Benthic Profile Data Documentation

Introduction

During OMEX I, a total of 223 parameters were determined as profiles along sediment cores by 10 investigators using a number of different protocols. The data set includes profiles measured on sediment cores and in-situ profiles obtained by benthic landers. The aim of this document is to allow the protocol used to obtain any particular value in the COREPROF table to be determined with ease.

To help you find the information you require quickly, the document is subdivided into sections that describe groups of closely related parameters. These are listed below as a series of hot links. Each section starts with the definition of the parameter codes covered, followed by a list of who measured one or more of those parameters by cruise. Next, there is a protocol section describing the methods used by each principal investigator. Finally, there may be comments on data quality that have been noted by BODC or have come to our attention.

<TIP> If you want to find out a how a particular parameter was measured and know the parameter code then the fastest way to find the information you require is to use the *Acrobat* 'find' tool to search for the parameter code. Then use the 'find' tool again to search for the name of the principal investigator. This will take you straight to the protocol description you require.

Document Index

Sediment Organic Carbon, Inorganic Carbon and Nitrogen

Classical carbon and nitrogen profiles.

Carbon, Nitrogen and Oxygen Isotopes

Profiles of the stable isotopic composition of both bulk sediments and picked foraminifera tests.

Lead and Caesium Isotopes

Profiles of the radioisotope concentration in bulk sediments.

Solid Phase Chemistry

Sediment bulk chemistry profiles, including determinations on either total sediment or the silt fraction.

Dry Bulk Density, Porosity and Water Content

Profiles of water content by both weight and volume (porosity) plus dry bulk density (sediment dry weight per unit wet volume) profiles.

Magnetic Susceptibility

Includes both raw magnetic susceptibility profiles and mass-normalised parameters.

Sediment Amino Acid Content

Profiles of total hydrolysable amino acid content plus the proportions of individual acids.

Sediment Mineralogy

X-ray diffraction mineralogy profiles including individual clay minerals.

Sediment Grain Size

A wide range of sediment grain size parameters determined by classical methods and particle sizers.

Pore Water Dissolved Oxygen and Resistivity

Includes parameters measured on deck and in-situ profiles obtained by the TROL lander.

Pore Water Nutrients, Dissolved Carbon, Sulphate and Metals

Profiles of a wide range of pore water chemistry parameters.

Nitrous Oxide in Pore Waters

This is an additional parameter that has been documented separately to simplify the text.

Foraminiferal Coiling

These parameters specify the proportion of selected species of foraminifera that have their tests coiled in the dextral sense.

Radiocarbon Dating

Absolute ages of sediment samples.

Pigments

Pigment concentrations in the sediment resulting from phytoplankton detritus.

Organic Biomarkers

A wide range of parameters that provide indirect measurements of the level of microbial activity within the sediment.

Kasten Coring Protocols

This section describes the handling protocols used for samples collected with the Kasten corer. The information has been included as a separate section to prevent unnecessary duplication between other sections of the document. Links are provided in the text where appropriate.

Box Coring Protocols

This section provides brief descriptions of the different types of box corer used. Again, this has been separated out to reduce information duplication and appropriate links are provided in the text.

References

Full references for the papers cited in the protocol descriptions.

Sediment Organic Carbon, Inorganic Carbon and Nitrogen

Parameter Code Definitions

CALCACWF Carbonate content by weight (<63 micron size fraction)
Wet sieving then weight loss on acidification
Per cent

CALCACXT Carbonate content by weight (bulk sediment)
Weight loss on acidification
Per cent

ICCNCIWF Inorganic carbon content (<63 micron size fraction)
Wet sieving, ignition at 400 °C then C/N analyser
Per cent

ICCNCIXT Inorganic carbon content (bulk sediment)
Ignition at 400 °C then C/N analyser
Per cent

OCCNCAXT Organic carbon content (bulk sediment)
Acidification then carbon/nitrogen analyser
Per cent

OCCNCIWF Organic carbon content (<63 micron size fraction)
Wet sieving then difference of C/N analyser readings before and after 400 °C ignition
Per cent

OCCNCIXT Organic carbon content (bulk sediment)
Difference of carbon/nitrogen analyser readings before and after 400 °C ignition
Per cent

TCCNCNXT Total carbon content (bulk sediment)
Carbon/nitrogen analyser
Per cent

TCCNCNWF Total carbon content (<63 micron size fraction)
Wet sieving then carbon/nitrogen analyser
Per cent

TNCNCNXT Total nitrogen content (bulk sediment)
Carbon/nitrogen analyser
Per cent

TNCNCNWF Total nitrogen content (<63 micron size fraction)
Wet sieving then carbon/nitrogen analyser
Per cent

Originator Code Definitions

Charles Darwin cruises CD84 and CD94

15	Prof. Nick McCave	Cambridge University, UK

Pelagia cruise PLG93

11	Dr. Wim Helder	NIOZ, Texel, the Netherlands

Pelagia cruise PLG95B and Charles Darwin cruise CD86

11	Dr. Wim Helder	NIOZ, Texel, the Netherlands
75	Dr. Tjeerd van Weering	NIOZ, Texel, the Netherlands

Pelagia cruise PLG95A

87	Prof. Carlo Heip	NIOO, the Netherlands

Cruises Cote d'Aquitaine NAOX1, NAOX2 and NAOX3, Auriga cruises PLUTUR2, PLUTUR3 and Andromeda cruise PLUTUR6

98	Dr. J-M Jouanneau	University of Bordeaux, France

Originator Protocols

Professor Nick McCave

Samples were collected using a Kasten core or box core. See the sections on Kasten Coring Protocols and Box Coring Protocols for details of the corer and sample handling. The fine (<63 micron) and coarse fractions of the sediment were separated by wet sieving.

Calcium carbonate was determined on the bulk sediment and the fine fraction by gravimetric measurement of the quantity of carbon dioxide liberated by the addition of 5% v/v sulphurous acid.

The method used for organic carbon and nitrogen was based on the Carlo Erba EA1106 CHN-OS analyser. The essence of the technique was the determination of total carbon and nitrogen followed by elimination of the organic component by heating at 400 °C for 3 hours. The inorganic

component was then determined and the organic component computed by difference. This method and possible alternatives are discussed in detail in Manighetti (1993), including quantitative assessment of errors. It was concluded that incomplete destruction results in organic carbon being underestimated by up to 0.1% for the samples collected during the BOFS programme in 1989 and 1990.

Dr. Wim Helder

Samples were taken with either a box corer or a multicorer of standard design (Barnett et al., 1984). See the section on Box Coring Protocols for details of the corer and sample handling.

Dried and homogenised sediment samples taken from a sectioned sub-core were analysed by on-line combustion in a Carlo-Erba NA-1500 elemental analyser before and after acidification (sulphurous acid) following the protocols of Verardo et al. (1990) to give total carbon, total nitrogen and organic carbon. Total nitrogen was determined in a separate run from carbon.

Dr. Tjeerd van Weering

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols). 9 cm diameter sub-cores were taken.

Dried and homogenised sediment samples taken from a sectioned sub-core were analysed by on-line combustion in a Carlo-Erba NA-1500 elemental analyser after removal of inorganic carbon by treatment with sulphurous acid following the protocols of Verardo et al. (1990) to give total nitrogen and organic carbon. Carbon and nitrogen were analysed on separate runs.

Professor Carlo Heip

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols). 10 cm diameter sub-cores were taken.

The sub-cores were sectioned on board ship and stored frozen at -25 °C until analysed.

The samples were analysed using a Carlo-Erba NA-1500 elemental analyser according to the protocol of Nieuwenhuize et al., 1994. Carbon was partitioned into organic and inorganic fractions by acidification with 25% HCl in-situ within silver sample cups. Carbon and nitrogen were analysed on the same sample.

Dr. Jean-Marie Jouanneau

Samples were collected with either a box corer or a 'MARK 1 minicorer'. Total carbonate content was determined using a 'LECO Carbon

Determinator'. Organic carbon was determined following the protocol of Strickland and Parsons (1972) as modified by Etcheber (1981). The analyses were carried out on a LECO CS-125 analyser after carbonates had been removed by addition of 2N HCl.

Comments on Data Quality

An organic carbon intercalibration exercise was co-ordinated by NIOZ as part of the OMEX study. Included in the exercise were Cambridge University, NIOO, NIOZ, Bordeaux University, Algarve University, GEOMAR and IFREMER. This list includes all of the above investigators. Those who are not included above have not supplied sediment carbon and nitrogen data but are amongst those who have supplied POC/PON data from water bottle or trap samples.

A single core slice (10-12 cm depth) from each of three sites (Galician Margin, Meriadzek Terrace and Goban Spur OMEX-F) was dried, homogenised and subdivided into 2 g aliquots in sealed glass vials. Each laboratory was supplied with three vials of each sample.

No values for total carbon or nitrogen were returned by Bordeaux. No organic carbon data were returned by Algarve.

Agreement of total carbon results between all laboratories was excellent with the overall variability of all samples from all laboratories less than 3%. The mean values and standard deviations for the three samples were 7.231 (SD 0.039), 2.311 (SD 0.061) and 8.219 (SD 0.132). The relative variations between the laboratories were 1.3%, 2.6% and 1.6%.

The organic carbon results showed more variation. The mean values and standard deviations for the three samples were 0.402 (SD 0.020), 0.090 (SD 0.031) and 0.280 (SD 0.014). The Cambridge results for two of the three samples (Galician Margin and Meriadzek Terrace) were significantly different from the overall mean. One of these was lower than the mean whilst the other was higher. Significant differences were also observed between GEOMAR and NIOO for one of the samples (Galician Margin). The relative variations between the laboratories were 4.9%, 34.7% and 4.9%.

The poorest agreement was in the total nitrogen data. The results obtained for the three samples were 0.051 (SD 0.016), 0.015 (SD 0.006) and 0.041 (SD 0.011). Significant differences were reported between Cambridge and NIOZ/NIOO and GEOMAR and NIOZ/NIOO data. The relative variations between the laboratories were 31.6%, 41.3% and 28.2%.

Carbon, Nitrogen and Oxygen Isotopes

Parameter Code Definitions

D13CMOBX Bulk sediment organic carbon ^{13}C enrichment (delta-^{13}C)
Mass spectrometry on acidified then combusted sample
Parts per thousand

D13CMTBX Bulk sediment total carbon ^{13}C enrichment (delta-^{13}C)
Mass spectrometry on combusted sample
Parts per thousand

D13CMXFA Globigerina bulloides test ^{13}C enrichment (delta-^{13}C)
Mass spectrometry on combusted sample
Parts per thousand

D13CMXFB Neoglobigerina pachyderma test ^{13}C enrichment (delta-^{13}C)
Mass spectrometry on combusted sample
Parts per thousand

D13CMXFF Uvigerina spp. test ^{13}C enrichment (delta-^{13}C)
Mass spectrometry on combusted sample
Parts per thousand

D15NMTBS Bulk sediment total nitrogen ^{15}N enrichment (delta-^{15}N)
Mass spectrometry on combusted sample
Parts per thousand

D18OMXFA Globigerina bulloides test ^{18}O enrichment (delta-^{18}O)
Mass spectrometry on combusted sample
Parts per thousand

D18OMXFB Neoglobigerina pachyderma test ^{18}O enrichment (delta-^{18}O)
Mass spectrometry on combusted sample
Parts per thousand

D18OMXFF Uvigerina spp. test ^{18}O enrichment (delta-^{18}O)
Mass spectrometry on combusted sample
Parts per Thousand

Originator Code Definitions

Cruise Charles Darwin CD84

15 Prof. Nick McCave Cambridge University, UK

Cruises Pelagia PLG93 and Charles Darwin CD86

11 Dr. Wim Helder NIOZ, Texel, the Netherlands

Originator Protocols

Professor Nick McCave

Samples were collected using a Kasten core. See the section on Kasten Coring Protocols for details of the corer and sample handling.

Oxygen and carbon isotopes were determined on foramanifera tests taken from the conduit samples. The samples were disaggregated into distilled water, and washed through 150mm or 63mm sieves. The coarse fraction was washed and dried in an oven at 60 °C and then split using a Soiltest CL-242A splitter until a sample containing approximately 300 whole foraminifera tests was obtained. The final split was strewn into a picking tray and individual species were extracted.

Test tubes (washed in Decon 90) were soaked in aqueous NaOCl for an hour and washed in distilled water. In these the samples were left in 5ml aqueous NaOCl for 3 hours which was then pipetted off and replaced by 3ml of 20% aqueous HCl. The solution was ultrasonicated for 5 minutes, left overnight to digest and ultrasonicated for a further 5 minutes.

The resulting solution was transferred using a fine funnel into 500 MWCO dialysis tubing, sealed using medical clips and placed into 2.5 litres of distilled water. This water was changed at least 8 times and left overnight. The process was deemed complete when no change in pH was detected an hour after the water was changed.

The solution was then transferred into chromic acid washed 9mm Pyrex sample tubes and centrifuged at 10,000 rpm for 5 hours. The sample was frozen overnight and dried in a vacuum oven at room temperature. CuO, Cu and silver wire were added to the sample and the tubes were then evacuated, sealed and heated to 450 °C for at least 14 hours.

The sample tubes were broken in the vacuum line of a VG Isotech SIRA series II mass spectrometer using a stainless steel cracker. Carbon dioxide was cryogenically separated from other gases and analysed. A mass scan from 28-55 was made to ensure that the sample had not been contaminated.

The sample was compared with a reference gas and the isotopic ratio calibrated to PDB.

Dr. Wim Helder

Samples were taken with either a box core or a multicorer of standard design (Barnett et al., 1984). See the section on Box Coring Protocols for details of the corer and sample handling.

Dried and homogenised sediment samples taken from a sectioned sub-core were analysed by on-line combustion in a Fisons CHN analyser followed by isotopic analysis in a Fisons-Optima stable isotope mass spectrometer. Results are given relative to the isotopic ratio in a PDB reference standard such that:

$$\text{Isotopic enrichment} = [(\text{sample ratio} / \text{standard ratio}) - 1] * 1000$$

Analyses were done on both bulk samples and samples that had had carbonate removed by acidification to give $\delta^{13}C$ data for both total sediment and the organic carbon fraction.

Lead and Caesium Isotopes

Parameter Code Definitions

C37CGSXX Caesium 137 content
Gamma-ray spectroscopy
Bequerels per kilogram

L210IGXX Solid phase ^{210}Pb content
Alpha spectroscopy on plated samples
Bequerels per kilogram

S37CGSXX Caesium 137 content standard error
Gamma-ray spectroscopy
Bequerels per kilogram

SL10IGXX Solid phase ^{210}Pb content standard error
Alpha spectroscopy on plated samples
Bequerels per kilogram

SX10GSXX Solid phase excess (wrt steady state) ^{210}Pb content standard error
Gamma-ray spectroscopy of compressed sediment pellets (^{210}Pb-^{226}Ra)
Bequerels per kilogram

SX10IGXX Solid phase excess (with respect to steady state) ^{210}Pb content standard error
Alpha spectroscopy on plated samples
Bequerels per kilogram

X210GSXX Solid phase excess (wrt steady state) ^{210}Pb content
Gamma-ray spectroscopy of compressed sediment pellets (^{210}Pb-^{226}Ra)
Bequerels per kilogram

X210IGXX Solid phase excess (with respect to steady state) ^{210}Pb content
Alpha spectroscopy on plated samples
Bequerels per kilogram

Originator Code Definitions

Charles Darwin cruises CD84 and CD94

15	Prof. Nick McCave	Cambridge University, UK

Charles Darwin cruise CD86 and Pelagia cruises PLG93 and PLG95B

75	Dr. Tjeerd van Weering	NIOZ, Texel, the Netherlands

Andromeda cruise PLUTUR6

98	Dr. J-M Jouanneau	University of Bordeaux, France

Originator Protocols

Professor Nick McCave

Samples were collected using a box core. See the sections on Box Coring Protocols for details of the corer and sample handling. The sub-cores were extruded into a nitrogen atmosphere at 5 °C and cut into 1 or 2 cm slices. Pore water was removed in a refrigerated centrifuge and the solid material was stored refrigerated.

Back on land, the samples were dried by heating for 24 hours at 105 °C in a VSL drying oven and cooled in a desiccator. The dried samples were ground in a Tema laboratory disc mill for four minutes using a large agate barrel and puck, then stored in airtight plastic containers.

Approximately a gram of sediment was accurately weighed into a 250 ml PTFE beaker. A little distilled water was used to moisten the sediment and 30 ml of 6N HCl was added along with 5 ml of H_2O_2. 0.75 ml of ^{209}Po tracer was pipetted into the beaker.

After cooling, the solution was filtered through a 0.4 micron Nuclepore membrane filter and the residue was removed from the filter with a fresh charge of 6N HCl, returned to the hot plate, refluxed for a further two hours, cooled and filtered as before.

The two filtrates were combined, evaporated down to approximately 5 ml and transferred to a polonium plating cell. Distilled water was added to achieve a total volume of 40 ml and an HCl concentration of 0.8N. Approximately a gram of ascorbic acid was stirred into the cell and a freshly burnished silver disc inserted. The disc was plated for 24 hours at room temperature with continuous stirring. At the end of plating, the disc was removed, washed with water and acetone then set aside for at least 24 hours.

The discs were counted for between 24 and 72 hours in a Canberra alpha spectrometer giving at least 1000 counts for each peak. Counting accuracy was checked using a standard reference material. Precision was checked by analysing replicates of a subset of samples. Replicate results consistently fell within the theoretical standard error for the method.

Dr. Tjeerd van Weering

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols). 9 cm diameter sub-cores were taken.

The sub-cores were cut into slices and frozen. Back in the laboratory, ^{210}Pb was determined by alpha spectrometry through the measurement of ^{210}Po which was assumed to be in equilibrium with ^{210}Pb.

A 0.5 g aliquot of dried, homogenised sample was spiked with ^{208}Po as a yield tracer. Samples were then dissolved in concentrated acids (12 ml HNO_3 and 2 ml HF). The resulting solution was evaporated to dryness and the residue dissolved in molar HCl at 80 °C. Trivalent iron was reduced by the addition of ascorbic acid. The isotopes were then plated from this solution onto silver discs and then assayed by alpha spectrometry.

Dr. Jean-Marie Jouanneau

Sediment cores were collected with either a box corer or a MARK I minicorer. The radioisotope levels were assayed on dried bulk sediment samples by counting on an EGSP 2200-25-R high resolution gamma-ray spectrometer.

Solid Phase Chemistry

Parameter Code Definitions

AGCNICXT Silver content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

ALCNAAWF Fine (<63 micron) sediment aluminium content
Wet sieving, acid digestion then atomic absorption
Per Cent

ALCNXMXT Bulk sediment aluminium content
X-ray fluorescence on flux diluted fused bead
Per Cent

ASCNICXT Bulk sediment arsenic content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

ASCNXTXT Bulk sediment arsenic content
X-ray fluorescence on pressed powder
Parts per million

BACNICXT Bulk sediment barium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

BACNXTXT Bulk sediment barium content
X-ray fluorescence on pressed powder
Parts per million

BECNICXT Beryllium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

BICNICXT Bismuth content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

BRCNXTXT Bulk sediment bromine content
X-ray fluorescence on pressed powder
Parts per million

CACNAAWF Fine (<63 micron) sediment calcium content
Wet sieving, acid digestion then atomic absorption
Per Cent

CACNXMXT Bulk sediment calcium content
X-ray fluorescence on flux diluted fused bead
Per Cent

CDCNAAWF Fine (<63 micron) sediment cadmium content
Wet sieving, acid digestion then atomic absorption
Parts per million

CDCNICXT Cadmium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

CECNICXT Bulk sediment cerium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

CECNXTXT Bulk sediment cerium content
X-ray fluorescence on pressed powder
Parts per million

CLCNXTXT Bulk sediment chlorine content
X-ray fluorescence on pressed powder
Per Cent

COCNAAWF Fine (<63 micron) sediment cobaltcontent
Wet sieving, acid digestion then atomic absorption
Parts per million

COCNICXT Cobalt content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

CRCNAAWF Fine (<63 micron) sediment chromium content
Wet sieving, acid digestion then atomic absorption
Parts per million

CRCNICXT Bulk sediment chromium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

CRCNXTXT Bulk sediment chromium content
X-ray fluorescence on pressed powder
Parts per million

CSCNICXT Caesium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

CUCNAAWF Fine (<63 micron) sediment copper content
Wet sieving, acid digestion then atomic absorption
Parts per million

CUCNICXT Bulk sediment copper content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

CUCNXTXT Bulk sediment copper content
X-ray fluorescence on pressed powder
Parts per million

DYCNICXT Dysprosium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

ERCNICXT Erbium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

EUCNICXT Europium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

FECNAAWF Fine (<63 micron) sediment total iron content
Wet sieving, acid digestion then atomic absorption
Per Cent

FECNXMXT Bulk sediment total iron content
X-ray fluorescence on flux diluted fused bead
Per Cent

GACNICXT Bulk sediment gallium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

GACNXTXT Bulk sediment gallium content
X-ray fluorescence on pressed powder
Parts per million

GDCNICXT Gadolinium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

HFCNICXT Hafnium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

HOCNICXT Holmium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

IDCNICXT Indium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

IXCNXTXT Bulk sediment iodine content
X-ray fluorescence on pressed powder
Parts per million

KXCNXMXT Bulk sediment potassium content
X-ray fluorescence on flux diluted fused bead
Per Cent

LACNICXT Bulk sediment lanthanum content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

LACNXTXT Bulk sediment lanthanum content
X-ray fluorescence on pressed powder
Parts per million

LICNICXT Lithium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

LUCNICXT Lutetium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

MGCNXMXT Bulk sediment magnesium content
X-ray fluorescence on flux diluted fused bead
Per Cent

MNCNAAWF Fine (<63 micron) sediment total manganese content
Wet sieving, acid digestion then atomic absorption
Per Cent

MNCNXTXT Bulk sediment total manganese content
X-ray fluorescence on pressed powder
Per Cent

MNCNXMXT Bulk sediment total manganese content
X-ray fluorescence on flux diluted fused bead
Per Cent

MOCNICXT Molybdenum content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

NACNXMXT Bulk sediment sodium content
X-ray fluorescence on flux diluted fused bead
Per Cent

NBCNICXT Bulk sediment niobium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

NBCNXTXT Bulk sediment niobium content
X-ray fluorescence on pressed powder
Parts per million

NDCNICXT Neodymium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

NICNAAWF Fine (<63 micron) sediment nickel content
Wet sieving, acid digestion then atomic absorption
Parts per million

NICNICXT Bulk sediment nickel content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

NICNXTXT Bulk sediment nickel content
X-ray fluorescence on pressed powder
Parts per million

PBCNICXT Bulk sediment lead content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

PBCNXTXT Bulk sediment lead content
X-ray fluorescence on pressed powder
Parts per million

PDCNICXT Palladium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

PRCNICXT Praseodymium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

PXCNXMXT Bulk sediment phosphorus content
X-ray fluorescence on flux diluted fused bead
Per Cent

RBCNICXT Bulk sediment rubidium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

RBCNXTXT Bulk sediment rubidium content
X-ray fluorescence on pressed powder
Parts per million

SBCNICXT Antimony content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

SCCNICXT Scandium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

SECNICXT Selenium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

SICNXMXT Bulk sediment silicon content
X-ray fluorescence on flux diluted fused bead
Per Cent

SMCNICXT Samarium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

SNCNICXT Tin content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

SRCNICXT Bulk sediment strontium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

SRCNXTXT Bulk sediment strontium content
X-ray fluorescence on pressed powder
Parts per million

SXCNXMXT Bulk sediment sulphur content
X-ray fluorescence on flux diluted fused bead
Per Cent

SXCNXTXT Bulk sediment sulphur content
X-ray fluorescence on pressed powder
Per Cent

TACNICXT Tantalum content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

TBCNICXT Terbium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

THCNICXT Bulk sediment thorium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

THCNXTXT Bulk sediment thorium content
X-ray fluorescence on pressed powder
Parts per million

TICNICXT Bulk sediment titanium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

TICNXMXT Bulk sediment titanium content
X-ray fluorescence on flux diluted fused bead
Per Cent

TICNXTXT Bulk sediment titanium content
X-ray fluorescence on pressed powder
Per Cent

TLCNICXT Thallium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

TMCNICXT Thulium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

UXCNICXT Bulk sediment uranium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

UXCNXTXT Bulk sediment uranium content
X-ray fluorescence on pressed powder
Parts per million

VXCNICXT Bulk sediment vanadium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

VXCNXTXT Bulk sediment vanadium content
X-ray fluorescence on pressed powder
Parts per million

WXCNICXT Tungsten content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

YBCNICXT Ytterbium content of bulk sediment
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

YXCNICXT Bulk sediment yttrium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

YXCNXTXT Bulk sediment yttrium content
X-ray fluorescence on pressed powder
Parts per million

ZNCNICXT Bulk sediment zinc content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

ZNCNXTXT Bulk sediment zinc content
X-ray fluorescence on pressed powder
Parts per million

ZRCNICXT Bulk sediment zirconium content
Inductively-coupled plasma mass spectrometry (ICP-MS)
Parts per million

ZRCNXTXT Bulk sediment zirconium content
X-ray fluorescence on pressed powder
Parts per million

Originator Code Definitions

Cruises Charles Darwin CD84 and CD94

86	Dr. Rachel Mills	Southampton Oceanography Centre, UK

Auriga cruises PLUTUR2, PLUTUR3 and PLUTUR4 and Andromeda cruise PLUTUR6

98	Dr. J-M Jouanneau	University of Bordeaux, France

Meteor cruise M30_1

51	Prof. Wolfgang Balzer	University of Bremen, Germany

Originator Protocols

Dr. Rachel Mills

Samples were collected using a box core. See the sections on Box Coring Protocols for details of the corer and sample handling. The sub-cores were extruded into a nitrogen atmosphere at 5 °C and cut into 1 or 2 cm slices. Pore water was removed in a refrigerated centrifuge and the solid material was stored refrigerated.

Back on land, the samples were dried by heating for 24 hours at 105 °C in a VSL drying oven and cooled in a desiccator. The dried samples were ground in a Tema laboratory disc mill for four minutes using a large agate barrel and puck, then stored in airtight plastic containers.

For XRF major element analysis, fused glass beads were produced using 2 grams of sample in a 5:1 dilution with a eutectic borate flux. The data were matrix corrected (for both absorption and enhancement) using theoretically derived alpha or influence coefficients. The data were delivered to BODC as oxide percentages but have been re-computed as element percentages prior to loading in the database.

For XRF trace element analysis, 40 mm diameter discs were prepared by pressing 5 g of ground sediment with a PVA binder at 10 tons for 30 seconds in a hand press. Matrix corrections were applied to all samples. The presence of the binder caused problems with the analysis of some elements, particularly Br, I and V, which were overcome by re-grinding the samples and making up the pellets again until acceptable results were obtained. The data were supplied to BODC as parts per million except for sulphur and chlorine which were as element percentages. The data for Mn and Ti were converted to percentages (divided by 10,000) to conform to database standard units.

Dr. Jean-Marie Jouanneau

Sediment samples were collected using either a box corer or a MARK I minicorer. Major and trace elements were measured on dried, pulverised powders of bulk sediment by X-ray fluorescence spectrometry. The analyses were carried out on a Philips PW1500-10 instrument. Interference corrections were applied using the equations of Lachance and Trail, 1966. The results were calibrated against international synthetic rock standards provided by USGS and ANRT.

On selected samples, a much larger suite of trace elements were determined by ICP-MS.

Professor Wolfgang Balzer

Cores were collected using a multicorer of standard design (Barnett et al., 1984). The core tubes were transferred to a refrigerated laboratory and cut into sections. The solid samples were wet sieved through a 62 micron sieve to remove the sand fraction, dried at 110 °C and carefully ground.

Approximately 25-35 mg of sediment was weighed into a small Teflon beaker and 0.5 ml of nitric acid added. The beaker was placed in a Teflon digestion bomb together with 2 ml nitric, 5 ml hydrofluoric and 2 ml hydrochloric acids. The bomb was placed in a microwave oven and the sediment digested under pressure.

The resulting solution was evaporated to near dryness on a hotplate and dissolved in 4 ml 0.5M sub-boiled nitric acid. The resulting solution was analysed for trace metals (Co, Ni, Cu, Cd, Mn and Cr) by flameless atomic absorption spectrometer employing Zeeman background correction. Major elements (Al, Ca and Fe) were analysed using a flame atomic absorption spectrometer.

Reagent blanks were subjected to the full analytical procedure. The resulting corrections were always less than 10% of the sample concentrations.

The data were supplied to BODC in units of μmol/g. These were converted to μg/g by multiplying by the appropriate atomic weights and then scaled to per cent or ppm as appropriate.

Dry Bulk Density, Porosity and Water Content

Parameter Code Definitions

DBDXCSXX Dry bulk density
Salt-corrected particle mass and volume computed from mass
Grams per cubic centimetre

DBDXMNXX Dry bulk density
Uncorrected particle mass and measured volume
Grams per cubic centimetre

POROWVXX Porosity (water content by volume)
Weight of water liberated on oven/freeze drying per unit volume of wet sediment
Per Cent

WCWTDRXX Sediment water content by weight
Mass difference on oven/freeze drying per unit mass of wet sediment
Per cent

Originator Code Definitions

Charles Darwin cruises CD84 and CD94

15	Prof. Nick McCave	Cambridge University, UK

Charles Darwin cruise CD86 and Pelagia cruise PLG93

75	Dr. Tjeerd van Weering	NIOZ, Texel, the Netherlands
82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Pelagia cruise PLG95A

87	Prof. Carlo Heip	NIOO, the Netherlands

Pelagia cruise PLG95B

11	Dr. Wim Helder	NIOZ, Texel, the Netherlands
75	Dr. Tjeerd van Weering	NIOZ, Texel, the Netherlands

Meteor cruises M27_1 and M30_1 and Valdivia cruise VLD137

82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Cote d'Aquitaine cruises NAOX1, NAOX2 and NAOX3, Auriga cruises PLUTUR2 and PLUTUR3 and Anromeda cruise PLUTUR6

98	Dr. J-M Jouanneau	University of Bordeaux, France

Discovery cruise DI216 and Meteor cruise M30_1

51	Prof. Wolfgang Balzer	University of Bremen, Germany

Originator Protocols

Professor Nick McCave

Samples were collected using a Kasten core or box core. See the sections on Kasten Coring Protocols and Box Coring Protocols for details of the corer and sample handling.

Water content was determined on the syringed samples by weight loss after drying at 60 °C for 48 hours. Because it was difficult to accurately measure sample volume, dry bulk density was determined by assuming an average particle density of 2.65 g/cm^3, a salinity of 35 g/kg and a water density of 1.025 g/cm^3 to calculate the salt-corrected particle weight (from dry mud weight) and the total sample volume. Detailed equations are given in Manighetti (1993).

Dr. Tjeerd van Weering

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols) or piston corer. 9 cm diameter sub-cores were taken from the box cores.

Samples of 6 ml volume were taken at regular intervals along the core using a cut-off syringe. The weight of the samples before and after freeze drying was determined. Dry bulk density was calculated as dry weight divided by the sample volume. Porosity (assumed to be water content by volume) was computed as the difference between wet weight over dry weight divided by the sample volume. No correction was made for salt.

Dr. Wim Helder

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols). Sub-cores were taken using 54 mm id Plexiglas tubes. The

cores were extruded and sectioned and the samples collected in glass vials. Porosity was calculated from the weight loss after drying for 24 hours at 105 °C.

Dr. Thomas Soltwedel

Samples were usually collected using a multicorer of standard design (Barnett et al., 1984). However, for some stations on cruises Charles Darwin CD86 and Pelagia PLG93 samples were collected using the NIOZ box corer (see the section on Box Coring Protocols).

Sub-sampling was achieved using 5 ml or 20 ml disposable syringes as small piston corers to take samples down to 10 cm depth. Between three and five replicate samples were taken from each core.

The syringe contents were sectioned into approximately 1 cm slices back in the laboratory. Porosity was computed from the weight loss per unit volume of sediment on oven drying at 60 °C for 48 hours.

Professor Carlo Heip

Samples were collected using the NIOZ box corer (see the section on Box Coring Protocols). 10 cm diameter sub-cores were taken using plastic tubes and cut into sections. Porosity was determined from the weight loss on drying the sediment.

Dr. Jean-Marie Jouanneau

Core samples were collected using either a box corer or a MARK I minicorer. Water content was determined by measuring the difference between the wet and dry weights of the sediment.

Professor Wolfgang Balzer

Cores were collected using a multicorer of standard design (Barnett et al., 1984). Fixed volume samples for porosity determination were taken from along one of the subcores and the porosity determined by measuring the weight loss on drying.

Magnetic Susceptibility

Parameter Code Definitions

NMGSWSCT Mass-normalised magnetic susceptibility (63-1000 micron total sediment)
Wet sieving then magnetic susceptibility detector
Magnetic susceptibility cgs units / gram

NMGSWSFT Mass-normalised magnetic susceptibility (<63 micron total sediment)
Wet sieving then magnetic susceptibility detector
Magnetic susceptibility cgs units / gram

XMGSWSCT Magnetic susceptibility (63-1000 micron bulk sediment)
Wet sieving then magnetic susceptibility detector
Magnetic susceptibility cgs units

XMGSWSFT Magnetic susceptibility (<63 micron bulk sediment)
Wet sieving then magnetic susceptibility detector
Magnetic susceptibility cgs units

XMGSXPBT Magnetic susceptibility (bulk wet sediment)
Magnetic susceptibility probe
Magnetic susceptibility cgs units

Originator Code Definitions

Cruise Charles Darwin CD84

15	Prof. Nick McCave	Cambridge University, UK

Originator Protocols

Professor Nick McCave

Samples were collected using a Kasten core. See the section on Kasten Coring Protocols for details of the corer and sample handling.

Bulk magnetic susceptibility measurements were made on board ship using a Bartington Instruments MS2 meter with a probe-type detector. The measurements were made on the core slabs taken for X-Ray. The probe was

held against the sediment slabs at 2 cm intervals. The readings were corrected for background susceptibility and instrumental drift by taking alternate measurements with the detector held away from the slab.

Conduit samples were wet sieved to obtain coarse (63mm-1mm) and fine (<63mm) fractions. The magnetic susceptibility of the dried sediment fractions was determined using a Bartington MS2B sensor with an internal diameter of 36mm. The upper limit of the coarse fraction was restricted to 1mm to eliminate the influence of occasional large, ice-rafted pebbles.

Each sample was measured twice, with a blank container introduced between every sample to correct for drift. The mass normalised parameters were derived by dividing the averaged corrected magnetic susceptibility value by the appropriate fraction weight.

Sediment Amino Acid Content

Parameter Code Definitions

ALACHPXT Alanine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

ARGCHPXT Arginine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

ASPCHPXT Aspartic acid content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

GLUCHPXT Glutamic acid content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

GLYCHPXT Glycine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

HAACHPXT Total hydrolysable amino acid (THAA) content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

ILECHPXT Isoleucine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

LEUCHPXT Leucine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

PHECHPXT Phenylalanine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

SERCHPXT Serine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

THRCHPXT Threonine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

TYRCHPXT Tyrosine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

VALCHPXT Valine content of bulk sediment
HPLC on hydrolysed sample
Micrograms per gram of dry sediment

Originator Code Definitions

Cruises Charles Darwin CD84 and Discovery DI216

89 Dr. Tomasz Boski Universidade do Algarve, Portugal

Originator Protocols

Dr. Tomasz Boski

Samples were collected using either a box corer or a multicorer of standard design (Barnett et al., 1984). See the section on Box Coring Protocols for general details of the corer and sample handling. Box cores were taken on cruise CD84. One of these, 5B, was kept at -25 °C until analysed. The other two cores analysed, 3B and 4B, were stored at 2 °C. On DI216, the multicorer was used and all sub-sampling was done on board. To avoid contamination, the outer rims of the cores that had been in contact with non-sterile objects were discarded. All sample handling was done with sterilised instruments. The samples were freeze dried in organic-free vials and subsequently homogenised through gentle mortar grinding.

Aliquots (0.5g) of homogenised sediment were introduced into vacuum vials with 2.5 ml of 6N suprapure HCl and 1 ml of 1.3×10^{-3} M norvaline as recovery standard. After flushing with nitrogen for 5 minutes, the sealed vials were placed in an aluminium heating block and maintained at a temperature of 110 °C for 24 hours to complete hydrolysis of protinaceous material. The hydrolysate was dried in a roto-evaporator at 90 °C. The analyte was thoroughly re-dissolved in organic-free HPLC grade distilled water and made up to a volume of 25 ml.

A standard stock solution was obtained by dissolving the 14 quantitatively most important amino acids in slightly acid HPLC grade water. The stock solution aliquots were kept at -20 °C and thawed as required to make up working standards.

HPLC analysis for amino acids was carried out on their fluorescent derivatives with orthophtaldialdehyde (OPA) made by dissolving 135 mg of OPA in 5 ml of HPLC grade methanol, adding 100 µl of 2-mercaptoethanol and making up to volume (25 ml) with borate buffer. This solution was freshly prepared for each series of HPLC runs and always kept refrigerated in the dark. The derivatisation reaction was carried out with constant agitation for 120 seconds on 1 ml of sample or standard with 400 µl OPA and borate buffer.

Six minutes after the reaction was started, the sample was injected through an Anotop filter (Merck) into the HPLC loop. Gradient elution was carried out in seven steps over 60 minutes as follows:

Time (minutes)	Eluent A (%)	Eluent B (%)
0	100	0
19	100	0
21	60	40
28	60	40
36	40	60
43	40	60
47	0	100
60	0	100
61	100	0

Eluent A was 78% 0.1 M acetate buffer at pH 6.9 plus 18.5% MeOH and 3.5% tetrahydrofurane. Eluent B was 35% of the acetate buffer with 65% MeOH.

The instrument used was a JASCO chromatograph equipped with two pumps, Lichrosorb 10 (Merck) inverse phase 25 cm RP-18 column enclosed in a thermostatic unit, Rheodyne 7125 injector, high pressure solvent mixing module, UV/VIS detector and 821-FP spectrofluorometer. The instrument was coupled to a computer running either Jones Chromatography JCL6000 which was subsequently replaced by a Borwin Access data system providing peak integration, baseline adjustment and calibration facilities.

Individual amino acid concentrations were computed using the formula of Lindsay (1992).

Analytical error was estimated on 5 replicates from core 5B to be between 7 and 10% relative.

Sediment Mineralogy

Parameter Code Definitions

CHLOXDXT Chlorite content of bulk sediment
X-ray diffraction
Per cent

CLAYXDXT Total clay mineral content of bulk sediment
X-ray diffraction
Per cent

CLCTXDXT Calcite content of bulk sediment
X-ray diffraction
Per cent

ILLMXDXT Illite mixed layer (10-14V) content of bulk sediment
X-ray diffraction
Per cent

ILLPXDXT Pure Illite content of bulk sediment
X-ray diffraction
Per cent

KAOLXDXT Kaolinite content of bulk sediment
X-ray diffraction
Per cent

ORTHXDXT Orthoclase feldspar content of bulk sediment
X-ray diffraction
Per cent

PLAGXDXT Plagioclase feldspar content of bulk sediment
X-ray diffraction
Per cent

QRTZXDXT Quartz content of bulk sediment
X-ray diffraction
Per cent

SMECXDXT Smectite content of bulk sediment
X-ray diffraction
Per cent

Originator Code Definitions

Cruises Charles Darwin CD84 and Discovery DI216

89 Dr. Tomasz Boski Universidade do Algarve, Portugal

Originator Protocols

Dr. Tomasz Boski

Samples were collected using either a box corer or a multicorer of standard design (Barnett et al., 1984). See the section on Box Coring Protocols for general details of the corers and sample handling.

The X-ray diffraction analysis was carried out on unoriented powder mounts for overall mineralogical analyses and on oriented aggregates for the determination of the principal clay mineral species.

The unoriented samples were prepared by gentle hand mortar grinding to obtain a powder with a particle size <37 microns. No pre-treatment or pressure was applied during mounting to enhance the random orientation of the particles. The preparations were quantified by selecting specific reflections and applying the correction factors of Cook et al. (1975) as modified by Remy and Ferrel (1989).

The oriented aggregate was prepared according to the following protocol to minimise possible damage to the clay structure. Carbonate was destroyed by a mild (0.2N) HCl attack and chlorides were removed by washing and resuspension in distilled water. The oriented aggregates, from the <2 micron size fraction, were obtained through triple resuspension, decantation and centrifugation at 3000 rpm.

Three distinct series of oriented aggregates were prepared. The first series were run air-dried, treated with ethylene glycol vapour and after heating to 500 °C. The second series were analysed after Li saturation and run air-dried, after heating to 300 °C and treated with ethylene glycol vapour. The third series were run air-dried, after heating to 110 °C and treated with ethylene glycol vapour. The relative proportions of clay minerals were determined from the changes in the diffractograms produced by the various treatments.

The diffraction data were obtained using a Philips PW1390 diffractometer with Cu Kα radiation (30 kV, 30 mA) and a Philips PW1050 vertical goniometer equipped with a 4 degree divergence slit, a 0.2 mm receiving slit, 4 degree scatter slit, graphite monochromator and a proportional detector. The scanning velocity was 1° 2θ per minute.

Sediment Grain Size

Parameter Code Definitions

MDGSPPXX Median grain size
Pipette method for fines with optical microscopy for coarse fraction
Micrometers (microns)

MDGSPSXX Median grain size
Particle sizer
Micrometers (microns)

MDGSSSXX Median grain size
Sieving and/or sedimentation tube method
Micrometers (microns)

MNGSSSXX Mean grain size
Sieving and settling tube method
Micrometres (microns)

MOGSSSXX Grain size mode
Sieving and settling tube method
Micrometres (microns)

PC05SSXX Grain size of the 5th percentile
Sieving and settling tube method
Micrometres (microns)

PC50SSXX Grain size of the 50th percentile
Sieving and settling tube method
Micrometres (microns)

PC90SSXX Grain size of the 90th percentile
Sieving and settling tube method
Micrometres (microns)

PRSCSSMO Proportion of sediment in the mode size class
Sieving and settling tube method
Per Cent

PRSCSSSA Proportion of sediment in the >125 micron size class
Sieving and settling tube method
Per Cent

PRSCSSSB Proportion of sediment in the 63-125 micron size class
Sieving and settling tube method
Per Cent

PRSCSSSC Proportion of sediment in the 30-63 micron size class
Sieving and settling tube method
Per Cent

PRSCSSSD Proportion of sediment in the 15-30 micron size class
Sieving and settling tube method
Per Cent

PRSCSSSE Proportion of sediment in the <15 micron size class
Sieving and settling tube method
Per Cent

PRSCSSSF Proportion of sediment in the >250 micron size class
Sieving and settling tube method
Per Cent

PRSCSSSG Proportion of sediment in the 125-250 micron size class
Sieving and settling tube method
Per Cent

PRSCSSSH Proportion of sediment in the <30 micron size class
Sieving and settling tube method
Per Cent

SDGSSSXX Standard deviation of the grain size
Sieving and settling tube method
Micrometres (microns)

SKGSSSXX Grain size skewness
Sieving and settling tube method
Dimensionless

SPRPWSXC Dry weight proportion of coarse (63-1000 micron) size fraction
Wet sieving
Per cent

SPRPWSXF Dry weight proportion of fine (<63 micron) size fraction
Wet sieving
Per cent

Originator Code Definitions

Charles Darwin cruise CD84

15	Prof. Nick McCave	Cambridge University, UK
89	Dr. Tomasz Boski	Universidade do Algarve, Portugal

Discovery cruise DI216

89	Dr. Tomasz Boski	Universidade do Algarve, Portugal

Pelagia cruise PLG93

82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Pelagia cruise PLG95A

87	Prof. Carlo Heip	NIOO, the Netherlands

Pelagia cruise PLG95B and Charles Darwin cruise CD86

75	Dr. Tjeerd van Weering	NIOZ, Texel, the Netherlands

Meteor cruise M27_1

82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Cote d'Aquitaine cruises NAOX1, NAOX2 and NAOX3, Auriga cruises PLUTUR2 and PLUTUR3 and Anromeda cruise PLUTUR6

98	Dr. J-M Jouanneau	University of Bordeaux, France

Meteor cruise M30_1

51	Prof. Wolfgang Balzer	University of Bremen, Germany

Originator Protocols

Professor Nick McCave

Samples were collected using a Kasten core. See the sections on Kasten Coring Protocols and Box Coring Protocols for details of the corer and sample handling. The fine (<63 micron) and coarse fractions of the sediment were separated by wet sieving and weighed.

Dr. Tomasz Boski

Samples were collected using either a box corer or a multicorer of standard design (Barnett et al., 1984). See the section on Box Coring Protocols for general details of the corer and sample handling.

The box core samples from CD84 were treated with peroxide to destroy organic matter then washed on a 63 micron sieve. The fine fraction was agitated and sampled by pipette over a two hour period to determine 7 classes of grain size. The coarse fraction was examined using a binocular microscope.

The multicore samples from DI216 were separated into coarse and fine fractions by washing on a 63 micron sieve. These were then analysed using a Micrometrics SediGraph 5000ET particle sizer with a computer interface devised by Jones et al. (1988).

Dr. Tjeerd van Weering

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols) or piston corer. 9 cm diameter sub-cores were taken from the box cores.

The sub-cores were extruded and cut into slices. Untreated samples were sent to the Netherlands Geological Survey in Haarlem for grain size analysis. The samples were dispersed into suspension using ultrasound and assayed using a Malvern MasterSizer X Version 1.2a. A focal length of 1000 mm was used for coarse grained sediments (cores CD86-01, CD86-02 and CD86-11) and 300 mm was used for all other samples.

The median grain size from this analysis was included in the database. However, full size spectra were supplied and may be obtained from BODC on request.

Dr. Thomas Soltwedel

Samples were usually collected using a multicorer of standard design (Barnett et al., 1984. However, for some stations on Pelagia PLG93, samples were collected using the NIOZ box corer (see the section on Box Coring Protocols).

Sub-sampling was achieved using 5 ml or 20 ml disposable syringes as small piston corers to take samples down to 10 cm depth. Between three and five replicate samples were taken from each core.

The syringe contents were sectioned into approximately 1 cm slices back in the laboratory. The sediment grain size distribution was obtained by sieving sediment through mesh sizes of 32, 63, 125, 250, 500 and 1000 microns. The

median grain size was estimated from the resulting cumulative frequency curve plotted from the weights retained on each sieve.

Professor Carlo Heip

Samples were collected using the NIOZ box corer (see the section on Box Coring Protocols). 10 cm diameter sub-cores were taken using plastic tubes and cut into sections.

Sediment grain size distribution was determined using a Malvern Particle Sizer 3600 EC.

Dr. Jean-Marie Jouanneau

Cores were taken using either a box corer or a MARK I minicorer. Grain size analysis was performed using the classic method of sieving and settling tubes.

Professor Wolfgang Balzer

Cores were collected using a multicorer of standard design (Barnett et al., 1984). The core tubes were transferred to a refrigerated laboratory and cut into sections. The solid samples were wet sieved through a 62 micron sieve, dried at 110 °C and weighed.

Pore Water Dissolved Oxygen and Resistivity

Parameter Code Definitions

DOXYMETX Micro-electrode oxygen
Oxygen micro-electrode usually mounted on a micro-manipulator
Micromoles/litre

DOXYWITX Winkler oxygen
Winkler titration
Micromoles/litre

REFFMEXX Resistivity formation factor
Pt micro-electrode (ratio of resistivity/resistivity of overlying water)
Dimensionless

Originator Code Definitions

Pelagia cruise PLG93 and Charles Darwin cruise CD86

11	Dr. Wim Helder	NIOZ, Texel, the Netherlands
82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Pelagia Cruise PLG95B

11	Dr. Wim Helder	NIOZ, Texel, the Netherlands

Meteor cruises M27_1 and M30_1

82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Originator Protocols

Dr. Wim Helder

Pore water oxygen and resistivity profiles were either measured on deck by profiling a box core (see Box Coring Protocols) or in-situ using the Temperature Resistivity Oxygen Lander (TROL) benthic lander. TROL is

equipped with five mono-cathodic oxygen micro-electrodes (Revsbech and Jørgensen, 1986) and a resistivity probe (Andrews and Bennett, 1981). Details of TROL characteristics are given in Tengberg et al. (1995) and Epping and Helder (1996).

Box core samples were profiled as soon as possible after the sample arrived on deck using Commercial Clark type electrodes (Diamond corp., Ann Abor, USA, Type 737) with a 60 micron tip and a four-wired platinum resistivity electrode (Andrews and Bennett, 1981) mounted on a micro-manipulator. Profile measurements were made every 100 microns unless a high penetration depth was to be expected in which case the measurements were taken every 1 or 5 mm. The oxygen electrodes were calibrated by comparing their outputs in the water overlying the sediment against Winkler titration measurements.

The multiple electrode profiles were examined and any deemed faulty were rejected. Data from the remaining electrodes were averaged.

Resistivity data are presented as the formation factor which is defined as the ratio of the resistivity of the pore water over the resistivity of the overlying water.

Dr. Thomas Soltwedel

Samples were collected using either a multicorer of standard design (Barnett et al., 1984) or, on cruise CD86, the NIOZ box corer. See the section on Box Coring Protocols for more details.

Unlike other data sets in this section, the data documented here are not pore water oxygen concentration profiles but oxygen concentrations of the water overlying the sediment. These were obtained by siphoning off aliquots of the bottom water overlying the sediment in the core tube which were analysed for dissolved oxygen by the Winkler technique.

Pore Water Nutrients, Dissolved Carbon, Sulphate and Metals

Parameter Code Definitions

AMONAAD2 Dissolved ammonium
Colorometric autoanalysis (0.4/0.45 μm pore filtered)
Micromoles/litre

CORGCOD2 Dissolved organic carbon
High temperature Pt catalytic oxidation (0.4/0.45 μm pore filtered)
Micromoles/litre

FEDVAAD2 Dissolved ferrous (divalent) iron
Colorometric autoanalysis (Ferrospectral 550nm) (0.4/0.45 μm pore filtered)
Nanomoles per litre

MNDVAAD2 Dissolved manganous (divalent) manganese
Colorometric autoanalysis (Formaldoxime 480nm) (0.4/0.45 μm pore filtered)
Nanomoles per litre

MNXXAAD2 Dissolved total manganese
Atomic absorption (0.4/0.45 μm pore filtered)
Nanomoles per litre

NTRIAAD2 Dissolved nitrite
Colorometric autoanalysis (0.4/0.45 μm pore filtered)
Micromoles/litre

NTRZAAD2 Dissolved nitrate + nitrite
Colorometric autoanalysis (0.4/0.45 μm pore filtered)
Micromoles/litre

NTRZAADC Dissolved nitrate + nitrite
Colorometric autoanalysis (centrifuged)
Micromoles/litre

PHOSAAD2 Dissolved phosphate
Colorometric autoanalysis (0.4/0.45 μm pore filtered)
Micromoles/litre

PHOSAADC Dissolved phosphate
Colorometric autoanalysis (centrifuged)
Micromoles/litre

SLCAAAD2 Dissolved silicate
Colorometric autoanalysis (0.4/0.45 μm pore filtered)
Micromoles/litre

SLCAAAD2C Dissolved silicate
Colorometric autoanalysis (centrifuged)
Micromoles/litre

SPHTMAD2 Dissolved sulphate
Manual colorometric analysis after barium/gelatine treatment (0.4/0.45 μm pore filtered)
Micromoles/litre

TCO2CAD2 Total dissolved inorganic carbon (TCO_2)
Quantification of acid-liberated CO_2 using a CO_2 analyser
Micromoles/litre

UREAAAD2 Dissolved urea
Colorometric autoanalysis (0.4/0.45 μm pore filtered)
Micromoles/litre

Originator Code Definitions

Cruises Pelagia PLG93 and PLG95B and Charles Darwin CD86

11	Dr. Wim Helder	NIOZ, Texel, the Netherlands

Cruises Discovery DI216 and Meteor M30_1

51	Prof. Wolfgang Balzer	University of Bremen, Germany

Originator Protocols

Dr. Wim Helder

Sediment samples were collected using either the NIOZ box corer or a multicorer of standard design (Barnett et al., 1984). See the section on Box Coring Protocols for general details of the corer and sample handling.

Both corer types used were designed to preserve the sediment/water interface and to provide intact samples of overlying bottom water. Aliquots of this were routinely analysed for nutrients and oxygen and compared. The

data were compared with those from near-bed CTD rosette bottle samples. If significant differences were observed, it was assumed that mixing had occurred between the overlying water and the pore waters and pore water profiles were not determined.

Sub-cores were taken using 54 mm diameter Plexiglas tubes, leaving a depth of approximately 4 cm of overlying water. The sediment was extruded out of this tube and cut into slices. The pore water was extracted from each slice using nitrogen at 2 atmospheres pressure in a Teflon Reeburgh squeezer (Reeburgh, 1967).

The samples were filtered through 0.45 micron acrodisc filters prior to analysis.

Nutrients

The following chemistries were used:

Ammonium:	Phenol method
Phosphate:	Ammonium molybdate / ascorbic acid method
Nitrate / nitrite:	Sulphanylamide / napthylethylenediamine method using a Cu/Cd coil (efficiency >98%) for reduction
Silicate:	Ammonium molybdate / ascorbic acid method

Samples were always analysed from the surface to the bottom to minimise the risk of cross-sample contamination.

Working standards were freshly prepared daily by diluting stock standards to the required concentration with natural, aged, low-nutrient seawater. The nutrient concentrations in this were determined by manual colorometric analysis. The low-nutrient seawater was also used as a wash between samples. A second mixed nutrient stock, poisoned with 0.2% chloroform or 20 mg/l $HgCl_2$, was used as an independent check. Pipettes and volumetric flasks were calibrated before each cruise and standard batches were intercalibrated.

Accuracy of analyses is reported as about 1% of the full scale value for nitrate, nitrite and silicate and 2% of the full scale for phosphate and ammonium.

The oxidised nitrogen data were reported as nitrate and nitrite, the nitrate values having been computed by subtracting nitrite from nitrate plus nitrite. BODC practice is to store nitrate plus nitrite and the values in the database have been determined by summing the nitrate and nitrite values supplied.

Sulphate

Sulphate was determined by precipitation with barium chloride following the method of Vosjan and Beke (1971), NIOZ-report 1971-11.

Metals

Mn^{2+} and Fe^{2+} concentrations were determined colorometrically. Iron was coloured with Ferrospectral and measured at 550 nm following the protocol of Stookey, 1970. Manganese was coloured by the Formaldoxime reaction and measured at 480 nm following the method of Brewer and Spencer, 1971.

Professor Wolfgang Balzer

Cores were collected using a multicorer of standard design (Barnett et al., 1984). Only samples judged at the time of recovery to have an undisturbed sediment-water interface were used for pore water extraction. The cores used had clear overlying water and often intact biological structures were present on the sediment surface.

The cores were transferred immediately to a constant temperature laboratory set at sea floor temperature. The sediments were extruded, sectioned and the samples packed into centrifuge tubes. Pore waters were extracted by centrifuging under refrigeration for 25 minutes at 5000 rpm. for some stations, separate sub-cores were sectioned and the pore waters extracted by squeezing. On Discovery DI216 these operations were performed in an argon atmosphere.

The pore waters were analysed as follows:

Nutrients

Nutrients were either determined on board ship or determined back in the laboratory on frozen samples. On Discovery DI216, the analyses were done on the ship using a ChemLab autoanalyser employing standard colorometric techniques. On land, standard colorometric techniques as described in Grasshoff et al. (1983) were employed.

Manganese

The supernatant from centrifuging was taken up in a plastic syringe and filtered through acid-cleaned 0.4 micron Nuclepore or Teflon filters. The samples were acidified to a pH of about 2 using sub-boiled nitric acid and stored in acid-cleaned polypropylene tubes. Analysis was done on diluted samples by direct injection into a Zeeman-GFAAS.

Dissolved Organic and Inorganic Carbon

The samples for DOC analysis were obtained by squeezing out the pore waters through 0.45 micron Teflon filters using nitrogen. The samples were acidified and sealed in clean glass vials. These were stored refrigerated and analysed on board ship within hours to days using a high temperature oxidation technique (Sharp et al., 1994).

Dissolved inorganic carbon was determined by quantification of the amount of carbon dioxide liberated upon acidification of the filtered pore water samples.

Nitrous Oxide in Pore Waters

Parameter Code Definitions

DN2OGCTX Dissolved nitrous oxide
Gas chromatography (unfiltered water)
Nanomoles per litre

Originator Code Definitions

Cruise Meteor M30_1

51 Prof. Wolfgang Balzer Bremen University, Germany

Originator Protocols

Professor Wolfgang Balzer

Cores were collected using a large box corer, known as the 'Giant Box Core'. A series of small Plexiglas tubes were pressed into the sediment and removed with a few cm of overlying water and some air to prevent the water becoming anoxic.

The sub-cores were transferred to a refrigerated laboratory for processing. The sediment was extruded and cut into slices which were placed in 50 ml glass jars. These were sealed with brass lids having central rubber septa to allow sampling of the head space.

Nitrous oxide was determined immediately using GC-ECD and a packed Poropak Q column for separation.

Foraminiferal Coiling

Parameter Code Definitions

GHDXSRBS Proportion of Globorotalia hirsuta tests with dextral coiling
Hand picking from >150 micron fraction (shipboard: variable sample size)
Per Cent

GHDXFVBS Proportion of Globorotalia hirsuta tests with dextral coiling
Hand picking from >150 micron fraction (laboratory: fixed volume sample)
Per Cent

GTDXSRBS Proportion of Globorotalia truncatulinoides tests with dextral coiling
Hand picking from >150 micron fraction (shipboard: variable sample size)
Per Cent

GTDXFVBS Proportion of Globorotalia truncatulinoides tests with dextral coiling
Hand picking from >150 micron fraction (laboratory: fixed volume sample)
Per Cent

NPDXSRBS Proportion of Neogloboquadrina pachyderma tests with dextral coiling
Hand picking from >150 micron fraction (shipboard: variable sample size)
Per Cent

NPDXFVBS Proportion of Neogloboquadrina pachyderma tests with dextral coiling
Hand picking from >150 micron fraction (laboratory: fixed volume sample)
Per Cent

Originator Code Definitions

Pelagia cruises PLG93 and PLG95B and Charles Darwin cruise CD86

75 Dr. Tjeerd van Weering NIOZ, Texel, the Netherlands

Originator Protocols

Dr. Tjeerd van Weering

The objective of these measurements was the biostratigraphic correlation and the determination of relative ages of sediment cores by looking at the relative proportion of foraminifera tests that are right-coiled. In the Bay of Biscay area, N pachyderma was predominantly left-coiled during the late Glacial (19-10 ka BP), but dominantly right-coiled in the Holocene (10-0 ka BP). G. truncatulinoides was dominantly right-coiled in the late Glacial and in the late Holocene (7-0 ka BP), but showed a shift to left-coiling in the early Holocene (10-7 ka BP). G. hirstula was left-coiled in the late Glacial and early Holocene, but dominantly right-coiled in the late Holocene.

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols) or piston corer. 9 cm diameter sub-cores were taken from the box cores.

The foraminifera populations were examined quickly on board ship. Small sediment samples were taken every 5 or 10 cm down the core and passed through a 150 μm sieve. The coiling sense of approximately 50 specimens of each species was determined in the total sieve residue. Where possible, determinations were made on replicate box cores.

More accurate determinations were done back in the laboratory on fixed volume samples. These were dried, weighed and then washed through a set of 150 μm and 63 μm sieves. The coiling sense of approximately 50 specimens per species was then determined in splits of the >150 μm residue.

Radiocarbon Dating

Parameter Code Definitions

SAGEMCGB Corrected AMS ^{14}C sediment age (Globigerina bulloides tests)
Accelerated mass spectrometry on picked tests then Stuiver and Reimer correction
Years before present (1950)

SAGEMCNP Corrected AMS ^{14}C sediment age (Neogloboquadrina pachyderma tests)
Accelerated mass spectrometry on picked tests then Stuiver and Reimer correction
Years before present (1950)

SAGEMSGB AMS ^{14}C sediment age (Globigerina bulloides tests)
Accelerated mass spectrometry on picked tests
Years before present (1950)

SAGEMSNP AMS ^{14}C sediment age (Neogloboquadrina pachyderma tests)
Accelerated mass spectrometry on picked tests
Years before present (1950)

SESAMCGB Corrected AMS ^{14}C sediment age (Globigerina bulloides tests) standard error
Accelerated mass spectrometry on picked tests then Stuiver and Reimer correction
Years

SESAMCNP Corrected AMS ^{14}C sediment age (Neogloboquadrina pachyderma tests) standard error
Accelerated mass spectrometry on picked tests then Stuiver and Reimer correction
Years

SESAMSGB AMS ^{14}C sediment age (Globigerina bulloides tests) standard error
Accelerated mass spectrometry on picked tests
Years

SESAMSNP AMS ^{14}C sediment age (Neogloboquadrina pachyderma tests) standard error
Accelerated mass spectrometry on picked tests
Years

Originator Code Definitions

Charles Darwin cruise CD86 and Pelagia cruise PLG95B

75 Dr. Tjeerd van Weering NIOZ, Texel, the Netherlands

Originator Protocols

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols) or piston corer. 9 cm diameter sub-cores were taken from the box cores.

The cores were cut into sections and the sediment washed through a 150 μm sieve. Specimens of Globigerina bulloides or, if these were unavailable, left-coiled Neogloboquadrina pachyderma were hand picked from the >150 μm size fraction. These were ultrasonically cleaned and aliquots of approximately 20 mg were sent to the R.J. van der Graaff Laboratory, Utrecht for age determination by accelerated mass spectrometry.

The raw ages were corrected using a reservoir age of 402 years and the calibration program of Stuiver and Reimer, 1993.

Pigments

Parameter Code Definitions

CPCNHPXT Chlorophyll-a plus phaeophorbides content of bulk sediment
HPLC assay of acetone extract
Micrograms per gram of dry sediment

SCPEFLTX Total chloroplastic pigment in sediment
Fluorometric assay of acetone extraction
Migrograms per ml of wet sediment

SDSPFLTX Standard deviation of total chloroplastic pigment in sediment
Fluorometric assay of acetone extraction
Micrograms per ml of wet sediment

Originator Code Definitions

Charles Darwin cruise CD86 and Pelagia cruises PLG93 and PLG95A

95	Prof. Peter de Wilde	NIOZ, Texel, the Netherlands
82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Meteor cruises M27_1 and M30_1, Pelagia cruise PLG95B and Valdivia cruise VLD137

82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Originator Protocols

Dr. Thomas Soltwedel

Samples were usually collected using a multicorer of standard design (Barnett et al., 1984). However, for some stations on the Pelagia cruises and CD86, samples were collected using the NIOZ box corer (see the section on Box Coring Protocols).

Sub-sampling was achieved using 5 ml disposable syringes as small piston corers to take samples down to 10 cm depth. Between three and five replicate samples were taken from each core. The syringes containing the

sediment were wrapped in aluminium foil to protect them from light, shock frozen at -80 °C and stored at -18 °C.

Back in the laboratory, the sediment from the syringes was cut into 1 cm sections. Each sample was mixed with 8 ml of 90% acetone in test tubes sealed with a lamella stopper and placed in a cell grinder for two minutes to extract the pigments. After this, the samples were centrifuged for 30 minutes at 4000 rpm.

The liquid extract was removed with a pipette and assayed in a Turner Designs fluorometer in a thin-walled cuvette. The instrument was calibrated against chlorophyll standards.

Professor Peter de Wilde

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols). The samples were freeze dried before being extracted into acetone containing a fixed volume of water. Pigments were assayed by HPLC using eluents, gradients and column similar to those described in Wright et al., 1991. Detection was by a photodiode array coupled with a fluorometer and the pigments were quantified as described in Tahey et al., 1994.

Organic Biomarkers

Parameter Code Definitions

DNACHPXT Deoxyribonucleic acid (DNA) content of bulk sediment
HPLC assay of acidified EDTA/SDS extract
Micrograms per gram of dry sediment

PHLPPHTX Total phospholipids in sediment
Photometric assay (NH4molybdate/malachite green) of chloroform extract
Nanomoles/ml wet sediment

RNACHPXT Ribonucleic acid (RNA) content of bulk sediment
HPLC assay of acidified EDTA/SDS extract
Micrograms per gram of dry sediment

SDHAFLTX Standard deviation of hydrolytic activity within sediment
Incubation of sample doped with fluorescine diacetate
Nanomoles/ml wet sediment/hour

SDNADSTX Deoxyribonucleic acid (DNA) in sediment
Fluorescence determination of DAPI stain
Micrograms per ml of wet sediment

SDPLPHTX Standard deviation of total phospholipids in sediment
Photometric assay (NH4 molybdate/malachite green) of chloroform extract
Nanomoles/ml wet sediment

SDPRPHTX Standard deviation of total protein in sediment
Photometric assay of NaOH hydrolysed sample indicated by Coomassie blue
Micrograms per ml of wet sediment

SDSDDSTX Standard deviation of DNA in sediment
Fluorescence determination of DAPI stain
Micrograms per ml of wet sediment

SDTAELTX Standard deviation of total adenylates as ATP
Firefly enzyme luminescence of sediment plus pore water
Nanograms per ml of wet sediment

SHTAFLTX Hydrolytic activity within sediment
Incubation of sample doped with fluorescine diacetate
Nanomoles/ml wet sediment/hour

SPROPHTX Total protein in sediment
Photometric assay of NaOH hydrolysed sample indicated by Coomassie blue
Micrograms per ml of wet sediment

TADNELTX Total adenylates as ATP
Firefly enzyme luminescence of sediment plus pore water
Nanograms per ml of wet sediment

Originator Code Definitions

Charles Darwin cruise CD86, Pelagia cruises PLG93 and PLG95A

95	Prof. Peter de Wilde	NIOZ, Texel, the Netherlands
82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Meteor cruises M27_1 and M30_1, Pelagia cruise PLG95B and Valdivia cruise VLD137

82	Dr. Thomas Soltwedel	Alfred Wegener Institut, Bremerhaven, Germany

Originator Protocols

Dr. Thomas Soltwedel

Samples were usually collected using a multicorer of standard design (Barnett et al., 1984). However, for some stations on the Pelagia cruises and CD86, samples were collected using the NIOZ box corer (see the section on Box Coring Protocols).

Sub-sampling was achieved using 20 ml disposable syringes as small piston corers to take samples down to 10 cm depth. Between three and five replicate samples were taken from each core. The syringes containing the sediment were wrapped in aluminium foil to protect them from light, shock frozen at -80 °C and stored at -18 °C.

Back in the laboratory, the sediment from the syringes was cut into 1 cm sections for analysis.

DNA

The sediment samples were placed in a test tube with 3 ml of phosphate buffer and the cells broken down by ultrasound. The homogenisation was centrifuged for 20 minutes at 6000 rpm at 0 °C.

A 1 ml aliquot of sample was drawn off and 0.96 ml of Tris buffer added. The fluorescence (excitation 372 nm, measurement at 454 nm) of this mixture was determined to give a blank value. 40 µl of DAPI (2 mg/l) were then added and the fluorescence immediately measured again. The detection limit of this method was estimated at 1 µg of DNA in the sample.

Proteins

The samples for protein analysis were dried for at least 48 hours at 60 °C until completely dry. 3 ml of 0.5 N sodium hydroxide was then added and the samples were allowed to hydrolyse for two hours in a water bath at 60 °C.

After hydrolysis, the samples were centrifuged at 3500 rpm for 10 minutes. The supernatant liquid was decanted off and the remaining sediment was washed with 2 ml of 0.5N sodium hydroxide. This was again centrifuged and the supernatant added to the aliquot previously obtained.

The full hydrolysis procedure was repeated five more times and the extracts from all stages were combined.

Test tubes were filled with 2 ml of Coomassie blue, taking care to keep the reagent cool for as long as possible, together with an aliquot of the hydrolate. After a short (2 minutes measured with a stop watch) delay to allow the hydrolysed proteins to react, the samples were assayed at a wavelength of 595 nm.

Frequent measurements were made of reagent blanks and the instrument was calibrated using a series of standards up to 150 µg of globulin per ml on each day when measurements were made. If sample concentrations exceeded the top standard, they were brought into range by quantitative dilution.

Adenylates

The adenylate extractions were carried out on board ship. The sediment samples were washed by suspending into 3 ml of cold phosphate buffer solution. The suspension was centrifuged at 3500 rpm for 20 minutes at 0 °C and the supernatant liquid was siphoned off and discarded.

The sediment was then added to 3 ml of hot (90 °C) glycine buffer and left to extract for 90 minutes in a water bath. The samples were then centrifuged at

3500 rpm for 10 minutes. The sample extracts were decanted into Greisner test tubes, sealed with lamella stoppers and frozen.

Calibration standards were prepared in parallel with the samples, frozen and stored under the same conditions as the samples.

Back on land, total adenylate was determined by adding the following reagents to 1 ml aliquots of the extracts:

0.2 ml Tris-HCl buffer
0.1 ml K^+, Mg^{2+} solution
0.01 ml 0.6 N sulphuric acid
0.01 ml Phosphoenolpyruvat (PEP) solution

After thorough mixing, 0.02 ml of pyruvatkinase and 0.02 ml of myokinase were added and the extracts incubated at 30 °C for 45 minutes in a water bath.

Aliquots of 100 µl of incubated sample were placed in cuvettes filled with 1 ml of arsenate buffer. After mixing, 100 µl of Firefly enzyme was added and the luminance measured on a BioOrbit 1250 Luminometer. Precautions were taken (through careful control of procedure times) against errors due to variation in the activity of the Firefly enzyme.

Hydrolytic Activity

Hydrolytic activity was determined on board ship on fresh samples as soon as possible after the core was taken. The sediment was suspended into 4 ml of filtered sea water and 100 µl of fluorescine-diacetate in acetone was added. The doped solution was incubated with agitation at 2 °C.

At half-hourly intervals, 500 µl of sample was taken from the incubation, diluted with 3 ml of filtered sea water and cold centrifuged at 4000 rpm for 10 minutes. The concentration of fluorescine liberated by hydrolytic activity was determined fluorometrically. This process was repeated over an incubation time of four hours to determine the quantity of fluorescine produced per unit sediment per unit time.

Phospholipids

The method for determination of phospholipids was based on the protocol of Findlay et al., 1989. The phospholipids were extracted by adding wet sediment to a mixture of chloroform, methanol, distilled water and phosphate buffer over several days. The chloroform phase was pipetted off, filtered and evaporated to dryness under nitrogen.

The residue was treated with potassium peroxodisulphate solution and incubated in a sealed vial at 95 °C for a day. Ammonium molybdate and malachite green solution were then added and the solution assayed at a

wavelength of 610 nm on a photometer after allowing time for the reactions to complete.

Professor Peter de Wilde

Samples were taken using the NIOZ box corer (see the section on Box Coring Protocols). The samples were deep frozen in liquid nitrogen on board ship and stored at -80 °C.

Back in the laboratory, the nucleic acids were extracted in a Tris-HCl buffer containing 10% EDTA and 0.2% SDS. The mixture was sonicated and centrifuged at room temperature. The supernatant was filtered and injected directly into an HPLC coupled to an absorbance detector (260 nm). The column, flow rate, eluents and gradient were similar to those described by Copella et al., 1987.

RNA and DNA were quantified using standards (MERCK) from calf thymus and baker's yeast. Detector output was linearly proportional to concentration over the range of standards used. Recovery of standards added to sediments varied around 90%.

Kasten Coring Protocols

Cambridge University

The Kasten Corer used was built to the specifications of Kuehl et al. (1985). The barrel (3m long and 15 cm square) had one removable side, which acted as a lid, and a mechanism to allow the core to be extruded sideways and sub-sampled into slabs of any desired size.

A sliding weight provided extra stability to keep the barrel vertical during penetration. During recovery, a perforated plate was fitted into the barrel and pressed onto the core surface with a rod before the barrel was brought on board to prevent slumping. This tended to destroy the integrity of the top 2cm of the core, but produced a better result than a slumped core.

The corer successfully produced cores up to 2.6m long, providing a sedimentary record from the present day back to the late Pleistocene. The large size of the core (15 cm square) provided adequate material for sub-samples to be provided for several research groups.

The core was sub-sampled by pressing square section PVC conduit (1m long and 6cm square) into the opened surface of the core. Samples (approximately 10 cubic centimetres volume) were taken, using a syringe, from the 4cm spaces between the conduit and stored in airtight containers for water content analysis.

The core was then raised by 6 cm and the conduit was detached from the core using a cheese wire. The samples were then closed, sealed and stored in wooden trays.

A layer of plastic trays (33 cm by 15 cm and 2 cm deep) was then pressed into the core surface, the core raised again and the trays detached using the cheese wire. These were X-rayed on board (images included on the CD-ROM) and later used for bulk magnetic susceptibility determinations. Following this, the trays were frozen and stored. A further set of conduit sub-samples was then taken.

The conduit samples were used for isotopic measurements and various size-fractionated determinations. The syringe samples were used to determine water content, dry bulk density and total and inorganic carbon.

Box Coring Protocols

Cambridge University and Southampton Oceanography Centre

The box cores were obtained using an IOS box corer (Peters et al. (1980)) which takes 50x50 cm square cores up to 0.5m in length. Sub-cores of various diameters were taken using plastic tubes and sectioned using a scheme appropriate to length of profile expected.

Universidade do Algarve

The box cores were obtained using an IOS box corer (Peters et al. (1980)) which takes 50x50 cm square cores up to 0.5m in length. Sub-samples of approximately 35cm length were obtained by pressing tightly closed, cooled perspex tubes into the sediment. The cores were sectioned back at the laboratory into 1 and 2 cm slabs which were immediately centrifuged and freeze dried.

NIOZ

The box corer consisted of a cylindrical (30 or 50 cm diameter) box equipped with a hydraulically dampened closing lid constructed in the NIOZ workshops. Sub-samples of the core were taken by insertion of Plexiglas liners of various diameters after siphoning off the overlying water. All sub-core procedures were performed in a constant temperature laboratory maintained at sea floor temperature.

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