

# Benthic Data Documentation

## Introduction

The benthic data are stored in the database as **along-core profiles** or, in cases where this is not appropriate, as **whole-core properties**. The documentation in this section has been structured in the same manner to parallel the data.

# Benthic Profile Data Documentation

## Introduction

During LOIS (SES) 167 parameters were determined as profiles along sediment cores by 5 investigators using a number of different protocols. The data set includes profiles measured on sediment cores obtained by Multicorers or Sholkovitz Gravity corers. 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 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.

# Benthic Profile Index

## Sediment Organic Carbon, Carbonate, Organic Matter and Nitrogen

Organic carbon, carbonate, organic matter and nitrogen profiles along multicores.

## Carbon Isotopes

Profiles of the stable isotopic composition of organic and inorganic carbon in bulk sediment.

## Radioisotopes

Profiles of the lead, caesium and americium radioisotope concentrations in bulk sediments.

## Radiocarbon Dating

Absolute ages of sediment samples.

## Solid Phase Chemistry

This includes inorganic sulphur (TRIS), XRF major and trace elements plus trace metal speciation data.

## 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.

## Sediment Grain Size

A wide range of statistical sediment grain size parameters computed from size spectra determined by a particle sizer. Some surface area data, determined by nitrogen adsorption, are also included.

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Profiles of total hydrolysable amino acid content.

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Alkanoic acid data in surface sediment for chain lengths from C-11 to C-34.

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Alkanol data in surface sediment for chain lengths from C-11 to C-34.

### **Sediment n-Alkane Content**

Alkane data in surface sediment for chain lengths from C-17 to C-38.

### **Sediment $\alpha,\omega$ -Alkanadioic Acid Content**

$\alpha,\omega$ -alkanadioic acid data in surface sediment for chain lengths from C-10 to C-27.

### **Sediment Pollen Content**

Profiles of sediment pollen counts.

### **References**

Full references for the papers cited in the benthic profile protocol descriptions.

# Sediment Organic Carbon, Carbonate, Organic Matter and Nitrogen

## Parameter Code Definitions

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

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

STOMLIBS Bulk sediment total organic matter  
Weight loss on sample ignition  
Percentage of dry sediment

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

## Originator Code Definitions

### Charles Darwin cruise CD92A

116	Dr. George Wolff	University of Liverpool
118	Dr. Martyn Harvey	CCMS Dunstaffnage Marine Laboratory

### Charles Darwin cruise CD93B and Challenger cruises CH120, CH121C, CH123B, CH124, CH126B and CH128B

118	Dr. Martyn Harvey	CCMS Dunstaffnage Marine Laboratory
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## Originator Protocols

### Dr. George Wolff

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm deep cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site. Two of the cores were immediately frozen (-20°C) for organic matter and metal analysis.

The sediment sample (150mg) was weighed into a pre-weighed vial (7ml) and wetted with Milli-Q water (18 M $\Omega$  cm<sup>-1</sup> resistivity; 2 to 3 drops). HCl (1M) was added dropwise (2 ml) and once evolution of CO<sub>2</sub> had subsided, the sample was sonicated (5 min) and any reaction was allowed to continue (60 min).

The sample was then centrifuged (2000 rpm; 10 min) and the supernatant decanted. Fresh HCl (1M; 2 ml) was added and the sample placed on a mechanical shaker (30 min). This step was repeated if necessary. Following centrifugation, the sample was washed with Milli-Q water until the washings were neutral. The samples were then frozen and freeze-dried.

The determined weight loss was taken as total carbonate. The TOC and TN determinations were carried out on the decarbonated residues using a Carlo Erba 1106 CHN Elemental Analyser. Reproducibility was determined by replicate analyses (x8) of a homogenised sample; the coefficients of variation from the mean concentration for both TOC and TN were less than 3%.

TOC and TN values are weight percentages of dry sediment.

#### **Dr. Martyn Harvey**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). Samples were also collected from site S5 on cruise Challenger CH128B. The samples were separated into 5 cm sections down to 20 cm sediment depth. The organic matter content of the sediment was measured as the weight loss after ignition of the sample.

# Carbon Isotopes

## Parameter Code Definitions

D13CMIBX Bulk sediment inorganic carbon  $^{13}\text{C}$  enrichment (delta- $^{13}\text{C}$ )  
Mass spectrometry on acid liberated  $\text{CO}_2$   
Parts per thousand

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

## Originator Code Definitions

### Cruise Charles Darwin CD92A

78	Dr. A. McKenzie	Scottish Universities Reactor Centre
116	Dr. George Wolff	University of Liverpool
117	Dr. Lynda Mitchell	CCMS Dunstaffnage Marine Laboratory

### Charles Darwin cruise CD93B and Challenger cruises CH120, CH121A, CH121C, CH123B, CH124, CH126B, CH128B.

117	Dr. Lynda Mitchell	CCMS Dunstaffnage Marine Laboratory
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## Originator Protocols

### Dr. Lynda Mitchell

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm deep cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

The 4.5cm long cores were separated onboard the ship into 1cm segments and frozen. Samples were washed with hydrochloric acid, then cleaned by dialysis in distilled water. The sediment samples were then freeze-dried.

Oxidation with  $\text{CuO}$  at approximately  $800^\circ\text{C}$  initiated carbon dioxide production. The carbon dioxide was cryogenically separated before the samples were analysed for sediment organic carbon using a mass spectrometer.

### **Dr. A. McKenzie**

Samples were taken using a Sholkovitz Gravity corer at two sites (N1500 and R1000) during April 1995. The cores were sliced into various segment lengths of between 0.5 cm and 4 cm. Radiocarbon dating was undertaken on the samples, using benzene liquid scintillation. Stable mass spectrometry on acid liberated carbon dioxide gave the inorganic  $^{13}\text{C}$  enrichment. The delta  $^{13}\text{C}$  value was used, with the  $^{14}\text{C}$ -sediment age, to indicate the likely carbon reservoir of the sample.

### **Dr. George Wolff**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm deep cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

The bulk organic carbon-13 compositions of the samples were determined by analysis of  $\text{CO}_2$  gas using a VG Isogas SIRA 12 triple collecting mass spectrometer. Carbon dioxide was generated in evacuated quartz tubes by oxidation of organic matter in the decarbonated sediments ( $\text{CuO}$ ,  $850^\circ\text{C}$ , 2 hours). The  $\text{CO}_2$  was subsequently separated from  $\text{H}_2\text{O}$  and  $\text{NO}_x$  by cryogenic distillation. The isotopic composition of the samples is expressed as  $\delta^{13}\text{C}$  relative to the v-PDB international standard (precision  $\pm 0.05\text{‰}$  or better).

# Radioisotopes

## Parameter Code Definitions

A41CGSXT	Bulk sediment $^{241}\text{Am}$ content Gamma-ray spectroscopy Bequerels per kilogram
C37CGSXT	Bulk sediment $^{137}\text{Cs}$ content Gamma-ray spectroscopy Bequerels per kilogram
L210GSXX	Solid phase $^{210}\text{Pb}$ content Gamma-ray spectroscopy of compressed sediment pellets Bequerels per kilogram
L210IGXX	Solid phase $^{210}\text{Pb}$ content Alpha spectroscopy on plated samples Bequerels per kilogram
S37CGSXT	Bulk sediment $^{137}\text{Cs}$ content standard error Gamma-ray spectroscopy Bequerels per kilogram
S41CGSXT	Bulk sediment $^{241}\text{Am}$ content standard error Gamma-ray spectroscopy Bequerels per kilogram
SL10GSXX	Solid phase $^{210}\text{Pb}$ content standard error Gamma-ray spectroscopy of compressed sediment pellets Bequerels per kilogram
SL10IGXX	Solid phase $^{210}\text{Pb}$ content standard error Alpha spectroscopy on plated samples Bequerels per kilogram
SX10GSXX	Solid phase excess (wrt steady state) $^{210}\text{Pb}$ content standard error Gamma-ray spectroscopy of compressed sediment pellets Bequerels per kilogram
X210GSXX	Solid phase excess (wrt steady state) $^{210}\text{Pb}$ content Gamma-ray spectroscopy of compressed sediment pellets Bequerels per kilogram

## Originator Code Definitions

### Charles Darwin cruise CD92A

40	Dr. Graham Shimmield	CCMS Dunstaffnage Marine Laboratory
78	Dr. A. McKenzie	Scottish Universities Reactor Centre

### Challenger cruise CH128B

78	Dr. A. McKenzie	Scottish Universities Reactor Centre
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## Originator Protocols

### Dr. Graham Shimmield

Sediment cores from four sites (S700, R1000, N1500 and N2000), on a transect across the Hebridean margin, were collected in April 1995. The cores, obtained from a multicorer of standard design (Barnett et al., 1984), were up to 30cm in length. The cores were sliced into 0.5cm (to 10cm depth) and 1cm portions to allow high resolution  $^{210}\text{Pb}$  analysis.

In the laboratory samples were dried at 60°C and salt-corrected dry bulk densities calculated. The samples were ground using a tungsten carbide Tema mill and then completely digested in a mixture of nitric, hydrofluoric, and perchloric acids and a  $^{208}\text{Po}$  (activity approx. 1Bq/ml) tracer added. The solutions were pH adjusted and  $^{210}\text{Po}$  and the  $^{208}\text{Po}$  tracer were plated onto silver discs and counted on EG&G Ortec<sup>TM</sup> surface barrier alpha detectors.  $^{210}\text{Pb}$  activity was determined via measurement of its granddaughter,  $^{210}\text{Po}$ , with which it was assumed to be in secular equilibrium. Standard error values of  $^{210}\text{Pb}$  content were also calculated.

### Dr. A. McKenzie

Sediment cores from sites S700, R1000, N2000 were collected, using a multicorer of standard design (Barnett et al., 1984), during April 1995; cores from site S5 were collected during July 1996. The 10cm deep multicorer cores were separated into 1cm sections. A Sholkovitz Gravity corer was also used at sites N1500 and R1000 during April 1995. The 10cm deep Sholkovitz cores were separated into 0.5 cm sections

$^{210}\text{Pb}$ ,  $^{226}\text{Ra}$ ,  $^{137}\text{Cs}$  and  $^{241}\text{Am}$  values were obtained by preparing the dried sediment samples as compressed discs using a hydraulic press to apply a pressure of 20 tonnes. The discs were sealed in plastic containers and stored for a month to allow equilibration of  $^{222}\text{Rn}$  with  $^{226}\text{Ra}$ . They were then counted using a low-background LEGe detector.

Unsupported  $^{210}\text{Pb}$  concentrations in the sample were determined by subtraction of the  $^{226}\text{Ra}$  concentration from that of total  $^{210}\text{Pb}$ .

Detection efficiencies were determined using sediment samples spiked with known activities of the radionuclides of interest.

# Radiocarbon Dating

## Parameter Code Definitions

SAGESCBX <sup>14</sup>C sediment age (bulk sediment)  
Scintillation counting on purified and trapped acid liberated CO<sub>2</sub>  
Years before present (1950)

SESASCBX <sup>14</sup>C sediment age (bulk sediment) standard error  
Scintillation counting on purified and trapped acid liberated CO<sub>2</sub>  
Years before present (1950)

## Originator Code Definitions

### Cruise Charles Darwin CD92A

78 Dr. A. McKenzie Scottish Universities Reactor Centre

## Originator Protocols

### Dr. A. McKenzie

Sediment cores from sites R1000 and N1500 were collected, using a Sholkovitz Gravity corer, during April 1995. The cores were sliced into various segment lengths of between 0.5cm and 4cm. The profile of sediment ages for the sample from site N1500 was obtained to a depth of 31cm. The depth of the core from site R1000 was 50cm.

The radiocarbon ages were determined using liquid scintillation spectroscopy. The carbonate component of the sediments was hydrolysed using hydrochloric acid on a sample large enough to produce a gram of elemental carbon. The carbon dioxide thus liberated was cryogenically trapped and purified by prolonged vacuum pumping.

Thereafter, the carbon dioxide was sublimed and passed over molten lithium contained in a stainless steel vacuum reaction vessel. The reaction between the carbon dioxide and the lithium yielded lithium-carbide, which was cooled to room temperature.

Distilled water was added to the vessel to generate acetylene, which was passed through a series of purification traps to remove moisture and oxides of nitrogen before being cryogenically trapped and pumped on.

The acetylene was then sublimed and passed over a chromium based catalyst (120°C) on an alumina support. Cyclotrimerisation of the acetylene

yielded benzene, which was removed from the catalyst using a cryogenically cooled finger.

The benzene was stored for a period of around 3 weeks to allow any  $^{222}\text{Rn}$  that might have been present to decay.

A fixed volume of benzene (2g) was added to a scintillation vial containing fixed weights of a primary and secondary fluor (butyl-PBD and bis-MSB respectively). Samples containing less than 2g were made up to the fixed weight using  $^{14}\text{C}$ -free scintillation grade benzene. The samples were counted on a Packard 2000CA/LL together with modern reference standards, background standards and known age samples.

Radiocarbon ages were determined using a form of the first order decay equation:

$$T = \lambda^{-1} \ln (A_0/A_t)$$

where  $T$  = radiocarbon age,  $\lambda$  = decay constant,  $A_0$  = equilibrium living activity (a function of the activity of the modern reference standard) and  $A_t$  = activity of the sample.

# Solid Phase Chemistry

## Parameter Code Definitions

- ALCNXMXT Bulk sediment aluminium content  
X-ray fluorescence on flux diluted fused bead  
Per Cent
- BACNXTXT Bulk sediment barium content  
X-ray fluorescence on pressed powder  
Parts per million
- BRCNXTXT Bulk sediment bromine content  
X-ray fluorescence on pressed powder  
Parts per million
- CACNXMXT Bulk sediment calcium content  
X-ray fluorescence on flux diluted fused bead  
Per Cent
- CECNXTXT Bulk sediment cerium content  
X-ray fluorescence on pressed powder  
Parts per million
- CRCNXTXT Bulk sediment chromium content  
X-ray fluorescence on pressed powder  
Parts per million
- CUCNS1XT Loosely held bulk sediment copper content  
AA assay of Chester et al. (1988) Stage 1 leachate (1M ammonium acetate at pH 7)  
Parts per million
- CUCNS2XT Carbonate and surface associated bulk sediment copper content  
AA assay of Chester et al. (1988) Stage 2 leachate (1M sodium acetate at pH 5)  
Parts per million
- CUCNS3XT Easily reducible bulk sediment copper content  
AA assay of Chester et al. Stage 3 leachate (1M Na acetate (pH5)/0.25M hydroxylamine hydrochloride)  
Parts per million
- CUCNS4XT Moderately reducible bulk sediment copper content  
AA assay of Chester et al. Stage 4 leachate (0.25M hydroxylamine hydrochloride/25pc acetic acid)  
Parts per million

CUCNS5XT Organic matter and sulphide associated bulk sediment Cu content  
AA assay of Chester et al. (1988) Stage 5 leachate (H<sub>2</sub>O<sub>2</sub>/1M ammonium acetate (pH2))  
Parts per million

CUCNSTXT Bulk sediment copper content  
Sum of AA determinations on Chester et al. (1988) staged leachates  
Parts per million

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

FECNS2XT Carbonate and surface associated bulk sediment iron content  
AA assay of Chester et al. (1988) Stage 2 leachate (1M sodium acetate at pH 5)  
Per Cent

FECNS3XT Easily reducible bulk sediment iron content  
AA assay of Chester et al. Stage 3 leachate (1M Na acetate (pH5)/0.25M hydroxylamine hydrochloride)  
Per Cent

FECNS4XT Moderately reducible bulk sediment iron content  
AA assay of Chester et al. Stage 4 leachate (0.25M hydroxylamine hydrochloride/25pc acetic acid)  
Per Cent

FECNS5XT Organic matter and sulphide associated bulk sediment Fe content  
AA assay of Chester et al. (1988) Stage 5 leachate (H<sub>2</sub>O<sub>2</sub>/1M ammonium acetate (pH2))  
Per Cent

FECNSTXT Bulk sediment iron content  
Summation of AA determinations on Chester et al. (1988) staged leachates  
Per Cent

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

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

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

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

MNCNS1XT Loosely held bulk sediment manganese content  
AA assay of Chester et al. (1988) Stage 1 leachate (1M  
Ammonium acetate at pH 7)  
Per Cent

MNCNS2XT Carbonate and surface associated bulk sediment Mn content  
AA assay of Chester et al. (1988) Stage 2 leachate (1M sodium  
acetate at pH 5)  
Per Cent

MNCNS3XT Easily reducible bulk sediment manganese content  
AA assay of Chester et al. Stage 3 leachate (1M Na acetate  
(pH5)/0.25M hydroxylamine hydrochloride)  
Per Cent

MNCNS4XT Moderately reducible bulk sediment manganese content  
AA assay of Chester et al. Stage 4 leachate (0.25M  
hydroxylamine hydrochloride/25pc acetic acid)  
Per Cent

MNCNS5XT Organic matter and sulphide associated bulk sediment Mn  
content  
AA assay of Chester et al. (1988) Stage 5 leachate (H<sub>2</sub>O<sub>2</sub>/1M  
ammonium acetate (pH2))  
Per Cent

MNCNSTXT Bulk sediment manganese content  
Sum of AA determinations on Chester et al. (1988) staged  
leachates  
Per Cent

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

MOCNXTXT Bulk sediment molybdenum content  
X-ray fluorescence on pressed powder  
Parts per million

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

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

NDCNXTXT Bulk sediment neodymium content  
X-ray fluorescence on pressed powder  
Parts per million

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

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

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

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

SCCNXTXT Bulk sediment scandium content  
X-ray fluorescence on pressed powder  
Parts per million

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

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

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

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

TRISMBBS Bulk sediment total reduced inorganic sulphur  
Methylene blue colorimetry  
Milligrams per gram of dry sediment

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

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

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

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

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

## **Originator Code Definitions**

### **Cruise Charles Darwin CD92A**

40	Dr. Graham Shimmield	CCMS Dunstaffnage Marine Laboratory
116	Dr. George Wolff	University of Liverpool
118	Dr. Martyn Harvey	CCMS Dunstaffnage Marine Laboratory

### **Charles Darwin cruise CD93B and Challenger cruises CH120, CH121C, CH123B, CH124, CH126B and CH128B**

118	Dr. Martyn Harvey	CCMS Dunstaffnage Marine Laboratory
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## **Originator Protocols**

### **Dr. Martyn Harvey**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). Samples were also collected from site S5 on cruise Challenger CH128B. The samples were separated into 5cm sections down to a sediment depth of 20cm.

Sub-cores from each depth horizon were injected with  $^{35}\text{S}$ -sulphate and incubated for 24 hours before being frozen. In the laboratory the sediment was acidified and heated to release the sediment sulphide. An aliquot of this sample was taken for sulphide (TRIS) determination by methylene blue method (Cline, 1969). Briefly, this involves adding a couple of reagents to the sample. The reagents react with the TRIS in the sample to produce a blue colouration in proportion to the amount of TRIS present. The level of colouration was measured spectrophotometrically.

### **Dr. Graham Shimmield**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The cores were sliced into 0.5cm intervals for 10cm and 1cm for the remainder of the core (all cores were less than 30cm in length). Three replicate samples were collected from separate deployments of the multicorer at each site. Trace metal analysis was completed on cores from all four sites. Analysis for the major elements was not carried out on the core from site N2000.

#### Major elements

1g of dried sediment was accurately weighed into a platinum crucible and an ultra-pure lithium tetraborate flux, Spectroflux 105 (Johnson-Matthey Chemicals Ltd.) added in a ratio 5:1 (flux to sample). This was placed in a muffle furnace at 1100°C for 20 minutes. After cooling the slight weight loss was made up with the addition of Spectroflux 105. The mixture was reheated on a gas burner until the glass melted.

The molten liquid was poured onto a graphite mould contained inside a brass sleeve (on a hotplate at 220°C) and pressed with an aluminum plunger to produce a 45mm diameter glass disc approximately 1mm thick. After cooling, the discs were stored in a desiccator.

They were analysed for a series of major elements (Si, Al, Fe, Mg, Ca, Na, K, Ti, Mn and P) using a Phillips PW 1450 sequential automatic X-ray spectrometer.

#### Trace elements

3g of dried sediment was accurately weighed and placed in a stainless steel sleeve resting on a highly polished tungsten carbide disc. Both the sleeve and cylinder were enclosed within a larger stainless steel cylinder. A Perspex plunger was inserted into the sleeve and the powder was compacted into a semi-competent disc using hand pressure.

The Perspex plunger and sleeve were removed and sufficient boric acid to cover the sediment disc was added. A large stainless steel plunger was lowered onto the sample and placed under a hydraulic press where 10 tons of pressure was used to compress the sample.

This pressed powder disc with boric acid backing was analysed for a series of trace elements (Sc, Ba, V, La, Ce, Nd, Cr, Ni, Cu, Zn, Pb, Th, U, Rb, Sr, Y, Zr, Nb, Mo, I and Br) using a Phillips PW 1450 sequential automatic X-ray spectrometer.

Both major and trace elements were corrected for the presence of residual sea-salt.

### **Dr. George Wolff**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site. Two of the cores were immediately frozen (-20°C) for organic matter and metal analysis.

Trace metal concentrations and solid state speciation for copper, iron and manganese were determined using a five stage sequential leaching method (Chester et al., 1988). The total authigenic metal concentrations were obtained from the sum of the individual five stages. The principal components associated with each stage in the sequential scheme are as follows:

Stage 1: elements 'loosely held' or 'exchangeable' associations. This was obtained using the reagent 1M ammonium acetate at pH 7.

Stage 2: elements in 'carbonate' and in 'surface associations' with phases such as hydrous iron and manganese oxides, clays and organic matter. This was obtained using the leaching reagent 1M sodium acetate at pH 5.

Stage 3: elements in 'easily reducible' associations, e.g. in 'new' oxides and oxyhydroxides of manganese and amorphous iron oxides. This was obtained using 1M sodium acetate plus 0.25M hydroxylamine hydrochloride.

Stage 4: elements in 'moderately reducible' associations, e.g. in 'aged' manganese oxides and crystalline iron oxides. This was obtained using leaching reagent 0.25M hydroxylamine hydrochloride plus 25% acetic acid.

Stage 5: elements in organic matter and sulphide associations. This was obtained using leaching reagent hydrogen peroxide plus 1M ammonium acetate (pH 2).

Metals were determined using a Perkin-Elmer 2380 flame-AAS. Detection limits were taken to be 3 x standard deviation of the sample blanks. Reproducibility of the sequential leaching scheme (monitored using a standard River Mersey sediment) was generally better than 10%.

Data for iron and manganese were scaled at BODC from the original units ( $\mu\text{g/g}$  or  $\text{mg/g}$  dry sediment) to percentage to conform with database standard units.

# Dry Bulk Density, Porosity and Water Content

## Parameter Code Definitions

- DBDXCMXX Dry bulk density  
Salt-corrected 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
- WCWTD RXX Sediment water content by weight  
Mass difference on oven/freeze drying per unit mass of wet  
sediment  
Per cent

## Originator Code Definitions

### Charles Darwin cruise CD92A

- |     |                      |                                     |
|-----|----------------------|-------------------------------------|
| 40  | Dr. Graham Shimmield | CCMS Dunstaffnage Marine Laboratory |
| 116 | Dr. George Wolff     | University of Liverpool             |
| 118 | Dr. Martyn Harvey    | CCMS Dunstaffnage Marine Laboratory |

### Charles Darwin cruise CD93B and Challenger cruises CH120, CH121C, CH123B, CH124, CH126B and CH128B

- |     |                   |                                     |
|-----|-------------------|-------------------------------------|
| 118 | Dr. Martyn Harvey | CCMS Dunstaffnage Marine Laboratory |
|-----|-------------------|-------------------------------------|

## Originator Protocols

### Dr. Martyn Harvey

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). Samples were also collected from site S5 on cruise Challenger CH128B. The samples were separated into 5cm sections down to a depth of 20cm. The sediment porosity was calculated from the weight of water liberated on oven/freeze drying the sample.

### **Dr. George Wolff**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site. Two cores were immediately frozen (-20°C) for organic matter and metal analysis. The sediment water content by weight was calculated from the mass difference on oven/freeze drying the sample.

### **Dr. Graham Shimmield**

Sediment cores from four sites (S700, R1000, N1500 and N2000), on a transect across the Hebridean margin, were collected in April 1995. The cores, obtained from a multicorer of standard design (Barnett et al., 1984), were up to 30cm in length. The cores were sliced into 0.5cm (to 10cm depth) and 1cm portions. In the laboratory samples were dried at 60°C and salt-corrected dry bulk densities calculated.

# Sediment Grain Size

## Parameter Code Definitions

KRTSPSXX	Grain size kurtosis Particle sizer Dimensionless
MDGSPSXX	Median grain size Particle sizer Micrometres (microns)
MNGSPSXX	Mean grain size Particle sizer Micrometres (microns)
MOGSPSXX	Grain size mode Particle sizer Micrometres (microns)
PC10PSXX	Grain size of the 10th percentile Particle sizer Micrometres (microns)
PC25PSXX	Grain size of the 25th percentile Particle sizer Micrometres (microns)
PC50PSXX	Grain size of the 50th percentile Particle sizer Micrometres (microns)
PC75PSXX	Grain size of the 75th percentile Particle sizer Micrometres (microns)
PC90PSXX	Grain size of the 90th percentile Particle sizer Micrometres (microns)
PRSCPSSN	Proportion of sediment in the 3-38 micron size class Particle sizer Per Cent
SDGSPSXX	Standard deviation of the grain size distribution Particle sizer Micrometres (microns)

SKGSPSXX Grain size skewness  
Particle sizer  
Dimensionless

SSARNAXT Bulk sediment surface area  
Nitrogen adsorption  
Square metres per gram of dry sediment

## **Originator Code Definitions**

### **Charles Darwin cruise CD92A**

116	Dr. George Wolff	University of Liverpool
117	Dr. Lynda Mitchell	CCMS Dunstaffnage Marine Laboratory

### **Challenger cruise CH120**

117	Dr. Lynda Mitchell	CCMS Dunstaffnage Marine Laboratory
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## **Originator Protocols**

### **Dr. George Wolff**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

A Coulter LS130 Particle size counter was used to obtain grain size spectra after pre-treatment of the samples with hydrogen peroxide and "Calgon" dispersing agent. Only data for mean and mode grain size are included. The full spectra are available on request from the originator.

Sediment specific surface areas were measured by nitrogen adsorption, using a five-point BET method on a Micromeritics ASAP 2010. Salt was removed from the samples by washing with Milli-Q water adjusted to pH 8 with ammonium hydroxide. Prior to analysis the freeze-dried samples were outgassed under vacuum at 175°C overnight or until all adsorbed water had been removed.

### **Dr. Lynda Mitchell**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 5cm long cores were sliced into 1cm sections. Grain size data was obtained using a Coulter LS100

particle size counter. Grain size statistics were calculated for the size range 0.4 $\mu\text{m}$  to 900 $\mu\text{m}$ .

# Sediment Amino Acid Content

## Parameter Code Definitions

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

## Originator Code Definitions

### Cruise Charles Darwin CD92A

116 Dr. George Wolff University of Liverpool

## Originator Protocols

### Dr. George Wolff

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

Hydrolysable amino acids were determined by the method of Horsfall and Wolff (1997). Identified amino acids in hydrolysed samples were quantified by comparison of their peak areas with those of the appropriate internal standard (see Horsfall and Wolff, 1997). Detection limits were calculated as 4 x maximum blank values and were in the sub-picomole level (eg. glutamic acid, 0.2 pmoles).

Full procedural blanks using an ashed sediment sample (800°C; 24 h) were carried out periodically throughout the course of the work. Replicate analyses (4) were carried out on a standard mixture to determine the reproducibility of the HPLC analysis. The coefficient of variation (CV) between replicates was <2.5%. In order to determine the analytical reproducibility of the method, a homogenised sediment sample was hydrolysed on six separate occasions and the extracts analysed. The CVs for identified acids were <10% (eg. valine CV = 5.3%, leucine CV = 9.3%).

THAA data were converted by BODC to micrograms per gram of dry sediment from  $\mu\text{mol}$  per gram of dry sediment using an average molecular weight of 122.5, see Patience et al. (1990). BODC also holds amino acid carbon and nitrogen as percentage contributions to TOC and TN (%TaaC and %TaaN; see Cowie and Hedges, 1992) supplied by the originator. However, these

have not been included in the database as they may be easily computed from the data held.

# Sediment Alkanoic Acid Content

## Parameter Code Definitions

- CF11GCTX Bulk sediment 11-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF12GCTX Bulk sediment 12-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF13GCTX Bulk sediment 13-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF14GCTX Bulk sediment 14-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF15GCTX Bulk sediment 15-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF16GCTX Bulk sediment 16-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF17GCTX Bulk sediment 17-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF18GCTX Bulk sediment 18-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF19GCTX Bulk sediment 19-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF20GCTX Bulk sediment 20-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF21GCTX Bulk sediment 21-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF22GCTX Bulk sediment 22-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF23GCTX Bulk sediment 23-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF24GCTX Bulk sediment 24-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF25GCTX Bulk sediment 25-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF26GCTX Bulk sediment 26-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF27GCTX Bulk sediment 27-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF28GCTX Bulk sediment 28-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CF29GCTX Bulk sediment 29-carbon alkanolic acid (unspecified structure)  
content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

- CF30GCTX Bulk sediment 30-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF31GCTX Bulk sediment 31-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF32GCTX Bulk sediment 32-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CF34GCTX Bulk sediment 34-carbon alkanolic acid (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

## Originator Code Definitions

### Charles Darwin cruise CD92A

116 Dr. George Wolff University of Liverpool

## Originator Protocols

### Dr. George Wolff

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

#### Extraction

The extraction method was modified from Wolff et al. (1995). An aliquot of the freeze-dried sediment (2-3g) was extracted in a soxhlet apparatus with dichloromethane (DCM) and methanol (MeOH; 1:1; 100 ml) for 36 hours. 2,21-dimethyldocosane was added to the soxhlet thimble as an internal standard. 5 $\alpha$ (H)-cholestane was added to the round-bottom flask as a recovery standard.

The solvent was evaporated *in vacuo* (to ca. 2ml) and the resulting extract was hydrolysed with 6 % KOH/MeOH (30ml for 12 hours). The neutral fraction

was recovered with *n*-hexane (3 x 30 ml). The aqueous fraction was evaporated *in vacuo* (to 0.5 ml) and mixed with pre-extracted water (DCM, 25 ml).

The mixture was then acidified to pH ~2 with aqueous HCl (6N) and the acid fraction was recovered by extraction with DCM (3 x 30 ml). The acid fraction was cleaned-up over a short silica column (Pasteur pipette) with DCM as eluent. A quantification standard (2,21-dimethyldocosane) was added to the acid fraction after removal of the solvent under nitrogen.

Samples were derivatised by silylation with bis-(trimethylsilyl)-trifluoroacetamide (BSTFA; 50 ml; 80 °C; 30 min) prior to gas chromatography-mass spectrometry.

### Analysis

Gas chromatography-mass spectrometry (GC-MS) was carried out according to Wolff et al. (1995), using a Hewlett-Packard 5890A gas chromatograph fitted with an on-column injector and interfaced to a VG TS-250 mass spectrometer.

The samples were analysed using a DB-5 silica column (30 m x 0.32 mm *i.d.*; 0.25 mm film, J&W Scientific) with helium as carrier gas. The oven temperature was programmed from 40 to 320°C at 6°C min<sup>-1</sup> after 1 min and was held at 320°C for 20 minutes. Typical GC-MS operating conditions were: ionisation potential 70 eV, source temperature 230 °C and trap current 200 mA. Full mass data were acquired from 50-600 D every second, at a mass resolution of 500.

Data were collected on a VAX 3500 workstation, and processed using VG-Opus software.

### Identification and Quantification of individual lipids

Lipids were identified and quantified using GC and GCMS. Comparison of the relative retention indices and mass spectra of the analytes with literature data enabled lipid identification. Semi-quantitative data were determined by comparison of peak areas of the internal standard and the compounds of interest in the reconstructed total ion current (TIC) chromatograms. Reproducibilities of extraction (%RSD) are tested regularly in the University of Liverpool laboratories and are generally better than 10% (see Santos et al., 1994; Prartono and Wolff, in press).

The data are quoted as µg g<sup>-1</sup> dry sediment, and are for the 0 to 1cm surface sediment section only. Whole profile lipid data are available on request from the originator for single cores from the R1000 and N1500 sites.

## Molecular Parameters.

Carbon Preference Index (CPI) and Regional Source Index (RSI) values for *n*-alkanes, *n*-alkanols and *n*-alkanoic acids were determined according to Pelzer and Gagosian (1989). The CPI and RSI values are held by BODC.

# Sediment n-Alkanol Content

## Parameter Code Definitions

- CA11GCTX Bulk sediment 11-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA12GCTX Bulk sediment 12-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA13GCTX Bulk sediment 13-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA14GCTX Bulk sediment 14-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA15GCTX Bulk sediment 15-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA16GCTX Bulk sediment 16-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA17GCTX Bulk sediment 17-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA18GCTX Bulk sediment 18-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA19GCTX Bulk sediment 19-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA20GCTX Bulk sediment 20-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CA21GCTX Bulk sediment 21-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA22GCTX Bulk sediment 22-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA23GCTX Bulk sediment 23-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA24GCTX Bulk sediment 24-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA25GCTX Bulk sediment 25-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA26GCTX Bulk sediment 26-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA27GCTX Bulk sediment 27-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA28GCTX Bulk sediment 28-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA29GCTX Bulk sediment 29-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA30GCTX Bulk sediment 30-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA31GCTX Bulk sediment 31-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA32GCTX Bulk sediment 32-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

CA34GCTX Bulk sediment 34-carbon alkanol (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

## Originator Code Definitions

### Charles Darwin cruise CD92A

116 Dr. George Wolff University of Liverpool

## Originator Protocols

### Dr. George Wolff

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

#### Extraction

The extraction method was modified from Wolff et al. (1995). An aliquot of the freeze-dried sediment (2-3g) was extracted in a soxhlet apparatus with dichloromethane (DCM) and methanol (MeOH; 1:1; 100 ml) for 36 hours. 2,21-dimethyldocosane was added to the soxhlet thimble as an internal standard. 5 $\alpha$ (H)-cholestane was added to the round-bottom flask as a recovery standard.

The solvent was evaporated *in vacuo* (to ca. 2ml) and the resulting extract was hydrolysed with 6 % KOH/MeOH (30ml for 12 hours). The neutral fraction was recovered with *n*-hexane (3 x 30 ml). The aqueous fraction was evaporated *in vacuo* (to 0.5 ml) and mixed with pre-extracted water (DCM, 25 ml).

The mixture was then acidified to pH ~2 with aqueous HCl (6N) and the acid fraction was recovered by extraction with DCM (3 x 30 ml). The acid fraction was cleaned-up over a short silica column (Pasteur pipette) with DCM as eluent. A quantification standard (2,21-dimethyldocosane) was added to the acid fraction after removal of the solvent under nitrogen.

Samples were derivatised by silylation with bis-(trimethylsilyl)-trifluoroacetamide (BSTFA; 50 ml; 80 °C; 30 min) prior to gas chromatography-mass spectrometry.

#### Analysis

Gas chromatography-mass spectrometry (GC-MS) was carried out according to Wolff et al. (1995), using a Hewlett-Packard 5890A gas chromatograph fitted with an on-column injector and interfaced to a VG TS-250 mass spectrometer.

The samples were analysed using a DB-5 silica column (30 m x 0.32 mm *i.d.*; 0.25 mm film, J&W Scientific) with helium as carrier gas. The oven

temperature was programmed from 40 to 320°C at 6°C min<sup>-1</sup> after 1 min and was held at 320°C for 20 minutes. Typical GC-MS operating conditions were: ionisation potential 70 eV, source temperature 230 °C and trap current 200 mA. Full mass data were acquired from 50-600 D every second, at a mass resolution of 500.

Data were collected on a VAX 3500 workstation, and processed using VG-Opus software.

#### Identification and Quantification of individual lipids

Lipids were identified and quantified using GC and GCMS. Comparison of the relative retention indices and mass spectra of the analytes with literature data enabled lipid identification. Semi-quantitative data were determined by comparison of peak areas of the internal standard and the compounds of interest in the reconstructed total ion current (TIC) chromatograms. Reproducibilities of extraction (%RSD) are tested regularly in the University of Liverpool laboratories and are generally better than 10% (see Santos et al., 1994; Prartono and Wolff, in press).

The data are quoted as µg g<sup>-1</sup> dry sediment, and are for the 0 to 1cm surface sediment section only. Whole profile lipid data are available on request from the originator for single cores from the R1000 and N1500 sites.

#### Molecular Parameters.

Carbon Preference Index (CPI) and Regional Source Index (RSI) values for *n*-alkanes, *n*-alkanols and *n*-alkanoic acids were determined according to Pelzer and Gagosian (1989). The CPI and RSI values are held by BODC.

# Sediment n-Alkane Content

## Parameter Code Definitions

- CX17GCTX Bulk sediment 17-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX18GCTX Bulk sediment 18-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX19GCTX Bulk sediment 19-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX20GCTX Bulk sediment 20-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX21GCTX Bulk sediment 21-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX22GCTX Bulk sediment 22-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX23GCTX Bulk sediment 23-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX24GCTX Bulk sediment 24-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX25GCTX Bulk sediment 25-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX26GCTX Bulk sediment 26-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX27GCTX Bulk sediment 27-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

- CX28GCTX Bulk sediment 28-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX29GCTX Bulk sediment 29-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX30GCTX Bulk sediment 30-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX31GCTX Bulk sediment 31-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX32GCTX Bulk sediment 32-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX33GCTX Bulk sediment 33-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX35GCTX Bulk sediment 35-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CX38GCTX Bulk sediment 38-carbon alkane (unspecified structure) content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

## **Originator Code Definitions**

### **Charles Darwin cruise CD92A**

116 Dr. George Wolff University of Liverpool

## **Originator Protocols**

### **Dr. George Wolff**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

## Extraction

The extraction method was modified from Wolff et al. (1995). An aliquot of the freeze-dried sediment (2-3g) was extracted in a soxhlet apparatus with dichloromethane (DCM) and methanol (MeOH; 1:1; 100 ml) for 36 hours. 2,21-dimethyldocosane was added to the soxhlet thimble as an internal standard. 5 $\alpha$ (H)-cholestane was added to the round-bottom flask as a recovery standard.

The solvent was evaporated *in vacuo* (to ca. 2ml) and the resulting extract was hydrolysed with 6 % KOH/MeOH (30ml for 12 hours). The neutral fraction was recovered with *n*-hexane (3 x 30 ml). The aqueous fraction was evaporated *in vacuo* (to 0.5 ml) and mixed with pre-extracted water (DCM, 25 ml).

The mixture was then acidified to pH ~2 with aqueous HCl (6N) and the acid fraction was recovered by extraction with DCM (3 x 30 ml). The acid fraction was cleaned-up over a short silica column (Pasteur pipette) with DCM as eluent. A quantification standard (2,21-dimethyldocosane) was added to the acid fraction after removal of the solvent under nitrogen.

Samples were derivatised by silylation with bis-(trimethylsilyl)-trifluoroacetamide (BSTFA; 50 ml; 80 °C; 30 min) prior to gas chromatography-mass spectrometry.

## Analysis

Gas chromatography-mass spectrometry (GC-MS) was carried out according to Wolff et al. (1995), using a Hewlett-Packard 5890A gas chromatograph fitted with an on-column injector and interfaced to a VG TS-250 mass spectrometer.

The samples were analysed using a DB-5 silica column (30 m x 0.32 mm *i.d.*; 0.25 mm film, J&W Scientific) with helium as carrier gas. The oven temperature was programmed from 40 to 320°C at 6°C min<sup>-1</sup> after 1 min and was held at 320°C for 20 minutes. Typical GC-MS operating conditions were: ionisation potential 70 eV, source temperature 230 °C and trap current 200 mA. Full mass data were acquired from 50-600 D every second, at a mass resolution of 500.

Data were collected on a VAX 3500 workstation, and processed using VG-Opus software.

## Identification and Quantification of individual lipids

Lipids were identified and quantified using GC and GCMS. Comparison of the relative retention indices and mass spectra of the analytes with literature data enabled lipid identification. Semi-quantitative data were determined by comparison of peak areas of the internal standard and the compounds of interest in the reconstructed total ion current (TIC) chromatograms.

Reproducibilities of extraction (%RSD) are tested regularly in the University of Liverpool laboratories and are generally better than 10% (see Santos et al., 1994; Prartono and Wolff, in press).

The data are quoted as  $\mu\text{g g}^{-1}$  dry sediment, and are for the 0 to 1cm surface sediment section only. Whole profile lipid data are available on request from the originator for single cores from the R1000 and N1500 sites.

#### Molecular Parameters

Carbon Preference Index (CPI) and Regional Source Index (RSI) values for *n*-alkanes, *n*-alkanols and *n*-alkanoic acids were determined according to Pelzer and Gagosian (1989). The CPI and RSI values are held by BODC.

## Sediment $\alpha,\omega$ -Alkanadioic Acid Content

### Parameter Code Definitions

- CD10GCTX Bulk sediment 10-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD12GCTX Bulk sediment 12-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD13GCTX Bulk sediment 13-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD14GCTX Bulk sediment 14-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD15GCTX Bulk sediment 15-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD16GCTX Bulk sediment 16-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD17GCTX Bulk sediment 17-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD18GCTX Bulk sediment 18-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD19GCTX Bulk sediment 19-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD20GCTX Bulk sediment 20-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD21GCTX Bulk sediment 21-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

- CD22GCTX Bulk sediment 22-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD23GCTX Bulk sediment 23-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD24GCTX Bulk sediment 24-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD25GCTX Bulk sediment 25-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD26GCTX Bulk sediment 26-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment
- CD27GCTX Bulk sediment 27-carbon alpha-omega alkanadioic acid content  
Gas chromatography-mass spectrometry  
Micrograms per gram of dry sediment

## **Originator Code Definitions**

### **Charles Darwin cruise CD92A**

116 Dr. George Wolff University of Liverpool

## **Originator Protocols**

### **Dr. George Wolff**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

### **Extraction**

The extraction method was modified from Wolff et al. (1995). An aliquot of the freeze-dried sediment (2-3g) was extracted in a soxhlet apparatus with dichloromethane (DCM) and methanol (MeOH; 1:1; 100 ml) for 36 hours. 2,21-dimethyldocosane was added to the soxhlet thimble as an internal

standard. 5 $\alpha$ (H)-cholestane was added to the round-bottom flask as a recovery standard.

The solvent was evaporated *in vacuo* (to ca. 2ml) and the resulting extract was hydrolysed with 6 % KOH/MeOH (30ml for 12 hours). The neutral fraction was recovered with *n*-hexane (3 x 30 ml). The aqueous fraction was evaporated *in vacuo* (to 0.5 ml) and mixed with pre-extracted water (DCM, 25 ml).

The mixture was then acidified to pH ~2 with aqueous HCl (6N) and the acid fraction was recovered by extraction with DCM (3 x 30 ml). The acid fraction was cleaned-up over a short silica column (Pasteur pipette) with DCM as eluent. A quantification standard (2,21-dimethyldocosane) was added to the acid fraction after removal of the solvent under nitrogen.

Samples were derivatised by silylation with bis-(trimethylsilyl)-trifluoroacetamide (BSTFA; 50 ml; 80 °C; 30 min) prior to gas chromatography-mass spectrometry.

#### Analysis

Gas chromatography-mass spectrometry (GC-MS) was carried out according to Wolff et al. (1995), using a Hewlett-Packard 5890A gas chromatograph fitted with an on-column injector and interfaced to a VG TS-250 mass spectrometer.

The samples were analysed using a DB-5 silica column (30 m x 0.32 mm *i.d.*; 0.25 mm film, J&W Scientific) with helium as carrier gas. The oven temperature was programmed from 40 to 320°C at 6°C min<sup>-1</sup> after 1 min and was held at 320°C for 20 minutes. Typical GC-MS operating conditions were: ionisation potential 70 eV, source temperature 230 °C and trap current 200 mA. Full mass data were acquired from 50-600 D every second, at a mass resolution of 500.

Data were collected on a VAX 3500 workstation, and processed using VG-Opus software.

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Lipids were identified and quantified using GC and GCMS. Comparison of the relative retention indices and mass spectra of the analytes with literature data enabled lipid identification. Semi-quantitative data were determined by comparison of peak areas of the internal standard and the compounds of interest in the reconstructed total ion current (TIC) chromatograms. Reproducibilities of extraction (%RSD) are tested regularly in the University of Liverpool laboratories and are generally better than 10% (see Santos et al., 1994; Prartono and Wolff, in press).

The data are quoted as  $\mu\text{g g}^{-1}$  dry sediment, and are for the 0 to 1cm surface sediment section only. Whole profile lipid data are available on request from the originator for single cores from the R1000 and N1500 sites.

#### Molecular Parameters

Carbon Preference Index (CPI) and Regional Source Index (RSI) values for *n*-alkanes, *n*-alkanols and *n*-alkanoic acids were determined according to Pelzer and Gagosian (1989). The CPI and RSI values are held by BODC.

# **Sediment Pollen Content**

## **Parameter Code Definitions**

SPOLMWXT Bulk sediment pollen content  
Moore and Webb (1978) protocols  
Number per gram

## **Originator Code Definitions**

### **Charles Darwin cruise CD92A**

116 Dr. George Wolff University of Liverpool

## **Originator Protocols**

### **Dr. George Wolff**

Samples were collected using a multicorer of standard design (Barnett et al., 1984), at four sites (S700, R1000, N1500, N2000). The 20cm long cores were sliced into 1cm sections. Three replicate samples were collected from separate deployments of the multicorer at each site.

Samples were prepared for pollen analysis using procedures described in (Moore and Webb, 1978).

Briefly, this involved treating the samples with hydrochloric acid, potassium hydroxide, hydrofluoric acid, and sodium pyrophosphate. Acetolysis was performed to remove cellulose. The samples were then stained with safranin and made up in glycerol.

The exotic marker method was used to determine absolute pollen concentrations with >250 counts taken per sample.

## References

- Barnett P.R.O., Watson J. and Connelly D., 1984. A multiple corer for taking virtually undisturbed samples from shelf, bathyal and abyssal sediments. ***Oceanol. Acta***, 7, 399-408.
- Chester R., Thomas A., Lin F.J., Basaham A.S. and Jacinto G., 1988. The solid state speciation of copper in surface water particulates and oceanic sediments. ***Marine Chemistry*** 24, 261-292.
- Cline J.D., 1969. Spectrophotometric determination of hydrogen sulfide in natural waters. ***Limnol. Oceanogr.*** 14, 454-458
- Cowie G.L. and Hedges J.I., 1992. Sources and reactivities of amino acids in a coastal marine sediment. ***Limnology and Oceanography*** 37, 703-724.
- Horsfall I.M. and Wolff G.A., 1997. Hydrolysable amino acids in surficial sediments from the Porcupine Abyssal Plain, northeast Atlantic Ocean. ***Organic Geochemistry***, 26, 311-320.
- Moore P.D. and Webb J.A., 1978, An illustrated guide to pollen analysis. ***Biological Science Texts***, Hodder and Stoughton, London.
- Patience R.L., Clayton C.J., Kearsley A.T., Rowland S.J., Bishop A.N., Rees A.W.G., Bibby K.G. and Hopper A.C., 1990. An integrated biochemical, geochemical and sedimentological study of organic diagenesis in sediments from Leg 112. In Suess E. et al. ***Proc. Ocean Drilling Prog., Scientific Results***, 112, 135-153.
- Pelzer E.T. and Gagosian R.B., 1989, Organic geochemistry of aerosols over the Pacific Ocean. In Duce R.A. et al. ***Chemical Oceanography***, 10, 281-338. Academic Press, London.
- Prartono T. and Wolff G.A., in press. Organic geochemistry of lacustrine sediments: evidence for the changing trophic status of the lake, Rostherne Mere, UK. ***Organic Geochemistry***.
- Santos V., Billett D.S.M., Rice A.L. and Wolff G.A., 1994. The fate of organic matter in deep sea sediments from the Porcupine Abyssal Plain: I. Lipids. ***Deep Sea Research I***. 41, 787-819.
- Wolff G.A., Boardman D., Sutton I., Horsfall I., Ripley M., Lewis C.A., Rowland S.J., Patching J., Ferrero T., Lamshead J., Davis N., Chester R. and Rice A.L., 1995, The biogeochemistry of sediments from the Madeira Abyssal Plain. ***Internationale Revue der gesamten Hydrobiologie***, 80, 333-349.

# Whole Core Measurements Data Documentation

## Introduction

Most benthic data collected during SES were profiles along the length of cores. However, a small number of data sets were obtained using grabs, box corers or were properties of the sediment/water interface. These data are stored in the database CORETOT table and the aim of this document is to allow the protocol used to obtain any particular data value within this 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 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

## Americium and Caesium Isotopes

$^{241}\text{Am}$  and  $^{137}\text{Cs}$  content of bulk sediment.

## Sediment Oxygen Demand

In-situ determinations of the water to sediment oxygen flux.

## Benthic Macrofauna

Macrofauna wet weight biomass

## Reference

Full reference for the paper cited in the benthic whole-core protocol descriptions.

# Americium and Caesium Isotopes

## Parameter Code Definitions

A41CGSXT	Bulk sediment $^{241}\text{Am}$ content Gamma-ray spectroscopy Bequerels per kilogram
C37CGSXT	Bulk sediment $^{137}\text{Cs}$ content Gamma-ray spectroscopy Bequerels per kilogram
S37CGSXT	Bulk sediment $^{137}\text{Cs}$ content standard error Gamma-ray spectroscopy Bequerels per kilogram
S41CGSXT	Bulk sediment $^{241}\text{Am}$ content standard error Gamma-ray spectroscopy Bequerels per kilogram

## Originator Code Definitions

### Challenger cruise CH124

78 Dr. A. McKenzie Scottish Universities Reactor Centre

## Originator Protocols

### Dr. A. McKenzie

Samples were collected using a Day Grab in January 1996, during cruise Challenger CH124. A sample was taken from each of five sites (4G, 7G, 9G, 13G and T).

$^{137}\text{Cs}$  and  $^{241}\text{Am}$  values were obtained by preparing the dried sediment samples as compressed discs using a hydraulic press to apply a pressure of 20 tonnes. The discs were sealed in plastic containers and counted using a low-background LEGe detector.

# Sediment Oxygen Demand

## Parameter Code Definitions

SODMODXX Sediment oxygen demand (water to sediment oxygen flux)  
Measurement of the change in overlying water oxygen  
concentration during an on-deck incubation  
Millimoles/m<sup>2</sup>/day

## Originator Code Definitions

**Charles Darwin cruises CD92A and CD93B and Challenger cruises CH120, CH121C, CH123B, CH124, CH126B, CH128B.**

118 Dr. Martyn Harvey CCMS Dunstaffnage Marine Laboratory

## Originator Protocols

### Dr. Martyn Harvey

Sediment cores were collected from sites N1500, N2000, R1000, and S700 between April 1995 and August 1996. Cores were also taken from sites S5, S1000, N1000 and P1000 on cruise Challenger CH128B. Samples were collected using three deployments of the DML multicorer (Barnett et al., 1984) at each site.

Aerobic respiration was determined by measuring the oxygen uptake rate of the sediment. Approximately 100 litres of seawater, for incubation of the sediment, was obtained from within 15 metres of the seabed. Two cores from the same multicorer drop were used in each experiment.

The constant temperature room was used for these incubations so that *in situ* temperatures could be maintained. Temperatures were either determined from the CTD or a reversing thermometer.

Water samples obtained at the beginning and end of the incubation period were fixed and titrated using the Winkler technique to measure the dissolved oxygen content.

# Benthic Macrofauna

## Parameter Code Definitions

MFWWSANM	Nematode benthic macrofauna wet weight biomass Sieving (300 micron mesh), picking and weighing Grams/square metre
MFWWSAPC	Polychaete benthic macrofauna wet weight biomass Sieving (300 micron mesh), picking and weighing Grams/square metre
MFWWSACR	Crustacean benthic macrofauna wet weight biomass Sieving (300 micron mesh), picking and weighing Grams/square metre
MFWWSAMO	Mollusc benthic macrofauna wet weight biomass Sieving (300 micron mesh), picking and weighing Grams/square metre
MFWWSAEC	Echinoderm benthic macrofauna wet weight biomass Sieving (300 micron mesh), picking and weighing Grams/square metre
MFWWSATL	Total benthic macrofauna wet weight biomass Sieving (300 micron mesh), picking and weighing Grams/square metre

## Originator Code Definitions

### Challenger cruise CH120

117 Dr. Lynda Mitchell Dunstaffnage Marine Laboratory

## Originator Protocols

### Dr. Lynda Mitchell

The samples were collected using the DML boxcorer during Challenger cruise CH120. The box had dimensions of 50cm by 50cm. The entire sample was sieved using a mesh size of 300µm. Once sieved the sample was sorted into phyla or groups. The content of each group was then weighted. The data were converted to grams per metre square by multiplying the weights by four.

## Reference

Barnett, P.R.O., Watson, J. and Connelly, D., 1984. A multiple corer for taking virtually undisturbed samples from shelf, bathyal and abyssal sediments. ***Oceanol. Acta***, 7, 399-408.