µL of sample diluted with 1950 µL H_2O for anion analysis (ion chromatography), 1 mL of sample diluted with 9 mL of 1% HNO_3 for cation and element analysis (inductively coupled plasma optical emission spectrometry), 1.8 mL of sample preserved with zinc acetate solution for DIC analysis (infrared spectrometry), 0.5–2 mL of sample preserved with zinc acetate solution for sulfide (HS^-) analysis (spectral photometry). The remainder of the original sample was kept without addition of preservatives.

5.5.4 Water-Soluble Organic Metabolites

Objectives - In order to investigate carbon flow in microbial communities, we aim to decipher information encoded in low-molecular-weight organic compounds in pore-waters that act as central intermediates or terminal products in metabolic processes. We are particularly interested in the distribution, abundance, and isotopic composition of volatile fatty acids (VFAs) such as acetate (Heuer et al., 2006, 2009) and took samples for shore-based analysis. Furthermore, volatile alcohols such as methanol and ethanol, generated during microbial degradation of organic matter, could also be potentially important to carbon cycling in marine sediment (Zhuang et al., 2014). For the interpretation of compound-specific data additional information on bulk parameters are crucial and lead us to take samples for bulk analysis of contents and stable carbon isotopic compositions of dissolved inorganic carbon (DIC), dissolved organic carbon (DOC) and total organic carbon (TOC) in the sediment's solid phase. Sampling focused on pore-waters but also included water column samples retrieved by CTD-rosette.

Onboard sample processing – Three subsamples were taken from the pore-water samples retrieved together with the Inorganic Geochemistry Group from closed MUC cores and gravity cores by rhizon micro suction samplers (see 5.5.2): 2 mL for quantitative and stable carbon isotopic analysis of VFAs, 6 mL for analysis of alcohols, and 2 mL for stable carbon isotopic analysis of DIC. All samples were stored in glass vials at -20°C. Samples for stable carbon isotope analysis of DIC were stored without headspace in 2 mL vials.

5.5.5 Molecular Characterization of Dissolved Organic Matter (DOM)

Objectives - DOM in deep ocean sediments contains important information about the preservation and remineralization of organic matter. Organic matter preservation is strongly dependent on the chemical composition and the resistance or susceptibility to microbial decomposition. Therefore, knowledge about the composition and structures of DOM in sediment pore-waters allows insights into the types of compounds utilized by benthic archaea. We sampled sediment pore-waters in order to apply Fourier Transform Ion Cyclotron Resonance Mass Spectrometry (FT-ICR-MS) which recently has delivered first extensive molecular insights into the poly-disperse and complex nature of this material (Schmidt et al., 2009). In addition, samples were taken to quantify DOC concentrations. Samples were taken from selected depths of the split core after visual core description. All analyses will be conducted shore based.

Onboard sample processing - Pore waters in a volume of 20 to 50 ml were extracted with rhizons (0.15 µm pore size) from selected sediment depths of MUC and gravity cores at all sites. All samples were acidified to pH 2 with hydrochloric acid in a glove bag (nitrogen atmosphere) in order to prevent oxidative decay of samples from reductive sediments. Solid phase extraction (SPE) was carried out for the enrichment and desalting of DOM on pre-cleaned SPE cartridges (PPL bond elut, 200 mg

sorbent, Varian). Afterwards, DOM was eluted with 0.5 ml methanol and stored under nitrogen atmosphere in combusted glass vials at -20°C. Aliquots were taken from the original sample and the SPE extract for measurements of DOC concentrations.

5.6 Sampling of Sediments for Sedimentological and Geochemical Solid Phase Analyses

(I. Aiello, M. Elvert, V. Heuer, X. Liu, J. Wendt, G. Zhuang)

5.6.1 Introduction

The shipboard visual description of all sediment cores served to characterize the depositional environment and guided our geochemical and microbiological sampling program. Solid phase sampling was conducted on MUC and gravity cores. In addition, suspended matter was collected from the water column by ISP.

5.6.2 Lithological Core Description

Core description included visual observation of sedimentary structures, lithologic boundaries, trace fossils, authigenic minerals and any other feature visible at naked eye. Sediment color was determined using the Munsell Color Chart (Munsell Color Company, Inc., 1994). Sediment composition was determined using smear slide petrography. For the smear slide preparation, a small amount of sediment was taken with a wooden toothpick and put on a 2.5 cm x 7.5 cm glass slide. The sediment sample was homogenized with a drop of deionized water and evenly spread across the slide to create a very thin (approximately <50 µm), uniform layer of sediment grains for quantification. The dispersed sample was dried on a hot plate. A drop of Norland optical adhesive was added as mount medium to a cover glass. The smear slide was then fixed in an ultraviolet light box. Smear slides were examined with a transmitted-light petrographic microscope equipped with a standard eyepiece micrometer. Biogenic and mineral components were identified and their percentage abundances were visually estimated under petrographic microscope using Rothwell (1989).

The principal name applied to a sediment was determined by the component or group of components (e.g., total biogenic carbonate) that comprise(s) >60% of the sediment, except for subequal mixtures of biogenic and siliciclastic material. The main principal names are:

- **Siliciclastic Sediments:** if the total siliciclastic plus volcaniclastic content was >50% and the sediment had a higher proportion of siliciclastic grains, the main name was determined by the relative proportions of sand-, silt-, and clay sized siliciclastic grains when plotted on a modified Shepard (1954) ternary classification diagram (Examples of siliciclastic principal names are clay, silt, sand, silty clay, sandy clay, clayey silt, sandy silt, clayey sand, and silty sand).

- **Biogenic Sediments:** if the total biogenic content was >50% then the principal name applied was ooze. Biogenic components were not described in textural terms. Thus sediment containing 65% sand-sized foraminifers and 35% siliciclastic clay is called foraminifer ooze, not foraminifer sand.
- **Mixed Sediments:** Sediments containing subequal mixtures of biogenic, siliciclastic grains. In mixtures of biogenic and non-biogenic material where the biogenic content is 40%–60% (termed “mixed sediments” in the IODP classification), the name consists of two parts: (1) major modifier(s) consisting of the name(s) of the major fossil group(s), with the least common fossil listed first, followed by (2) the principal name appropriate for the siliciclastic components (e.g., foraminifer diatom silty clay).
- Prefixes: If a biogenic component represented between **10% and 40%** of the sediment, it was indicated with a minor modifier that consists of the component name hyphenated with the suffix “**rich**” (e.g., diatom-rich clay). When a component made up **5%–10%** of the sediment, it was indicated with a minor modifier that consists of the component name hyphenated with the word “**bearing**” (e.g., diatom-bearing clay). When two minor components were present, minor modifiers were listed in order of increasing abundance before the principal name.
- Examples of nomenclature for mixed sediment: A sediment with 15% foraminifer, 40% coccoliths, and 45% clay is a foraminifer-rich coccolith clay; a sediment with 5% diatoms, 10% radiolarians, and 85% clay is a diatom- and radiolarian-bearing clay.

5.6.3 Sampling Procedures

For basic geological, geochemical and microbiological characterization of the sediments, three samples, each 2-3 mL, were collected by cut-off syringes for (a) grain size analysis, (b) elemental and isotopic analysis of bulk sedimentary carbon and nitrogen, that will provide information about the sources, preservation and remineralization of organic matter, and (c) cell counts (see *5.7 Sampling of Seawater and Sediments for Microbiological Investigations)*. Samples were taken in regular intervals every 10-20 cm, but a higher resolution was chosen at interesting lithological boundaries. Sample were stored in 12 mL plastic vials for grain size analysis and elemental and isotopic analysis and stored at room temperature and -20°C, respectively.

For the structural and isotopic analysis of microbial membrane lipids, ~100 mL of sediment were sampled from gravity and MUC cores. In addition, particulate matter was filtered from the water column using ISP (see *5.2 Sampling of the Water Column with CTD/Rosette and In-Situ-Pumps (ISP))*. Intact polar lipids (IPLs) are important building blocks of microbial cell membranes. IPL analysis leads to general taxonomic information on the active microbial community and simultaneous semi-quantification of both archaeal and bacterial lipids, which can be used for estimation of extant biomass (Lipp et al., 2008). In addition, comparison of carbon isotopic values of IPLs

with $\delta^{13}C$ of other carbon pools such as methane, DIC, and total organic carbon will provide a first indication of microbial metabolism and the carbon source used for building biomass (Biddle et al., 2006). Sediment samples for lipid analysis were stored in 150 mL HDPE-vials. ISP-filters were wrapped in combusted tin foil and stored in amber glass vials. The samples were stored at -20°C. Shorebased, IPLs will be analyzed using high-performance liquid chromatography/electrospray ionization multistage mass spectrometry following the protocols described in Sturt et al. (2004), Biddle et al. (2006), and Schubotz et al. (2009). Isotopic characterization of individual IPL groups can be achieved using preparative high-performance liquid chromatography followed by isotope ratio monitoring mass spectrometry (Biddle et al., 2006).

5.7 Sampling of Seawater and Sediments for Microbiological Investigations

(N. Goldenstein, V. Heuer, M. Könneke, G. Zhuang)

5.7.1 Introduction

Samples were taken to investigate microbial communities in sediments and the overlying water column (Tables 3 and 4) based on cell counts, metagenomic analysis of microbial diversity, structural and isotopic analysis of sedimentary microbial membrane lipids (see above), incubation experiments, enrichments, and rate measurements.

5.7.2 Sampling Procedures

DAPI cell counts - For DAPI cell counts, 0.5 ml sediment samples were taken with syringes, fixed in 1% PBS-buffered paraformaldehyde, washed in ethanol/PBS buffer, and stored at -20°C. With DAPI cell counts all microbial cells with double-stranded DNA are quantified. The fluorochrome DAPI reacts with DNA by intercalation into the stacked basepairs within the double helix. Under UV excitation light in epifluorescence microscopy, DAPI-stained cells reveal a distinct blue fluorescence and can be counted on a per ml basis. DAPI cell counts deliver the 100% baseline for cell number quantification and comparisons with other approaches that target specific populations like Fluorescence in-situ hybridization (FISH) or quantitative polymerase chain reaction (q-PCR); they can be combined with FISH to visualize microbial cells with group-specific 16S rRNA-targeted gene probes (see Teske et al., 2009).

Metagenomic analysis of microbial diversity - For metagenomic analysis, about 20 mL of wet sediment were sampled with cut-off syringes, stored in sterile Falcon Tubes, immediately frozen at -32°C (i.e. the coldest temperature available), shipped on dry ice and stored at -80°C in the laboratories on shore. Samples will be subjected to DNA and RNA extraction using specially optimized protocols that optimize recovery while at the same time minimize PCR inhibition (Lloyd et al., 2010); the combination is essential for successful PCR amplification, whole-genome amplification, and q-PCR from often DNA-limited subsurface sediments.

Subsequently, the microbial community composition will be determined by next generation sequencing techniques.

Incubation experiments - Sediment samples were taken for shore based incubation experiments. 100 – 500 mL of wet sediment were collected from MUC and gravity cores as soon as possible after core opening to minimize exposure to oxygen. Sterile spoons or syringes were used to transfer sediment from cores into glass bottles which were sealed with gas-tight butyl stoppers. The headspace gas of the bottles was immediately replaced with N_2 before samples were stored at 4°C. Various shore based incubation experiments are planned to investigate biogeochemical processes in benthic microbial communities and to enrich selected microorganisms.

Enrichments – For enrichment culturing 50 – 200 mL seawater, from every depth sampled during the CTD casts, were filled into sterile 100 to 250 mL Schott bottles and stored at 4°C. The seawater samples will be sterile filtered using Whatman syringe filters (poresize 0.7 µm) and spiked with NH_4Cl or urea in addition to streptomycine for selective enrichment of archaea from the water column. From MUC deployments one core was designated for enrichment culture sampling each time. For this, 5 cm of overlying bottom water were mixed with the surficial sediments inside the core tube, resulting in a liquid sediment slurry. The slurry material was thereafter sampled from the cores, using sterile, cut-open 20 mL plastic pipettes and transferred to sterile 100 mL Schott bottles, which were stored at 4°C for further shore-based treatment. In the lab the surface sediment slurries will be diluted in 8 steps with sterile-filtered seawater, obtained from CTD casts during the cruise. The slurries of the dilution series will be spiked with NH_4Cl or urea in addition to streptomycine. Each of the slurries will subsequently be used to inoculate a solid agar medium, providing the same nutrient and energy sources as the slurries. This setup will be used to potentially enrich specific archaea from surface sediments on solid media.

Rates of methanogenesis – To investigate the relative contribution of different methanogenic pathways, sediment samples were collected from selected cores for shore based rate measurements of methanogenesis from different substrates (HCO_3^- , acetate, methanol, methylamine). The rate measurements will employ ^{14}C labeled substrates and the conversion of these substrates to methane will be identified with radiotracer techniques. Samples were taken from two gravity cores (GeoB17306-2 and GeoB17308-4) and one MUC (GeoB17314-1). In each core, quadruplicate samples of ~2 mL of sediment were taken for each substrate from selected depths. The samples were stored in gas-tight vials without headspace at 4°C. The vials were modified Hungate tubes, in which the lower end was cut off and closed by a plunger made from butyl rubber. The samples were introduced into the modified Hungate tubes through the cut-end, the plunger was added from the cut-end to move the sediment to the threaded end of the tube, where a septum was placed onto the sediment and tightened with a screw cap.

6. Preliminary Results

6.1 Results of CTD/Rosette Deployments

(N. Goldenstein, M. Könneke)

In situ measurements of temperature [°C], salinity [psu], oxygen [mL/L], turbidity and chlorophyll [mg/m^3] were conducted during CTD-rosette deployments (see 5.2).

Stations GeoB17301 – GeoB17303 showed ~0.5°C elevated surface compared to deeper seawater temperatures of about 13°C (Fig. 6). The deep temperature was constant from a depth of 300 – 400 m to the bottom of the profile. The other parameters measured exhibited a similar separation between surface and deep water-masses, with salinity changing from ~38.3 to 38.5 psu and oxygen content shifting from ~5.5 mL/L to 4 mL/L within the upper 400 m of the water column. Turbidity of ~1 and chlorophyll contents of ~2.5 mg/m^3 at surface dropped at <100m water depth, with minimum values of 0.8 and 0 mg/m^3, respectively. CTD measurements at the deepest station GeoB17304 suffered from harsh weather conditions which resulted in lateral movement of the vessel and correspondingly the rosette during the CTD cast. This lateral, in addition to the regular vertical, motion might be responsible for the scatter and drift in the data recorded by the sensors at this site. However, the separation in shallow and deeper waters and the values of the individual parameters described for GeoB17301 – 17303 are also observed for GeoB17304 (Fig. 6).

The riverine influence from the adjacent River Rhône on the stations GeoB17305 – GeoB17308 becomes visible due to a large salinity shift from ~36 to ~38 psu in the upper 20 m of the water column (Fig. 7). The other parameters change in the same part of the cast profiles, indicating a surface water current of colder less saline water, possibly influenced by snow-melt derived water masses, transported with the River Rhône.

The warmer water draining from the Moulouya river triggers a shift from ~16°C surface water temperature to ~14°C at depth for stations GeoB17310 – GeoB17313 (Fig. 8). Changes in salinity from lower to higher values with increasing water depth as well as the shift in temperature occur gradually over depth compared to the profiles obtained in front of the River Rhône mouth where shifts were completed in the upper 20 m of the water column. Hence, the water column offshore the Moulouya probably undergoes deeper mixing, preventing the formation of a clearly stratified water column. The deep station GeoB17315 in contrast to the other deep offshore stations still exhibits the influence by warm freshwater up to 100 m water depth with a shift to conditions resembling the other deep stations (GeoB17301 – GeoB17304) at >100m water depth (Fig. 9).

CTD-data are available at the PANGAEA World Data Center MARE (http://doi.pangaea.de/10.1594/PANGAEA.835609).

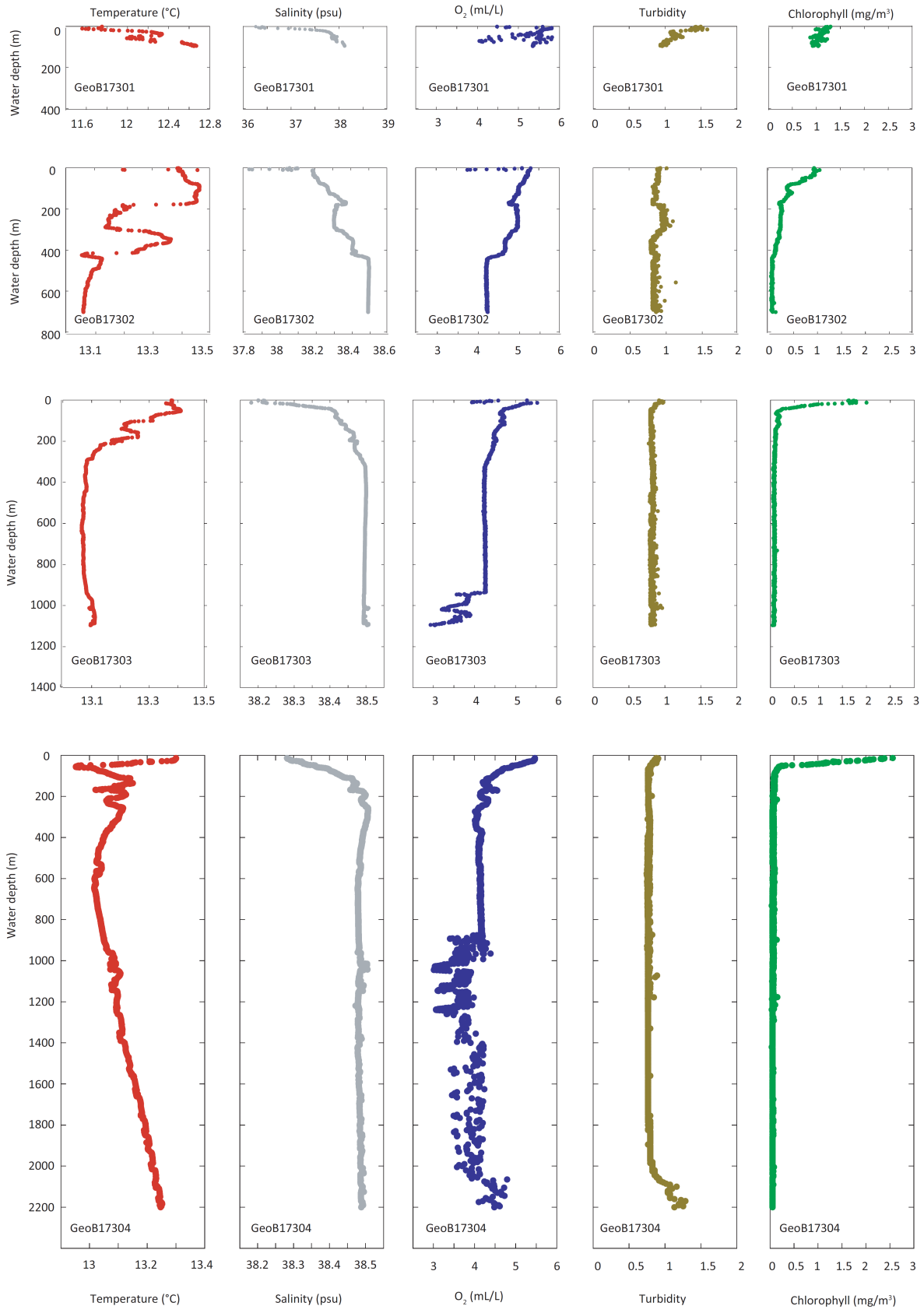

Fig. 6: Results of CTD-deployments in the Cap de Creus Canyon.

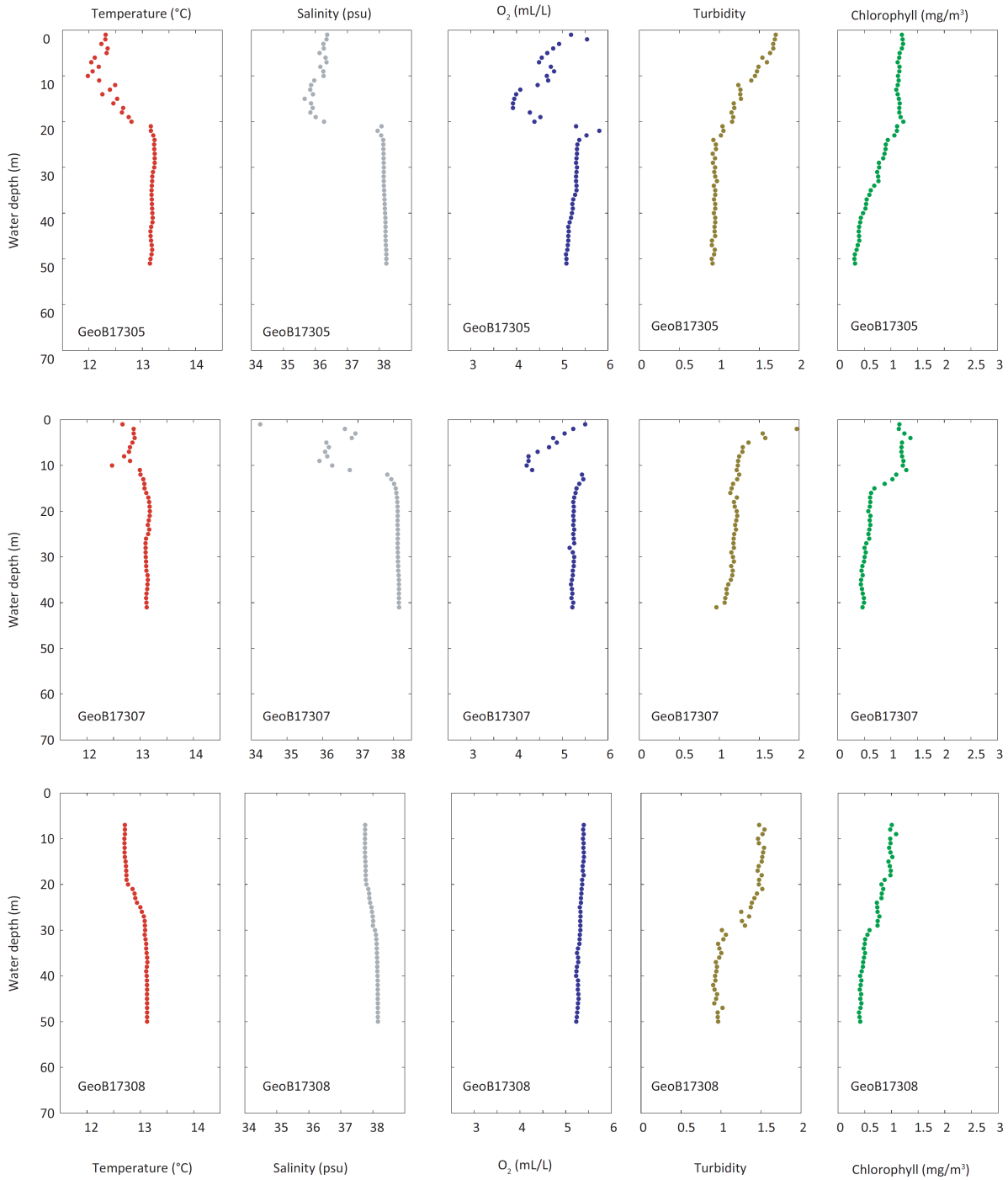

Fig. 7: Results of CTD-deployments off the River Rhône.

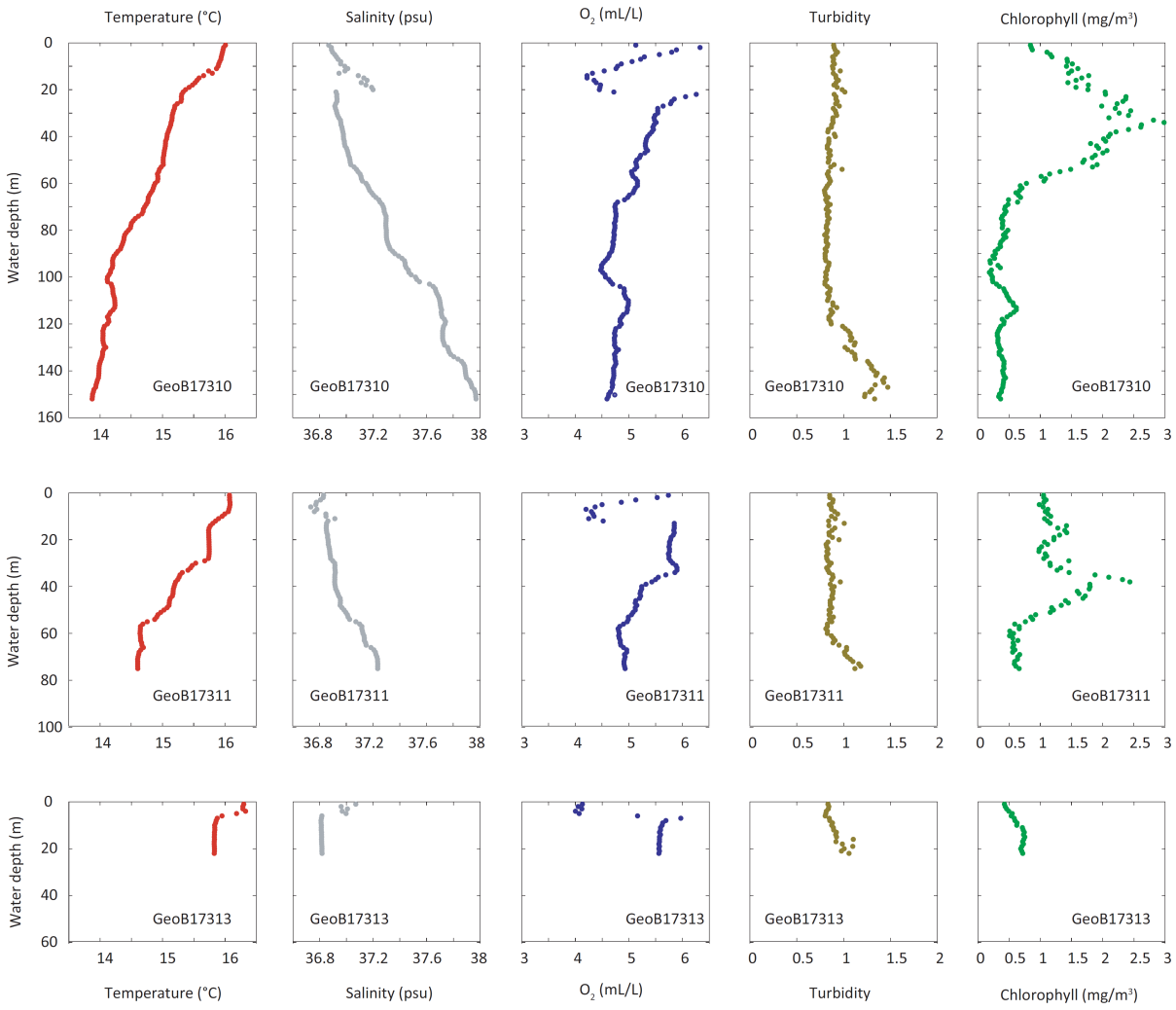

Fig. 8: Results of CTD-deployments off the Moulouya River

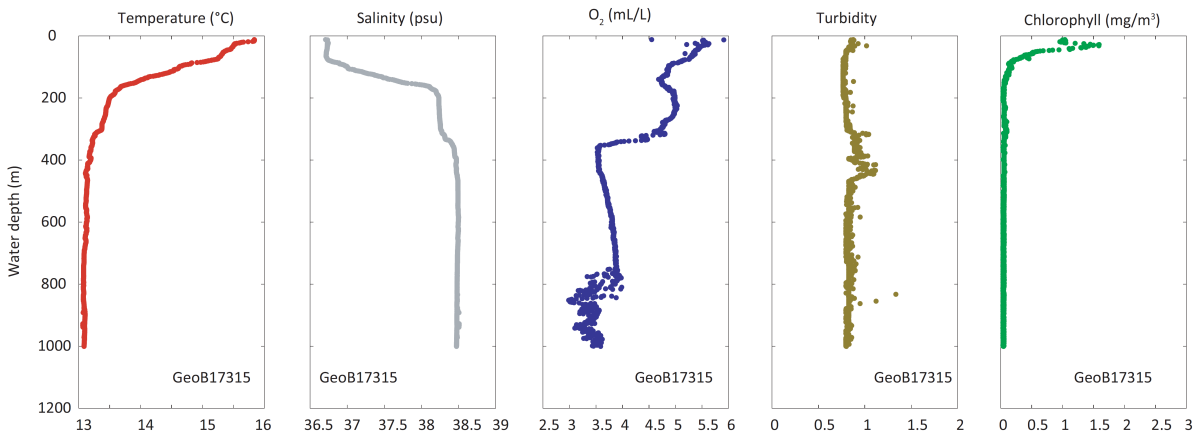

Fig. 9: Results of CTD-deployments at Site GeoB17315

6.2 Results of Gas Analysis

(M. Elvert, V. Heuer, J. Wendt)

We observed high concentrations of methane in sediments on the shelf off the River Rhône. At Sites GeoB17306, GeoB17307, and GeoB17308, methane concentrations of pore-waters reached up to 6.6 mM, 11 mM and 3 mM, respectively, in the upper 4 - 6.5 m of sediment (Fig. 10). In the prodelta of the River Rhône (GeoB17306 and GeoB17307), the sulphate-methane interface was encountered at ~1 m below seafloor (mbsf). At Site GeoB17308, methane concentrations started to increase with depth at ˜2.5 m below seafloor. At all other sites sampled during POS450, methane concentrations were ≤4 µM in the recovered sediments. At Site GeoB17304, gas analysis was delayed by rough weather conditions. The work deck was no longer accessible for the scientific party when the tool deployment was completed, and the recovered intact gravity core was secured on the work deck for several hours (ambient air temperature ~15°C). We cannot exclude that dissolved gases were lost from the core during storage.

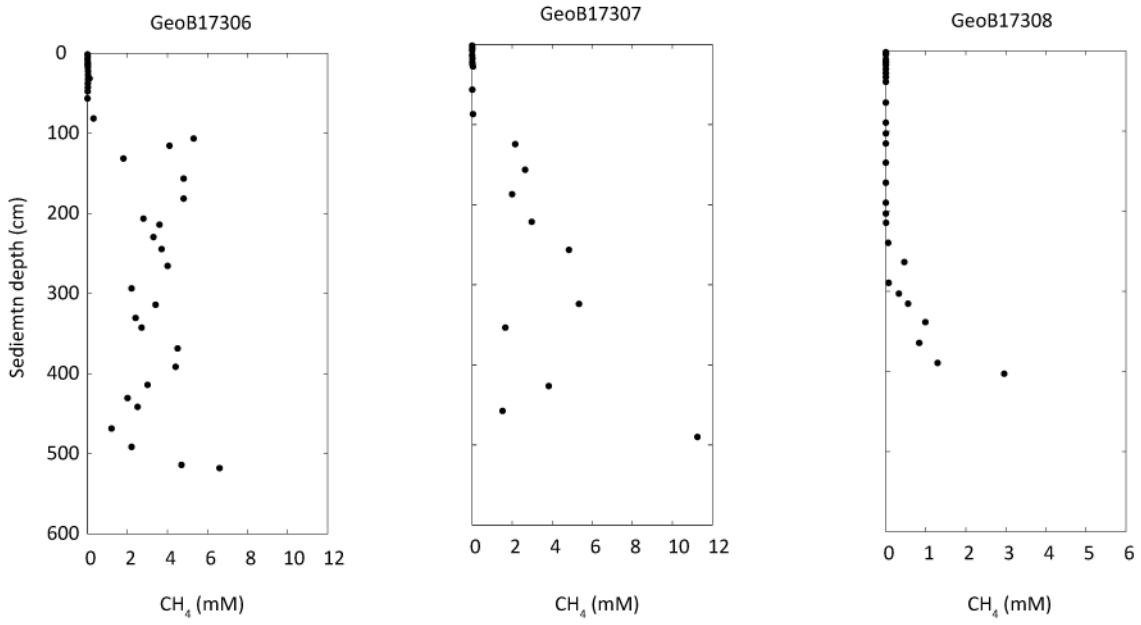

Fig. 10: Concentrations of dissolved methane in sediment cores recovered from Sites GeoB17306, GeoB17307, and GeoB17308.

6.3 Results of Sediment Pore-Water Analysis

(T. Goldhammer, S. Pape)

Pore-water concentrations of Fe^{2+} were analyzed in undisturbed multi– and gravity cores. The sites from the Rhône transect were rich in dissolved Fe^{2+} as expected from the deposition of terrestrial material. All sites revealed characteristic pattern in Fe^{2+} concentrations. While sites GeoB17307, GeoB17308 and GeoB17305 showed more or less continuous profiles, site GeoB17306 surprised with heavy downcore

scatter (Fig. 11). At the moment, we rule out sampling artifacts and attribute this pattern to strong variability in the Fe content of the deposited material. As GeoB17306 is the site closest to the river mouth, it is likely that exceptionally high sedimentation rates exceeded turnover via reductive dissolution of Fe–bearing sediment particles, and thus a steady state of Fe^{2+} production and diffusional transport has not yet been achieved in these immature sediments. Site GeoB17307 is characterized by lower Fe^{2+} concentrations in the upper sediment column, a pronounced, broad peak from 220 to 400 cm depth, followed by a much smaller peak in the lowermost sediment layers. Site GeoB17308 exhibits here a somewhat inverse pattern, with highest concentrations in the surface and deep layers, and a prominent minimum from 150 to 350cm core depth. The short core of site GeoB17305, in contrast, displays the lowest Fe^{2+} concentrations of all sites, with a slight maximum in the upper 50 cm and rather constant decline towards core bottom – indicative of minor sedimentary Fe supply at the site most distant from the river mouth.

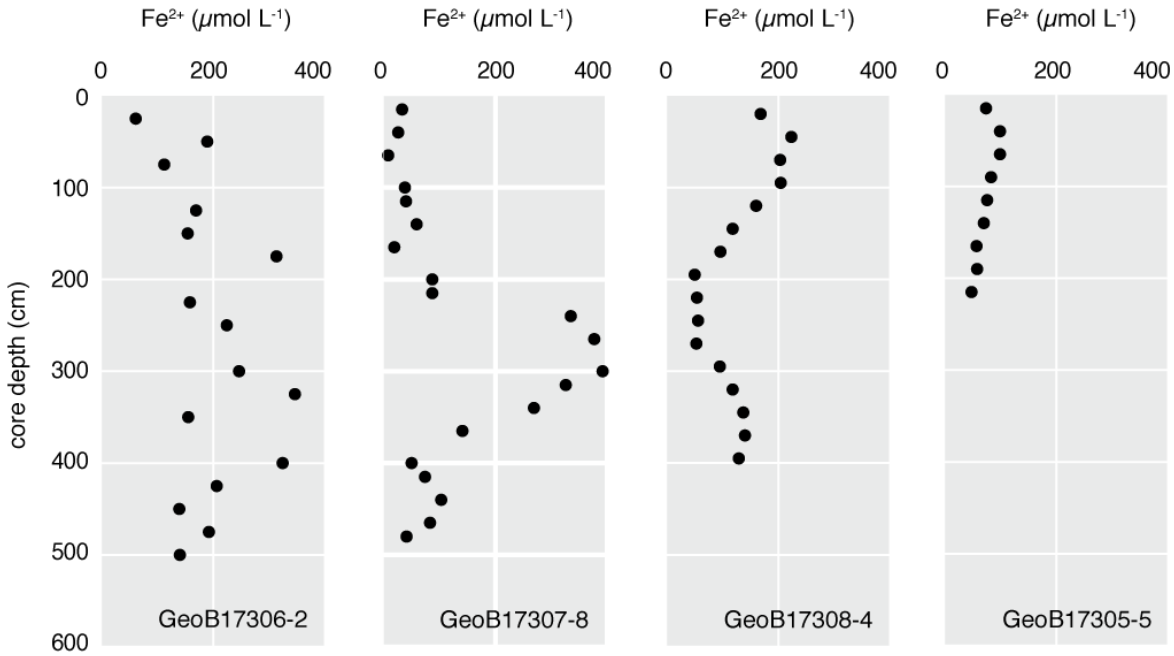

Fig. 11: Onboard pore-water concentrations of dissolved iron (Fe^{2+}) in gravity cores from sites GeoB17306, GeoB17307, GeoB17308 and GeoB17305 (left to right), with increasing distance from the Rhône river mouth.

6.4 Results of Visual Core Description and Smear Slide Analysis

(I. Aiello)

6.4.1 Cap de Creus Canyon

Location of sites - Four sites were sampled in the Cap de Creus Canyon and Ligurian-Provençal Basin to investigate the transport of sediments from the shelf to the deep sea (Fig. 3): GeoB17301 – GeoB17304. Site GeoB17301 is located on the shelf area next to the head of the Cap de Creus submarine canyon in a water depth

of 115 m (Fig. 3). Site GeoB17302 is situated midslope (746 m water depth) about 500 m south of the thalweg of the Cap de Creus Canyon in a mega-scale furrow field described by Lastras et al. (2007) (Fig. 12) which are visible on side scan sonar maps as NW-SE seafloor striations. Site GeoB17303 is located downslope (1146 m water depth) next to the thalweg of the Cap de Creus Canyon and is also in a mega-scale furrow field (Fig. 13). Site GeoB17304 represents the deep-water end member and is located off the Cap de Creus Canyon in a water depth of 2291 m (Fig. 14). More information on sites, tool deployments, and core recovery is given in Table 2 and in the station list below (*7 Station List POS450*).

Core description GeoB17301 – Three MUC deployments failed but 30 cm of sediment were recovered with gravity core GeoB17301-7. The sediment consisted mainly of coarse sand and shell debris which probably correspond to relict deposits from the last glacial maximum (LGM) (cf. Fig. 2 in Rabineau et al., 2005).

Core description GeoB17302 - Two gravity cores were successfully collected from this site and the longer core GeoB17302-5 was used for visual description. The 1.86 m long gravity core GeoB17302-5 is composed of two units:

Unit 1:
0-25 cm: ASH-BEARING COCCOLITH-RICH CLAY
The top 10 cm is dark greyish brown, with higher moisture content; the bottom 15cm is bioturbated with cm-wide sub-vertical burrows in a dark gray massground. Smear slides show that the sediment is composed of a subequal mix between coccoliths and brownish clay (coccolith clay). Larger siliciclastic grains, volcanic glass and foraminifer fragments also occur but only in trace amounts. Long (cm) hair like plant remains were also observed.

Unit 2:
25-185 cm: FORAM- AND ASH-RICH COCCOLITH CLAY and FORAM- AND SAND-BEARING COCCOLITH-RICH CLAY
The majority of the core is a homogenous, consolidated, dark gray sediment with black sulfide streaks and small black pockets with coarser, sandier material. The sediment is moderately sorted and is a mix of siliciclastics (sand, silt and clay size) and coccoliths, although volcanic shards and silt-size foraminifer fragments and whole tests are also present. The majority of the lithogenic clasts is angular and subangular. The clasts are pleiochroic, several show cleavage. The composition of the siliciclastic fraction is arkose and quartz is rare.

173-174 cm: FORAM-BEARING COCCOLITH-AND CLAY-RICH SAND
Near the bottom of the core, a ~1 cm thick coarser and wetter interval is present. The layer is a foram-bearing sand with clast size ranging between silt and medium sand. This is the only sedimentary structure preserved.

Interpretation GeoB17302 - Sediments are hemipelagic. The main biogenic component (coccoliths) reflects oligotrophic conditions. Extensive bioturbation indicates oxic conditions although the preservation of a thin sandy layer at the bottom of the core suggests that less- oxic conditions can also develop. In Unit 1, brownish clay is the main terrigenous component while silt to medium sand siliciclastics dominate Unit 2. The observation that much of the clasts are moderately sorted angular to subangular points to a compositionally/texturally immature sediment. Sorting might be reflecting the fact that the sediment is bioturbated. A possible source for the material is flooding events from the main axis of the canyon. Volcanic ash shards and fine sand to silt size siliciclastic occur scattered throughout the core, and only in one case a sandy layer is present. In the original sediment they were probably deposited in discrete layers that were then reworked by bioturbation.

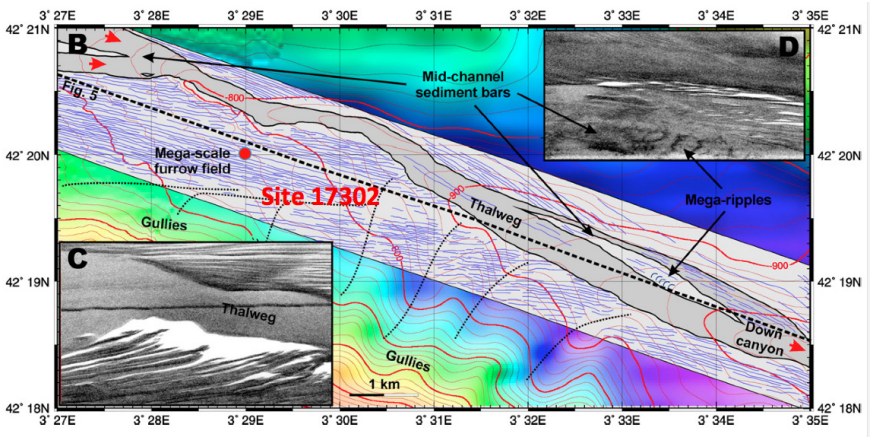

Fig. 12: Location of Site GeoB17302, modified from Lastras et al. (2007)

Core description GeoB17303 – Gravity coring operations were not successful at site GeoB17303.

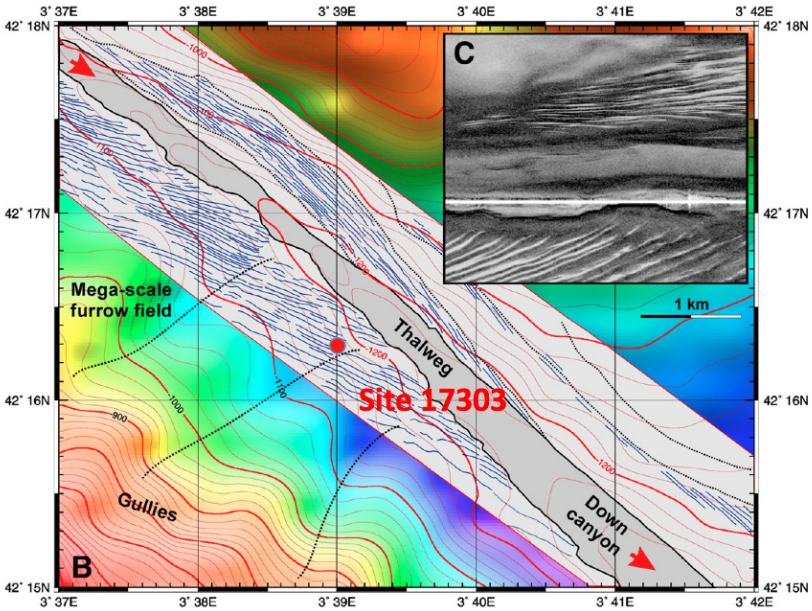

Fig. 13: Location of Site GeoB17303, modified from Lastras et al. (2007)

Core description GeoB17304 - Site GeoB17304 is located on the Rhône Neofan previously investigated by Beaudouin et al. (2004) and Bonnel et al. (2005) (Site MD99-2344). The site is in a channel-levee shaped unit of the Petit-Rhône Neofan, which is a 80-m-thick, lenticular shaped, sedimentary body created after the last avulsion of the Petit-Rhône deep-sea turbiditic channel (Droz, 1983). The 2.34m long gravity core GeoB17304-3 correlates with the upper unit (0 to 2.32 m) of core MD99-2344 (Beaudouin et al., 2004) and can be described as composed of three different lithologic units:

SURFACE SEDIMENT: SILT- AND FORAM-BEARING ASH-RICH COCCOLITH CLAY

The surface samples contain a variety of biogenic particles besides coccoliths including: radiolarians (both nassellaria and spumellaria), juvenile forams and sponge spicules.

Unit 1:

0-44 cm: COCCOLITH-RICH CLAY, FORAMINIFER OOZE and LIMONITE

The top 18 cm is brown, while it is very pale brown between 18 cm (redox boundary?) and 34 cm. Between about 16 cm and 30 cm, the sediment is laminated and white foraminifer tests are visible with naked eye. Iron precipitation is evident between 34 and 44 cm where the sediment is brownish yellow mud with prominent limonite staining ($FeO(OH) \cdot nH_2O$; probably goethite).

Unit 2:

44-75 cm: FINE TO COARSE SAND AND CLAY-BEARING SILT

This unit is characterized by prominent siliciclastic sedimentation of turbidites, at least two. Description of the unit is from bottom to top:

Between 75 and 68-68 cm there is a first patchy, micro-faulted sandy layer embedded in harder clay-rich silt which probably correspond to a first turbidite bed.

An erosional surface (66-68 cm) marks the bottom of the overlying turbidited bed. It starts with ~3 cm coarse sand showing graded bedding (facies Ta of Bouma), followed by ~10 cm of parallel laminated coarse to medium sand (Tc?), and ~8 cm of medium-fine sand laminated towards the base and bioturbated towards the top (Td). The upper part of the turbidite is a ~5 cm dark gray mud (Te).

Unit 3:

75-234 cm: ALTERNATIONS BETWEEN GRAY CLAY-RICH SILT/CLAY AND DARK GRAY SAND

The unit is characterized by alternations between 15 softer, dark gray, fine-sand-to-silt, gray clay-rich silt/clay beds and harder, gray, often banded, clay-rich interbeds. The sandy beds are more pronounced and thicker in the upper

part and become thinner and fainter (less obvious) in the lower part of the unit. The majority of the clay interbeds appears banded and the banding is given by the occurrence of few-mm-thick sandy layers.

Interpretation GeoB17304 - As previously described by Beaudouin et al. (2004) and Bonnel et al. (2005), the sequence recorded in the collected gravity core describes two very different depositional settings within the Rhône Neofan. Described here in chronological order: The lowermost Units 3 and 2 record sedimentation on a levee of a submarine fan with an upward trend of increasing frequency of turbidites and/or decreasing distance from the channel. The hemipelagic clay at the top of the uppermost, thickest and coarsest turbidite marks the end of this depositional environment as a consequence of the rerouting (avulsion) of the Rhône depositional system following post-LGM sea level rise. Unit 1 at the top represents pelagic and hemipelagic sedimentation characterized by much lower sedimentation rates than Units 2 and 3, and only marginally influenced by sediment input from the Rhône River.

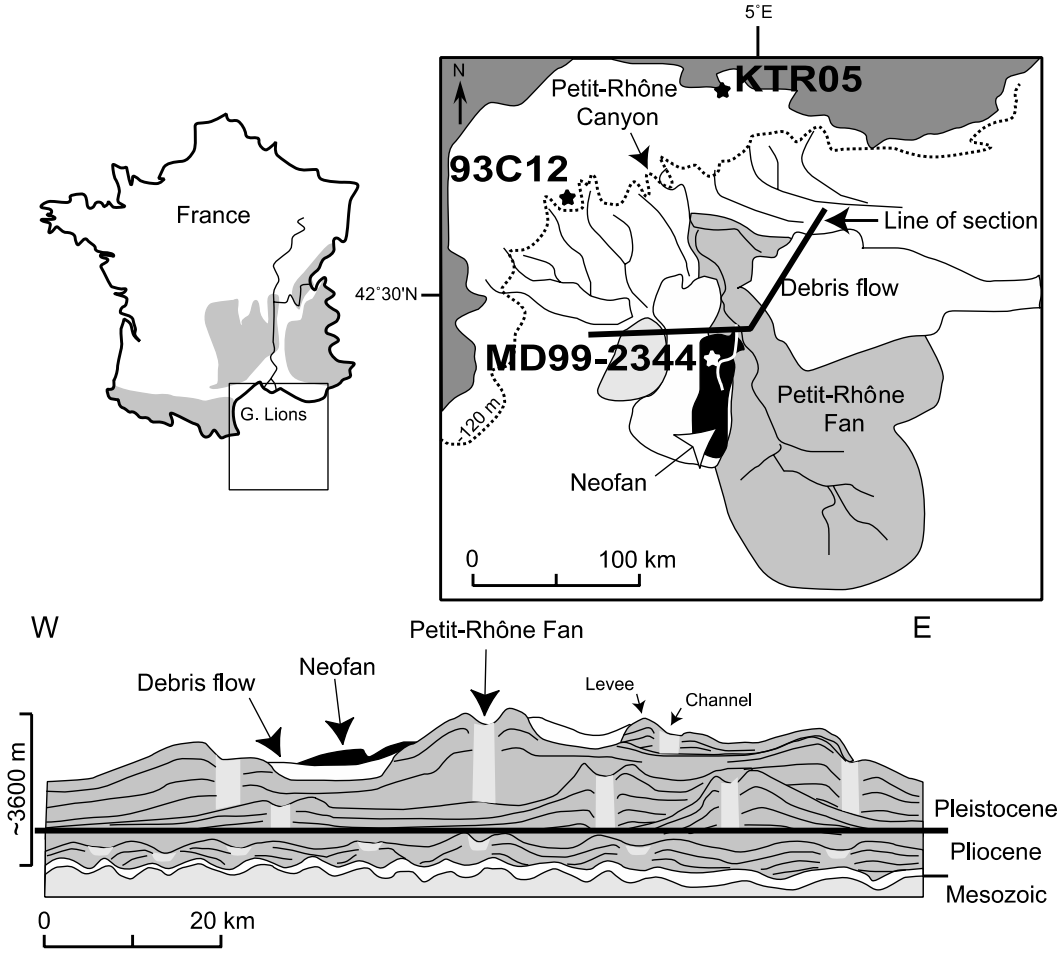

Fig. 14: Site GeoB17304 resembles core MD99-2344 on the Petit-Rhône Fan (from Beaudouin et al., 2004)

6.4.2 Shelf off the River Rhône in the Gulf of Lions

Location of sites – Four sites were sampled in the Gulf of Lions to track the riverine input of the River Rhône to the shelf (Fig. 4): GeoB17305 – GeoB17308. Sites GeoB17306 and GeoB17307 represent the pro-delta of the River Rhône, while site GeoB17308 and GeoB17305 are located 6.3 NM and 16 NM, respectively, west of the river mouth. Site GeoB17305 is a typical mud-belt site and represents the marine end-member. All sites are located in shallow water depth of 30-62 m. More information on sites, tool deployments, and core recovery is given in Table 2 and in the station list below (*7 Station list*).

Core description GeoB17306 - The site was established in the pro-delta in the closest possible proximity to the mouth of the River of Rhône at a water depth of 30 m. The core description is based on a 0.52 m long core of MUC GeoB17306-1. The core is composed of two units distinguishable by color:

Unit 1:
0-31 cm: ORGANIC-RICH DIATOM-BEARING SILT-RICH COCCOLITH CLAY and COCCOLITH-RICH CLAY
The top 7 cm is dark greyish brown, with high moisture content open voids (burrows); below the sediment is firmer. The sediment is mainly clay mixed in different proportions with coccoliths and secondarily diatoms (mainly penates, secondarily centric) and silt-size siliciclastic particles. Organic material in the form of plant debris and brown organic particles mixed with clay is common.

Unit 2:
31-52 cm: ASH- BEARING COCCOLITH-RICH SILTY CLAY and COCCOLITH-RICH CLAY
The bottom part of the multicore is dark gray and firmer clay mixed with coccoliths. The organic component seems less abundant than in the upper part as well as the occurrence of plant material. Glass shards occur in trace amounts.
Interpretation GeoB17306 - The sediment is a hemipelagic mix of clay and coccoliths. The abundance of other, secondary components (silt, ash, and diatoms) varies through the core probably reflecting changing conditions of both terrestrial input and primary productivity, with the diatoms reflecting higher productivity conditions. Extensive bioturbation indicates oxic conditions. Organic material in form of plant debris and mixed clay and organics is particularly abundant in the upper ~30cm of the core which is also characterized by a brownish color as opposed to the darker bottom part.

Core description GeoB17307- The site was established in the pro-delta in close proximity to the mouth of the River Rhône at a water depth of 52 m. The description

is based on a 0.34 m long core recovered from MUC GeoB17307-5. The core is composed of two units distinguishable by color:

0-5 cm: COCCOLITH CLAY
The top 5 cm is very dark greyish brown, with high moisture content;

5-28 cm: CLAY RICH COCCOLITH OOZE
Dark greenish gray with several dark (sulfide?) streaks;

28-34 cm: ORGANIC-RICH ASH-BEARING SAND
Very dark gray to black bottom layer with coarser silt and charcoal-looking material inside.

Core description GeoB17308 - The site was established on the shelf, 6.3 NM west of the mouth of the River Rhône, in a water depth of 62 m. The core-description is based on a 0.34 m long core recovered by MUC GeoB17308-1. The core is composed of two units distinguishable by color:

0-3 cm: ORGANIC-RICH COCCOLITH-BEARING CLAY
The top 3 cm is very dark greyish brown, with high moisture content;

3-33 cm: COCCOLITH-BEARING CLAY
Dark greenish gray with dark streaks.

Core description GeoB17305 – The site was established on the shelf, 16 NM west of the mouth of the River Rhône, in a water depth of 61 m. Site GeoB17305 is a typical mud-belt site and represents the marine end-member. The 229 cm long gravity core GeoB17305-5 is very homogenous and appears highly bioturbated safe for a few faint lighter-colored laminae. Shell debris (coquina layers) is abundant throughout the core either dispersed in the mud or clustered in small pockets. The bulk of the sediment is coccolith-bearing clay.

6.4.3 Shelf and Slope Offshore the Moulouya River, Morocco

Introduction - The mouth of the Moulouya River is a bar built estuary located just a few km west of the border between Algeria and Morocco. The mouth is located in an emergent margin characterized by a 10 m high marine terrace indicating uplift. The Moulouya River is the largest river on the Moroccan Mediterranean coast with a high sediment yield (250 t/km^2/yr) (Ludwig et al., 2003). Although the main surface water current system is the anticyclonic Eastern Alboran Gyre, Millot (1999) shows local variations in the gyre structure including an eastward, cyclonic flow just northwest of the Melilla peninsula. However, an aerial photo from the Moulouya mouth (Fig. 15) shows that the plume of sediments jets north and then curves to the west just north of the island of Chafarinas. As shown by the sediment recovered (and non recovered) during our cruise, the shelf- upper slope in the region is essentially an

erosional/condensed surface strongly controlled by winnowing due to high current velocities creating lag (coarse material) deposits.

The area is also influenced by the complex 'wrench' tectonics of the basement and the Mio-Pliocene sediments and unconformities draping it. As shown by Ammar et al. (2007), the continental margin in front of the Moulouya River is characterized by a NNW-SSE oriented basement high which borders the Habibas Basin to the NW. The basement is probably made of calc-alkaline rocks of Tortonian age similar to the volcanic rocks exposed at Cap de Trois Fourches to the west. Seismic profiles show that this high like other basement highs in the region has a flat top indicating that it was eroded by waves. The profiles show a prograding layer (probably a Messinian-Tortonian carbonate platform; Cornée et al., 2006) above the basement overlain by a Messinian erosional surface. A reflector indicating the presence of a volcanic sill is also present in the subsurface of our study area probably related to the Pliocene volcanism of the near Chefrina Island.

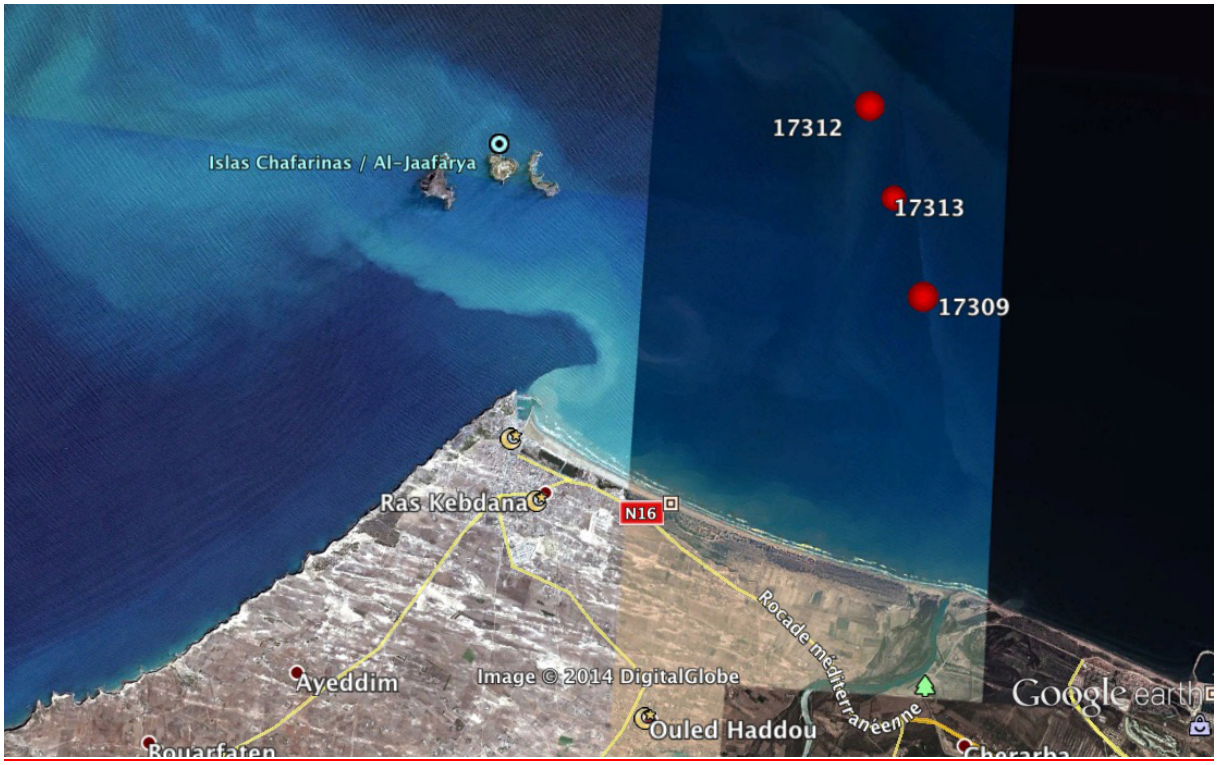

Fig. 15: Picture form Google Earth showing the area south of the Moulouya River off Morocco, and the location of Sites GeoB17309 - GeoB17312 and GeoB17313. The mouth of the river is visible in the bottom right. A sediment plume moving away from shore towards the northwest and crossing the location of Isla Chafarinas is also visible.

Location of Sites – Six sites were investigated on the shelf north of the Moulouya River (Fig. 5): GeoB17309 – GeoB17314. Site GeoB17309 was established offshore the Moulouya River in the closest possible distance from the river mouth at a water depth of 16 m. The other sites were chosen based on an aerial photograph of the river plume. Site GeoB17313 and GeoB17312 are located north of Site GeoB17309 in the middle of the river plume at water depth of 28 m and 52 m, respectively. No

sediment cores could be recovered from these sites. Site GeoB17311 is just north of Chafarinas Island, at ~88 m water depth within the river plume as imaged by aerial photographs. Site GeoB17310 is located further north on the shelf, outside the river plume at bathyal (~161 m) water depth. Site GeoB17314 is located at ~65 m water depth on the shelf west of Chafarinas Island. According to the tectonic map of Ammar et al. (2007), the site is located over a local basement depression. We chose to explore this site because it could act as depocenter and has a higher preservation potential for sediments than the other sites.

Core description GeoB17309 – The gravity core GeoB17309-1 returned bent and recovered only 74 cm of sediment.

Unit 1:
0-5 cm: COARSE SAND WITH SHELLS
The top 5 cm is more unconsolidated and coarser than the majority of the core below. The layer is sandy and includes several, broken and preserved, shells. The unconsolidated sand layer seems to lie unconformably on top of the more consolidated and probably much older unit below.

Unit 2:
5-74 cm: COCCOLITH-BEARING TO COCCOLITH-RICH CLAY
Below the sand unit the sediment is dark greenish gray between 5 and 10 cm and is composed of a coccolith-bearing clay and it is reddish between 10 and 16 cm. Sediment is dark gray for the rest of the core between 16 and 74 cm. Between 16 and 33 cm, the stiffest part of the core is composed of sand-bearing coccolith-rich clay. At 44 cm there is a reddish lamina and a gray band between 56 and 58cm.

Interpretation GeoB17309 - The core shows clearly two very distinct lithologies that indicate very different genesis and depositional environments. The thin top Unit 1 is a modern unconsolidated coarse sand and shell deposit typical of the nearshore, high-energy zone in which the core has been retrieved. It is separated from the unit below by a sharp change in physical properties and grain size. The more consolidated unit below is probably an inner shelf deposit with abundant clay size material and variable amounts of coccoliths. The presence of banding and some lamination indicate deposition under conditions of variable oxygen content. The stiffness of Unit 2 also suggests that these could be older sediments that have been buried, uplifted and eroded by wave action and therefore the boundary between Units 1 and 2 is an unconformity.

Core description GeoB17311 – MUC deployment GeoB17311-2 recovered ~45 cm long cores, reddish at the top ~10 cm and more reddish brown towards the bottom. The main composition is ORGANIC-RICH SANDY CLAY WITH TRACE AMOUNTS OF COCCOLITHS.

Interpretation GeoB17311 - The redness and oxidization of the sediment, the presence of brown clay and organic material are very distinct from other cores collected on the nearby shelf and suggest that the core is a river plume deposit.

Core description GeoB17310 - The MUC deployment GeoB17310-1 recovered ~20 cm of homogenous, greenish gray FORAM-BEARING COCCOLITH-RICH CLAY, similar to the Unit 2 at site GeoB17309 although it is not banded and seems lees stiff. Gravity core GeoB17310-5 is 130 cm long, homogenously greenish gray and can be divided in two major units:

Unit 1:
0-82 cm: COCCOLITH-BEARING SAND-RICH CLAY
Homogenous in the upper 60 cm, a few preserved, shells occur scattered between 60 and 83 cm.

Unit 2:
83-130 cm: COARSE SAND TO GRANULES AND COCCOLITH-BEARING FORAM-RICH CLAYEY SAND
The top of this coarse unit, between 83 and 92 cm is transitional with the previous one showing and progressive down-core increase in sand and shell fragments. The rest of the unit is characterized by broken-up shells (coquina layers) mixed with coarse sand and granules in a poorly-sorted matrix made of foram-rich clayey sand.

Interpretation GeoB17310 - The sedimentologic characteristics of Unit 1 suggest deposition in the inner- outer-shelf (similar to the modern depth environment). The coarse Unit 2 can be tentatively interpreted as a lag deposit from a nearshore depositional environment related to the LGM sea level low stand

Core description GeoB17314 - The MUC deployment GeoB17314-1 recovered ~30 cm of reddish brown ORGANIC-RICH COCCOLITH-BEARING SAND-RICH CLAY

7 Station List POS450

(V. Heuer)

Station #	GeoB #	Gear	Date (UTC)	Time (UTC)	Latitude	Longitude	Water depth (m)	Recovery
Cap de Creus Canyon and Ligurian-Provençal Basin								
POS450/575-1	17301-1	CTD-ro	02.04.2013	23:13	42° 24.74' N	3° 17.72' E	115.3	5 bottles
POS450/575-2	17301-2	ISP	03.04.2013	00:09	42° 24.75' N	3° 17.71' E	115.2	2 pumps
POS450/578-1	17301-3	ISP	04.04.2013	02:21	42° 24.77' N	3° 17.69' E	114.4	2 pumps
POS450/578-2	17301-4	MUC	04.04.2013	07:05	42° 24.76' N	3° 17.70' E	114.9	failed
POS450/578-2	17301-5	MUC	04.04.2013	07:19	42° 24.77' N	3° 17.69' E	114.6	failed
POS450/578-3	17301-6	MUC	04.04.2013	07:38	42° 24.78' N	3° 17.70' E	114.9	failed
POS450/578-4	17301-7	GC	04.04.2013	10:07	42° 24.34' N	3° 16.55' E	101.9	30 cm
POS450/576-1	17302-1	CTD-ro	03.04.2013	06:26	42° 19.98' N	3° 29.01' E	725.8	10 bottles
POS450/576-2	17302-2	ISP	03.04.2013	07:15	42° 19.99' N	3° 29.00' E	725.8	2 pumps
POS450/576-3	17302-3	MUC	03.04.2013	11:14	42° 19.99' N	3° 28.99' E	746.4	25 cm
POS450/576-4	17302-4	GC	03.04.2013	12:35	42° 20.00' N	3° 29.00' E	745.4	121 cm
POS450/576-5	17302-5	GC	03.04.2013	13:51	42° 20.00' N	3° 29.00' E	741.4	186 cm
POS450/577-1	17303-1	ISP	03.04.2013	16:36	42° 16.39' N	3° 39.00' E	1135.4	3 pumps
POS450/577-2	17303-2	CTD-ro	03.04.2013	22:46	42° 16.34' N	3° 38.95' E	1133.4	12 bottles
POS450/577-3	17303-3	CTD-ro	03.04.2013	23:21	42° 16.39' N	3° 39.01' E	1141.4	only data
POS450/579-1	17303-4	GC	04.04.2013	14:09	42° 16.38' N	3° 39.02' E	1146	failed
POS450/580-1	17304-1	CTD-ro	04.04.2013	23:59	41° 59.40' N	4° 50.11' E	2281.4	12 bottles
POS450/580-2	17304-2	ISP	05.04.2013	01:54	41° 59.41' N	4° 50.11' E	2281.3	4 pumps
POS450/580-3	17304-3	GC	05.04.2013	09:28	41° 59.41' N	4° 50.13' E	2291.3	234 cm
Shelf off the River Rhône								
POS450/581-1	17305-1	CTD-ro	05.04.2013	22:13	43° 13.79' N	4° 30.64' E	60.8	4 bottles
POS450/581-2	17305-2	ISP	05.04.2013	22:30	43° 13.80' N	4° 30.63' E	62.3	1 pump
POS450/581-3	17305-3	MUC	06.04.2013	06:14	43° 13.80' N	4° 30.61' E	60.9	40 cm
POS450/588-1	17305-4	GC	07.04.2013	12:16	43° 13.81' N	4° 30.64' E	61.4	failed
POS450/588-2	17305-5	GC	07.04.2013	12:56	43° 13.81' N	4° 30.61' E	61.6	229 cm
POS450/582-1	17306-1	MUC	06.04.2013	10:07	43° 18.95' N	4° 52.18' E	29.6	50 cm
POS450/586-1	17306-2	GC	07.04.2013	07:37	43° 18.97' N	4° 52.16' E	29.4	516 cm
POS450/583-1	17307-1	MUC	06.04.2013	12:07	43° 18.23' N	4° 51.54' E	52.1	failed
POS450/583-2	17307-2	MUC	06.04.2013	12:38	43° 18.24' N	4° 51.53' E	51.9	failed
POS450/583-3	17307-3	MUC	06.04.2013	13:37	43° 18.24' N	4° 51.53' E	52.1	failed
POS450/583-4	17307-4	MUC	06.04.2013	13:52	43° 18.24' N	4° 51.52' E	52.1	failed
POS450/583-5	17307-5	MUC	06.04.2013	14:34	43° 18.23' N	4° 51.54' E	52.1	35 cm
POS450/585-1	17307-6	ISP	07.04.2013	01:05	43° 18.24' N	4° 51.54' E	51.6	1 pump
POS450/585-2	17307-7	CTD-ro	07.04.2013	04:47	43° 18.24' N	4° 51.52' E	52.4	6 bottles
POS450/585-3	17307-8	GC	07.04.2013	06:08	43° 18.24' N	4° 51.55' E	52.4	492 cm
POS450/584-1	17308-1	MUC	06.04.2013	16:16	43° 16.20' N	4° 43.79' E	61.9	35 cm
POS450/584-2	17308-2	CTD-ro	06.04.2013	19:55	43° 16.56' N	4° 43.68' E	57.4	6 bottles
POS450/584-3	17308-3	ISP	06.04.2013	20:11	43° 16.54' N	4° 43.68' E	58.1	1 pump
POS450/587-1	17308-4	GC	07.04.2013	10:06	43° 16.21' N	4° 43.80' E	61.4	405 cm

Station #	GeoB #	Gear	Date (UTC)	Time (UTC)	Latitude	Longitude	Water depth (m)	Recovery
Shelf and slope offshore the Molouya River and Alborán Sea								
POS450/589-1	17309-1	GC	10.04.2013	10:00	35° 10.00' N	2° 21.02' W	15.6	74 cm
POS450/590-1	17310-1	MUC	10.04.2013	12:04	35° 16.23' N	2° 21.91' W	161.6	20 cm
POS450/593-1	17310-2	ISP	10.04.2013	23:07	35° 16.15' N	2° 21.84' W	152.4	4 pumps
POS450/593-2	17310-3	CTD-ro	11.04.2013	02:51	35° 16.23' N	2° 21.90' W	159.4	8 bottles
POS450/595-1	17310-4	CTD-ro	11.04.2013	08:59	35° 16.23' N	2° 21.94' W	160.4	only data
POS450/595-2	17310-5	GC	11.04.2013	09:17	35° 16.24' N	2° 21.94' W	161.6	failed
POS450/591-1	17311-1	MUC	10.04.2013	13:14	35° 13.32' N	2° 26.07' W	87.9	failed
POS450/591-2	17311-2	MUC	10.04.2013	13:42	35° 13.31' N	2° 26.09' W	86.9	40 cm
POS450/594-1	17311-3	CTD-ro	11.04.2013	03:46	35° 13.30' N	2° 26.13' W	88.1	6 bottles
POS450/594-2	17311-4	ISP	11.04.2013	04:09	35° 13.31' N	2° 26.11' W	87.1	3 pumps
POS450/594-3	17311-5	GC	11.04.2013	07:44	35° 13.32' N	2° 26.10' W	86.9	failed
POS450/592-1	17312-1	MUC	10.04.2013	15:03	35° 11.74' N	2° 21.72' W	51.9	failed
POS450/592-2	17312-2	MUC	10.04.2013	15:22	35° 11.74' N	2° 21.71' W	51.6	failed
POS450/592-3	17312-3	MUC	10.04.2013	15:41	35° 11.74' N	2° 21.71' W	51.6	failed
POS450/592-4	17313-1	CTD-ro	10.04.2013	16:21	35° 10.89' N	2° 21.40' W	27.9	4 bottles
POS450/592-5	17313-2	ISP	10.04.2013	16:40	35° 10.88' N	2° 21.37' W	27.5	2 pumps
POS450/596-1	17313-3	MUC	11.04.2013	13:16	35° 10.90' N	2° 21.39' W	27.6	failed
POS450/596-2	17313-4	MUC	11.04.2013	13:23	35° 10.87' N	2° 21.40' W	27.1	failed
POS450/597-1	17314-1	MUC	11.04.2013	15:38	35° 7.99' N	2° 31.98' W	65.6	35 cm
POS450/598-1	17315-1	CTD-ro	12.04.2013	00:45	35° 43.46' N	3° 12.39' W	1055.1	12 bottles
POS450/598-2	17315-2	ISP	12.04.2013	06:00	35° 43.50' N	3° 12.54' W	1053.3	4 pumps
POS450/598-3	17315-3	MUC	12.04.2013	07:21	35° 43.44' N	3° 12.34' W	1052	40 cm

8 Data and Sample Storage and Availability

All metadata were delivered to the PANGAEA World Data Center MARE and to the German Oceanographic Data Centre (DOD) at the Bundesamt für Seeschifffahrt und Hydrographie (BSH). The ship station list is published together with the summary cruise report in SeaDataNet, the Pan-European infrastructure for marine and ocean data management. Reference geological cores are stored in the MARUM core repository. The cores and all samples have obtained international geological sample numbers (IGSN). CTD-data are available at the PANGAEA World Data Center MARE (http://doi.pangaea.de/10.1594/PANGAEA.835609), and the submission of further shipboard and post-cruise data to PANGAEA is in progress.

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