Cruise Report

RV Pelagia August 31-September 17, 2015
Istanbul-Varna
64PE401

Black Sea– Fe-Vici cruise

Caroline Slomp (Chief Scientist)
with contributions from participants

Universiteit Utrecht

Koninklijk Nederlands Instituut voor Zeeonderzoek

Nederlandse Organisatie voor Wetenschappelijk Onderzoek

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Front page: View from the ship on September 5

Figure 1. Sediment trap (lost in 2014) retrieved from the seafloor through dredging

1. Introduction and participants

This cruise forms part of the Vici Project "Response of the Iron Biogeochemical Cycle on Continental Shelves to Seawater Deoxygenation" funded by the Netherlands Organisation for Scientific Research (NWO). The project involves partners in the Netherlands (Utrecht University, UU and the Royal Netherlands Institute for Sea Research; NIOZ), Romania (GeoEcoMar) and the United States (Rutgers University). The project builds on knowledge gained during the PHOXY project which included research cruises to the Black Sea in 2013 (with RV Pelagia) and in 2014 (with Mare Nigrum).

Specific goals of this cruise:

The aim of this cruise was to gain insight into the mechanisms and rates of iron release from sediments on the northwestern shelf of the Black Sea and the lateral transport of iron (and other trace metals) over the shelf to the adjacent anoxic basin. We are specifically interested in (1) the relationship of sediment iron release with changes in bottom water oxygen concentrations (2) the role of bioirrigating fauna for iron release (3) the potential role of cable bacteria in the sediment for iron release to the porewater (4) the form in which Fe is transported over the shelf. Sampling took place along the same redox transect sampled during the PHOXY cruise to the Black Sea in 2013 with some additional stations on the shelf.

GEOTRACES

The Fe Vici cruise has been endorsed as a GEOTRACES process study and will follow International GEOTRACES protocols relating to intercalibration, methodology and data management.
http://www.geotraces.org/science/intercalibration/945-intercalibration-procedures
http://www.geotraces.org/libraries/documents/Intercalibration/Cookbook.pdf

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Acknowledgements:
We thank the master of the Pelagia, John Ellen, and his crew and NIOZ Marine Research facilities for the excellent support during our cruise.

Cruise blog: http://geoblog.weebly.com/expedition-black-sea-2015

Collaborators outside the Netherlands:

GeoEcoMar

THE STATE UNIVERSITY OF NEW JERSEY RUTGERS

Table 1. Fe-Vici cruise-Participants and NIOZ-technicians and their contact information and activities.

Name & affiliation	Contact information	Main activity
1. Caroline Slomp, UU	c.p.slomp@uu.nl	Chief scientist
2. Peter Kraal, UU	p.kraal@uu.nl	Porewater, Radio-isotope experiments, traps
3. Nikki Dijkstra, UU	n.dijkstra@uu.nl	Water column sampling, in-situ pumps, Radio-isotope exp. traps
4. Wytze Lenstra, UU	W.K.Lenstra@uu.nl	CH4, incubations, porewater, alkalinity
5. Martijn Hermans, UU	M.Hermans@uu.nl	Micro-electrodes, sediment collection, alkalinity
6. Marie Seguret, UU	M.J.M.Seguret@uu.nl	water column sampling
7.Niels van Helmond	n.vanhelmond@uu.nl	Porewater, micro-electrodes, alkalinity, incubations
8. Rob Witbaard, NIOZ	Rob.Witbaard@nioz.nl	Landers, whole core incubations
9 Adrian Teaca, GeoEcoMar	adrianxteaca@yahoo.com	Macro-fauna shelf stations
10. Silke Severmann Rutgers University, USA	silke@marine.rutgers.edu	Water column sampling, in-situ pumps, rhizon sampling
11. Amy Anderson, Rutgers University, USA	anderson@marine.rutgers.edu	Water column sampling, in-situ pumps, rhizon sampling
12. Santiago Gonzalez	Santiago.Gonzalez@nioz.nl	Radio-isotope experiments
13. Ruud Groenewegen	Ruud.Groenewegen@nioz.nl	Landers, CTD, electronics
14. Sharyn Ossebaar	Sharyn.Ossebaar@nioz.nl	Nutrient analyses, sampling from Niskins
15. Karel Bakker	Karel.Bakker@nioz.nl	Nutrient analyses, sampling from Niskins
16. Lorendz Boom	Lorendz.Boom@nioz.nl	Sediment coring, traps (NIOZ technician)
17. Barry Boersen	Barry.Boersen@nioz.nl	Sediment coring, traps (NIOZ technician)

Figure 2: The crew and scientists of the Fe-Vici cruise on September 15 (missing in the photograph: Adrian Teaca).

2. Cruise Track and overview of activities

A total of 11 stations were visited in the northeastern Black Sea. Stations 2-8 are the same as during the PHOXY cruise in 2013. Stations 10, 11 and 12 were removed from the schedule because of lack of time (delay in the harbour of Istanbul) and are not included in the table below. Station 17, which was located between sites 3 and 4, was added to obtain a third off-shelf water column profile.

Table 2. Stations visited during the Black Sea cruise. Key stations are shaded. The sequence of the stations in the table is based on the water depth and depositional environment (coastal, shelf, slope and deep basin).

Station number	Latitude	Longitude	water depth (m)	bottom water redox, location
2	42°53.8'N	30°40.7'E	2107	anoxic, deep basin
1	43°21.05'N	30°22.3'E	1596	anoxic, deep basin
3	43°31.8''N	30°15.5'E	1100	anoxic, deep basin
17	43°36.06'N	30°11.41'E	818	anoxic, slope
4	43°40.6'N	30°07.5'E	380	anoxic, slope
5	43°42.6'N	30°06.1'E	190	anoxic, slope
6	43°42.8'N	30°05.1'E	130	hypoxic, shelf edge
14	43°45.9'N	30°04'07"E	114	hypoxic, shelf edge
7	43°53.8'N	29°58.6'E	78	oxic, shelf
8	44°14.7'N	29°43.4'E	64	oxic, mid shelf
13	44°36.45'N	29°27.4'E	39	oxic, coastal
9	44°34.9'N	29°11.38'E	27	oxic, coastal

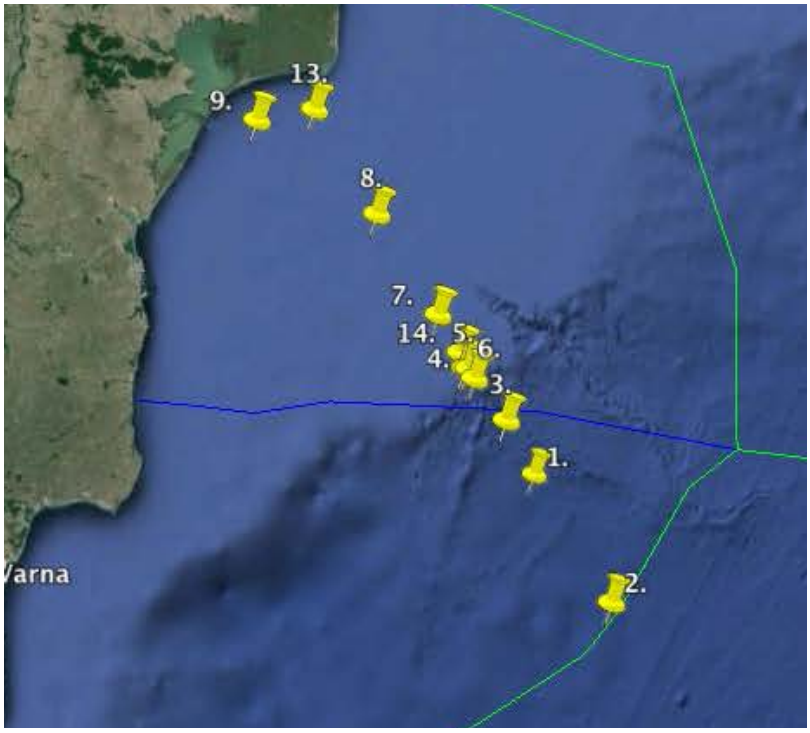

Figure 3. Map with stations in the Black Sea. Note that stations 2-8 are the same as in 2013. Note that stations 10-12 are not included and were not visited due to lack of time.

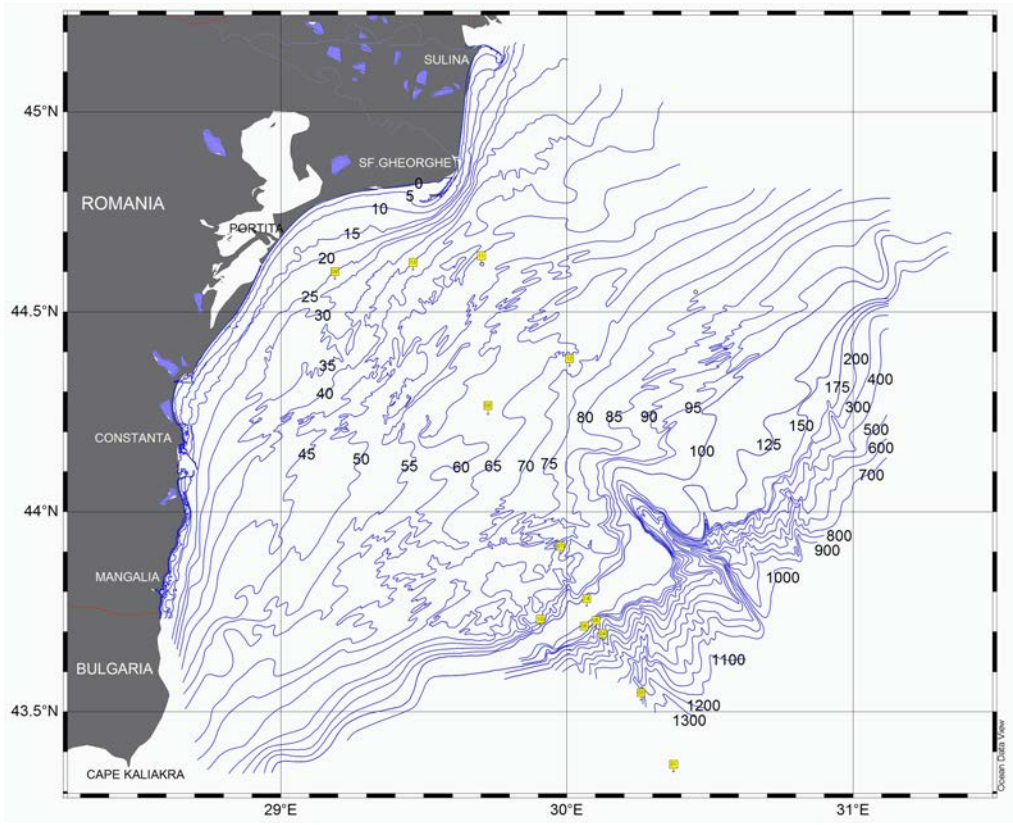

Figure 4. Map with stations in Black Sea including contourlines showing water depths (courtesy: Adrian Teaca)

Table 3. Overview of activities at each station (CTD, MC), box cores, and deployments (lander, 4 pumps, 4 traps) where relevant. Where more boxcores and multicores were taken than were used (because they were e.g. too full) the total number of deployments is included in brackets.

site	Ultra Clean CTD	CTD	Multi-core casts	Landers	Box Core	In-situ pumps	Traps
1	1	-	1	-	-		-
2	2		3	-	-	4 x 4	4 out, 4 in
3	1	-	-	-	-	-	-
4	1	-	2	1	-	-	-
5	1	-	2 (4)	1	-	1 x 4	-
6	1	-	2	2	2 (4)	1 x 4	-
7	1	-	2 (3)	2	2 (4)	1 x 4	-
8	1	-	2	1	2	1 x 4	-
9	-	1	4	1	2 (4)	1 x 2	-
13	-	1	3	1	2 (3)	1 x 2	-
14	1	-	1	1	2	1 x 4	-
17	1	-	-	-	-	1 x 4	-

Overview of sample collection:

1. UltraClean CTD (Ruud, Roald, Marie, Silke, Nikki, Amy)
   - Sensor data for temperature, salinity, oxygen, transmissivity, fluorescence
   - Collection of water samples, typically:
     - 24 water depths at deep stations 1-6
     - at least 15 water depths at shelf stations 7-14
     - Collection of samples for nutrients (NH_4, NO_3, NO_2, PO_4, silica, sulfide and DIC for UU (Karel Bakker/Sharyn Ossebaar, NIOZ)
     - Collection of water and particulate samples in clean container:
       - Total Fe and other (unfiltered sample)
       - Dissolved Fe (<0.2 um filter)
       - Truly Dissolved Fe (<0.02 um filter) (selected samples)
       - Suspended material > 0.2 um, 0.2> and >0.02 um (selected samples)
       - Samples for isotopes
       - Samples sample request Anne Roepert
       - Samples sample request Ian Snowball
       - Samples sample request Jordia Garcia-Orellana

2. In-situ pumping to collect suspended material at selected sites for mineralogical, geochemical and isotope analyses (Marie, Silke, Nikki, Amy)
   - Four pumps, 3 with one filter (NIOZ), 1 with a dual filter (UU)

3. Sediment traps (material processed by Nikki, Peter, others):
   - retrieval of 1 trap
   - dredging for 3 traps
   - CTD sampling to fill trap bottles
   - deployment of new trap

4. Lander and other flux incubations:
   - sediment cores: total flux (oxic, anoxic) and Br- (Wytze)
   - seafloor: landers (Rob, Ruud), shelf sites and one deep site

5. Sieving for meso- and macrofauna (Adrian)

6. Porewater and sediment collection:
   - multicore slicing in glovebox and centrifugation, subsampling
   - rhizon sampling
   - oxic slicing of sediment for 210Pb
   - frozen core for solid phase analyses
   - core for microelectrode profilng (Martijn)
   - sediment cores for storage and experiments (various sites)
   -
7. Alkalinity titrations (Martijn)
8. Radio-isotope incubations (Nikki, Santiago)

Cruise acronym for lables on cores: 64PE401 (Fe-Vici)
First number: station (up to 14); Second number: sample (up to 50)
CTD: water sample
PW: porewater
BW: bottom water
SED: sediment: numbered continuously

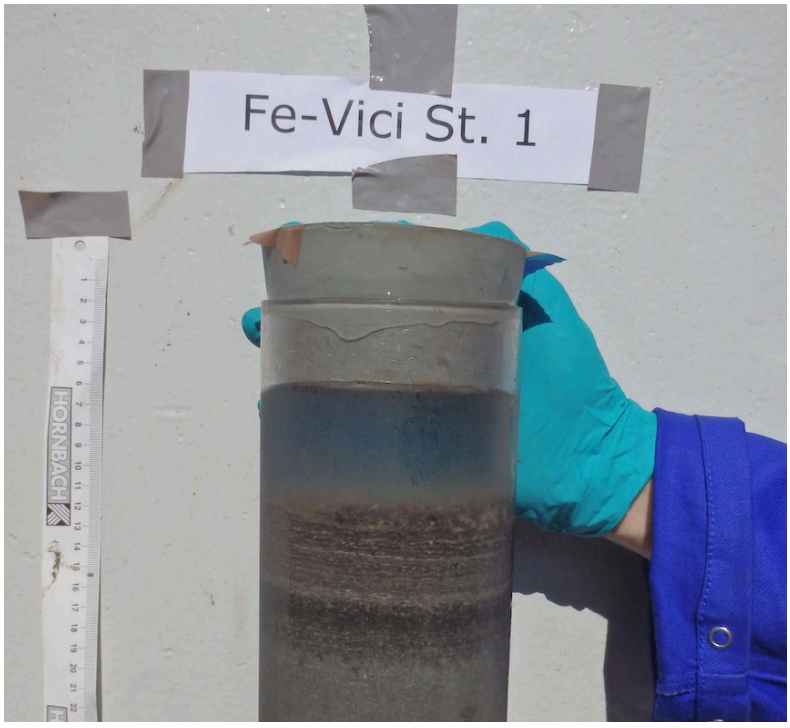

Figure 5. Example of laminated surface sediments (site 1).

3. Day-to-day activities (narrative)

Transit

All cruise participants arrived in Istanbul on **Sunday August 30** and boarded the ship in Istanbul on the morning of **Monday August 31**. The transit through the Bosporus began at 13h on **Tuesday September 1**. Station 1 was reached at about 7 am on

Figure 6. Safety drill in the Bosporus

Day 1 - Wednesday September 2

Ultraclean CTD

Deployment of the CTD at station 1 began at 9h. Bottles were closed according to the CTD sample list (Appendix I). The CTD was on deck around 11h and was directly transferred to the clean container where sampling began as soon as possible.

Figure 7 Deployment of ultraclean CTD

Multicores
Two multicore casts were taken at this station. The weights were: 2 in the central part, 2 times 3 weights on top. The sediments were laminated and contained unit I and part of unit II. The first deployment was only partially successful with several cores being too full, not full enough or leaking water during removal of the cores from the multicorer. The second deployment was completely successful. Cores were distributed according to the sample plan (Appendix II). The station was abandoned at ca. 15h.

Figure 8. Multicore cast collected at station 1

Arrival at station 2 at ca. 19h.

Day 2 - Thursday September 3

Ultraclean CTD
Deployment of the CTD began at 6h. Bottles were closed according to the CTD sample list (Appendix I). The CTD was on deck around 8.30h and was directly transferred to the clean container where sampling began as soon as possible.

In-situ pump deployments
Four deployments, each with 4 pumps, were carried out, with the final 4th deployment (running overnight) being retrieved the next day (Day 3 - Friday September 4) at 6h (see section 7).

Ultraclean CTD
Deployment of the CTD began at 8h. Bottles were closed according to the CTD sample list, which included duplicate samples for total and dissolved metals, water to fill the sediment trap bottles and various sample requests.

Multicores
Three casts were taken (9.30-11h, 11.30-13h, 13.30h-15.00h). Cores were distributed according to the sample plan (Appendix II). Coring was completed at ca. 15h.

Sediment trap retrieval
The sediment trap that was deployed at 214 m in 2014 was retrieved successfully between 15.30 and 16.30h (24 bottles). The position of the three sediment traps on the seafloor was determined and preparations were made to retrieve the traps through dredging.

Day 4 - Saturday September 5

Sediment trap retrieval by dredging
The seafloor was dredged to retrieve the 3 sediment traps lost in 2014. The traps were retrieved successfully from 2100 meter water depth in an operation that was completed at 18h. Preparations were made until 21.30h to allow for deployment of 4 new sediment traps the next day.

Figure 9. Preparations for dredging of the sediment traps

Day 5 - Sunday September 6

Sediment trap deployment
Four sediment traps were deployed at station 2 at depths of 214 m, 448, 932 and 1716 meter (coordinates N 42 53.82', E 30.40.75'). All trap bottles were filled with anoxic water taken previously with the ultraclean CTD. Sampling will start on September 10.

Transit to station 3
Station 2 was abandonned at ca. 16h in the afternoon. After a 5h transit, an ultraclean CTD was taken at station 3 from 21.15 to 23h in the evening.

Transit to station 6.

Day 6 - Monday September 7

Ultraclean CTD

Sampling at Station 6 began early at 6h with a CTD cast that was on deck at a few minutes after 7h.

In-situ pumping

At 7.30h the pumps were overboard and in-situ pumping was performed from 7.45-8.45h.

Lander deployment

A lander was deployed that was at the seafloor at 9.30h.

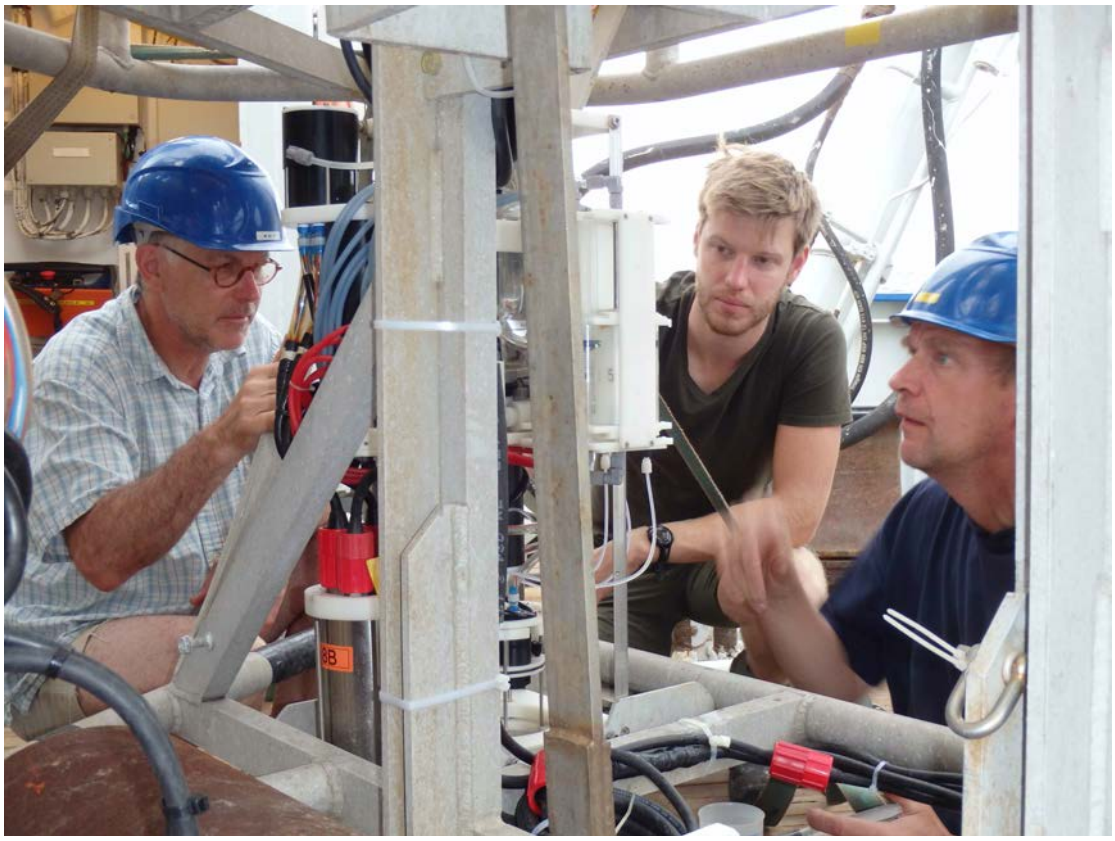

Figure 10. Preparing one of the two landers for a deployment

Multicoring and boxcoring

Two multicore casts were taken and two box cores (for macrofauna). At 13.30h the last box core was on deck. No micro-electrode work was done because of the large number of shells on top of the sediment. No living macrofauna were observed. The lander was retrieved at 17h.

Day 7 - Tuesday September 8

Ultraclean CTD

Sampling at Station 7 began at 8h with an Ultraclean CTD, which came on deck again at 8.45h.

In-situ pumping

A deployment of 4 in-situ pumps was completed at 11h and a lander deployment was then completed at 11.30h.

Multicoring and box coring
Multicoring followed using the weights of station 6. Because the first cast did not penetrate very far into the sediment, the cast (number 1) was not used and the weights were increased to a total of 10 at each side at the top. Cast 2 was then used. The weights were maximized for the second cast (2 more in the central part). Cast 3 was used for rhizon sampling. Two box cores were then obtained for macrofauna (completed at 15.30h).

Lander
The lander was retrieved at 18.45h and transit to station 8 began at 19.00h. Station 8 was reached at 21.15h and the second lander was deployed.

Day 8 – Wednesday September 9

Ultraclean CTD
Sampling at station 8 began at 8h with an ultraclean CTD. The CTD was on deck at 8.45h.

In-situ pumping
A deployment of 4 in-situ pumps was completed at 11h (pumping from 9.45 to 10.45h).

Multicoring and box coring
Multicoring followed using the weights of station 7. A distinct layer of shells was present at the top of the core. The cores were distributed according to plan, however, no micro-electrode profiling was done because of the shells. A novel measurement at this site was the inclusion of Br incubations for bioirrigation rates. The sediment no longer had a sulfide smell upon slicing of the core. Two boxcores were taken and living macrofauna were observed. There were very large shells at the bottom of the core. The sedimentation rate at this site is likely very low.

Lander recovery and deployment
The lander that was deployed the day before was recovered at 14h.

Transit to station 9
The lander was deployed again at site 9 at 19h, directly after arrival.

Day 9 – Thursday September 10

CTD and in-situ pumping
At site 9, no ultraclean sampling was performed. Instead, we used the CTD with Go-flow bottles and 2 in-situ pumps in single deployments. The bottom of the CTD frame was in contact with the sediment and this influenced the composition of the deepest sample, both for the CTD and the in-situ pumped filters.

Multicoring and box coring
Four multicore casts were taken with two breaks for boxcoring and retrieval of the lander. The sediments were highly bioturbated, especially the surface sediment, and a distinct brown surface layer was present at the top of the core, suggesting the presence of a thin layer of Fe-oxides at the surface. At this site, the weights on the multicorer were set at their maximum. Two box cores were taken.

Lander recovery and deployment.
The lander deployed the previous evening at site 9 was recovered at ca. 13.20h. A transit to station 13 followed and a lander was deployed.

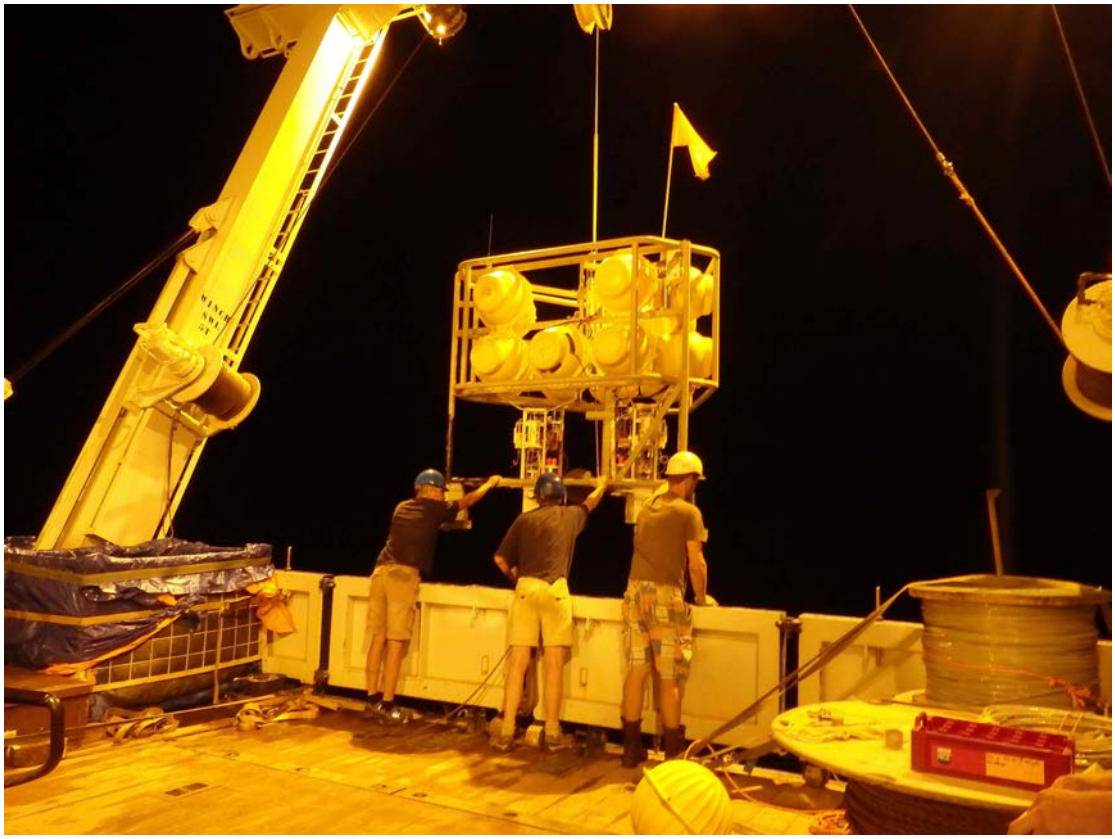

Figure 11. Lander deployment in the evening

Day 10 – Friday September 11

CTD and in-situ pumping

At site 13, no ultraclean sampling was performed. Instead, we used the CTD with Go-flow bottles and 2 in-situ pumps in single deployments. Sampling began at 7h and completed at 8h.

Multicoring and box coring

Three multicore casts were taken at this location followed by three boxcores (1 was unsuccesful). The sediments at this site were also highly bioturbated.

Lander recovery and deployment.

The lander deployed the previous evening at site 13 was recovered at ca. 13.20h. A transit to station 7 followed and a lander was deployed at ca. 19.20h.

Day 11 – Saturday September 12

Ultraclean CTD

The CTD was deployed at station 5 at 6.15h and was on deck again at 7.15h. Station 5 is apparently located near a shipping route; various ships, including military vessels, passed. This is a station where the deeper waters are anoxic and sulfidic.

In-situ pumping

Four pumps were deployed at station 5 and they pumped from 8.00-9.30h.

Multicoring

4 multicore casts were taken; the first was too full and the third did not trigger. The cores from casts 2 and 4 were divided as indicated in the appendix. The ideal weights for this site are: 2 x 2 at the top and 4 in the middle.

Lander operations
A lander was deployed at station 5 at 13.30h. A transit to station 7 followed where a lander was retrieved from the seafloor at ca. 15.30h. After a transit to station 14, a lander was deployed there again at 19h.

Day 12 – Sunday September 13

Ultraclean CTD
The CTD was deployed at station 14 at 6.15h. The water depth at this station was slightly deeper than estimated (112 m). The weather was rather rough all night and waves continued to be high during the whole day. The first part of the CTD profile (20 m) on the downcast was not recorded.

Multicores and boxcores
Two multicores were taken between 8h and 9h. Two boxcores were then taken. The sediments were very similar to those obtained at stations 6 and 7. In contrast to station 6, the fauna was alive at this site. Two boxcores were taken, and boxcoring was completed at 9.25h.

Lander retrieval – part I
The lander from site 14 was retrieved at ca. 10.30h.

In-situ pumping
The ship then moved 500m up current and in-situ pumping using 4 pumps was then started. The pumps were back on deck at 13.15h.

Lander retrieval and deployment – part II
A transit to site 5 followed and the lander was retrieved at ca. 14.30h
A transit to site 6 followed and the lander was deployed. Transit to site 4.

Today was Sharyn's birthday and there was cake in the morning and a party in the bar in the evening.

Day 13 – Monday September 14

Ultraclean CTD
The CTD was deployed at station 4 at 8h and was on deck at 9.10h. It was Wytze's birthday and there was cake in the morning.

Multicores
Two multicore casts were taken between 10h and 11.15h and a total of 13 cores were processed as indicated in the Appendix.

Transit to station 6 and retrieval of lander.
A lander was retrieved from station 6 at 13.30h.

Transit to station 4 and deployment of lander
A lander was deployed at station 4 at 17h.

Day 14 – Tuesday September 15

Ultraclean CTD

The CTD was deployed at station 17 at 8h and was on deck again at 10h.

In-situ pumping

Four pumps were deployed at station 17 and they pumped from 10h-12h.

Hasssan celebrated his birthday with cake and coffee.
The cruise picture was taken at ca. 10.15h

After a transit to station 4, a lander was retrieved from the seafloor at 13.30h. Because of the soft sediment the lander overfilled and the syringes were clogged with mud. Only the bottom water samples were analysed.

END OF SAMPLING at ca. 14h. Start of transit to Varna & Barbecue

Figure 12. Preparing a sediment trap for deployment

4. NUTRIENTS – Fe-VICI Cruise 64PE401 on R.V. Pelagia

Karel Bakker[1] and Sharyn Ossebaar[1]
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Summary
Nutrients were analysed in two thermostated lab containers equipped with QuAAtro Gas Segmented Continuous Flow Analysers, measuring approximately 4600 samples during the cruise for the different parameters. Measurements were made simultaneously on three channels for Ammonium, Nitrite, and Nitrate with Nitrite together. In the other lab container, Phosphate and Silicate were simultaneously analysed, and Dissolved Inorganic Carbon (DIC) and Sulphide were each separately measured. All measurements were calibrated with standards diluted in low nutrient seawater (LNSW) or artificial seawater in the salinity range of the stations of the Black Sea at approximately 20$^{o}/_{oo}$.

Equipment and Methods
Sample Handling
The oxic and anoxic samples were collected in 60ml high-density polyethylene syringes with a three way valve to make it possible to sample air free water from the ultra-clean PVDF bottles of the CTD. The syringes with a three way valve were first rinsed three times with a small amount of the sample taken directly from the CTD (Ultra Clean & Rosette) bottles before being completely filled.
After sampling on deck, the samples were processed immediately in the lab; samples were filtered over 0.2µm and instantly sub-sampled for Sulphide in a glass vial already containing 40µl 1N NaOH and filled with a round meniscus before being capped and stored upside down in a refrigerator and typically analysed within 2 hours. DIC was sampled as the second sub-sample in the same way as Sulphide, however without any addition of hydroxide and normally measured within two hours after sampling. Two more vials made out of high density polyethylene, also known as 'pony-vials', were used for storing NH4 and NO3 plus NO2 as one sample and the other containing 15µl 5N HCl for storing the PO4 combined with Si sample. The NH4 and NO3 plus NO2 samples were simultaneously measured in the other lab container within 3 hours of sub sampling.
Pore-water samples were stored in the dark at 4°C until analysis and were generally measured within 12 hours after being processed. Incubation experiment samples from on-board flux experiments and in-situ experiments from the benthic lander were also measured in the same way as the CTD samples. Prior to analysis, all samples were brought to lab temperature in about one to two hours. To avoid gas exchange and evaporation during the runs with Sulphide, DIC and NH4 analysis, all vials including the calibration standards were covered with 'parafilm' under tension before being placed into the auto-sampler, so that the sharpened sample needle easily penetrated through the film leaving only a small hole. The QuAAtro uses an LED instead of a lamp as a light source as it is not affected by the movement of the ship giving a stable reading. The typical sampler rate of 60 samples per hour was used. Calibration standards were diluted from stock solutions of the different nutrients in 0.2µm filtered LNSW diluted with de-ionised water to obtain approximately the same salinity as the samples and were freshly prepared every day. This diluted LNSW was also used as the baseline water for the analysis in between the samples. The LNSW is surface seawater depleted of most nutrients. Each run of the system had a correlation coefficient of at least 0.9999 for 10 calibration points, but typical 1.0000 for linear chemistry, however the DIC and HS- were fitted using a quadratic calibration curve. The samples were measured from the lowest to the highest concentration in order to keep carry-over effects as small as possible, i.e. from surface to deep waters. Concentrations were recorded in 'µmol per liter' (µM/L) at the

container temperature of 22.5°C for the DIC, HS, PO4, Si container and at 23°C for the NH4, NO3, NO2 container. During the cruise, a freshly diluted mixed nutrient standard, containing silicate, phosphate and nitrate (a so-called nutrient cocktail), was measured. The cocktail sample was used as a guide to monitor the performance of the standards.

Pore-water was collected under anoxic conditions in glove boxes under nitrogen atmosphere and sub sampled for H2S, DIC, N, PO4 and Si. For the PO4 pore-water samples, an extra addition of 4µl 5N HCl per 1ml of sample was added to compensate for high DIC background levels, expected up to 20mM DIC, to keep the pH in between 1 and 2 to prevent any form of iron-phosphate precipitates. Sulfide samples were diluted using a dilution factor of 4 made with anoxic demineralised water containing 8ml 1N NaOH/L. DIC samples were also diluted using a dilution factor of 10 with anoxic demineralised water containing 24.5g NaCl/L, this ensuring that the samples remained with the same ionic strength as the Black Sea.

Analytical Methods
The colorimetric methods used are as follows:

Ortho-Phosphate (PO_4) reacts with ammonium molybdate at pH 1.0, and potassium antimonyltartrate is used as a catalyst. The yellow phosphate-molybdenum complex is reduced by ascorbic acid and forms a blue reduced molybdophosphate-complex which is measured at 880nm (Murphy & Riley, 1962).

Ammonium (NH_4) reacts with phenol and sodiumhypochlorite at pH 10.5 to form an indo-phenolblue complex. Citrate is used as a buffer and complexant for calcium and magnesium at this pH. The blue color is measured at 630nm. Koroleff, 1969 and optimized by W. Helder and R. de Vries, 1979.

Nitrate plus Nitrite (NO_3+NO_2) is mixed with an imidazol buffer at pH 7.5 and reduced by a copperized cadmium column to Nitrite. The Nitrite is diazotated with sulphanylamide and naphtylethylene-diamine to a pink colored complex and measured at 550nm. Nitrate is calculated by subtracting the Nitrite value of the Nitrite channel from the 'NO3+NO2' value. (Grasshoff et al, 1983)
Nitrite (NO_2) is diazotated with sulphanylamide and naphtylethylene-diamine to form a pink colored complex and measured at 550nm. (Grasshoff et al, 1983)

Silicate (Si) reacts with ammonium molybdate to a yellow complex and after reduction with ascorbic acid, the obtained blue silica-molybdenum complex is measured at 820nm. Oxalic acid is added to prevent formation of the blue phosphate-molybdenum (Strickland & Parsons, 1968).

Dissolved Inorganic Carbon (DIC):
Samples are acidified online after being oxidised by H_2O_2 to prevent H_2S being released before entering the silicon dialyser whereby the formed CO_2 is dialysed to a secondary flow. This secondary flow contains a slightly alkaline phenolphthalein solution giving a pink colour. The more CO_2 that is dialysed, the lower the pH and therefore some discolouration of the pink reagent is observed. This decolouring is measured at 520nm and is an inverse chemistry spectrophotometer method described by Stoll, Bakker, Nobbe and Haesse, 2001.

H_2S:
To keep the samples in the S_2^- form under alkaline conditions, a small aliquot of NaOH is added. The Hydrogen Sulfide in the sample reacts with para-aminodimethylaniline and ferric chloride to yield methylene blue which is measured at 660nm as described by Grasshof, K., 1969.

Calibration and Standards

Nutrient primary stock standards were prepared at the NIOZ as follows;
Phosphate: by weighing Potassium dihydrogen phosphate in a calibrated volumetric PP flask to make 1mM PO4 stock solution.
Silicate: by weighing Na_2SiF_6 in a calibrated volumetric PP flask to 19.84mM Si stock solution.
Ammonium: by weighing Ammonium Chloride in a calibrated volumetric PP flask to make 1mM NH4 stock solution.
Nitrate: by weighing Potassium nitrate in a calibrated volumetric PP flask set to make a 10mM NO_3 stock solution.
Nitrite: by weighing Sodium nitrite in a calibrated volumetric PP flask set to make a 0.5mM NO_2 stock solution.
DIC: by weighing Na_2CO_3 stock in a calibrated volumetric PP flask set to make a 200mM stock solution.
S_2^- : by weighing Na_2S in 0.5N NaOH set to make a 50mM Sulphide stock solution.

All standards were stored at room temperature in a 100% humidified box apart from the S_2^- standard which was stored in the refrigerator. The calibration standards were prepared daily by diluting the separate stock standards, using three electronic pipettes, into four 100ml PP volumetric flasks (calibrated at the NIOZ) filled with diluted LNSW. The blank values of the diluted LNSW were measured onboard and added to the calibration values to get the absolute nutrient values. In the case of Sulphide, calibration standards were made using anoxic demineralised-water with an addition of 0.8ml 1N NaOH/100ml to keep the Sulphide under alkaline conditions.

Statistics
Quality Control
Our standards have already been proven by inter-calibration exercises from ICES and Quasimeme, and last year the RMNS exercise organised by MRI, Japan.
Our cocktail standard was regularly measured and its value remained stable for all nutrient measurements during the cruise.

Mean Detection Limits
The method detection limit was calculated during the cruise using the standard deviation of ten samples containing 2% of the highest standard used for the calibration curve and multiplied with the student's value for n=10, thus being 2.82. (M.D.L = Std Dev of 10 samples x 2.82).

	µM	Used measuring ranges µM:
PO4	0.007	3
SiO2	0.02	80
NH4	0.015	10

NO3+NO2	0.006	15.5
NO2	0.003	0.5
HS-	0.26	400

Further Remarks

It is suggested that through diluting the samples by means of electronic pipettes, one for the sample and one for the dilution water, a small error of maximum 1.0% could be introduced.

Interference on the NH4, PO4 and Si channels was seen when analysing undiluted anoxic samples. Tests were carried out to determine the best procedure with anoxic samples while using these standard oxic sea water methods. It was concluded that for the CTD anoxic samples, a dilution factor of 11 was used for NH4, and for PO4 and Si samples were acidified (15µl 5N HCl per 5ml of sample) and then diluted using a factor of 5. For porewater samples the highest possible dilution factor that was analytically accurate was used, thus using a 101 dilution factor for NH4 and a factor of 11 for NO2, NO3, PO4 and Si. However, for NO2, when using this 101 dilution factor, the measured values were near the analytical detection limit of the channel. The reported pore-water results took into account the dilution steps that were made in the glovebox prior to analysis. The CTD data reported on board is currently preliminary as minor corrections are possible to be made due to re-calculation of the sub surface waters close to the detection value of the used LNSW.

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Figure 13. Sharyn and Karel in the two auto-analyser labs

5. Water column sampling incl. ultraclean CTD

Marie Séguret[1], Amy Andersen[2], Nikki Dijkstra[1], Silke Severmann[2]
[1]Utrecht University, [2]Rutgers University

Sampling of the water column was carried out by using an ultra-clean trace metal conductivity-temperature-depth (CTD) rosette consisting of an all-titanium frame with 24 sample bottles of 24 L each made of PVDF plastic (abbreviated uc CTD, made at NIOZ), arranged as two rows of twelve bottles, see Figure 14. Samples for dissolved components and particulates (>0.02 and 0.2 um) were collected (Table 4)

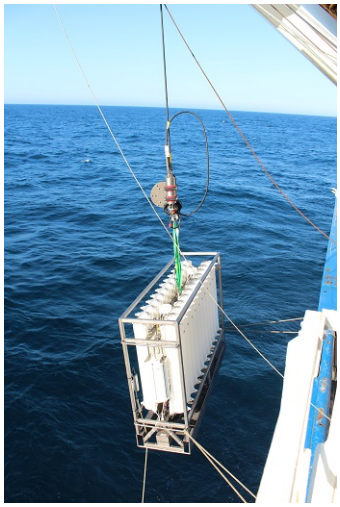
a)  b)  c) 

Figure 14. uc CTD a) upper view, b) deployment, c) clean CTD container with the uc CTD in the background.

A Kley France winch was used to deploy the uc CTD to deep waters by a 17.7 mm diameter Kevlar hydrowire with seven independent internal signal/conductor cables (Cousin Trestec S.A.). Sampling of the uc CTD occurred in a class 100 clean-room container specially designed to get the uc CTD in and out rapidly to avoid contamination. The container was also equipped with an anti-room separating the main part of the container of the outside by a plexiglass door to keep dust and other sources of contamination outside. Moreover the designed container allowed simultaneous sampling of 12 bottles covering both sides of the uc CTD. To prevent contamination and to keep the uc CTD safe and secure, the uc CTD was at all times placed inside the clean air container (meeting class 100 clean-room specifications) when not in use during casts.

The water column structure of Station 1 to 9 and Stations 13, 14 and 17 was analyzed and 24 samples were in general taken with the uc CTD with the exception of the shallow stations Station 9 and Station 13 that were collected with the conventional CTD. Station 2 was sampled on two consecutive days to answer the various sample requests but also for the GEOTRACES requirements, cast 1 on day 1 and cast 2 on day 2. Station 8 was sampled once but due to the lack of depth, duplicate bottles were taken and sampled as cast 1 referring to the odd numbers of the bottles of the uc CTD, eg 1, 3, 5 and cast 2 referring to the even numbers of the bottles, eg 2, 4, 6. Special emphasis was put on the transition zone between oxic and sulfidic waters (‘redoxcline’), which is prominent in the Black Sea basin. In this transition zone, water was collected at 5 m intervals. In addition, several samples below and above the redoxcline were collected. Anoxic samples were collected following the PHOXY cruise procedure, explained in the “Collection of trace metals” paragraph below. The sensors on the uc CTD collected continuous profiles of key physicochemical water column properties:

conductivity, temperature, fluorescence, beam transmission and dissolved O2, the latter with a Seabird oxygen electrode. The water collected in the uc CTD bottles at various depths was immediately sampled for trace metal analysis but also sampled and analyzed on-board for NH4+, NO3-, NO2-, PO43-, HS- and DIC with Quattro gas-segmented continuous flow analyzers.

a)  b) 

Figure 15. Filtration setup for the 0.2 um filtration and b) processing of the anoxic 0.2 um filter by Amy.

Figure 16. The clean sampling team

Table 4. Station, number of depths sampled and amount and type of samples collected.

Station	Depths sampled	Number and type of samples
Station 1	24 depths	144 total and dissolved
Station 2	24 depths	228 total and dissolved, 12 particulate
Stat. 2 cast 2	24 depths	116 total and dissolved, 62 particulate
Station 3	24 depths	118 total and dissolved
Station 4	24 depths	178 total and dissolved, 36 particulate
Station 5	24 depths	144 total and dissolved, 36 particulate
Station 6	24 depths	192 total and dissolved, 39 particulate
Station 7	14 depths	84 total and dissolved, 36 particulate
Station 8	12 depths	84 total and dissolved, 38 particulate
Stat. 8 cast 2	12 depths	84 total and dissolved
Station 9	5 depths	20 total and dissolved, 7 particulate
Station 13	8 depths	32 total and dissolved, 10 particulate
Station 14	24 depths	120 total and dissolved, 36 particulate
Station 17	24 depths	168 total and dissolved, 24 particulate

6. In-situ pump sampling incl. sample overview

Nikki Dijkstra[1], Silke Severmann[2]
[1]Utrecht University, [2]Rutgers University

In situ pumping was performed at 10 sites, when necessary using multiple casts. Four McClane pumps were used (3 x WTS-LV; 1 x WTS LV-Dual Filter). Each pump was programmed before deployment and material was collected on 0.8 um Supor filters (142 mm diameter). The programming consists of a sample volume, an initial flow rate, a minimum flow rate, a time limit, the pump data period and a count-down timer. The filters were collected immediately after the pumps were retrieved. The filters were photographed from above, placed in a petri-dish and packed in plastic bags (oxic samples) or aluminum bags (anoxic samples, handled in a glovebag, flushed with nitrogen) bags and stored at -20 C.

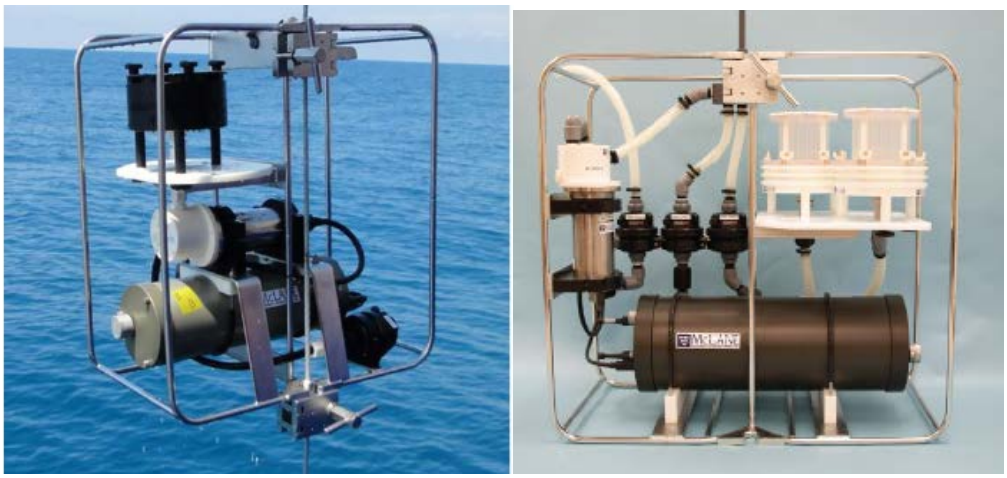


Figure 17. McClane pumps: Left: WTS-LV, Right: WTS-LV-DF

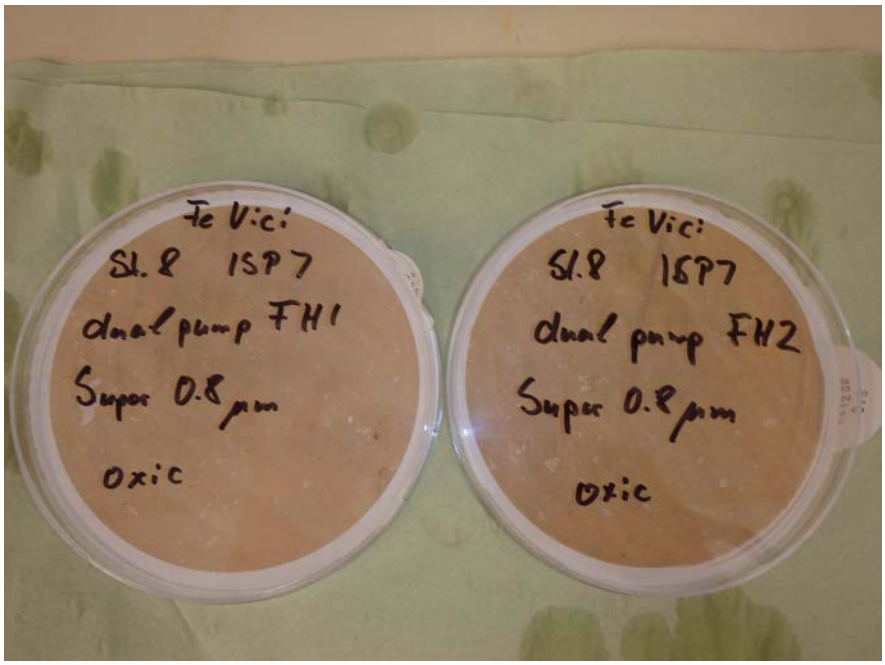


Figure 18. Example of filters collected through in-situ pumping (station 8, dual filter pump)

7. Micro-electrode profiling

Martijn Hermans, Utrecht University

High resolution depth profiling was performed with microsensors for O_2 (50-µm), H_2S (50-µm) and pH (100-µm) on multicores. These microsensors were connected to a four-channel Microsensor Multimeter (Unisense A/S). Calibrations for the sensors were performed daily prior to retrieving the multicores on deck.

O_2 calibration

The O_2 sensor was calibrated in seawater by using the calibration chamber (Unisense A/S, Denmark). The first calibration point was made in air-saturated seawater (100% saturation) by vigorously pumping air in the calibration chamber with an aquarium pump. The second calibration point was made by flushing the calibration chamber with nitrogen for about 10 minutes.

H_2S calibration

The H_2S sensor is light sensitive, since the electrolyte in the sensor gets photo-degraded by high light intensities, resulting in a false high background signal compared to the signal in the dark. Therefore, all calibrations were done in a dark room. Na_2S standards were used for a 5 point calibration (0, 5.031, 12.568, 25.106 and 50,086 µM). The Na_2S stock solution (≈ 0,01M ΣH_2S) was prepared anoxically by dissolving ~0,24 g Na_2S x 9 H_2O in 100mL of N_2-flushed water and subsequent storage in a nitrogen purged glovebox. Since the H_2S (50-µm) sensor detects the partial pressure of H_2S gas, which is only a fraction of ΣH2S, the calibration was performed quickly in N_2-flushed acidified seawater (pH ~3.5) to ensure there was no introduction of oxygen and that all ΣH_2S was available as H_2S.

pH calibration

Calibrations for pH were performed with three NIST (pH 4, 7 and 10) buffers (Hach) and a TRIS buffer to correct for salinity effects. The pH is reported on the total scale**.**

Figure 19. Micro-electrode profiling set-up

Measurements
The table below gives an overview of the sites that were selected for high resolution depth profiling and the different analyses that were performed on these sites. The cores were measured three times. There were no significant changes over time for the oxic sites (St. 9 and St. 13) in contrast to the anoxic sites (St. 1, 2, 4 and 5) which did show a significant change over time.

Table 5. Overview of micro-electrode profiles

Station	mbs	O_2	H_2S	pH	Temp	Date
St. 1	**1596**		✓	✓	✓	2-9-2015
St. 2	**2107**		✓	✓	✓	4-9-2015
St. 4	**380**		✓	✓	✓	14-9-2015
St. 5	**190**		✓	✓	✓	12-9-2015
St. 9	**27**	✓	✓	✓	✓	10-9-2015
St. 13	**39**	✓	✓	✓	✓	11-9-2015

The graphs below show the ΣH_2S and pH profiles for St. 5, 4, 1 and 2. The ΣH_2S concentrations increase with depth in the sediment at all four stations. ΣH_2S concentrations are highest at the deepest station. pH decreases with depth in the sediment. The deeper stations (St.1 and St.2) are characterised by the lowest pH.

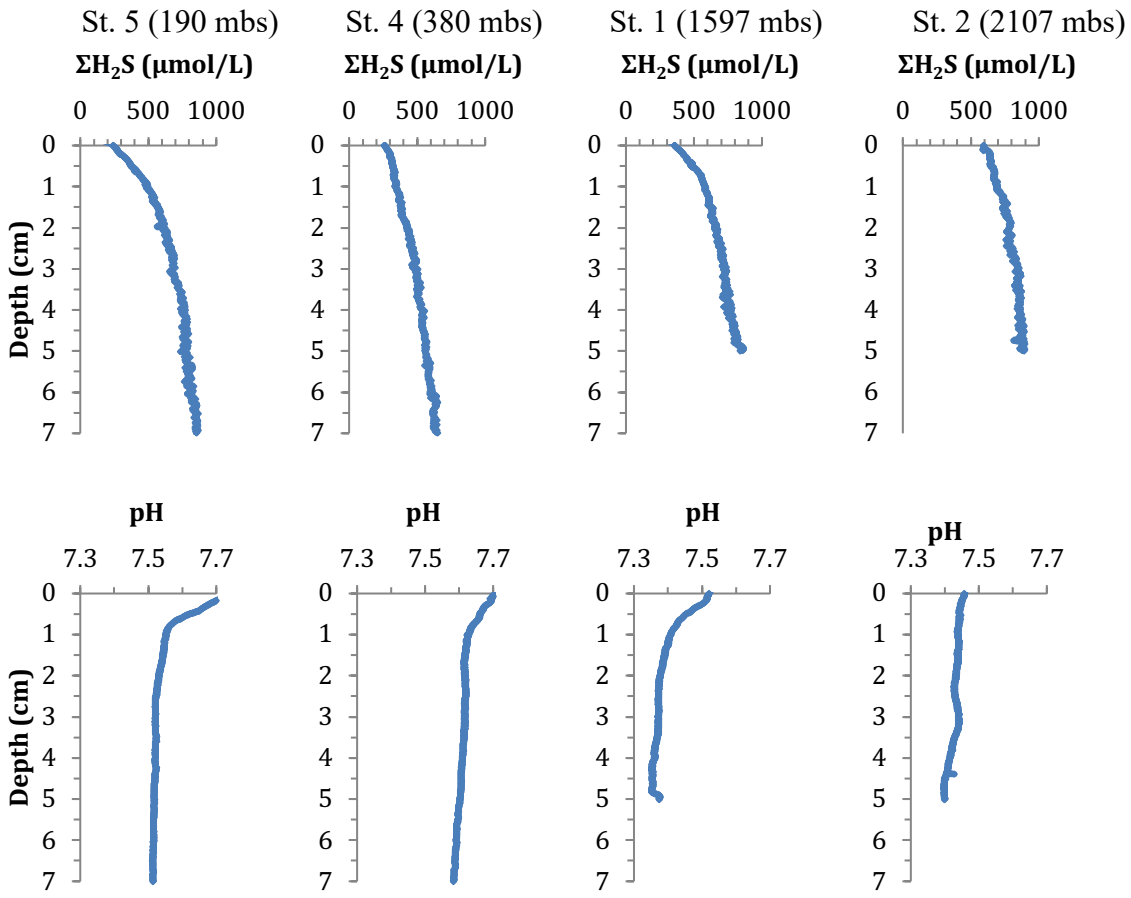


Figure 20. Micro-electrode profiles of sulfide and pH for stations 5, 4, 1 and 2.

8. Porewater and sediment collection

Niels van Helmond , Peter Kraal, Wytze Lenstra, Silke Severmann

At selected sites, pore waters were extracted from multicores for analysis of dissolved inorganic constituents. In combination with solid-phase analyses, these data will be used to assess the rates of diagenetic reactions in the sediments, and vertical fluxes of dissolved constituents between the sediments and the water column.

Sediment slicing and pore water sampling

Ten cm diameter multicores were recovered with an Oktopus multicoring apparatus (www.oktopus-mari-tech.de). Twelve cores were recovered per cast. Each core contained 30-60 cm of sediment, plus overlying water. Generally, sites below the redoxcline (1, 2) yielded longer cores, due to the softer sediment texture relative to the redoxcline (6) than oxic sites (7 - 14). The weighting system of the multicore was adjusted between oxic and anoxic sites to achieve optimum sediment recovery.

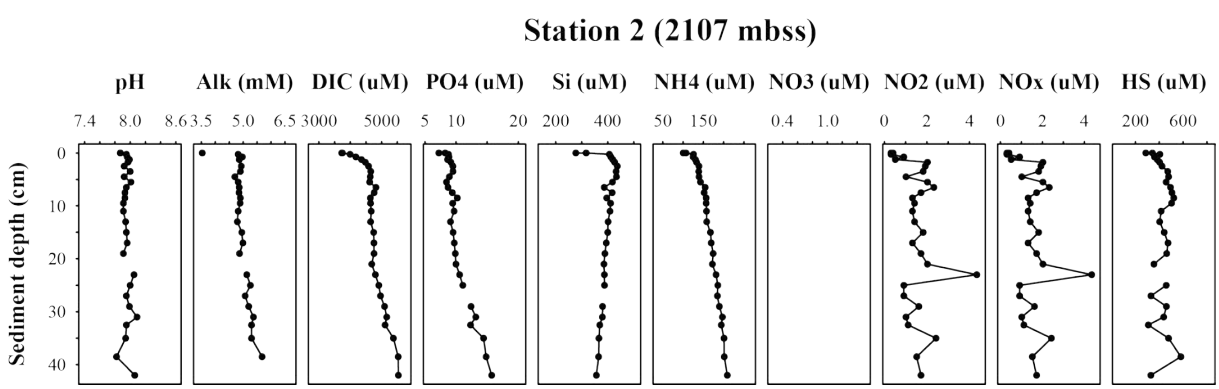
On deck, one core from each cast was stoppered at the top and base and transported to a temperature-controlled container (temperature set to match bottom water, 8 – 12 °C). Duplicate bottom water samples were extracted using 20 mL syringes positioned in the overlying water ~10 cm from the sediment surface. The filled syringes were transferred directly to a nitrogen-filled glovebox for later subsampling. The remaining water was drained from the core until ~2 cm of overlying water was left, and the core was inserted vertically into a nitrogen-filled glovebox inside the temperature-controlled container. The last overlying water was removed with a syringe and the core was sliced according to a general scheme, which was adjusted when needed to optimally separate contrasting sediment intervals:

Table 6. Sediment slicing intervals

Interval	Resolution
0–2 cm	0.5 cm
2–10 cm	1 cm
10–30 cm	2-3 cm
30cm–core base	4 cm

Porewater was collected through centrifugation. For sampling pore water methane in the multicores, one core from each cast was pre-drilled with 2 cm diameter holes and samples were taken with cutoff syringes.

Preliminary Results



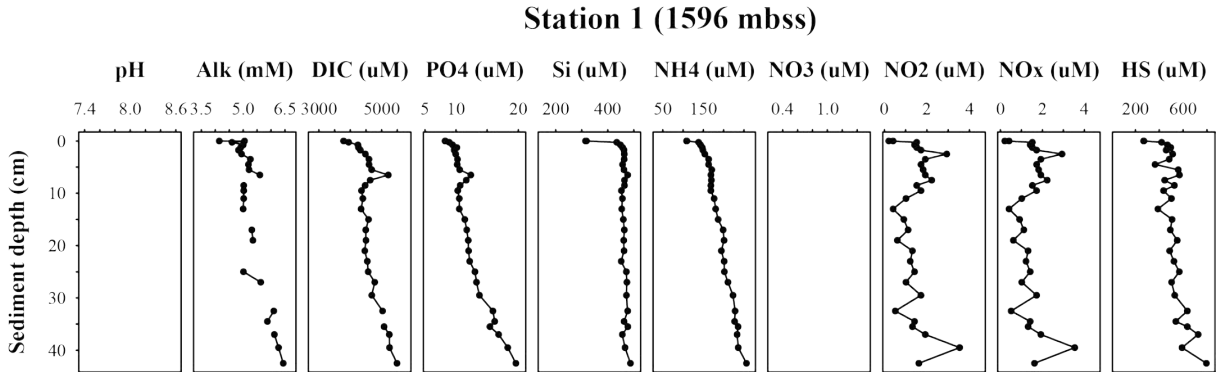


Figure 21. Porewater profiles for stations 2 and 1.

Multicores sliced for 210Pb dating
One MC per station was sliced for 210Pb dating. The core was sliced using the hydraulic core extruding device onboard the *RV Pelagia*.

9. Sediment trap recovery and deployment

Nikki Dijkstra[1], Peter Kraal[1], Wytze Lenstra[1], Ruud Groenewegen[2]
Utrecht University[1], NIOZ[2]

Sediment traps – recovery and deployment
During this expedition, there were two objectives regarding sediment traps. The first was to recover the lower part of a mooring (including three sediment traps deployed at 214, 448, 932 and 1716 mbss) that was deployed June 18, 2013. It sank to the bottom at 2013 station PHOX 2 during a partly failed recovery attempt in September 2014 with the R/V Mare Nigrum. The second objective was to deploy a mooring with four sediment traps (214, 448, 932 and 1716 mbss) at 2015 Station 2.

Sediment trap recovery
On September 5, 2015 a successful dredging attempt was undertaken to recover the 2013 mooring. Based on the location of the releases at the bottom of the mooring (determined by acoustic communication between a deck unit and the mooring) and calculation of the orientation of the complete mooring based on current velocities, a transect perpendicular to the estimated location of the mooring was determined. Using a weight, a construction with four ankers was dragged over the seafloor on the transect. The cable of the mooring was caught by the weight, and the complete mooring was brought on board. The carrousels of all three sediment traps contained bottles that clearly contained material, and the carrousels had all returned to their starting positions, indicating successful deployments. Analysis of the data loggers of the motors that turn the carrousels later confirmed this. The 24 sediment trap bottles were immediately removed from the carrousel and brought into a temperature-controlled container (9 °C) where they were packed in aluminum laminate bags that were flushed with N_2 prior to cool storage (4 °C).

Sediment trap deployment
On September 6, 2015 four sediment traps were deployed: at 1716 (Trap 1), 932 (Trap 2), 448 (Trap 3) and 214 (Trap 4) mbss. The 24 bottles for each trap were filled with water from the corresponding depth taken a day earlier with the ultra-clean CTD sampling device. The water was kept in the CTD bottles for as long as possible, then used to fill the 250 mL trap bottles,

which were capped and left in a temperature-controlled container up to the moment they were screwed into the trap carrousels.
Sampling started on September 10, 2015 and will continue for 24 times 15 days. The final sample will be taken on September 4, 2016.

10. Lander deployments

Rob Witbaard[1], Ruud Groenewegen[1], Wytze Lenstra[2]
NIOZ[1], Utrecht University[2]

The NIOZ ALBEX landers were deployed at 7 stations (Table 7). A total of 9 deployments were made. Station 6 and 7 were repeated as the first deployments were of insufficient duration.

Table 7. Overview of lander deployments

Date of deployment	Station	Deployment time
7 Sept	6	6 hours
8 Sept	7	6 hours
8 Sept	8	12 hours
9 Sept	9	12 hours
10 Sept	13	12 hours
11 Sept	7	12 hours
12 Sept	5	12 hours
12 Sept	14	12 hours
13 Sept	6	12 hours
14 Sept	4	12 hours

Figure 22. Benthic lander

Each lander was equipped with 3 measurement chambers with a surface area of 144 cm^2. The overlying watervolume was determined by the penetration depth of the chamber into the sediment. This depth is controlled by a resistivity probe mounted on the chamber. Watercolumn height was preset at 12 cm, meaning that the volume of overlying water was 1.72 liter.
After the deployments, waterheight was measured to check for deviations. At the end of each deployment winkler samples were taken from each chamber to get a first impression of the oxygen consumption rate. For this, the oxygen concentration in the measurement chamber at

the end was compared to the oxygen concentration of the overlying bottomwater as sampled by the CTD-rosette.
Bottom surface area of each box was photographed and sediments sieved over 0.500mm screen for fauna biomass and species determinations (Adrian Teaca).
During each measurement water samples were taken from the inside of the chamber as well as from the outside. Both samples were taken simultaneously.The first sample was taken 2 hours after chamber closure. The second sample was taken 2 hours after the first sample and all following samples were taken with a 3 hour interval. These water samples were processed by Wytze Lenstra to determine fluxes of metals and nutrients out of the sediment.

Oxygen consumption rates in the measurement chambers were measured directly with JFE Advantech optodes. These instruments measured in burst mode at a 1 minute interval. Based on these measurements a first estimate of the metabolic activity of the sea floor was made. This estimate is based on the decrease of the oxygen concentration over time.

The two coastal stations have a oxygen consumption rate which varies between 19 and 25 mmol $m^{-2}.day^{-1}$ The oxygen consumption rates at the deepest hypoxic stations ranged from 0.3 to 0.5 mmol $m^{-2}.day^{-1}$. A summary of the results is given in the figure below.

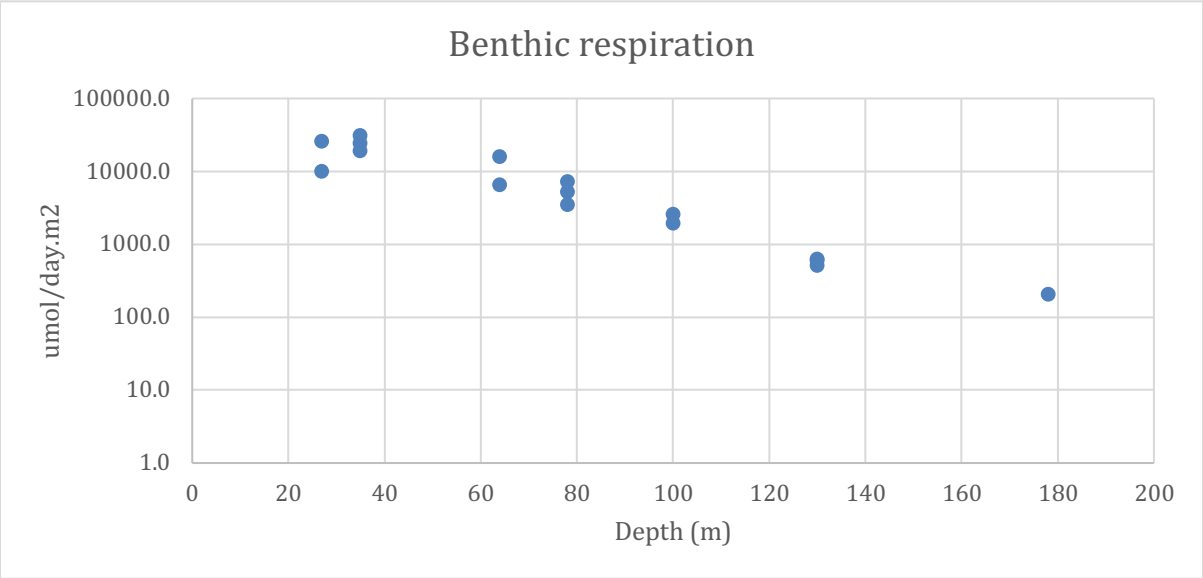


Figure 23. Benthic respiration as a function of water depth (preliminary results)

As an example, the dissolved Si concentration in a benthic lander incubation is shown below for station 5. The results indicate a release of Si from the sediment to the overlying water.

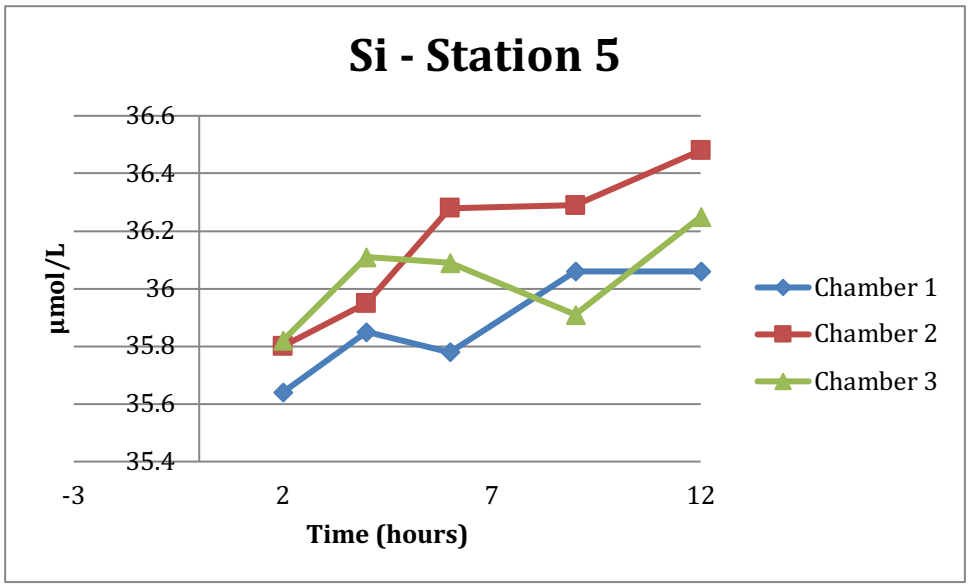


Figure 24: Results of benthic lander (Station 5). Silica was measured in the overlying water during an incubation of 12 hours. Concentrations of dissolved silica increased in all three chambers.

11. Macrofauna

Dr. Adrian Teacă, National Research and Development Institute for Marine Geology and Geoecology – GeoEcoMar, Constanta, Romania

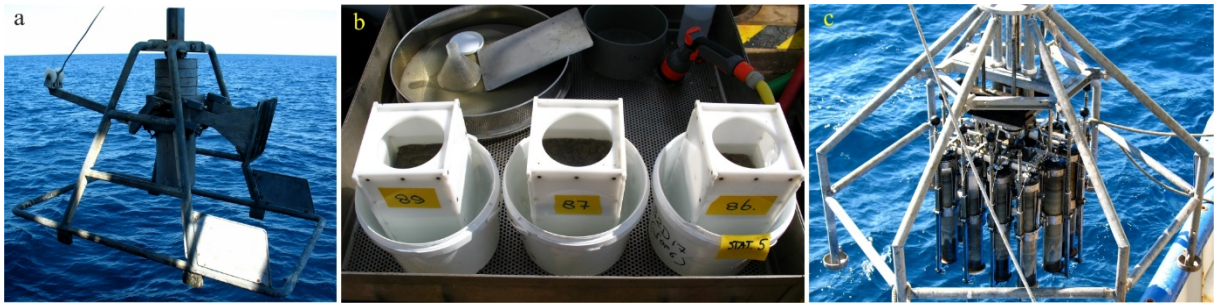
Report on the macro visual observations of macrozoobenthos community performed during the sampling program carried out on the Romanian continental platform of the Black Sea

A total number of 32 samples have been retrieved, out of which 12 macrozoobenthos (MZB) samples collected by means of Box Corer and 18 ones by lander box, and additionally, 2 meiobenthos samples by means of Multicorer.

1. a. Box corer and Lander MZB sampling and treatment procedure

In each station, two MZB samples were taken using the Box Corer (31 cm diameter of collecting cylinder, 0.0754 m^2 area, conversion factor for estimation of abundance of species at 1 square meter: 13.25) and three of them using the Lander Boxes (12x12 cm, 0.0144 m^2, 69.77 conversion factor to 1 square meter). The seawater overlying the sediments contained in the collecting cylinder and the lander boxes has been aspirated with a hose and then filtered through the 0.5 mm sieve.

Figure. 25. On board MZB sampling equipment: a – Box Corer, b – collecting boxes of the Lander, c – multicorer.



- The surficial part of the sediments collected within the **Box Corer** has been cut off in order to collect the biological material. Depending either of substrate type or the habitat, the thickness of the layer collected varied between 5 cm (in stations from depths greater than 100 m) and 35 cm (in coastal stations, where the fine terrigenous muds dominated). Subsequently, the part of sediments remained within the collecting tube has been double-checked in order to eliminate the possiblity of overlooking some polychaets or deep burrowing mollusks that could've been escaped from being accounted before.
- The samples from the **Lander** have been collected integrally.
- All MZB samples (Box Corer and Lander) were pre - washed on-board through 0.5 mm mesh size sieves, preserved and stored for subsequent laboratory analyses. A macroscopic screening of the samples for general characterization of benthos composition and type of sediments was also done on-board. In case of prevalence of shelly coarse sediments fraction within the samples (e.g., *Modiolula phaseolina* broken shells), after removal of fine sediments fraction by washing, the former has been sorted again to collect all the animals that could have been retained within.

1. b. Multicore meiobenthos sampling and treatment procedure

The meiobenthic samples have been collected within the Black Sea anoxic area (e.g., St. 05). Thus, the top 5 cm layer of sediments of the multicore tube samples have been taken out using a hydraulic extruder. Previously, the exceeding water within the tubes has been removed through a hose and then filtered and collected. The samples have been immediately preserved and stocked integrally in plastic containers.

2. Preservation

The material (including the matrix sediments remained after washing and living organisms) was preserved with 4% formaldehyde buffered and stored in plastic jars according to standard methods (Bubnova, Kholikova, 1980; Volodkovich, 1980).

Table 8. The list of MZB sampling stations

No. crt.	Date	Station ID	Depth, m	Coordinates		Box Corer (macro-benthos)	MUC (meio-benthos)	Lander	Habitat description
				Latitude	Longitude				
1	07.09.2015	06	~130	43°42.8`N	30°05.1`E	2	-	3	Deep circalittoral suboxic calcareous mud (periazoic zone with shelly mud constitute of *Modiolula phaseolina* shells and fero-manganese concretions)
2	08.09.2015	07	~78	43°53.8`N	29°58.6`E	2	-	3	Deep circalittoral oxic mixed sediments (shelly mud) with *Modiolula phaseolina, Terebellides stroemii* and *Amphiura stepanovi*
3	09.09.2015	08	~64	44°14.7`N	29°43.4`E	2	-	3	Deep circalittoral oxic mixed sediments (shelly mud) with *Modiolula phaseolina, Terebellides stroemii* and *Amphiura stepanovi*

No. crt.	Date	Station ID	Depth, m	Coordinates		Box Corer (macro-benthos)	MUC (meio-benthos)	Lander	Habitat description
				Latitude	Longitude				
4	10.09.2015	09	~27	44°34.93`N	29°11.38`E	2	-	3	Shallow circalittoral coastal terrigenous mud with *Mya arenaria* and *Upogebia pusilla*
5	11.09.2015	13	~40	44°36.4`N	29°27.72`E	2	-	3	Shallow circalittoral terrigenous mud with *Melinna palmata*
6	12.09.2015	05	~180	43°42.6`N	30°06.1`E	-	2	-	Deep circalittoral anoxic mud with surficial "fluffy layer"
7	12.09.2015	07L (B)	~78	43°53.8`N	29°58.6`E	-	-	3	Deep circalittoral oxic mixed sediments (shelly mud) with *Modiolula phaseolina, Terebellides stroemii* and *Amphiura stepanovi*
8	13.09.2015	05	~180	43°42.6`N	30°06.1`E	-	-	3	Deep circalittoral anoxic mud with surficial "fluffy layer"
9	13.09.2015	14	~110	43°45.9`N	30°4.07`E	2	-	3	Transition between Deep circalittoral oxic mixed sediments (shelly mud) with *Modiolula phaseolina,* and Deep circalittoral suboxic calcareous mud (periazoic zone with *Modiolula phaseolina,*

No. crt.	Date	Station ID	Depth, m	Coordinates		Box Corer (macro-benthos)	MUC (meio-benthos)	Lander	Habitat description
				Latitude	Longitude				
									hydroid *Bougainvillia muscus* Molgulidae tunicates and sponges *Suberites carnosus*
10	14.09.2015	06L (B)	~130	43°42.8`N	30°05.1`E	-	-	3	Deep circalittoral suboxic calcareous mud (periazoic zone with shelly mud constitute of *Modiolula phaseolina* shells and fero-manganese concretions)

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12. Radio-isotope experiments

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At three stations (Table 9), 3-10 multicores were sliced and well mixed (see table for the resolution). The sediment was mixed with CTD water from the deepest water depth ("bottom water"; 1:1 ratio). Abiotic control samples had a final formaldehyde concentration of 3.7%. The slurries were divided over 24 greiner tubes per experiment and the 33P or 55Fe was added to the slurries. The samples were shaken at 9 degrees until sample collection.

Table 9. Overview of Slurry incubations

Stations	Resolution	# of multicores	Sample handling	T1	T2	T3
2	0-7 cm (Fluff layer)	3	Anoxic: N2-gas lines & Al-bags	05-09-2015	10-09-2015	Still running
5	0-5 cm (Fluff layer)	3	Anoxic: glovebag & Al-bags	12-09-2015	15-09-2015	Still running
9	0-1 cm	10	Oxic	10-09-2015	13-09-205	15-09-2015 (Fe) Still running (P)

At each sampling intervals the sediment slurries were centrifuged at 3500-4500 rpm for 30 min. After centrifugation, a 500 ul sample was added to 1.5 NaOH solution for HS- measurements. Another 0.5 ml of sample was transported to a plastic vial acidified with 20 ul 0.5 M NaOH. Also an additional 0.5 ml water sample was collected in plastic pony vials for future analysis. To measure the activity in the supernatant 0.5 ml of sample was mixed with 7 ml Ultima Gold Scintillation Cocktail and measured onboard after approximately 6 hours. The sediment slurries were then again well mixed and shaken on the shaker table in the frozen until the next sampling interval. The HS samples were directly measured on board by Karel and Sharyn.

Besides the pore water analysis, also sediment slurries were frozen at -80 at specific time intervals (T1-T3; see table). The T1 samples were directly frozen after the addition of the tracer. Some of the incubations were still running during the flight back to Utrecht and will be collected in the week of 28 to 3 October. All samples were transported back to Utrecht at -20.

Results 33P incubations

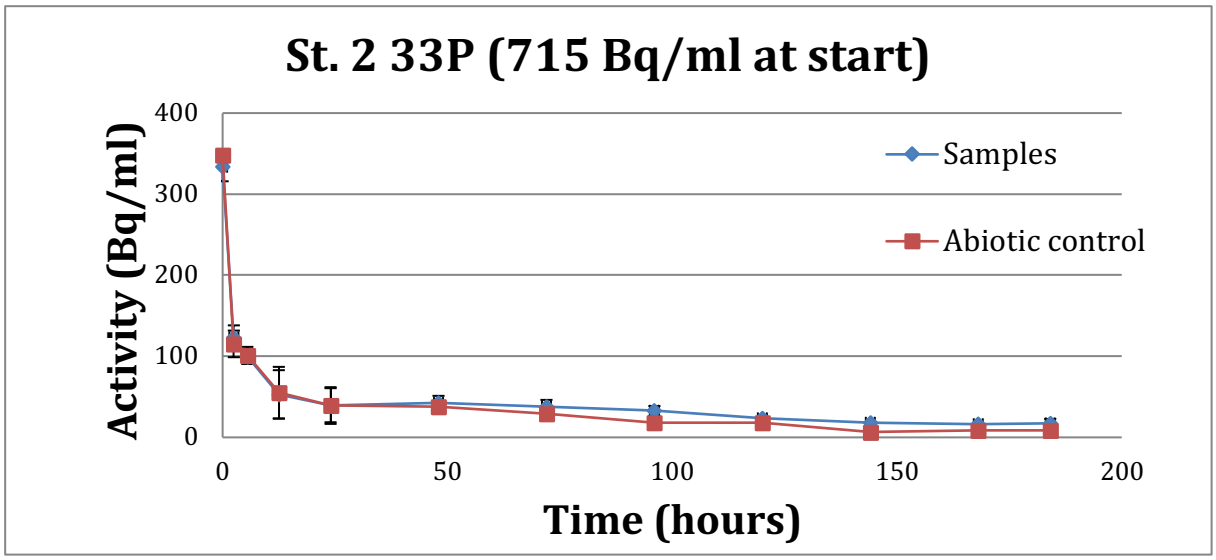

Figure 25. Temporal trend in 33P activity in slurry incubation for station 2.

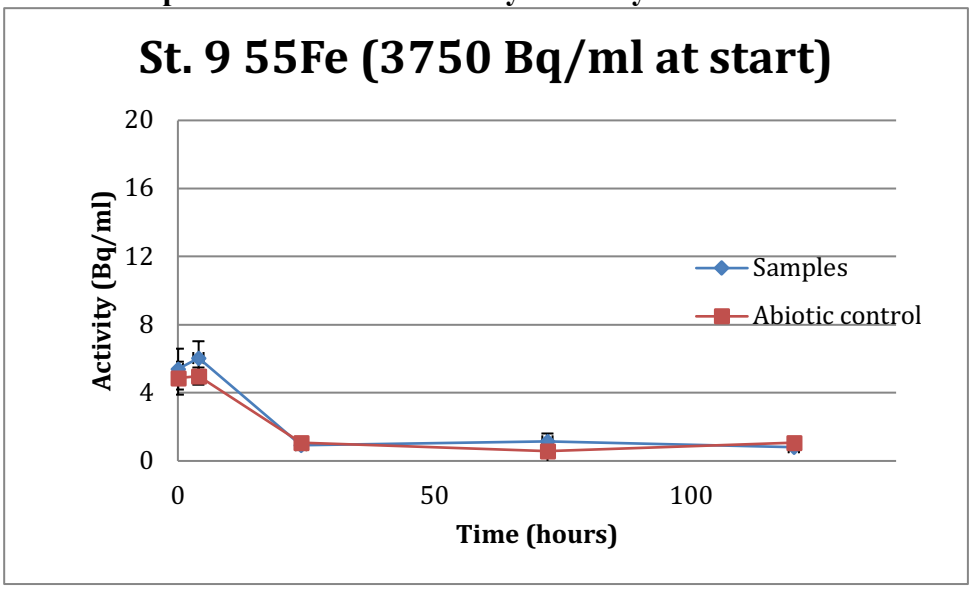

Figure 26. Temporal trend in 55Fe in slurry incubation for station 9.

Whole core incubations

We also performed whole core incubations with sediment cores from station 2. Three multicores that were used for the whole core incubations by Wytze Lenstra were placed in the radio isotope lab in the fridge. Due to the location in the fridge it was however difficult to stimulate anoxic conditions in the core.

13. Benthic flux incubations

Wytze Lenstra, Niels van Helmond, Utrecht University

To measure the benthic flux of iron and other chemical components 3 different incubation experiments were done:

- **Oxic incubation**
  To measure the benthic flux of Fe, Mn, DIC, PO4 , Si, NH_4^+ and NO_3^- under oxygen-saturated conditions in the overlying water.
- **Anoxic incubations**
  To measure the benthic flux of Fe, Mn, DIC, PO4 , Si, NH_4^+, NO_3^-, HS and oxygen respiration of sediment that is incubated with oxygen-depleted overlying water.
- **Bromide incubations**
  Where the incorporation of dissolved bromide from the overlying water into the sediment is measured to determine the rate of bio-irrigation.

Oxic incubations

Incubations of three cores per station were done for oxic stations. Cores were taken from the multicore and immediately brought to a temperature regulated room (temperature at bottom water temperature). Here the stoppers were removed and the overlying water was brought back to 550 ml. A tube connected to an aquarium pump was placed in the overlying water to saturate the water with oxygen. This small oxygen flow also keeps the water homogeneously mixed. Incubations had a duration of 8 hours, samples are taken at 7 moments in time. The volume lost when taken a sample is replaced with bottom water from the CTD.

Anoxic incubations

Anoxic incubations of three cores per station were done at oxic and anoxic stations. Cores were taken from the multicore and immediately brought to a temperature regulated room (temperature at bottom water temperature). Here the stoppers were removed and the cores were push up the tube until there is a volume of 550 ml of overlying water. After this a cap was placed on top of the tubes, so the overlying water is not in contact the air (see picture 14.1). An oxygen meter was placed in one of the caps to monitor the oxygen level, the decrease over time was used to calculate the rate of oxygen respiration. A stirrer in the cap mixes the overlying water. The incubation had a duration of 24 hours, samples were taken at 10 moments in time. The volume lost when a sample was taken was replaced with bottom water from the CTD.

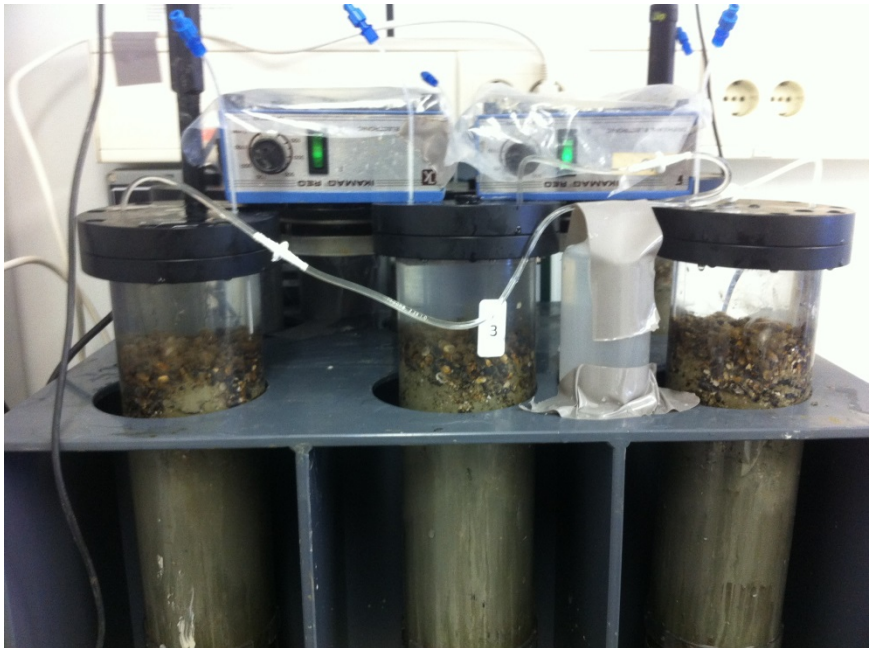

Figure 27. Setup of anoxic incubation experiment

Bromide incubations

Bromide incubations were performed to determine the rate of bio-irrigation in the sediment. Cores were taken from the multicore and immediately brought tino a temperature regulated room (temperature at bottom water temperature). The volume of the overlying water was adjusted to 550 ml. A tube connected to an aquarium pump was placed in the overlying water to establish homogeneous conditions. NaBr was added to the overlying water to increase the bromide concentrations approximately by 10 times. Incubations with bromide were done with 3 cores at 3 different days (table 10). After incubation the cores were sliced in high resolution (0.5 cm (0-5 cm), 1 cm (5-10 cm), 2 cm (10-20 cm) and 4 cm resolution after 20 cm). The three cores were incubated for three different times and sliced directly after incubations (1, 2 and 5 days). The samples were centrifuged for 20 minutes at 4500 rpm and filtered through a 0.45 um filter. The pore water was stored at 4 degrees. The solid phase of the core which was incubated for 5 days is stored at -20 degrees.

Table 10: Summary of multicore incubations

Date	Station	Bromide incubations	Anoxic incubations	Oxic Incubations
2 Sept	1		3 cores	
3 Sept				
4 Sept	2		3 cores	
5 Sept				
6 Sept				
7 Sept	6		3 cores	3 cores
8 Sept	7		3 cores	3 cores
9 Sept	8	3 cores	3 cores	3 cores
10 Sept	9	3 cores	3 cores	3 cores
11 Sept	13	3 cores	3 cores	3 cores
12 Sept				
13 Sept	14		3 cores	3 cores
14 Sept	4		3 cores	

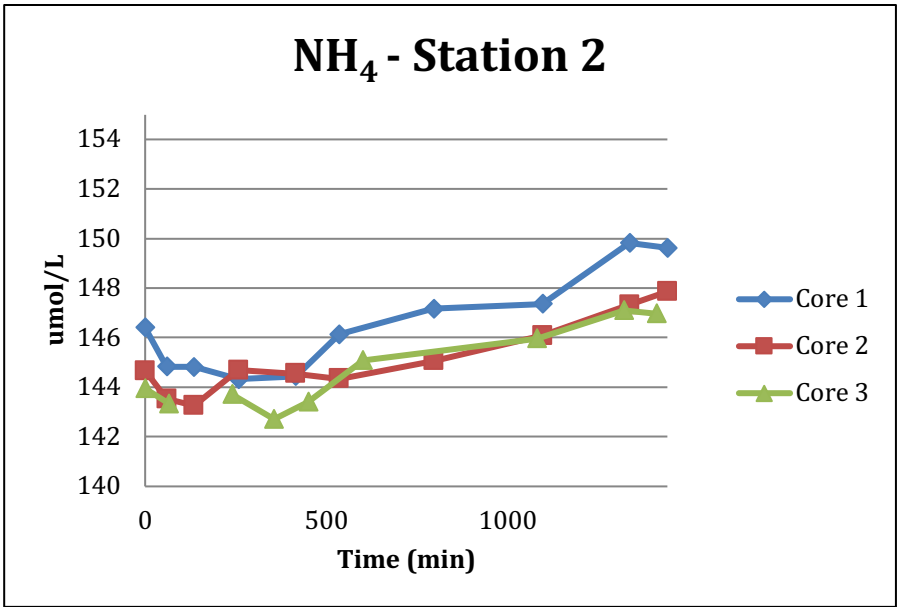

Figure 28: Concentrations of NH_4 in the overlying water measured for three anoxic incubations at station 2.

Appendix 1: CTD sampling depths.

Station 1	
uc CTD bottle	**Depth**
24	9
23	16
22	35
21	37
20	60
19	79
18	90
17	101
16	105
15	109
14	114
13	120
12	124
11	130
10	139
9	139
8	159
7	177
6	250
5	498
4	745
3	1000
2	1250
1	1550

Station 2	
uc CTD bottle	Depth
24	10
23	30
22	60
21	80
20	85
19	90
18	95
17	100
16	105
15	110
14	115
13	120
12	120
11	130
10	140
9	150
8	170
7	187
6	250
5	500
4	750
3	1000
2	1500
1	2077

Station 2 cast 2	
uc CTD bottle	Depth
24	10
23	30
22	60
21	75
20	80
19	90
18	95
17	100
16	105
15	110
14	115
13	120
12	140
11	170
10	215
9	250
8	450
7	500
6	750
5	930
4	1494
3	1715
2	2073
1	2073

Station 3

uc CTD bottle	Depth
24	10
23	30
22	50
21	60
20	70
19	80
18	85
17	90
16	95
15	100
14	105
13	110
12	115
11	120
10	125
9	129
8	135
7	140
6	150
5	200
4	251
3	502
2	753
1	1082

Station 4

uc CTD bottle	Depth
24	10
23	20
22	40
21	45
20	50
19	60
18	70
17	80
16	85
15	90
14	95
13	100
12	105

11	110
10	115
9	120
8	130
7	140
6	160
5	180
4	200
3	250
2	298
1	360

Station 5

uc CTD bottle	Depth
24	10
23	20
22	30
21	40
20	50
19	60
18	70
17	80
16	85
15	95
14	100
13	105
12	110
11	115
10	120
9	125
8	130
7	135
6	140
5	145
4	150
3	160
2	170
1	180

Station 6

uc CTD bottle	Depth
24	10
23	20

22	25
21	30
20	35
19	40
18	45
17	50
16	55
15	60
14	65
13	70
12	75
11	80
10	85
9	90
8	95
7	100
6	105
5	110
4	115
3	119
2	122
1	122

Station 7

uc CTD bottle	**Depth**
24	5
22	10
20	15
18	20
17	25
15	30
14	35
12	40
11	45
5	50
4	55
3	60
2	65
1	70

Station 8	
uc CTD bottle	Depth
23	5
21	10
19	15
17	20
15	25
13	29,8
11	35
9	37
7	39,3
5	44,6
3	50
1	55

Station 8 cast 2	
uc CTD bottle	Depth
24	5
22	10
20	15
18	20
16	25
14	29,8
12	35
10	37
8	39,3
6	44,6
4	50
2	55

Station 9	
uc CTD bottle	Depth
5	5
4	10
3	15
2	20
1	25

Station 13	
uc CTD bottle	Depth
1	5
4	10
7	15
8	20
9	25
16	30
17	35
18	40

Station 14

uc CTD bottle	Depth
20	5
19	10
18	15
17	20
16	25
15	30
14	35
13	40
12	45
11	50
10	55
9	60
8	65
7	70
6	75
5	80
4	85
3	90
2	95
1	100

Station 17

uc CTD bottle	Depth
24	10
23	30
22	35
21	40
20	45
19	50
18	55
17	60
16	70
15	80
14	90
13	100
12	110
11	120
10	130
9	140
8	145
7	150
6	155

5	160
4	200
3	250
2	500
1	813

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